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SCIENTIFIC  
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*Znanstveni članki*

- 84** *Liudmyla Melnyk, Olena Kyzymchuk, Liudmyla Zubkova*  
Ukrainian Folk Ornaments in Modern Knitting  
*Ukrajinski okrasni motivi v sodobnem pletenju*
- 96** *Boris Mahltig*  
High-Performance Fibres – A Review of Properties and IR-Spectra  
*Visokozmogljiva vlakna – pregled lastnosti in IR-spektrov*
- 119** *Ismail Hossain, Md. Hasib Uddin, Altab Hossain, Mohammad Abdul Jalil, Zulhash Uddin*  
Modelling the Effect of Resin-Finishing Process Variables on the Dimensional Stability and Bursting Strength of Viscose Plain Knitted Fabric Using a Fuzzy Expert System  
*Modeliranje vpliva spremenljivk postopka plemenjenja s smolo na dimenzijsko stabilnost in razpočno trdnost viskoznege pletiva z uporabo sistema mehke logike*
- 136** *Nataliia Ostapenko, Marina Kolosnichenko, Larysa Tretiakova, Tatyana Lutsker, Kalina Pashkevich, Alla Rubanka, Halyna Tokar*  
Definition of the Main Features of Material Assemblies for Thermal Protective Clothing During External High-temperature Effect Modelling  
*Opredelitev glavnih značilnosti modeliranja kompletov materialov za zaščitna oblačila pri visokih okoljskih temperaturah*
- 149** *Sushma Verma, Vinay Kumar Midha, Awadesh Kumar Choudhary*  
Effect of Parameters on the Runoff Erosion Control Performance of Structurally Modified Jute and Coir Geomeshes over Loamy Sand  
*Vpliv parametrov na površinsko spiranje ilovnatopeščene površine s pomočjo strukturno modificiranih geomrež iz jutnih in kokosovih vlaken*
- 159** *Manca Matičič Zver, Tina Vukasović*  
Consumers' Attitude Towards Eco Friendly Textile Products  
*Stališča potrošnikov do okolju prijaznih tekstilnih izdelkov*
- 172** *Subhankar Maity, Shivangi Pandey, Alok Kumar*  
Influence of Needle-punching Parameters for the Preparation of Polypyrrole-coated Non-woven Composites for Heat Generation  
*Vpliv parametrov iglanja na izdelavo s polipirolom prevlečenih kompozitnih vlaknovin za generiranje toplote*

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# Ukrainian Folk Ornaments in Modern Knitting

## *Ukrajinski okrasni motivi v sodobnem pletenju*

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### Abstract

National arts and crafts are not only the skills of an individual nation but an ethnic phenomenon that has unique features and serves as a source of information on national history and culture. Decorative and applied art in Ukraine combines the traditions and techniques of manufacturing and decoration, which have been developed and passed down from generation to generation. It has a clear national identity and numerous regional differences in ornamental motifs, compositions and favourite colours. Therefore, the Ukrainian folk art has a significant potential and is a source of ideas in creating modern clothing as well as interior items. Embroidery as a way of creating ornamental motifs on a textile material using various techniques is a common process for decorating clothes and interiors for the Ukrainians. Today, ornamental compositions of embroidered folk textiles could be transmitted into modern clothes using various technologies. However, the use of a knitting machine allows creating a pattern during the item production. The ornament transformation into a pattern for knitting can be carried out with graphic software by creating a grid with a cell size similar to the loop size of a knitted structure for corresponding interlooping. A number of clothing and interior items with ethnic motifs was created using the capabilities of flat knitting machines within the masters programme "Knitting Technology and Design" at the Textile Technology and Design Department of the Kyiv National University of Technologies and Design.

Keywords: national clothes, folk pattern, ethnic style, knitting, jacquard knitting, miss knit

### Izvleček

Narodna umetnost in obrt ne predstavljata samo veččin posameznega naroda, temveč sta etnični pojav, ki ima edinstvene značilnosti in je vir informacij o zgodovini in kulturi naroda. Dekorativna in uporabna umetnost v Ukrajini združuje tradicijo in tehnike izdelave in okraševanja, ki so se razvijale in prenašale iz roda v rod. Ima jasno nacionalno identiteto in številne regionalne razlike v okrasnih motivih, kompozicijah in najljubših barvah. Zato je ukrajinska ljudska umetnost velik potencial kot vir idej za ustvarjanje sodobnih oblačil in izdelkov za notranjo opremo. Vezenje je pri Ukrajincih kot način ustvarjanja okrasnih motivov na tekstilijah z različnimi tehnikami pogost postopek pri okraševanju oblačil in notranjih prostorov. Danes se okrasne kompozicije ljudskega vezenega tekstilij prenašajo v sodobna oblačila s pomočjo različnih tehnologij. Uporaba pletilnika omogoča ustvarjanje vzorca med samo izdelavo pletenine. Pretvorbo okrasnih motivov v vzorec za pletenje omogoča grafična programska oprema z izdelavo mreže z dimenzijami celic, podobnih zanki pletene strukture za ustrezno vezavo. V okviru magistrskega študijskega programa „Tehnologija pletenja in oblikovanje“ na Oddelku za tekstilno tehnologijo in oblikovanje na Nacionalni univerzi za tehnologije in oblikovanje v Kijevu so bila z uporabo zmogljivih ploskih pletilnikov ustvarjena različna oblačila in izdelki za notranjo opremo z etničnimi motivi.

Ključne besede: nacionalna oblačila, ljudski vzorec, etnični slog, pletenje, žakarsko pletenje, založena vezava

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## 1 Introduction

In the modern world, where cultural peculiarities of different nations and regions are smoothed out, the issues of reviving and developing the best national traditions are especially relevant. Traditional arts and crafts are the merits of people who preserve their spiritual experience and identity. The importance of national textiles for revealing people's worldview, their moral values and their own styles cannot be overestimated [1, 2]. Therefore, the recognition, preservation and development of national traditions should be an integral part of the new generations' education. National traditions instil love for the native land, develop the understanding of the need to study ethnic history and of the outstanding nation achievements. Therefore, ethnic folk textiles enrich the creativity and efforts of designers, being an invaluable source of ideas for modern textiles and clothing creation [3–5]. Modern clothes with ethnic motifs not only create our appearance and image, but also represent the inner world of people. They are a means of nonverbal communication in the society [6].

The Ukrainian folk textile heritage is an important part of the best national traditions, the art of individual regions of the country that contains various ideas aimed at preserving the national identity [77]. People have been creating products for many years, satisfying not only their daily practical needs, but also the aesthetic ones. The Ukrainian folk decorative and applied art is a special branch of the artistic activity that has a distinct national identity, and contains practical, ceremonial, ideological, semantic and educational functions. It is closely connected with the history and national traditions of regions and testifies the artistic endowments of folk artisans.

An important component of the Ukrainian folk art is embroidery, i.e. the process of decorating clothes and interiors. Embroidery is the artistic source that determines the specific character of the Ukrainian spiritual culture and the path to the national self-preservation. The artistic richness of embroidery is determined by numerous differences formed under the influence of historical and geographical conditions. Unique motifs and ornaments, colour scales and specific techniques have been formed in each region of Ukraine. With the help of embroidery, clothes and household items have been transformed into unique and inimitable works of art [8, 9]. The Ukrainian folk costume combines the art of decorating, patterning and sewing. The variety of forms

and imaging, the expediency of constructive and compositional solutions, the decor uniqueness, and the inseparable connection of these remain an inexhaustible source of creative ideas.

Apparently, this is the reason for fashion designers to constantly refer to the Ukrainian folk art for innovative ideas or a source of inspiration [10, 11]. They provide national characteristics into modern fashion that organically fit the world tendency [12]. The ethnic style is relatively common not only to the Ukrainian modern fashion, but throughout the world and especially to the European fashion [13].

The use of folk art motifs is popular today for interior design as well. The reference to folk interior solutions creates functional, aesthetic and comfortable housing for modern life. The ethnic Ukrainian houses have been decorated with embroidered towels and pillowcases, patterned carpets, paintings on the walls and furniture, etc. Such samples of national crafts create a wide field of activity for a designer in their introduction into the modern residential interior [14]. The ornament is an important element of the national Ukrainian style. The selection and alternation of patterns are organised in such a way that the kinship of symbols, unity of the world perception embodied in magical pattern amulets can be seen [10]. The embroidery symbols have never been random, conditional, since each element has its own purpose. They come from our ancestors, their knowledge and views on the nature [15]. In this case, the choice of colour is symbolic as well, which is considered a charm in combination with the ornament [16]. From the other side, symbolism is very diverse by regional grounds; therefore, it is an inexhaustible creative source for modern art [11].

The Ukrainian ethnic elements, which have been improved over the centuries and have absorbed the peculiarities of the centuries-old culture, give modern clothing and interior special flavour of those centuries. They increase their artistic emphasis and consumer attractiveness, as well as the national and regional identify [17].

### 1.1 Historical notes

Embroidery plays the leading role in the decoration of Ukrainian folk clothing, has a symbolic meaning of amulet and characteristic features in colour, technique and composition, honed over the centuries. The first mention of embroidery is known from the 4<sup>th</sup> century BC [18]. Since then, artistic and stylistic stitching has been formed, the sign system symbol-

ising the forces of nature and the structure of the world has emerged. There are motifs that have lived in embroidery for centuries, reminiscent of pagan beliefs. With Christianity, new art forms appeared. In particular, gold artistic stitching reached a high level in the Kyivan Rus and spread far beyond borders. Such products were mostly used by priests. Studies indicate that embroidery was a favourite art form among both the nobility and common people, and that the seamstresses mastered the gold stitching technique, creating rich and varied pattern texture. Information about folk embroidery is known only since the 18<sup>th</sup> to the beginning of the 19<sup>th</sup> century, since the study of works of art began as late as in the 1980s. In fact, the first results of the study of Ukrainian embroidery were summarised in the work

[19], which has been republished several times, and contains ornament samples and technique recommendations. The peculiarity of folk embroidery is its striking difference according to ethnographic regions, i.e. Middle Dnieper, Polissya, Carpathians and Prykarpattia, Podillya and South (cf. Figure 1). There were special ornaments, compositions and techniques in each region during the historical and cultural development in Ukraine. The colour scheme also depended on the Ukrainian region. Our ancestors created products that differed in the playful colours of Carpathian mountain valleys or Podillya plains, or their monochrome combination. The colour scheme of embroidery also depends on the purpose of the product – festival or casual. According to recent studies, the choice of colour is a factor that creates



Middle Dnieper



Carpathians and Prycarpathian



Polissya



Podillya

Figure 1: Embroidery of different regions of Ukraine (19<sup>th</sup>–beginning of 20<sup>th</sup> century) [18]

different emotions. Therefore, the choice of colour is an important factor in the clothes production [20]. Folk embroidery is reproduced worldview with artistic means. The Ukrainian embroidery is characterised by a combination of geometric and floral ornaments. The geometric elements such as rhombuses, rosettes and cruciform figures became the basis of the East Slavic pattern. The archaic forms of ornaments, e.g. miracle birds, griffins, outlines of a female figure are characteristic of the embroidery of Chernyhiy, Poltava, Podillya. In the 19<sup>th</sup> century, the interest in floral ornamentation increased [21].

The Hutsul region (Carpathians) attracts special attention since here, each district, even every village, has its favourite range of colours. The majestic beauty of the Carpathian nature contributed to the development of unique fine art. The Hutsul region is characterised by geometric ornaments and contrasting colour comparisons, whereas massiveness and densely placed patterns are typical of the Hutsul antic folk embroidery [22].

In the 20<sup>th</sup> century, the tradition of embroidery continued with a wide range of masters involved in the artistic life of the country, and their works were successfully exhibited in Berlin, Prague and London [18]. Experimental workshops were created where folk seamstresses and artists were searching for new forms and artistic styles. In the modern world, the Ukrainian embroidery is developing in the field of traditional household, amateur art and artists' creativity whose activities are aimed at studying the best embroidery samples and preserving its classical heritage (cf. Figure 2).

### 1.2 Embroidery techniques

Embroidery was placed into strictly defined places of clothing – sleeves, armpits, cuffs, hem of a shirt, since canvas ends were considered the best places for the penetration of evil forces [21, 23]. The beauty and perfection of embroidery largely depend on the execution technique. More than a hundred stitches are counted in the Ukrainian embroidery. These are various stitches: the top stitch is made on the drawing put on a cloth, the counting stitch is made by counting cloth threads. The oldest include “zavolikannia”, “zanyzuvannia”, “nyzyna” [8], “twisted stitch”, variation of “flat stitch”. Cross stitching appeared in Ukraine at the beginning of the 19<sup>th</sup> century. This stitch was quickly mastered by people and displaced more complex techniques from everyday life.

The names of many techniques come from the means of performance, e.g. “cutting”, “punching”, from the item embroidered, e.g. “towel stitch” for towels, or from place names, e.g. “Old Kyiv stitch”. In some cases, the technique name is associated with the appearance of the stitch, e.g. “chicken ford”, “nightingale eyes”, “buckwheat”, “oatmeal”. Most techniques are common in all regions of the country; however, each area has its favourite techniques and its traditional colour scheme. Thus, “nabiruvannia” is widespread in the Kyiv region, “lyshtva” in the Poltava region, and “zanyzuvannia” in the Volyn region. Stitches, spread almost all over Ukraine, are often made in different colours, hence creating their diversity. Folk embroidery is characterised by a variety of techniques in one product, which can contain up to 10–15 techniques at a time [9].



Figure 2: Ethnic clothing in 1980s

The hemstitch has been used in decoration for ages. This openwork stitch has decorative holes of a certain size. This is one of the most interesting, most ancient and simplest ways of embroidery. The pattern is formed due to the contrast of openwork holes and covers. It had both practical and decorative significance. The hemstitch was used for decorating women's and men's clothing, bed linens, tablecloths, towels etc., and for connecting different parts of clothes instead of seams. It was used for decorating pockets, neck, sleeves and skirt hems. White or light coloured threads (grey, pink, blue) are used for white fabrics and close to the tone if the fabric is coloured. Ornamental compositions can be both geometric and plant-geometric.

The hemstitch is usually made on a fabric from which threads are pulled out in one direction and the rest of the threads are combined in different ways into bundles ("twigs") that create patterns. The hemstitch is usually combined with different stitches, e.g. "nabiruvannia", "nyzyna", "zanyzuvannia", "cross stitch", "flat stitch", which enrich the pattern [24].

The hemstitch can be narrow and simple as well as complex, reminiscent of thin lace (cf. Figure 3). It is distinguished by type, technique, size and complexity. Depending on the formed components, there are the following types of hemstitch [25]:

- one-sided, when one side of the fabric strip with threads pulled out is embroidered, and double-sided when two sides of the fabric strip with threads pulled out are embroidered;
- simple and complex, containing one or more strips;
- narrow and wide (2 cm or more width is embroidered on the hoops).

### 1.3 Methods for creating ornaments on products

The development of modern technologies creates the conditions for obtaining decorative elements of embroideries using various methods. Following the hand embroidery tradition, machine embroidery can be used as well. It allows applying a pattern on different types of fabrics, clothing details even where other technologies are unacceptable. The main characteristic of machine embroidery is high-quality and aesthetic application of patterns, images and inscriptions on various textile materials. The machine embroidery technology compared to other methods allows creating an image in a clearly defined place, the embroidery being more stable and durable, even after repeated washing [26].

Printing is a well-known technique for decorating textile fabrics with the application of pigments, dyes or other related materials in the form of patterns. There is a number of printing methods that could be used for ethnic clothes decoration, e.g. block, roller, screen and heat transfer printing. These methods are used as a separate technological stage [4], which requires additional equipment, work force and premises.

Weaving is the oldest textile technique used to obtain interesting and intricate patterns in ancient times [27]. There are two main techniques of folk weaving in Ukraine, i.e. shuttle and sorting, which determine the nature of the ornament. The first is used for small patterns in the form of zigzags, obliques or rhombuses. The warp and weft threads in different directions of the covering create light and shadow tone transitions on the fabric surface. The sorting technique provides more opportunities for creating complex patterns on textiles. In addition



Figure 3: Hemstitch

to transverse stripes, there are geometrised motifs, e.g. rhombuses, stars, rosettes, stylised images of flowers, birds. This pattern technique prevails in the production of fabrics with straight contours [28–29]. Weaving is used in the manufacture of “zapasok”, i.e. a belt product, characterised by variety, sophistication and richness of artistic and aesthetic solutions. It has a significant variability in the combination of narrow ribbons with different thicknesses, in different rhythms of transverse-striped compositions, as well as shading – from warm, cold to contrasting colour range. Towels, tablecloths and carpets with the elements of the Ukrainian ornament are widely used for decorating interiors with weaving.

Knitting is one of the textile techniques that has the widest opportunities to create a variety of patterns and effects on fabric during its manufacture. The designed patterns are resistant to mechanical stress and weather conditions, and their quality depends generally on the quality of raw materials and the choice of colour. Changing the knitting machine gauge and the linear density of yarn, correspondingly, make it possible to create patterns from the finest ones to those simulating hand knitting. In addition, modern drawing design systems allow creating a variety of ornaments due to a different combination of local identity elements [30].

The first collections of knitted clothes in ethnic style were created in the last century (cf. Figure 4). A modern diversity of knitting equipment enables us to create both knitted goods with a small pattern repeat

and a pattern of unlimited repeat. The best places for folk decoration on modern clothes are the following: design of the neckline and collar, the top and bottom, the fasteners and sides, the waist and the pockets, etc. [31]. Thus, flat knitting machines with their knit-and-wear technology are the most perspective equipment. Details of different shapes can be knitted, with a pattern only in a certain part or throughout the whole length [21–23]. Such equipment has wide technological possibilities for any creative idea.

## 2 Materials and methods

The methods of a literary and visual-analytical review were used in the study of artistic decoration for Ukrainian folk clothes.

The reproduction of ornaments identical to the ornaments of traditional Ukrainian folk clothes was conducted by knitting. The knitted structures were made by openwork, jacquard, plated and miss knit. The developed knitted fabrics were used in children’s and women’s clothes as well as interior items. The ornaments are located in line with the tradition of embroidered folk products.

All knitted fabrics were produced on a 5-gauge flat knitting machine Brother, the technological capabilities of which allow making a pattern in a certain place of a product. 31 × 2 × 2 tex half wool yarn was used for jacquard knitting, 29 × 2 × 3 tex cotton yarn for openwork and interlooping with miss knit wales,



Figure 4: Knitted clothes in ethnic style in 1970s

while  $29 \times 2 \times 3$  tex cotton yarn as plated and 40 tex polyester yarn as ground were used for plated interlooping.

### 3 Results and discussion

The clothing creation with ethnic elements is an important part of the research work carried out at the Department of Textile Technology and Design. Students' involvement in their masters theses aimed at the study and development of arts and crafts contributes not only to their qualification as specialists in knitting, but also to the education of creative, active, initiative and of nationally conscious people with a strong intellectual potential, willing and able to work for their homeland prosperity. The main feature of folk decorative and applied arts and crafts is the understanding of creative work beauty. During the creation of ethnic collections, future specialists are exploring the design of arts and crafts, finding out the meaning of symbols' decoration, determining their influence on people and the environment. Such original work develops students' creative potential, their imagination and thinking. At the same time,

students show their knowledge capacity in the field of knitting equipment, production processes, interlooping types etc.

The variety of knitted fabrics allows reproducing colour, openwork, textured effects in products. Thus, jacquard and plated weaves are widely used for colour pattern creation, as well as open and split weaves for making cloth with holes.

#### 3.1 Cross stitching in knitting

Cross stitching is a traditional technique for the Ukrainian folk clothes decoration. The usage of the cross-stitch pattern with square units is not very appropriate for knitting. The pattern transformation onto small sections that correspond to the loop size is the main task for the pattern creation in knitting. The loop is the main and smallest element of the knitted structure that is characterised by wale (W) and course (C) spacing. The knitted structure can be inscribed in a square only with equal W and C. It forms a rectangle in all other cases: horizontal for  $W > C$  and vertical for  $C > W$ . This is a factor affecting the shape and size of the ornament. Therefore, to display the real dimension ratio of the future ornament, the pattern should consist of rectangles,

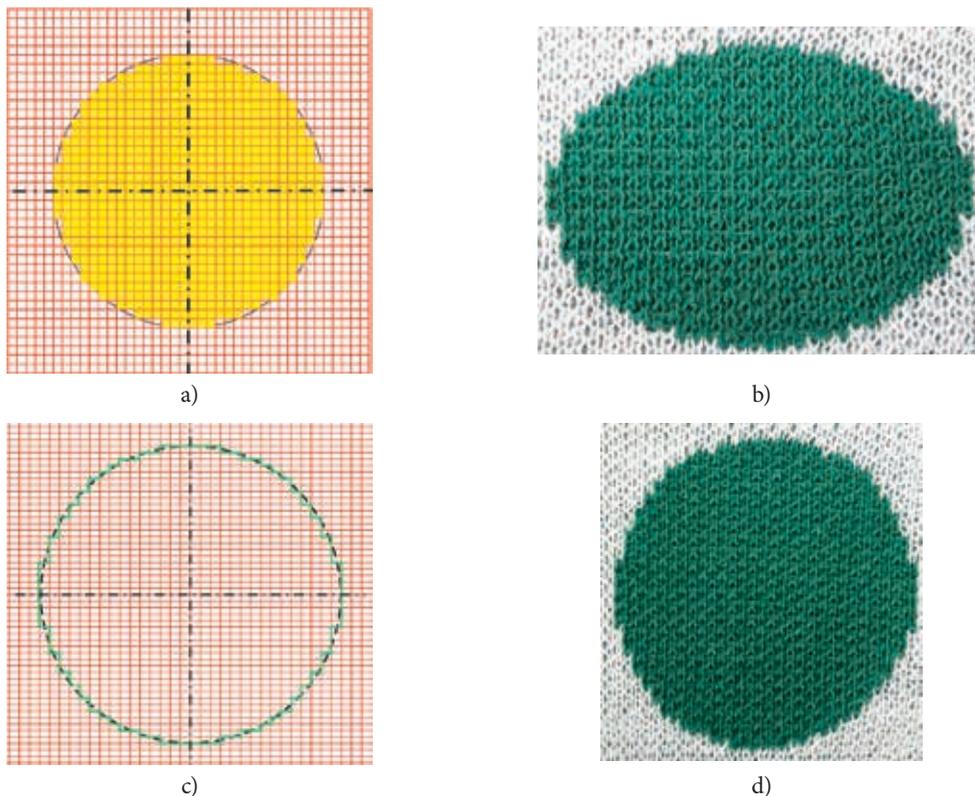


Figure 5: Circle in knitted samples

the size ratio of which is chosen according to the loop size. In this case, the knitting direction is an important factor.

To confirm this conclusion, a circle was chosen as the ornament unit for the jacquard sample. The pattern  $30 \times 30$  that forms a circle (cf. Figure 5a) with cross stitching turns into a horizontal ellipse (cf. Figure 5b) with 82 mm in width and 54 mm in height. It is proposed to use the applied graphics programs, e.g. CorelDraw, to reduce the time needed for pattern creation. It allows us to create a grid with desired cell sizes, superimposing it onto any original ornament. A grid with units  $W = 2.73$  and  $C = 1.78$  mm was used for the study. The circle (dashed line) with an 82 mm diameter was imposed on the grid and the outlines of the figure were drawn (green line) along the contours. In this case, the repeat size was 30 wales and 46 courses (cf. Figure 5c). The pattern corresponds to a circle in the knitted sample (Figure 5d). This method greatly simplifies and speeds up the

cross-stitch ornament implementation in knitting and was used in this work (cf. Figure 6).

It is very exciting to implement the traditional Ukrainian ornament into children's clothes. It should be noted that the choice of ornaments for children has always been very important. Ornaments that are mostly used in the Ukrainian children's folk costumes can be divided into two main groups, i.e. geometric and stylised figures.

A tree branch is considered one of the main symbols in the Ukrainian children's folk clothes. It has a special meaning, namely a tree branch is a symbol of the beginning of life. The branch frequently appears with berries, which symbolise the birth of future generations. A rose, as a symbol of beauty, is often depicted on girls' dresses. Generally, girls' shirts are embroidered with ornaments with phytomorphic or Scythian-type motifs. Geometric figures (rhombus, rectangle and circle) have been considered as ornaments for boys' clothes, rhombus and rectangle being



Figure 6: Cross-stitch pattern in knitted structure



Figure 7: Knitted clothes for kids (author: Oksana Zvereva)

symbols of wealth and fertility. In most cases, the ornament is adorned with moustaches that reflect male courage.

Both vegetative and geometric patterns from Ukrainian embroideries of different regions were chosen. A two-colour incomplete jacquard was used for the children’s collection. All fabric samples were produced in two colours, i.e. red and white. All ornaments were produced in two variants of embroidery imitation, i.e. red on white and white on red (cf. Figures 6 and 7).

As it has already been mentioned, the choice of an ornament has a tremendous value and is considered an amulet. Pysanka, for example, is the Ukrainian name for an Easter egg, ornamented with different folk patterns and motifs using wax and colourful dyes (cf. Figures 8a and 8b). The word “pysanka” originates from the Ukrainian verb “pysaty”, which means “to write”. The ceremonial egg decorating has a long history in the Ukrainian culture. Most motifs that survived to modern times are symbols of communication between our ancestors and nature. With these symbols, they protected themselves from evil forces. [32]

The ornament proposed for the interior goods decoration consists of two motifs. The first and the main one is a bird (cf. Figure 8c), which is centrally located.

The second one is the cross (cf. Figure 8d) which is located on the edge (cf. Figure 9). The bird is a symbol of birth, fertility, abundance. It is a semi-underground, semi-heavenly being, a symbol of love, loyalty and harmony. In Christianity, the bird is a symbol of the ascension to God. The cross is one of the sunny signs, the symbol of the Universe, the four sides of the world, four winds and four seasons of the year. It comes from the schematic ancient image of a bird, as the sun was a bird flying in the sky. The cross is also a symbol of life and immortality. Two colours were chosen for production, i.e. brown and ivory, symbolising reliability, devotion, stability.



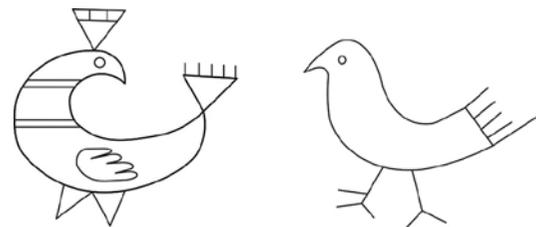
Figure 9: Interior knitted goods (author: Alina Kozhukhar)



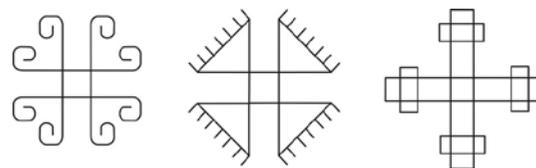
a)



c)



b)



d)

Figure 8: Pysanka with different motifs

### 3.2 Hemstitch in knitting

The variety of knitted fabrics allows reproducing not only coloured patterns, but also lace and textured effects. Thus, jacquard was used in the above mentioned work, as it reproduces the colour motifs of cross stitching very successfully. It is more interesting to create the hemstitch effect in knitwear. Several variants of interlooping can be used to reproduce such embroidery:

- plaiting, in which, according to the pattern, some loops are formed from two threads, and others from only one considerably thinner thread (cf. Figure 10a);
- openwork made by transferring certain loops to neighbouring needles (cf. Figure 10b);
- miss knit with switched off needles (cf. Figures 10c and 10d).

One-colour yarn was used. It corresponds to the principle of hemstitch formation in the Ukrainian folk embroidery. The yarns of different linear densities were used for the plated fabric and different raw materials to enhance the textured effect. The folk hemstitch was reproduced into women's dresses (cf. Figure 11) in many ways.

## 4 Conclusion

The ethnic style development in modern fashion is a relatively common trend throughout the world. The Ukrainian decorative and applied art as the ability of people to demonstrate the national culture, thinking and views is the inspiration source for the creation of modern clothes.

A promising way to reproduce folk motifs is knitting, which allows creating a pattern during the clothes production without an additional finishing process and technological transitions. The pattern placement is possible both, on the whole plane of the fabric and only on a certain place of a product for details. For this technique, the ornament transformation onto the smallest areas, i.e. the loop size, was successful. The use of modern pattern design systems permits creating ornaments of various compositions. The variety of knitted interlooping allows the reproduction of colour, openwork, relief effects in ornaments identical to the ornaments of traditional Ukrainian folk embroideries.

The knitted structures based on openwork, jacquard, plated and miss knit fabrics that reproduce the cross



a)



b)



c)



d)

Figure 10: Knitted hemstitch



a)



b)

Figure 11: Dress (a – author: Anna Konotop; b – author: Anna Kravchenko)

stitch and hemstitch folk patterns were developed. The developed knitted fabrics were used in children's and women's clothes, as well as for interior decoration. The use of national elements of the folk costume in the development of modern clothing gives them special colour, artistic expression, identity, consumer appeal. It is not only the source of centuries-old culture. It helps restoring historical and spiritual memory focusing on the aesthetics depth of the Ukrainian ethnicity. The use of ethnic elements not only expands the product range but also promotes national traditions, which are one of the sources for education to younger generations. The spiritual birth of a person occurs through the assimilation of the national culture, age-old traditions and customs. Involving students in the study of arts and crafts contributes to their education as creative, active, nationally conscious persons. It also leads to the expansion of professional skills for future textile technologists.

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# High-Performance Fibres – A Review of Properties and IR-Spectra

## *Visokozmogljiva vlakna – pregled lastnosti in IR-spektrov*

Scientific review/Pregledni znanstveni članek

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### Abstract

High-performance fibres are fibre materials that exhibit at least one extraordinary property compared to conventional fibre materials. That extraordinary property is frequently related to excellent fibre stability against certain influences such as fire, heat, chemicals or light. Also, a high mechanical strength is often a property of high-performance fibres. Nevertheless, it should be noted that high-performance fibres exhibit certain weaknesses in addition to their advantages. This review presents a broad overview of the most important high-performance fibres, with a special emphasis on their chemical structure and related infrared spectra (IR-spectra). The categorization of the fibres according to chemical substance classes was performed to make it easy for the reader to find a fibre of interest. The main categories are polyethylene (PE) fibres, polyacrylonitrile (PAN) fibres, polyvinylalcohol (PVAL) fibres, polyester-based fibres, polyamide-based fibres, polyetheretherketone (PEEK) fibres, polyimide (PI) fibres, halogen-containing fibres, polyphenylene sulfide (PPS fibres), resin-based fibres and finally inorganic fibres. Competing materials are also discussed, and structural related materials can be easily identified. In addition to discussing fibre properties and selected applications, one of the main aims is to present a various number of IR-spectra as a tool for structural understanding and to help identify unknown fibres. Here, beside the IR-spectra of high-performance fibres, the reference IR-spectra of common fibres are presented for comparison.

Keywords: High-performance fibres, synthetic fibres, inorganic fibres, infrared spectroscopy

### Izvleček

Visokozmogljiva vlakna so vlaknine, ki imajo v primerjavi s konvencionalnimi vlakninami vsaj eno izjemno lastnost. Izjemne lastnosti so pogosto povezane z odlično stabilnostjo vlakna pri nekaterih vplivih, kot so ogenj, toplota, kemikalije ali svetloba. Tudi visoka mehanska trdnost je pogosta lastnost visokozmogljivih vlaken. Kljub temu je treba upoštevati, da imajo visokozmogljiva vlakna poleg svojih prednosti tudi nekatere neprijetne slabosti. Članek podaja širši pregled najpomembnejših visokozmogljivih vlaken s posebnim poudarkom na kemični strukturi in z njo povezanih infrardečih spektrih (IR-spektri) vlaken. Vlakna so predstavljena v skupinah glede na njihovo kemično razvrstitev, tako da bralec laže najde vlakno, ki ga zanima. Glavne skupine so polietilenska vlakna, poliakrilonitrilna vlakna, polivinilalkoholna vlakna, vlakna na osnovi poliestrov in poliamidov, polietereterketonska vlakna, poliimidna vlakna, vlakna, ki vsebujejo halogene elemente, polietilensulfidna vlakna, vlakna na osnovi smole in na koncu anorganska vlakna. Prav tako so obravnavane konkurenčne vlaknine in tudi strukturno sorodnih vlaknin ni težko razpoznati. Poleg lastnosti vlaken in

izbranih področij uporabe je glavni cilj predstaviti številne IR-spektre kot orodje za strukturno razumevanje, pa tudi kot pomoč pri razpoznavanju neznanih vlaken. Tu so poleg IR-spektrov visokozmogljivih vlaken za primerjavo podani tudi referenčni IR-spektri tekstilnih vlaken.

Ključne besede: visokozmogljiva vlakna, sintetična vlakna, anorganska vlakna, infrardeča spektroskopija

## 1 Introduction

High-performance fibres are fibres with at least one outstanding property, i.e. a high-performance property. Beside a high mechanical strength, this high-performance property is often related to the high stability of the fibres against various influences such as heat, fire, chemicals, light, etc. [1-4]. However, each high-performance fibre has specific disadvantageous properties in addition to its advantageous high-performance property [4]. The best example to explain this issue is probably Kevlar fibre based on the polymer para-aramid. This fibre has a high strength and excellent flame-retardant property, but contains a very low light stability [5, 6].

The fields of application of high-performance fibres relate to their outstanding properties. In fields where these properties are demanded, these high-performance fibres are used. Fibres with high mechanical strength are used, for example, for the production of ropes, cut-resistant textiles and bullet-proof vests [7, 8]. They also find applications in fibre-reinforced composite materials [9]. Non-flammable fibres find applications, for example, in fire-protective clothes and in airplane interior textile materials [10, 11]. Fibres with excellent stability against heat and chemicals can be also be used for the production of protective clothes [12]. However, they are frequently used as filter materials for hot and aggressive gases.

The aim of this review is to give an overview regarding the most important high-performance and innovative fibres on the market. Some advantageous and disadvantageous properties are discussed in relation to the chemical structure of the polymers used to build up these fibres. Related to the chemical structure is the infrared spectrum, which enables the identification of specific functional chemical groups. The vibration of a chemical bond can be stimulated through the absorption of infrared light containing a specific energy. This specific energy corresponds to a certain wavelength of the infrared light. Beside using energy or wavelength, infrared spectra are usually shown in the function of wavenumber using the unit  $\text{cm}^{-1}$ . This wavenumber is

a reciprocal unit of the wavelength. A prominent example from the field of textile can be given with acrylic fibres. Acrylic fibres contain the nitrile group  $-\text{CN}$ , which contains a carbon-nitrogen triple bond. The stretching vibration of this carbon-nitrogen triple bond can be stimulated by infrared light in the wavenumber range of  $2,240 \text{ cm}^{-1}$  to  $2,260 \text{ cm}^{-1}$  [13, 14]. An infrared signal in this range of wavenumbers is quite unique for polyacrylonitrile. For this, acrylic fibres can be easily identified and distinguished from other fibres using IR-spectroscopy. In addition to the identification of specific chemical groups, IR-spectroscopy can be used to identify chemical compounds by comparison with spectra from literature or databases. Here, the spectral range from  $600 \text{ cm}^{-1}$  to  $1,750 \text{ cm}^{-1}$ , in particular, is compared. For this, this spectral range is also called the fingerprint area [13]. IR spectroscopy is an appropriate method for the quick identification of high-performance fibres without the need for sample destruction. Such a quick identification is often demanded in production processes to verify materials used. The identification of materials is also required during recycling of polymers and fibres.

In this review, fibres with similarities in the chemical structure are classified into the same category and described in the related sub-chapters. Competing fibre materials are named, as well as some advantageous and disadvantageous properties. The chemical structures of fibres are depicted and discussed according to some fibre properties, but also in relation to the recorded IR-spectra. All IR-spectra shown are exclusively measured for presentation in this review paper. For some fibre materials, the same fibre types from different products used in different applications are measured and compared. Instead of the fibre products, related bulk materials are examined using IR-spectroscopy for other fibres. One main reason for these alternative measurements is that several high-performance fibres are only available as compositions in blended yarns and fabrics. High-performance fibres are often quite expensive and, in the best-case scenario, their application in a small amount can lead to new and innovative products.

## 2 Parameters for description of fibre properties

The parameters to describe fibre properties can be put into several categories related to the area of interest. It is quite useful to use the following four categories: mechanical properties, temperature/flame stability, chemical stability and light stability. Mechanical properties used for fibre description include elastic modulus, tensile strength or elongation at break. For the description of the temperature stability of fibres, the values for glass temperature ( $T_g$ ), melting temperature ( $T_M$ ) and the temperature of decomposition ( $T_D$ ) are suitable. In addition, the maximum temperature of usage ( $T_{max}$ ) is quite frequently recommended by the supplier of a fibre. An overview of  $T_{max}$  values for a broad range of different fibres is given in Table 1. It is clear that there is a certain range of variation for each temperature value, even for the same type of fibre. This variation can be explained by different properties, even if the fibre is built up by the same type of polymer (e.g. different grade of crystallization, degree of polymerization, additives or co-polymer units). Also, the type of method for determining these values can have a certain influence, i.e. measuring the decomposition temperature under air or inert gas. In the case of  $T_{max}$ , the type of usage is also of relevance. The flame-retardant properties of a fibre material are usually described by the LOI (limited oxygen index) value. An overview of several LOI values is also given in Table 1. This value indicates the amount of oxygen that is necessary to set a fibre material on fire. Increasing LOI values also improves the flame-retardant properties of a fibre. Fibres with a low LOI of less than 21% can be easily ignited.

The description of the chemical stability of a fibre is almost more challenging and is determined through investigations comprising two parts. The first part is the influence of the chemical of interest on a fibre. Here, many process variations are possible, such as temperature, duration of contact or concentration. The second part is the fibre property that is measured before and after the chemical treatment, i.e. measuring the weight of the fibre material or its tensile strength. In a certain way, the testing of light stability of fibres is similar. Here, a certain fibre property is tested before and after illumination with light and is compared to reference materials. In combination with UV-stability, light stability is often called sun light stability or weathering stability. It should be

clear from this overview that the light stability of a fibre strongly depends on the type of light that is used for illumination and other surrounding parameters such as humidity or temperature.

Given this broad range of different parameters for describing high-performance fibre properties, the limitation of this review paper is clear. Only the most important points can be mentioned for each presented fibre.

## 3 Materials and methods

All infrared spectra presented in this review were measured especially for this paper. These measurements were performed using a IR Prestige 21 spectrometer supplied by Shimadzu, together with a Specac Golden Gate ATR unit. The fibre samples were taken from different sources and were tested as received without further treatments. Particularly worthy of mention are non-woven filter materials from the companies BWF Offermann, Waldenfels & Co (Offingen, Germany) and Zhejiang Rayjet Filter Media Technology (China). Several types of glass and silica fibres were obtained from Culimeta Textilglas Technologie GmbH (Bersenbrück, Germany). Sieve fabrics made from halogenated fibres were supplied by Reichelt Chemietechnik GmbH (Heidelberg, Germany). Several ropes and lines were obtained from the company Hamburger Tauwerk Fabrik (Hamburg, Germany). Polyethylene fibre non-woven, as reference material, was supplied by Modulor GmbH (Berlin, Germany). Several protein fibre materials used as reference materials were obtained from the company Das Wollschaf (Zweibrücken, Germany). Flame-retardant polyester and water-soluble yarns and fibre felt made from polyvinylalcohol fibres were obtained from the company StoffeDE (Schenefeld, Germany). Different fibre and polymer materials were obtained from Goodfellow GmbH (Friedberg, Germany). For some high-performance fibres, it was only possible to obtain this fibre in blended yarns and fabrics. In these cases, the IR-spectra were taken from the pure polymers supplied in other forms, such as plate, foil or bullet.

## 4 Synthetic fibres

Synthetic fibres are often referred to as chemical fibres and, by this distinguished form, natural fibres.

Table 1: Overview of the properties of several fibre materials (conventional and high-performance). Compared are the maximum temperatures for continuous usage, the LOI value and a selection of some properties that are typical for this type of fibre. The different mentioned values are compared from different reference sources. Some important brand names are given in brackets.

Type of fibre	Maximum temperature of usage (°C)	LOI (%)	Selected properties	References
Cellulosics	80	17	High humidity up-take	[15, 16]
Polypropylene PP	90	17	High chemical stability	[15, 17]
Polyethylene HPPE, (Dyneema)	90	18	High strength, high elastic modulus	[15, 18]
Wool	94	25	High humidity up-take	[15, 19]
Polyamide PA	110/115	24 (Nylon)	Mechanically stable, stable against abrasion	[15, 20]
Polyacrylonitrile PAN	125	18 (homo-PAN)	Stable against hydrolysis, light, weathering	[15, 21]
Polyester PET	150	21	Low cost	[15, 22]
Phenolic resin, novolac resin (Kynol)	150/200	30–34	Resistant against heat and fire	[15]
Polyetherimid, PEI	170	44	Resistant against heat and fire	[21]
Polyamidimid, PAI (Kermel)	180/200	30–32	Good temperature stability	[15]
Polyimid, PI (P84)	180/260	36/38	Good mechanical and thermal properties	[15, 23]
Polyphenylenesulfide PPS	190/200/280	40	Good chemical and thermal stability	[15, 23]
Meta-aramid (Nomex)	200	30–32	Good temperature stability	[15, 23]
Preoxidized PAN (Panox)	200	50	Good temperature stability, flame-retardant	[15]
Para-aramid (Kevlar)	190/200	28–30	Extraordinary mechanical properties, stability against contact heat	[15, 23]
Polyetheretherketone, PEEK	250	28/35	High temperature materials	[21, 23]
Polytetrafluoroethylene PTFE	250/260	95	Extraordinary chemical stability	[15, 23]
Polybenzimidazol, PBI	250/300	40/41	Extraordinary thermal stability	[15, 23]
Phenylene Benzobisoxazol, PBO (Zylon)	310/350	56–68	Extraordinary mechanical properties, stability against contact heat	[15, 21, 23]

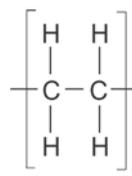
Synthetic fibres are also referred to as organic fibres and, by this distinguished form, inorganic fibres. If synthetic fibres are referred to as man-made fibres, they are, by this distinguished form, natural fibres. Below, synthetic fibres with high-performance properties are summarized and categorized for this purpose into groups related to their chemical composition. Later, in section 5, some inorganic high-performance fibres are presented.

#### 4.1 Polyethylene, PE

Polyethylene PE is probably one of the most used polymeric materials worldwide. Its chemical structure is the simplest of all synthetic polymers (Scheme 1). PE is used in many products from everyday life. Probably the most prominent PE product is the disposable plastic bag. PE is the first choice for many applications because it is a low-cost material. Nevertheless, there are also high-performance and

high-price PE products. High-performance polyethylene fibres (HPPE) are known for their high strength and their low specific weight (low density) [24]. Also, these HPPE fibres have good weathering stability and stability against salt water, which is important for marine applications. Manifold applications of HPPE fibres are found in protective clothing, composites, ropes, cables and in the field of medicine [24]. The many disadvantages of HPPE fibres include their low temperature resistance due to their low glass temperature  $T_g$  of around 90 °C and their high flammability, which is reflected in a low LOI value of around 18 [18]. It is also quite difficult to modify polyethylene fibres through chemical finishing because there are no functional groups on the fibre surface that could interact with the anchor groups of finishing agents [26]. The chemical structure of HPPE is the same as the structure of conventional polyethylene, if the structure of the monomer unit is considered (see Scheme 1). The main difference is the molecular weight, which is very high for HPPE, with values above one million g/mol. For this reason, the name UHMW-PE is used (ultrahigh molecular weight – PE) in addition to HPPE. For UHMW-PE, a molecular weight of more than 1 million g/mol means a polymerization degree in excess of 35,000. In comparison, conventional PE has a significantly lower molecular weight. For HDPE (high density polyethylene) materials, a molecular weight in the range of 100,000 g/mol is typical. LDPE (low density polyethylene) materials exhibit an even lower molecular weight and contain a branched polymeric structure that also contains side chains [27]. Several years ago, two companies were the main suppliers of HPPE fibres: DSM (Netherlands), with the brand Dyneema fibres, and Honeywell (USA) with the brand Spectra fibres. For this reason, often the name “Dyneema”

is typically used as synonym for HPPE fibres [2]. Nevertheless, there are many competing companies today that offer different types of HPPE fibres. The IR-spectrum of polyethylene is quite simple due to the simple chemical structure of this polymer (Figure 1). Only four main signals dominate the IR-spectrum of polyethylene. These signals are related to the stretching and deformation vibration of the C-H bonds and  $-CH_2-$  units that build up this polymer [13]. It is not possible to distinguish HPPE fibres from conventional PE materials using IR spectroscopy because their monomer units are chemically identical (Figure 1). One of the main applications of HPPE fibres are high-strength, light-weight ropes. For this reason, samples from ropes based on HPPE fibres were also taken and their IR-spectra were recorded (see comparison in Figure 1). One rope is a two-component system with a core made from grey-coloured Dyneema fibres and a braided surface made from PBO fibres. The other rope is made from high molecular weight polyethylene (HMPE), which contains an undefined black coloration. The both spectra of these commercial HPPE ropes exhibit the four main signals that can be attributed to the polyethylene structure. However, significant additional signals can be identified. The signal at  $1,726\text{ cm}^{-1}$  or  $1,728\text{ cm}^{-1}$  is typically attributed to a C=O stretching vibration, which of course cannot be caused by the polyethylene itself [13]. These HPPE ropes are probably modified or coated to improve their properties, i.e. to improve flame-retardant properties.



Scheme 1: Chemical structure of polyethylene (PE)

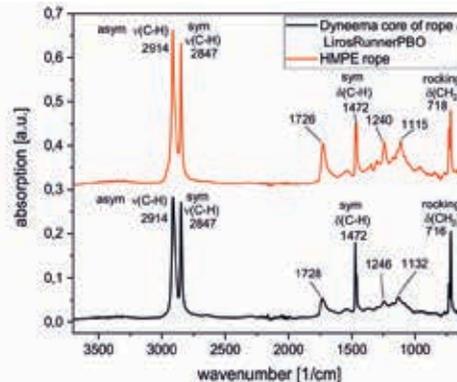
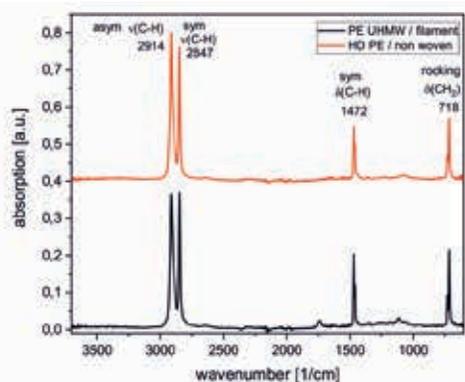
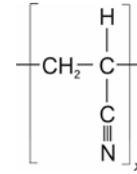


Figure 1: IR-spectra of two different fibre materials made from polyethylene (PE) (left) and IR-spectra of commercially available ropes based on HPPE (right)

#### 4.2 Polyacrylonitrile, PAN

Polyacrylonitrile (PAN) is the polymer used for the production of acrylic fibres. Besides polyester and polyamide fibres, acrylic fibres are the third important conventional chemical fibre and are used for a broad range of garments. In addition to this use for conventional clothing purposes, PAN fibres are also used as technical fibres, in particular because of their strength, their high stability against chemicals, and their light and weathering properties [28]. Applications include sun shades, tents or as filter materials [21, 28]. PAN fibres from high molecular weight polyacrylonitrile (UHMW-PAN) are used for the production of carbon fibres [29]. One disadvantage of PAN fibres is their high flammability as indicated by a LOI value of 17 (see Table 1) [20]. The chemical structure of polyacrylonitrile is shown in Scheme 2. The structure shown is a homopolymer, which is only built up by one type of monomer unit (the acrylonitrile unit). The main technical application of fibres from homopolymer PAN find is as filter materials for the filtration of hot and aggressive gases. In contrast, conventionally used acrylic fibres are not made from homopolymer PAN. In addition to an acrylonitrile monomer unit, they also contain other building units, up to a ratio of 50%. Fibres made from these copolymers are also referred to as acrylics or modacrylic fibres depending on the ratio of the co-unit. They are used in the garment industry. Copolymers of PAN are also used as filters for technical purposes [15].

The IR-spectra of a PAN homopolymer and PAN copolymer are compared in Figure 2. Both samples are taken from non-woven materials used for filtration purposes. Besides the C-H stretching vibration, the IR-spectra also indicate the signal at  $2,241\text{ cm}^{-1}$ , which is attributed to the stretching vibration of the CN triple bond. An IR-signal in this range of the spectrum is quite unique for fibre materials and can be used for the simple detection of PAN fibres [13, 30]. The finger print areas of both spectra are clearly different (Figure 2). The spectrum of the PAN copolymer shows a strong signal at  $1,735\text{ cm}^{-1}$  and an additional peak at  $1,635\text{ cm}^{-1}$ . These signals could be the result of the co-unit in the copolymer. The signal at  $1,735\text{ cm}^{-1}$  is probably caused by a C=O stretching vibration, which could, for example, be part of an ester group in a co-monomer unit. The signal at  $1,635\text{ cm}^{-1}$  can be attributed to the solvent dimethylformamide (DMF). DMF is frequently used in wet spinning processes for PAN fibre production and can remain as an impurity in PAN fibres after the spinning process.



Scheme 2: Chemical structure of polyacrylonitrile (PAN) (homopolymer)

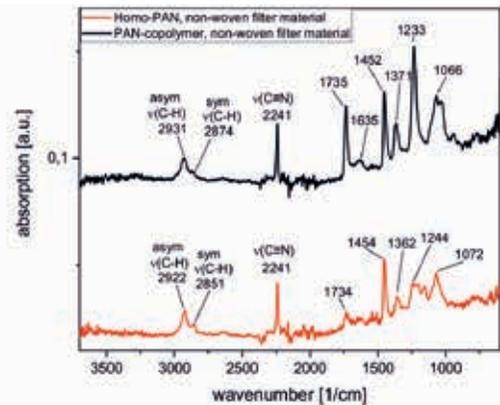


Figure 2: Two IR-spectra of polyacrylonitrile (PAN). Comparison of PAN homopolymer and PAN copolymer; both materials are used as non-woven for filter applications.

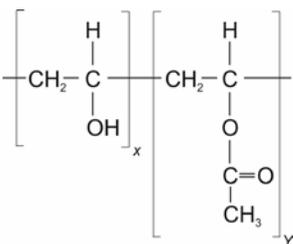
#### 4.3 Polyvinylalcohol, PVAL

Similar to PAN fibres, polyvinylalcohol fibres (PVAL) also exhibit a certain strength and stability against different chemicals [33]. Similar to PAN fibres, PVAL is represented by a simple chemical structure. In most cases, however, PVAL fibres are built up by a copolymer (see Scheme 3). Depending on the composition of the copolymer, a distinction must be made between water-insoluble and water-soluble PVAL fibres. Moreover, water-soluble types are distinguished by the temperature of water, which is necessary to produce the fibres. The copolymeric structure of PVAL fibres is the result of the production process of this polymer. Monomer vinylacetate is initially polymerized into polyvinylacetate. In the second step, re-esterification is performed and alcohol groups are formed in the polymer chain. Finally, the degree of re-esterification determines the ratio of both types of monomer units shown in Scheme 3.

Applications of water-soluble PVAL fibres include water-soluble sewing yarns or fibre felts, where removal after the production process is desired. PVAL

sewing yarns are also used in the medical field. These medical sewing yarns have the ability to decompose after a wound is healed and the yarn is no longer needed. Water-insoluble PVAL fibres find applications in the garment industry, where the chemical and mechanical stability of products are required. Also, a modified flame-retardant PVAL fibre was presented at the TechTextil 2019 fair in Frankfurt (Germany) and is supplied under the brand name Vinaryl. The supplier of this flame-retardant fibre product emphasizes chemical stability against acids and alkali relative to the lower stability of other FR fibres, such as aramid or FR-rayon.

The IR-spectra of two different water-soluble PVAL fibre products are compared in Figure 3. A sewing yarn and a fibre felt are compared. The IR-spectra of both materials have nearly the same shape. However, the maxima of some signals appear at different wavenumbers. The IR-spectrum of a water-soluble PVAL sewing yarn exhibits strong signals at  $3,339\text{ cm}^{-1}$  and  $2,909\text{ cm}^{-1}$ . These signals are related to O-H and C-H stretching vibrations, respectively. The strongest signal at  $1,085\text{ cm}^{-1}$  is attributed to the C=O stretching vibration. A signal at  $1,726\text{ cm}^{-1}$  can be attributed to a C=O stretching vibration [13]. This C=O bond is



Scheme 3: Chemical structure of polyvinyl alcohol (PVAL) with a co-monomer unit of vinylacetate

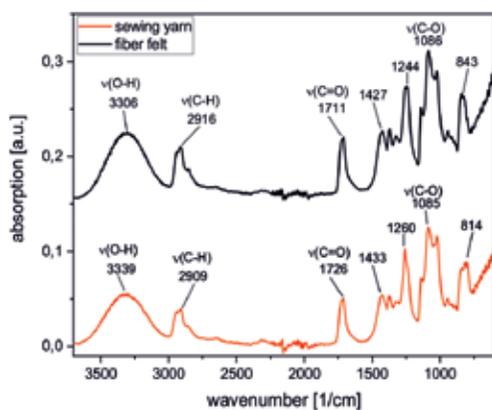


Figure 3: IR-spectra of two water-soluble fibre materials made from polyvinylalcohol. A sewing yarn and a fibre felt are compared.

probably part of the acetyl group of the comonomer unit in the PVAL structure.

#### 4.4 Polyesters and related materials

##### 4.4.1 Polyester types

Polyesters are generally polymers in which monomer units are connected by ester groups. An overview of some polyester types and their main properties is given in Table 2, in which a comparison with polyetheretherketone (PEEK) is also given. Polyethylene terephthalate (PET), as the most prominent polyester, enjoys the highest global market share in terms of the production of synthetic fibres [1]. Beside conventional PET fibres, there are several other types of polyester fibres with special, high-performance properties. An overview of some examples is given in Scheme 4. The polyester type with a structure most similar to PET is polybutylene terephthalate (PBT). In the case of PBT, the ethylene unit is replaced by a longer butylene unit. Because of this structural difference, fibres from PBT contain a higher elongation at break and higher flexibility than conventional PET fibres.

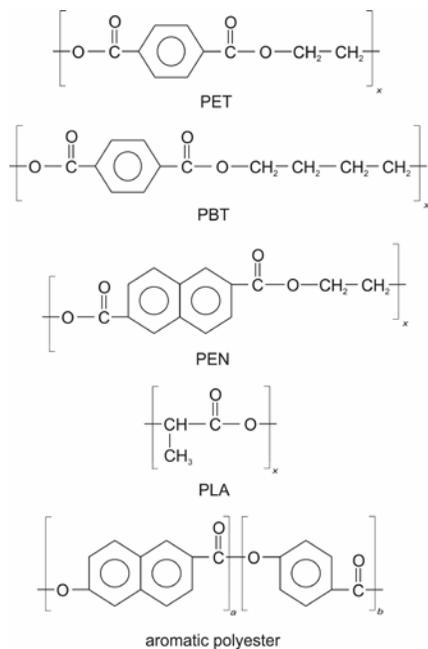
Poly(lactic acid) (PLA) is a biobased aliphatic polyester because the monomer unit lactic acid is produced through the fermentation of starch. From this it follows that PLA is usually produced through the ring-opening polymerization of the cyclic dimer of lactic acid [34–36]. PLA fibres are biodegradable and bioresorbable. Compared to PET fibres, this is a major advantage for fibre applications where a limited life-time is required. A prominent example is sewing yarn used in the medical sector, where the yarn must be degraded after a wound is completely healed [37]. However, PLA fibres are less biodegradable than natural fibres or fibres made from regenerated cellulose or protein.

High-performance materials with advanced properties include polyesters that are built up by two monomer units, both with an aromatic structure. An example of an aromatic polyester is shown in Scheme 4. Fibres from this aromatic polyester are distributed under the tradename Vectran. These fibres exhibit significantly higher thermal stability and stronger flame-retardant properties than conventional PET (see Table 2). Also, due to the aromatic nature of the polymer chain, aromatic polyester is a stiff polymer rod that is, in some ways similar, to para-aramid. For this, high strength materials are produced from Vectran fibres, including ropes and lines [38].

The IR-spectra of both PET and PBT polyester fibre materials are compared in Figure 4. The strong signals at  $1,713\text{ cm}^{-1}$  and  $1,240\text{ cm}^{-1}$  are related to

Table 2: Overview of some properties of different types of polyester and polyetheretherketone (PEEK).  $T_G$ ,  $T_M$  and LOI values, as reported from different literature sources, are compared.

Polymer type	Glass temperature $T_G$ (°C)	Melting temperature $T_M$ (°C)	LOI	Comments regarding selected properties	References
PET	74/80–85	253–260	21	Low cost, conventional polyester	[21, 22, 39–41]
PBT	20–40/ 47–52	212–220/ 221–226	20	Higher elasticity	[41–45]
PLA	54	170–175	20	Bio-based	[21, 46, 47]
Vectran; aromatic polyester	At 260 loss of crystal order	350	28/30	Rigid-rod polymer	[48–50]
PEEK	146–157	334	28/35	Thermal stability	[21, 23, 51]



Scheme 4: Chemical structures of different polyester types used for fibre production

the ester groups with the stretching vibration of the C=O bond and the C-O-C unit. The weak signal at  $2,968 \text{ cm}^{-1}$  is in line with the C-H stretching vibration [52]. All main signals of both materials occur at the same positions in the spectra. Because of the similar structure of both polyester types, the shape of their IR-spectra is nearly the same. Only in the fingerprint area, a few small features appear that are different.

As compared in Figure 4, the IR-spectrum of a flame-retardant polyester (from PET) is also presented. This material is supplied under the tradename Trevira FR and its flame-retardant properties are, according to supplier information, based in part on the phosphorous component, which is part of the polymer structure [53]. The IR-spectrum of this flame-retardant polyester is nearly the same as the spectrum of PET. The wavenumbers differ by only a few signals in a small range. Here, it can be stated that it is not possible to identify the flame-retardant component using IR-spectroscopy.

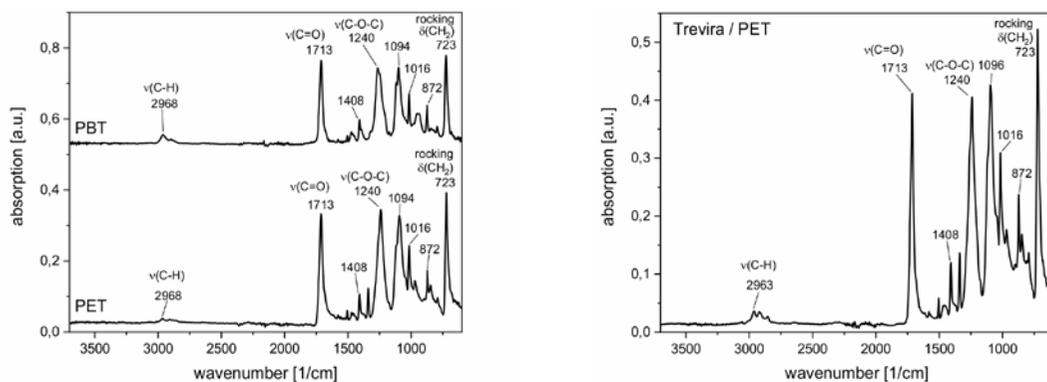


Figure 4: IR-spectra of two different polyester fibre materials made from polyethylene terephthalate (PET) and polybutylene terephthalate (PBT) (left) and the IR-spectrum of the commercially available fire-retardant polyester Trevira (right)

The spectra of the other two polyester fibre materials (PLA and Vectran) are presented in Figure 5. Both spectra are dominated by the strong signal related to the stretching vibration of the C=O double bond. For the PLA fibres, the C=O signal is detected at  $1,748\text{ cm}^{-1}$ , while this signal for the Vectra fibre occurs at a lower wavenumber of  $1,726\text{ cm}^{-1}$  [54, 55]. This appearance at a smaller wavenumber may be related to a weaker C=O bond in the Vectra fibre relative to the stronger C=O bond in PLA. The C=O bond is probably weakened due to the delocalization of the binding electrons of the C=O double bond in the attached aromatic ring systems. In contrast, no delocalization of bonding electron is possible in the aliphatic polyester PLA. In the IR-spectrum of PLA, the strongest signal appears at  $1,086\text{ cm}^{-1}$ , which is attributed to the stretching vibration of the C-O-C unit [55]. For both fibre types (PLA and Vectran), the signals according to the C-H stretching vibration are weak. For PLA fibres, these signals appear at  $2,947\text{ cm}^{-1}$  and  $2,997\text{ cm}^{-1}$ . The appearance of these signals in a range below the wavenumber of  $3,000\text{ cm}^{-1}$  is typical for C-H units, which are part of the aliphatic structure. In contrast, the signal of Vectran appears at  $3,071\text{ cm}^{-1}$ . This position in a range higher  $3,000\text{ cm}^{-1}$  is typical for a C-H unit that is part of an aromatic ring system.

For comparison purposes, the IR-spectrum of commercially available Vectran rope is also shown in Figure 5. To protect Vectran fibres in the rope, a block coating is present on the rope's surface. This coating should protect the Vectran against mechanical influences, but probably more important is protection against light and weathering. Vectran exhibits an aromatic polymer structure with a large conjugated

aromatic system. For this reason, it has the ability to take up UV-light easily and is this resistant to decomposition by sun light.

The IR-spectrum of the Vectran rope exhibits the same main signals at  $1,726\text{ cm}^{-1}$  and  $1,233\text{ cm}^{-1}$  as the other Vectran sample. The most prominent difference is the appearance of signals at  $2,851\text{ cm}^{-1}$  and  $2,918\text{ cm}^{-1}$ , which are probably related to the C-H stretching vibration of the aliphatic components. The broad signal at  $3,312\text{ cm}^{-1}$  could be attributed to a stretching vibration of an O-H bond or a N-H bond. From this it can be assumed that the black coating on the surface of the Vectran rope is based on Nylon.

#### 4.4.2 Polyetheretherketone (PEEK)

Fibres produced from polymer polyetheretherketone (PEEK) can be referred to as polyester-related material, even if the chemical structure of PEEK does not contain any ester group (Scheme 5). The properties and applications of PEEK fibres are quite similar to those of conventional PET fibres. However, due to its higher glass and melting temperature, the maximum temperature of usage of PEEK fibres is nearly  $100\text{ }^{\circ}\text{C}$  higher than that of PET fibres (see Tables 1 and 2) [23, 51]. For this reason, PEEK fibres are thermally more stable and flame-retardant than PET fibres. Also, the chemical stability against acids and alkaline solutions of PEEK fibres is better than that of PET fibres because of the stronger hydrolysis stability of ether groups in PEEK fibres relative to the ester groups of PET fibres. Besides PEEK, there are also other types of polyether ketones with different chemical structures, depending on the different number of keto- and ether-groups in the repeating unit of the polymer. The chemical structure of PEEK is shown

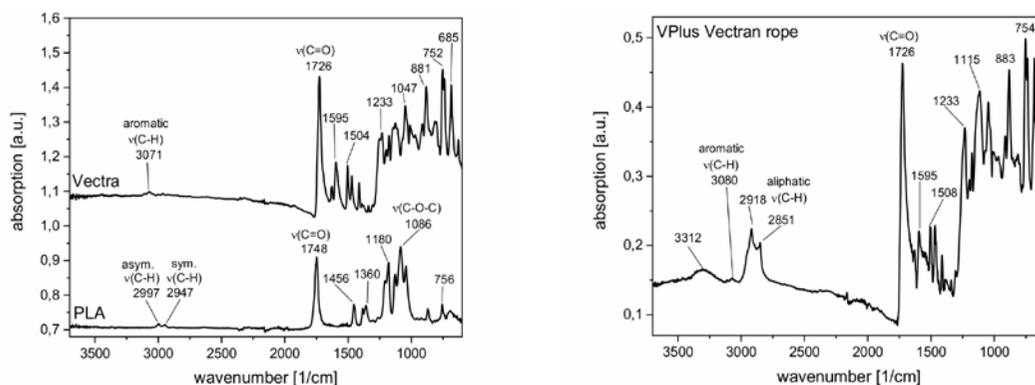
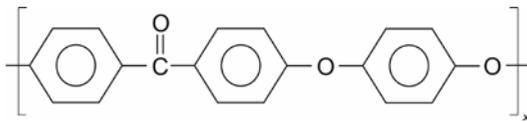


Figure 5: IR-spectra of two different polyester fibre materials made from an aromatic polyester supplied under the brand name Vectra and polylacticacid (PLA) (left) and the IR-spectrum of a commercially available Vectran rope (right)

in Scheme 5, and contains three benzene rings, two ether groups and one keto group in each repeating unit.

The IR-spectrum of PEEK is recorded from a PEEK monofilament (Figure 6). The weak signal at  $3,067\text{ cm}^{-1}$  is related to the C-H stretching vibration of C-H as part of the aromatic ring systems.

The signal at  $1,654\text{ cm}^{-1}$  is attributed to the C=O stretching vibration of the keto group. Signals at  $1,489\text{ cm}^{-1}$  and  $1,593\text{ cm}^{-1}$  can be attributed to the C=C vibration in the aromatic ring system. The strong vibration at  $1,223\text{ cm}^{-1}$  is related to the C=O stretching vibrations in the ether group [56]. The signal at  $833\text{ cm}^{-1}$  can be attributed to C-H vibration of two neighbouring hydrogen atoms that are placed at the benzene ring. This position and signal are typical for benzene rings with a 1.4 substitution [13].



Scheme 5: Chemical structure of polyetheretherketone (PEEK)

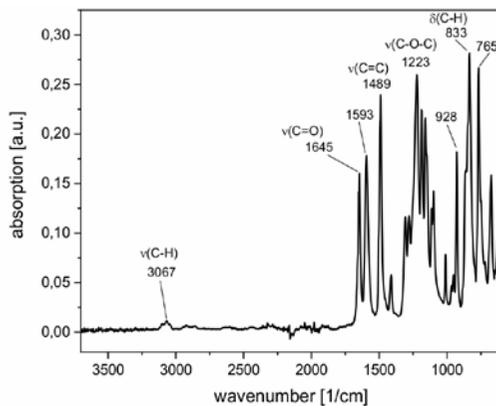


Figure 6: IR-spectrum of polyetheretherketone (PEEK)

## 4.5 Polyamides and related materials

### 4.5.1 Polyamide types

Polyamides are polymers in which monomer units are connected by amide groups. The chemical structure of three prominent polyamides is depicted in Scheme 6. Shown is the polyamide 6.6, PA6.6, as an example of an aliphatic polyamide. PA6.6 is also distributed as a conventional fibre under the name Nylon. Para-aramid and meta-aramid are shown as examples of aromatic polyamides. The related fibre products are distributed under different tradenames,

including Kevlar and Twaron for para-aramid, and Nomex for meta-aramid.

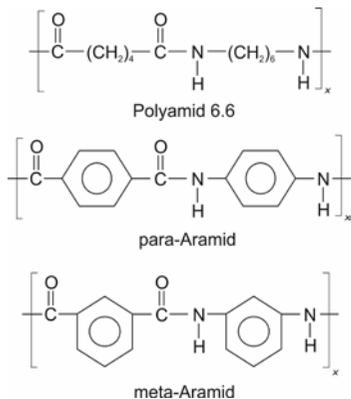
Polyamide 6.6 is built up by two monomer units, each containing six carbon atoms. Also available on the market are aliphatic polyamides with larger monomer units, including polyamide 6.10 or polyamide 10.10. Products in this area are supplied, for example, under the tradename Vestamid by Evonik. Besides other properties, fibres made from these special polyamides demonstrate higher flexibility, lower water up-take, better chemical resistance and lower melting points than conventional PA6.6 fibres. [57].

Aromatic polyamides such as para-aramid or meta-aramid are high-performance fibres with flame-retardant properties and LOI values in the range of 28 to 32 (see Table 1) [23]. Para-aramid is built up using rigid polymer rods. These rods are strongly connected with each other by hydrogen bridge bonds. As a result of this structure, fibres from para-aramid exhibit high strength and are used, for example, in ballistic protective textiles, high strength ropes or as reinforcement fibre in composite materials [23]. Meta-aramid has a lower strength than para-aramid, and is used for applications where a good chemical resistance is required [23]. There are also polyamides on the market containing an aromatic and an aliphatic monomer unit, such as the PA9T fibre supplied by Kuraray [58].

The IR-spectra of polyamide 6.6 and both the aromatic polyamides para-aramid and meta-aramid are compared in Figure 7. These spectra are dominated by signals typical for amide groups. The N-H stretching vibration is related to a signal at around  $3,300\text{ cm}^{-1}$ . Further, the amide groups are related to several signals in the IR-spectra, referred to as Amid I, Amid II and Amid III [14]. The Amid I signal is caused by the C=O stretching vibration of the C=O double bond in the amide group. Amide II is attributed to a combination of the stretching vibration of the C-N bond and a deformation vibration of the C-N-H unit [53]. Some publications assign a second signal of around  $3,086\text{ cm}^{-1}$  according to the overtone of the Amid II vibration [14].

For para-aramid, the Amid II signal at  $1,537\text{ cm}^{-1}$  appears as a double peak related to the C-N stretching vibration and N-H bending vibration [59]. For secondary amides, the weak Amide III signal in the range from  $1,200$  to  $1,330\text{ cm}^{-1}$  can also be attributed for polyamides. This Amide III signal is caused by a combination of the deformation vibrations from the N-H and OCN groups [14]. For PA6.6 fibres, the sig-

nals attributed to C-H stretching vibrations are of average intensity. The signals from the C-H stretching vibration are very weak compared to para-aramid and meta-aramid. For para-aramid, the signal at  $820\text{ cm}^{-1}$  can be attributed to the C-H deformation vibration of the two neighbouring hydrogen atoms that are placed at the benzene ring. This position and signal are typical for a benzene ring with a 1.4 substitution [61]. Analogously for meta-aramid, the signal at  $779\text{ cm}^{-1}$  can be attributed to the C-H deformation vibrations typical for a 1.3 substitution at the benzene ring [13].



Scheme 6: Chemical structures of polyamide 6.6, PA 6.6 (Nylon), para-aramid and meta-aramid

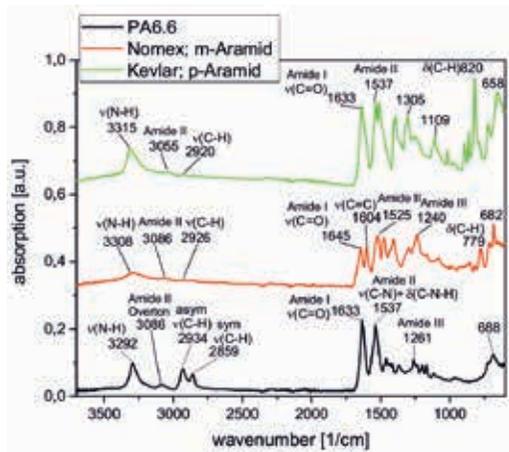


Figure 7: IR-spectra of three different polyamide fibre materials made from polyamide 6.6, para-aramid and meta-aramid

#### 4.5.2 Protein-based regenerated fibres

Similar to synthetic polyamides, protein-based fibres are also built up by polymers, in which monomer units are connected by amide groups. In contrast to synthetic polyamides, however, the monomer units of proteins are amino acids. Protein fibres are mainly

animal fibres such wool or silk. It is also possible to produce regenerated protein fibres from proteins of different sources such as soya (plant source) or casein from milk (animal source). The development of such protein-based regenerated fibres is quite old and even reported in patents from the 1940s [62, 63]. Today, particularly “milk fibre” is promoted as a kind of sustainable alternative fibre [64]. However, soya fibre has also made a comeback in recent years [41]. A recent and very innovative approach is the use of the biotechnological production of spider silk proteins for fibre production. These spider silk fibres exhibit a significantly higher elasticity than conventional silk fibres [65].

The IR-spectra of two different types of regenerated protein fibres are compared in Figure 8 using the IR-spectrum of mulberry silk as a reference for a natural fibre. The prominent signals related to the N-H stretching vibration and for Amid I and Amid II can be clearly identified for all three types of fibres, even if the signals appear at different positions on the spectra. The IR-spectra of these protein fibres exhibit the same main features as the spectra of the synthetic polyamides (compare Figures 7 and 8). Completely different are the compared IR-spectra of protein fibres in the fingerprint range below  $1,400\text{ cm}^{-1}$ . This difference is related to the different composition of these protein fibres. These protein fibres are built up using different combinations of amino acids containing different functional groups. Silk protein is known to contain a high ratio of the simplest natural amino acid glycine. In contrast, casein and proteins from soya are built up using a higher variety of different natural amino acids [66].

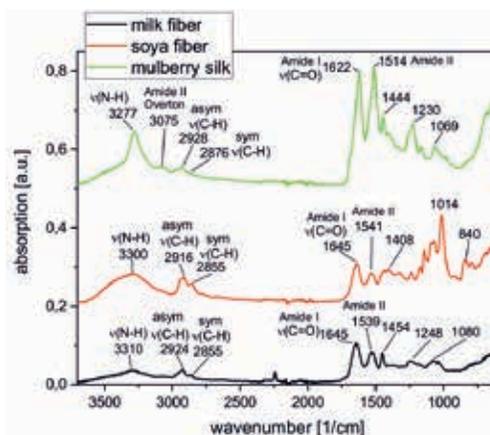


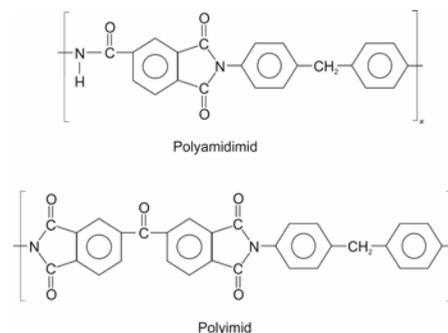
Figure 8: IR-spectra of three different protein-based fibre materials: mulberry silk and regenerated fibres from soya and milk proteins

#### 4.5.3 Polyamide-imide, PAI & Polyimide, PI

Polyimides (PI) are polymers in which monomer units are connected by imide groups. The structure of one commercially available polyimide is presented in Scheme 7. This polyimide is used for the production of high-performance fibres with excellent temperature stability. The maximum continuous working temperature is reported at 260 °C [67]. For this reason, the thermal stability is in the same range as that of PTFE fibres. Applications for polyimide fibres are found in filter materials for hot gases. The flame-retardant properties of polyimide fibres are also excellent. Thus, polyimide fibres are used in blended fabrics for fire-retardant clothes. Of course, there are different types and chemical structures possible for polyimides. One very prominent example is the polyimide shown in Scheme 7, which is also distributed under the tradename P84 [67]. Polyamide-imides (PAI) are polymers in which monomer units are connected by amide groups and imide groups. Some authors categorize polyamide-imide fibres to aramid fibres, in particular to meta-aramid because of some similarities in the structure. However, there is a significant chemical difference between meta-aramid and fibres from polyamide-imide, i.e. the presence of the imide group in PAI fibres. The structure of a polyamide-imide, which is used for production of a commercially available fibre, is presented in Scheme 7. This fibre is also distributed under the tradename Kermel and is promoted in particular for its flame-retardant properties [68]. Products are found often with blended fabrics, such as flame-protective underwear and workwear.

The IR-spectrum of polyimide fibres from type P84 is shown in Figure 9. This IR-spectrum from polyimide fibre is compared to the spectrum from polyamide-imide taken from a bulk sample. The spectrum of polyimide fibre is dominated by a strong signal at 1,715 cm<sup>-1</sup>, which is related to the stretching vibration of a C=O double bond. A second signal at 1,776 cm<sup>-1</sup> can also be attributed to the stretching vibration of a C=O bond. In fact, the structure of P84 exhibits two different types of C=O groups in the monomer unit (see Scheme 7). One C=O bond is part of a keto group, while the other four C=O bonds are parts of the imide groups. This explains the appearance of two signals for two different types of C=O bonds. Another explanation of these two signals is the assignment to the two different vibrations (symmetric and asymmetric) of the imide group. Imides, as part of five-membered aromatic rings, have been found to exhibit two signals around 1,700 cm<sup>-1</sup> and 1,770 cm<sup>-1</sup> [13]. Both of these signals relate to the symmetric and asymmetric stretching

vibrations of the two C=O bonds of the same imide group. Also, the IR-spectrum of polyamide-imide exhibits two signals at 1,709 cm<sup>-1</sup> and 1,776 cm<sup>-1</sup>, which can be attributed to the stretching vibrations of C=O bonds as part of the imide group, similar to the spectrum of polyimide. The signal for the C=O stretching vibration of the amide group usually appears at a lower wavenumber, while the signal at 1,658 cm<sup>-1</sup> can probably be attributed to the amide group. In contrast, the C-H stretching vibrations lead only to weak signals in the IR-spectrum of the polyimide P84. The signal at 3,057 cm<sup>-1</sup> can be attributed to the C-H vibration of H-atoms bonded to the aromatic ring systems. The other signal at 2,924 cm<sup>-1</sup> can be attributed to the C-H vibration of H-atoms that are part of the -CH<sub>2</sub>- unit. The signal at 718 cm<sup>-1</sup> can be attributed to the deformation vibration of the -CH<sub>2</sub>- unit. For the polyamide-imide sample, only a very weak signal at 2,924 cm<sup>-1</sup> can be identified, and can be attributed to the C-H stretching vibration.



Scheme 7: Chemical structures of a polyamide-imide (PAI) and a polyimide (PI). These types of polymers are used for fibre production. The PAI shown is distributed as a fibre under the tradename Kermel. The PI shown is distributed as a fibre material under the tradename P84 by the European company Evonik.

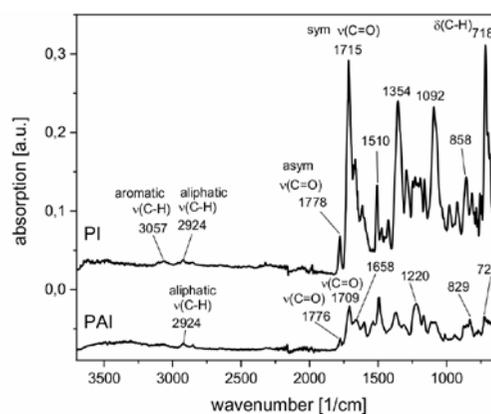


Figure 9: IR-spectra of the polyimide fibre P84 and a polyamide-imide bulk sample

#### 4.5.4 Elastane fibres

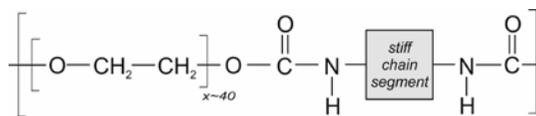
Elastane fibres are highly elastic synthetic fibres, and are supplied under the tradenames Lycra and Spandex. Elastane is broadly used in the clothing industry at ratios ranging from around 3 to 5 wt-% to increase the flexibility and the body fitting of garments. Elastane fibres can be referred to as high-performance fibres and as a polyamide-related material. The term high-performance traditionally relates to strong fibre stability against different parameters. In the case of elastane, however, the high-performance property is strong elasticity. The chemical structure of elastane is schematically presented in Scheme 8. This polymer is built up using repeating units containing two parts: a flexible polyethyleneoxide chain of around 40 ethyleneoxide units, and a stiff chain segment built up by connecting several aromatic ring systems and urethane groups [69]. This stiff segment is strongly bonded to the stiff segments of other elastane polymers inside the monofilament by hydrogen bridges. In some ways, this is similar to the attractive interaction between the polymer chains in Nylon or aramid fibres. In contrast, the polyethylene oxide unit forms a random polymer coil without strong attractive bonds to other neighbouring polymer chains. If a mechanical force pulls the polymer, the polymer coil from the polyethylene oxide is easily stretched to a multiple of its size. In this way, the elastane fibre becomes an elongated manifold. This type of elasticity is also referred to as entropic elasticity and is similarly found in natural rubber [70].

Beside these elastane fibres based on polyurethane/polyethyleneoxide copolymers, other types of elastic polymers and fibre materials have also been invented and discussed. An interesting approach offer polymers containing an inorganic backbone as polysiloxanes or polyphosphazenes [71, 72]. Polyphosphazenes are used in the production of nanofibres related to medical applications, but are also used as elastic polymeric material [73]. Additionally, polyphosphazenes are used for flame-retardant finishing processes [74]. For this reason, they can combine elastic properties with the high-performance property of fire retardance in one fibre material.

Because elastane fibres are only used in small amounts in a mixture with other fibres, it would be advantageous to separate a typical elastane fibre for investigation using IR spectroscopy. There are also different types of elastane fibre on the market, which primarily differ in terms of the construction and composition of the stiff chain segment. For this

reason, it is not possible to identify an IR-spectrum that can be referred to as a typical and original IR-spectrum of elastane.

Figure 10 presents the IR-spectrum of an elastane monofilament that is used as the core filament of a polyester yarn. This elastane core is removed from the polyester yarn and separately measured to identify, in particular, the IR-spectrum of elastane. The weak and broad signal at  $3,323\text{ cm}^{-1}$  can be attributed to the N-H stretching vibrations of the urethane groups. Three strong signals at  $2,801\text{ cm}^{-1}$ ,  $2,877\text{ cm}^{-1}$  and  $2,936\text{ cm}^{-1}$  are caused by the stretching vibrations of C-H bonds. However, the strongest signal is observed at  $1,105\text{ cm}^{-1}$ . This signal probably relates to the C-O stretching vibration of the ether unit in the polyethylene oxide PEO part of the polymer. Due to the size of the PEO unit, the ether group is the most dominant functional group in the elastane structure. The appearance of this strong signal can be deemed typical for elastane fibres. Other signals in the spectral range from  $1,400\text{ cm}^{-1}$  to  $1,732\text{ cm}^{-1}$  can be attributed to various types of C=O and C=C stretching vibrations that are part of the stiff monomer segment.



Scheme 8: Chemical structure of elastane

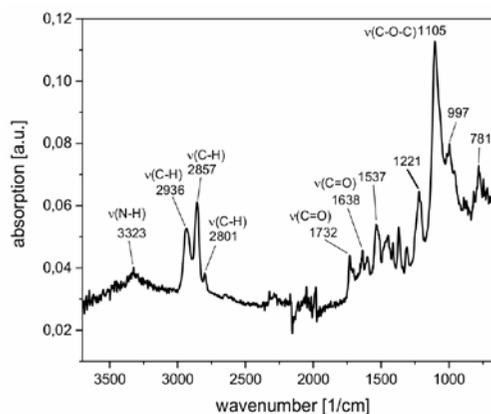


Figure 10: IR-spectrum of an elastic filament yarn

#### 4.6 Halogen-containing polymers

Halogen-containing polymers are polymers that contain at least one halogen atom in their monomer unit. Primarily polymers containing the halogens fluorine F and chlorine Cl are used for fibre production. These halogen-containing polymers are not flammable and exhibit LOI values above 34%, depending on the type

and number of halogen atoms in the monomer units of the polymers. In the case of thermal decomposition, however, combustion gases with toxic halogen-containing compounds are formed, e.g. HCl or HF. The chemical structure of some fluorinated and chlorinated polymers used for fibre production are shown in Scheme 9. The related IR-spectra are presented in Figures 11 and 12. The most important fluorinated polymer is polytetrafluoroethylene (PTFE), which is fully fluorinated and contains four fluorine atoms for each monomer unit. Due to the strong C-F bond, PTFE exhibits the strongest stability against chemicals among all synthetic fibres. At 95%, its LOI value is also the highest of all organic fibres [23]. The main disadvantages of PTFE are a high-cost factor and the lower strength of fibre material. Another unique feature of PTFE fibre material is its oleophobic property [75]. Polyvinylidene fluoride (PVDF) fibres can be used for the production of filter materials. Moreover, the production of piezoelectric PVDF fibres is also possible, with applications found in smart textiles or E-textiles [76]. Fibre materials from polyvinylidene chloride (PVDC) also exhibit excellent fire-retardant properties. Modacrylic modacrylic fibres with a co-unit of vinylidene chloride are frequently used. These fibres are also supplied under the tradename Kanekalon [77–79].

The IR-spectrum of a non-woven filter material made from PTFE fibres is presented in Figure 11. Due to the simple chemical structure of PTFE, its IR-spectrum exhibits only three significant signals. The strong

signals at 1,200  $\text{cm}^{-1}$  and 1,146  $\text{cm}^{-1}$  relate to the C-F stretching vibration, while the signal at 637  $\text{cm}^{-1}$  can probably be attributed to the deformation vibration of the segment  $-\text{CF}_2-$  [80]. The IR-spectrum of a monofilament sieve made from ethylene-tetrafluoroethylene-copolymer (ETFE) is also shown in Figure 11 and compared to the spectrum of PTFE. ETFE is a co-polymer with PE and PTFE units. However, the IR-spectrum of ETFE is not a simple combination of the single spectra of PTFE and PE (compare Figures 1 and 11). For ETFE, only a very weak signal at 2,978  $\text{cm}^{-1}$  is identified according to the C-H stretching vibration. Another signal at 1,452  $\text{cm}^{-1}$  relates to the deformation vibration of the segment  $-\text{CH}_2-$  [81].

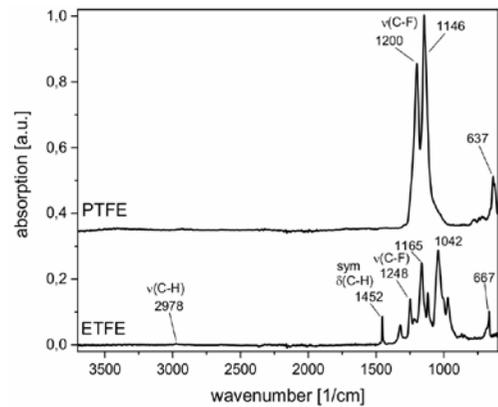
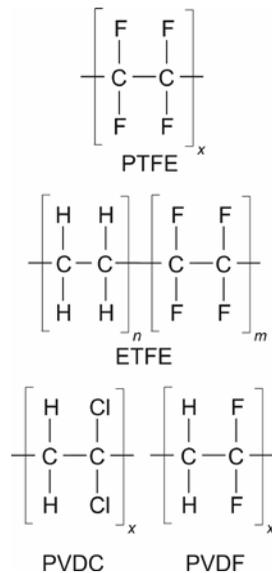


Figure 11: Comparison of the IR-spectra of the materials polytetrafluoroethylene (PTFE) and ethylene-tetrafluoroethylene-copolymer (ETFE)



Scheme 9: Chemical structures of different chlorine- and fluorine-containing polymers used for fibre production

IR-spectra of fibre materials from polyvinylidene fluoride (PVDF) and polyvinylidene chloride (PVDC) are compared in Figure 12. These materials were obtained as sieve fabrics for filter applications. A weak signal at around 3,000  $\text{cm}^{-1}$  is observed and relates to C-H stretching vibrations. For PVDF, a strong signal at 1,163  $\text{cm}^{-1}$  is observed, which can be attributed to the C-F stretching vibration [82]. For PVDC, a signal at 656  $\text{cm}^{-1}$  can be attributed to the C-Cl stretching vibration [83]. In this spectral range, however, other deformation vibrations related to the  $-\text{CH}_2-$  unit can also appear, making it difficult to accurately attribute the signal [13]. The medium signal at 1,743  $\text{cm}^{-1}$  could be caused by the stretching vibration of a C=O bond [13]. Due to the chemical structure of PVDC, there should be no C=O group with a related IR signal. However, chlorinated polymers such as PVDC or PVC are sensitive to photooxidation. For this reason, the appearance of C=O carbonyl groups can be the result of such photooxidative processes [83].

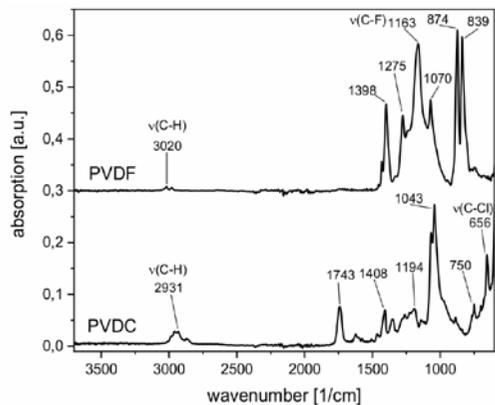


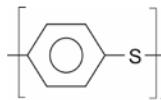
Figure 12: IR-spectra of the materials polyvinylidenefluoride (PVDF) and polyvinylidenchloride (PVDC) in comparison. Both materials were obtained as sieve fabrics for filter applications.

#### 4.7 Polyphenylene sulfide (PPS)

The high-performance properties of polyphenylene sulfide (PPS) fibres can be summarized by two statements: a high chemical stability, which is nearly as good as PTFE fibres, while the chemical stability of PPS fibres against high concentrated acids and alkali is extremely high, even near the boiling point of these liquids. Moreover, most organic solvents cannot damage PPS fibres [21, 23]. PPS fibres are only sensitive to oxidative agents such as hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) or nitric acid ( $\text{HNO}_3$ ). Also, oxygen from the atmosphere can damage PPS fibres at elevated temperatures. These oxidative agents attack the PPS polymer at the sulphur bridge -S- through the oxidation of the -SO- and -SO<sub>2</sub>- units. Filter materials made from PPS are used for the filtration of hot gases and aggressive media. PPS fibre materials are also used as sewing yarns for application with aggressive chemicals. The chemical structure of PPS is quite simple. PPS is the sulphur-containing, high-performance fibre with the greatest commercial relevance. Its chemical structure is depicted in Scheme 10. Other sulphur-containing, high-performance fibres are based on the polymer polysulfonamide (PSA), and are supplied under the tradename Tanlon. PSA fibres are especially known for their fire-retardant properties [84, 85].

An IR-spectrum taken from a filter material made from PPS non-woven material is shown in Figure 13. The weak signal at 3,065  $\text{cm}^{-1}$  can be attributed to C-H stretching vibrations of hydrogen atoms placed at the aromatic ring. The signals at 1,490  $\text{cm}^{-1}$  and 1,568  $\text{cm}^{-1}$  can be attributed to C=C vibrations from the benzene ring [86]. The signal at 804  $\text{cm}^{-1}$  can be attributed to the C-H deformation vibration, which

is typical for a 1,4 substitution of an aromatic ring system with two neighbouring hydrogen atoms [13].



Scheme 10: Chemical structure of polyphenylene sulfide (PPS)

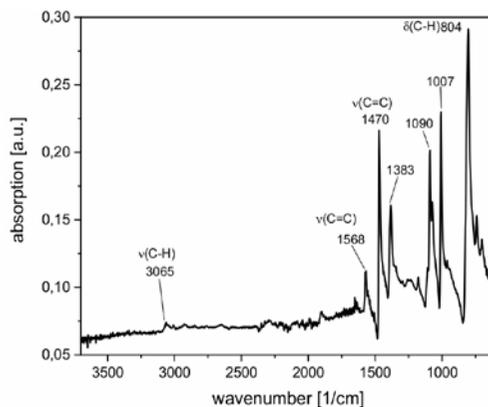


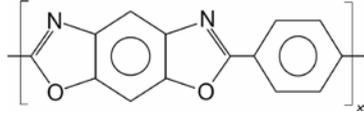
Figure 13: IR-spectrum of polyphenylene sulfide (PPS)

#### 4.8 Polyphenylene- benzobisoxazol (PBO)

Fibres made from polyphenylene-benzobisoxazol (PBO) combine several advantageous properties. The chemical structure of PBO fibres is depicted in Scheme 11. They are built up using an heteroaromatic rigid rod polymer chain. PBO fibres have the highest maximum temperature of usage of all commercially available synthetic fibres. The LOI value of PBO fibres is reported to be in the range of 56 to 68 [21, 23]. This high LOI value is especially remarkable because PBO does not contain any kind of halogen atom in its chemical structure. The mechanical strength of PBO fibres is high, so it is also used for the production of high-strength ropes. PBO fibres are also distributed under the tradename Zylon fibres.

Figure 14 shows the IR-spectrum of a PBO (Zylon) sample that was taken from a two-component rope. The core of the rope is braided from HPPE fibres, while the surface layer is braided from PBO fibres. The weak signal at 3,092  $\text{cm}^{-1}$  can be attributed to C-H stretching vibrations from H-atoms bonded to an aromatic ring system. The signal at 1,620  $\text{cm}^{-1}$  can be attributed to the C=N stretching vibration, which is part of the heteroaromatic system, while the signal at 1,053  $\text{cm}^{-1}$  can be attributed to the C-O stretching vibration of the -C-O-C- unit, which is also part of the heteroaromatic system [87]. The three signals at 1,412  $\text{cm}^{-1}$ , 1,495  $\text{cm}^{-1}$  and 1,568  $\text{cm}^{-1}$  can be

attributed to C=C vibrations from the aromatic ring systems. The signal at  $844\text{ cm}^{-1}$  can be attributed to the C-H deformation vibration, which is typical for a 1,4 substitution of an aromatic benzene with two neighbouring hydrogen atoms [13].



Scheme 11: Chemical structure of Polyphenylene-Benzobisoxazol (PBO), supplied as PBO fibres also under the tradename Zylon

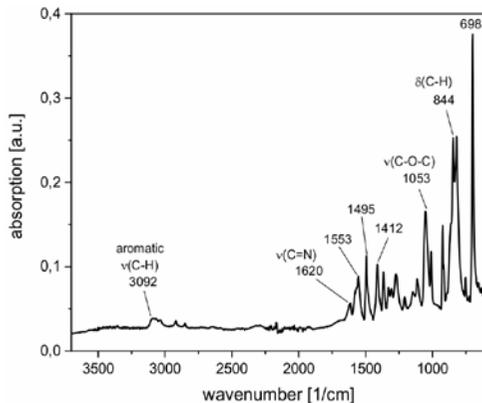


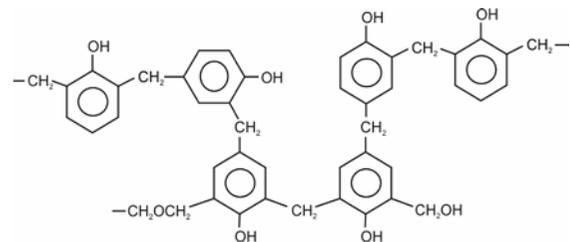
Figure 14: IR-spectrum of polyphenylene-benzobisoxazol fibres (PBO) taken from a braided fibre sample

#### 4.9 Resin-based fibres

Fibres can be also produced from resins. In the category of resin-based fibres, primarily two different types of resin-based fibre should be mentioned: melamine and phenol-based resins. Melamine fibres were previously distributed under the tradename Basofil. They are actually distributed as smart melamine fibres [88, 89]. Fibres from phenol resins are also referred to as novolac fibres and distributed under the tradename Kynol fibres [90]. These fibres exhibit flame-retardant properties, are self-extinguishing in contact with fire and exhibit low smoke formation. Nonwoven materials made from Kynol fibres can also be used as heat insulation material [91]. The chemical structure of resin-based fibres made from Kynol is given in Scheme 12. Resin-based fibres are built up using an irregularly crosslinked three-dimensional network. Textile materials made from Kynol fibres exhibit a typical orange coloration, which can be modified by laser treatment [92].

The related IR-spectrum of a novolac fibre is presented in Figure 15. The broad signal at a maximum of

$3,377\text{ cm}^{-1}$  can be attributed to the O-H stretching vibration of the hydroxy groups. The weak signals between  $2,849$  to  $3,009\text{ cm}^{-1}$  can be attributed to various C-H stretching vibrations. The signal at  $3,009\text{ cm}^{-1}$  can probably be attributed to C-H units that are part of the aromatic benzene rings. Both signals at  $2,916\text{ cm}^{-1}$  and  $2,849\text{ cm}^{-1}$  can be attributed to C-H stretching vibrations from the aliphatic  $-\text{CH}_2-$  unit [13]. The strongest signal at  $1,219\text{ cm}^{-1}$  probably relates to the stretching vibration of C-O bonds. The signals at  $1,601\text{ cm}^{-1}$  and the double signal at  $1,470\text{ cm}^{-1}$  can be attributed to C=C stretching vibrations in the aromatic ring [13]. However, the signal at  $1,470\text{ cm}^{-1}$  can be also attributed to the deformation vibration of the  $-\text{CH}_2-$  unit.



Scheme 12: Chemical structure of a resin-based fibres – novolac resin

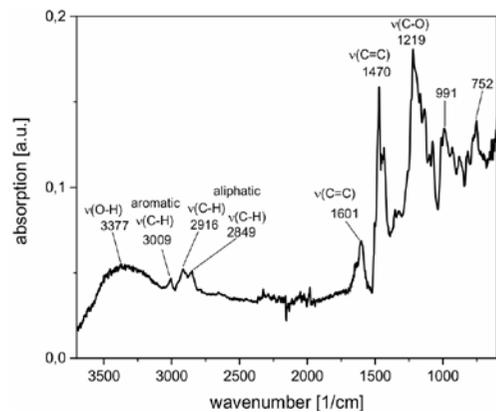


Figure 15: IR-spectrum of fibres made from novolac resin (Kynol fibres)

## 5 Inorganic fibres

Inorganic materials are materials that are not built up using carbon-hydrogen compounds. In accordance with this definition, inorganic fibres are all fibres that are not built up by the chemical elements carbon and hydrogen. The main categories of inorganic fibres are glass fibres, ceramic fibres, metal fibres and carbon fibres [93]. In comparison to organic synthetic

fibres, inorganic fibres contain a high thermal stability up to several hundred °C and even in excess of 1,000 °C even in presence of oxygen. Inorganic fibres from metal oxides are also not flammable and are thus used to produce fibres for high-temperature and flame-resistant applications.

The IR-spectra of some inorganic fibres built up using mixtures of different metal oxides are presented in Figure 16. These fibres were selected for this review because of their technical relevance and the fact that other inorganic fibres, such as metal fibres, SiC fibres and carbon fibres, do not exhibit significant signals in the studied spectral range of infrared light.

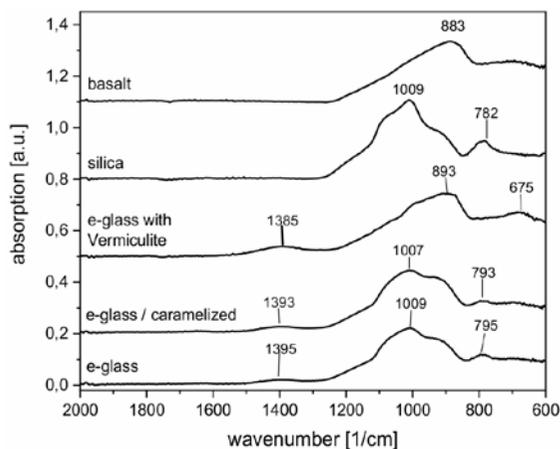


Figure 16: Comparison of the IR-spectra of different inorganic fibres

E-glass fibres are probably the most used glass fibre type and are mainly built up using silica ( $\text{SiO}_2$ ) and alumina  $\text{Al}_2\text{O}_3$  [94]. For E-glass fibres, the dominant signal in the infrared spectrum occurs at around  $1,000\text{ cm}^{-1}$  and can be attributed to the vibration of the group  $-\text{Si}-\text{O}-\text{Si}$  [95]. The second signal at around  $800\text{ cm}^{-1}$  can be also attributed to a  $-\text{Si}-\text{O}-$  vibration [96].

The weak signal at around  $1,390\text{ cm}^{-1}$  has been attributed by some authors to the absorption of other oxides such aluminium oxide or boron oxide [97]. However, this signal more likely relates to the presence of organic sizing agents on fibre surfaces. The surface of glass fibres is usually treated with small amounts of organic compounds to protect the glass fibres and to improve their mechanical properties. This mechanical protection is especially necessary during yarn and fabric production processes. Caramelized E-glass fibres are thermally treated under moderate conditions, so the organic compounds on the glass

fibre surface are partly decomposed. For this reason, a typical brown (caramelized) coloration of the glass fibre appears. The IR-spectra of E-glass fibres and caramelized E-glass fibres are nearly the same.

Beside caramelization, E-glass fibres can be also modified using natural inorganic compounds such as vermiculites. In this case, the IR-spectrum is determined by the composition of the inorganic vermiculite, which is placed on the surface of the E-glass fabrics. Vermiculite is a natural clay mineral that contains silicon and aluminium oxides, as well as magnesium oxides [98, 99]. The IR-spectra of E-glass fabric with vermiculite exhibit a broad signal with a maximum at  $893\text{ cm}^{-1}$  and a second broad signal at  $675\text{ cm}^{-1}$ . In literature, this second maximum at  $675\text{ cm}^{-1}$  is attributed to the vibration of  $\text{Mg}-\text{O}$  or to the stretch vibration of the group  $\text{Al}-\text{O}-\text{Si}$  [98, 99]. In contrast to E-glass fibres, silica fibre fabric only contains  $\text{SiO}_2$ . The IR signal at  $1,009\text{ cm}^{-1}$  is more precise, while there is no signal around  $1,390\text{ cm}^{-1}$ . Basalt fibres are produced by melting natural volcanic rocks (basalt stones). Beside silica and alumina, basalt fibres also contain significant amounts of other metal oxides, e.g. from iron or titanium [94, 100–103]. Basalt fibres can be also used as a coating additive to introduce UV-protective properties [103]. Due to the presence of iron oxide, basalt fibres exhibit a typical brown coloration. The IR-spectra of the basalt fibre presented in Figure 16 was recorded from a commercially available monofilament. This IR-spectrum shows one broad signal with a maximum at  $883\text{ cm}^{-1}$ . In comparison with the IR-spectra of basalt fibres reported in literature, this maximum appears at a lower wavenumber than in articles reporting a maximum of between  $1,000\text{ cm}^{-1}$  and  $1,100\text{ cm}^{-1}$  for basalt fibres [100, 104]. However, it should be clear that the IR-spectrum of a commercial basalt fibre product is influenced by the broad range of possible basalt composition and by the possible treatment of the fibre surface [100, 104].

## 6 Conclusions and future perspectives

In addition to conventional fibres (such as polyester (PET), polyamide (Nylon), cotton, etc.), there are many different high-performance fibres on the market that exhibit outstanding properties, giving them an advantage over conventional fibres. Of course,

it is not possible in the scope of this review to present information regarding each fibre type. For this reason, the focus was mainly on high-performance fibres with a significant commercial importance. The production rate of high-performance fibres is quite low compared to conventional fibres. Nevertheless, several high-performance fibres are of significant economic relevance, and are essential for many products and technical processes.

There is one main question for the future perspectives of high-performance fibres: Can new high-performance fibres establish a foothold on the market in the future? Actual trend materials in this respect are most likely fibres made from carbon nanotubes (CNTs) or graphene compounds [105–107]. Also, fibres developed using a bionic approach, such as spider silk, could gain in importance in the future. Another possible future trend could also be the reinvention and commercialization of fibre types developed in the past. Current examples of this trend are found for regenerated protein fibres or melamine-based resin fibres. Another possible candidate for reinvention could be fibres made from inorganic polymers, especially polyphosphazenes. A higher elasticity than elastane fibres has been reported for these fibres. Due to the high content of phosphorous and nitrogen, these fibres also exhibit flame-retardant properties. Such a combination of properties has made them highly interesting, especially as additive fibres in fire-protective garments [73, 108].

It should be clear, that market demand for high-performance fibres will continue to be high in the future, and there will, of course, be new material developments leading to advantageous materials with surprising properties.

### Acknowledgements

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## Modelling the Effect of Resin-Finishing Process Variables on the Dimensional Stability and Bursting Strength of Viscose Plain Knitted Fabric Using a Fuzzy Expert System

*Modeliranje vpliva spremenljivk postopka plemenitenja s smolo na dimenzijsko stabilnost in razpočno trdnost viskoznega pletiva z uporabo sistema mehke logike*

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### Abstract

The application of cross-linking resin is an effective method for improving and controlling dimensional stability, such as the shrinkage of viscose single jersey knits. However, such treatment often leads to a significant deterioration in the bursting strength of treated fabrics. In this regard, resin treatment using a softening agent can be an additional potential solution for retaining the bursting strength of treated fabrics. Resin treatment is one kind of chemical finishing process that inhibits cellulosic textile fibre swelling during wetting, provides fibre resistance to deformation and prevents shrinkage. The key objective of this study was to model the effect of resin-finishing process variables for predicting the shrinkage control and bursting strength of viscose single jersey knitted fabrics. The MATLAB (Version 8.2.0.701) fuzzy expert system was used to model the optimum resin and softener concentrations, as well as the best curing time for the prediction of maximum shrinkage control with a minimum loss in fabric bursting strength. The optimal process variables were found to be a resin concentration of 75 g/l, a softener concentration of 45 g/l and a curing time of 225 seconds. The fuzzy expert model developed in this study was validated using experimental data. It was found that the model has the ability and accuracy to predict fabric shrinkage and bursting strength effectively in the non-linear field.

Keywords: cellulose, modelling, FES model, fuzzy inference, dyeing, textiles

### Izvleček

Uporaba zamreževalne smole je ena izmed učinkovitih metod za izboljšanje in nadzor dimenzijske stabilnosti, kot je krčenje viskoznih levo-desnih pletiv. Obdelava s smolo je kemični postopek, ki zavira nabrekanje celuloznih tekstilnih vlaken v mokrem in zagotavlja odpornost vlaken proti deformiranju ter zavira krčenje. Vendar takšna obdelava po-

gosto močno poslabša razpočno trdnost obdelanih tkanin. Uporaba mehčalca je lahko ena od rešitev za ohranjanje razpočne trdnosti z zamreževalno smolo obdelanih pletiv. Ključni cilj raziskave je bil modelirati vplivne spremenljivke plemenitilnega postopka s smolo za napovedovanje nadzora krčenja in razpočne trdnosti enojnih pletiv iz viskoznih vlaken. Za napovedovanje nadzora maksimalnega krčenja ob minimalnem znižanju razpočne trdnosti pletiva je bil uporabljen mehki ekspertni sistem @ MATLAB (različica 8.2.0.701), s katerim so bili modelirani optimalni koncentraciji smole in mehčalca ter najugodnejši čas zamreževanja. Ugotovljene optimalne spremenljivke procesa so koncentracija smole 75 g/l, koncentracija mehčalca 45 g/l in čas strjevanja 225 s. Razviti mehki ekspertni model je bil potrjen z eksperimentalnimi podatki. Ugotovljeno je bilo, da ima model zmožnost in natančnost predvidevanja krčenja in razpočne trdnosti pletiva v nelinearnem območju.

Ključne besede: celuloza, modeliranje, model FES, mehko sklepanje, barvanje tekstilij

## 1 Introduction

Viscose is the first and foremost the oldest regenerated cellulose fibre produced using the wet spinning process. Viscose, however, is the most absorbent and highly reactive among all cellulose fibres. Globally speaking, knitted fabrics made of viscose fibres are very popular for fashionable apparel because of their lower price and amazing quality characteristics, such as rich brilliant colour, superior moisture absorbance, wear comfort, softness against the skin, and easy-care properties relative to cotton knit and woven fabrics [1–5]. However, poor dimensional stability is a well-known concern in viscose knitwear, even after decades of development in modern manufacturing methods [6]. Although almost all single-knit structures demonstrate a major propensity to shrink, single-knit structures made of viscose fibre are subject to extreme shrinkage, mainly in terms of length, because of its relatively lower crystalline and more amorphous structure than that of cotton fibre [1–3, 7]. Many studies have reported improvements in the poor dimensional stability of knitted fabrics. Reeves and Frank proposed that the shrinkage of cotton knitted fabrics could be reduced by any treatment that prevents cotton fibre swelling during wetting [8]. Candan and Onal reported that knitted fabrics made from open-end rotor yarns exhibit better dimensional stability than those made from ring-spun yarn [9]. However, open-end rotor yarns are not usually available in higher fineness and result in lower bursting strength than ring-spun yarn, thus limiting their use in knitted fabrics [10]. In another study, Candan and Onal reported that dimensional stability can be improved by decreasing the loop length of knitted fabrics. However, decreasing loop length in knitted fabrics is only practical to a certain extent, as knitting machines may not work properly after a further decrease. In the same study, Candan and Onal proposed

that the application of elastomeric yarns may be a good solution to poor dimensional stability. However, such an option is not always economical because of the cost considerations of elastane and the supplementary heat-setting process [9]. Moghassem and Tayebi mentioned that mercerization is one method used to improve the dimensional stability of cotton knits. Apart from the cost, the limited availability of a proper mercerization method for knits and quality control issues limit the mercerization process [11]. Safdar et al. mentioned that mechanical compaction using a compactor machine is a successful method for improving dimensional stability, but with limited shrinkage control, which may not last more than 4–5 washes [12]. Moreover, tumble drying is one way to control the dimensional stability of knitted garments [13–14]. However, lesser production and batch-to-batch quality variations of the tumble-drying method limit its use for improving dimensional stability [12]. In this regard, the application of cross-linking resin such as dimethylol dihydroxy ethylene urea (DMDHEU) is an additional potential solution for improving and controlling the dimensional stability problem of viscose jersey knits [3, 15]. A DMDHEU cross-linking agent is most widely-used for textile cellulosic fibre because it demonstrates good durable press properties at a low cost, is less detrimental to fabric strength, results in less discoloration, is post-curable, and yields low chlorine retention characteristics relative to other agents. Cross-linking happens within accessible fibre regions, providing fibre resistance to deformation, and improved elastic recovery from deformation. Cross-linking prevents fibre molecule movement during stress and prevents shrinkage. DMDHEU reacts with cellulose in the presence of a catalyst to form cross-links between individual cellulose chain molecules during resin treatment. In fact, cross-links occur between the four hydroxyl (-OH) groups of DMDHEU and single hydroxyl (-OH) group

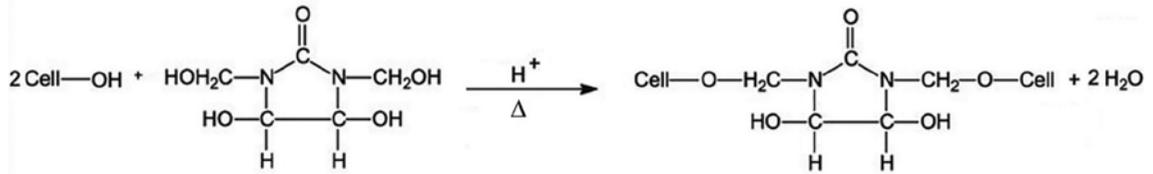


Figure 1: Cross-linking between DMDHEU and cellulose

of individual cellulose chain molecules. The reaction mechanism for cross-linking between DMDHEU and cellulose is shown in Figure 1 [16].

However, the use of such resin leads to a significant deterioration in the bursting strength of treated fabrics. This problem becomes even more severe when cross-linking products are used to control the shrinkage of open-structure fabrics, as well as high absorbent and more amorphous structure fibre fabric such as viscose single knit fabrics [2–3]. This is because a higher concentration of the cross-linking agent is required to control the high level of shrinkage in such structured fibre fabrics. Moreover, resins make the fabrics stiffer and harsh to the touch. In this regard, the application of softening agents through polyethylene emulsion can help to retain a fabric’s strength. Thus, in such cases, a critical balance must be maintained to attain the optimum dimensional stability in viscose knits with a minimum loss in fabric bursting strength [3]. Likewise, factors involving optimal shrinkage controlling with the desired bursting strength are resin concentration, softener concentration and curing time in the resin-finishing process. Therefore, the control of process parameters during the resin-finishing process is important for obtaining final products that meet customers’ requirement [17]. Moreover, all these factors perform non-linearly and interact with each other. It is thus very challenging for scientists and engineers to control resin-finishing processes. For this reason, it is not easy to create an exact model between process parameters and quality characteristics [4, 18, 19].

A conventional trial-and-error experimental approach did not succeed in this regard due to the major loss of time and resources [3]. Similarly, modelling based on mathematical and statistical techniques is not suitable because of its inability to capture the non-linear relationship between inputs and outputs. Alternatively, developing a prediction model using artificial neural network (ANN) and adaptive neuro-inference system (ANFIS) techniques is also a challenging and time-consuming process due to the large volume of trial data [18–21].

In this context, a fuzzy expert system (FES) is the scientific and engineering solution for quality modelling, as FES performs remarkably well with small amounts of experimental data in a non-linear, trial-and-error and complex textile domain [18–20, 22]. Moreover, a fuzzy logic model is more reasonable, cheaper in terms of design cost and often easier to apply than other models [18, 20–23]. Therefore, the main objective of this study was to develop a fuzzy resin model to achieve optimum dimensional stability in viscose knits with a minimum loss in fabric bursting strength as a function of resin concentration, softener concentration and curing time, which has not been reported in past studies.

## 2 Materials and methods

### 2.1 Fuzzy expert system

A fuzzy expert system is an artificial intelligence derived from fuzzy set theory established by Zadeh in 1965 [20, 21, 24]. The basic components of a fuzzy expert system are a fuzzifier, a fuzzy rule base, an inference engine and a defuzzifier as depicted in Figure 2.

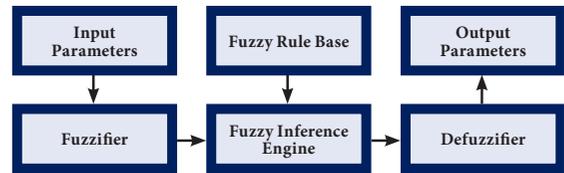


Figure 2: Basic structure of a fuzzy expert system [19]

#### Fuzzifier

A fuzzifier is the first block in fuzzy modelling, which converts all crisp numeric inputs into fuzzy numbers in a range from 0 to 1 using membership functions. A membership function is typically a curve that converts the numerical values of input variables into a fuzzy number in a range from 0 to 1. This value is called a membership value. Among various forms of membership functions, the triangle membership function is the simplest and most frequently used due to its accuracy [20, 24].

**Fuzzy rule base**

The fuzzy rules are the heart of fuzzy modelling and are expressed by if-then statements that narrate the input variables in the antecedent part and output variables in the consequent part, which determines the input-output relationship of the model [19, 20, 24]. As an expression, when a fuzzy model with two inputs and one output involves n fuzzy rules, the development of fuzzy rules can be presented as follows:

- Rule 1: If x is  $A_1$  and y is  $B_1$ , then z is  $C_1$
- Rule 2: If x is  $A_2$  and y is  $B_2$ , then z is  $C_2$
- ...
- Rule n: if x is  $A_n$  and y is  $B_n$ , then z is  $C_n$ ,

where x, y, and z are the linguistic variables representing the input variables and the output variable respectively, and  $A_i, B_i$  and  $C_i (i = 1, 2 \dots n)$  are the fuzzy numbers that represent the linguistic states.

**Inference engine**

The fuzzy inference engine is fundamentally a control mechanism that plays a central role in fuzzy modeling because of its human decision-making ability. Most commonly, the Mamdani max-min fuzzy inference mechanism is used to aggregate several fuzzy sets into a single fuzzy set because it assures a linear interpolation of the output between the rules [1, 20, 24]. For three-input and three-output fuzzy systems, the fuzzy inference mechanism can be presented graphically as bellows in Figure 3[1].

RC (resin concentration), SC (softener concentration) and CT (curing time) are in inputs side and LS (lengthwise shrinkage), WS (widthwise shrinkage) and BS (bursting strength) are on the output side.

**Defuzzifier**

A defuzzifier is the fourth and last block of the fuzzy expert system and converts the fuzzy inference out-

put into a non-fuzzy value z. Among various defuzzification methods, the centre of gravity method is most frequently used [2, 20, 24] and is calculated using equation 2 below.

$$z = \frac{\sum_{i=1}^n (\mu_i * b_i)}{\sum_{i=1}^n \mu_i} \tag{1}$$

where  $b_i$  represents the position of the singleton in the  $i$ th universe and  $\mu_i$  is equal to the firing strength of truth values of rule  $i (i = 1, 2 \dots n$  and  $n =$  number of observations). The firing strength of a rule is the product of the input membership grades. This value is passed to the membership grade of the output to the corresponding fuzzy set. A universe is the set of entities over which certain variables of interest in some formal treatment may range. A singleton is an individual member or thing distinct from others grouped with it.

*2.2 Development of a fuzzy resin model*

In order to develop a fuzzy resin model of viscose knitted fabrics, resin concentration (RC), softener concentration (SC) and curing time (CT) were used as input variables, while lengthwise shrinkage (LS), width-wise shrinkage (WS) and bursting strength (BS) were used as output variables. These resin-finishing process variables were chosen exclusively, as they have a significant effect on fabric shrinkage and bursting strength. A fuzzy logic toolbox from MATLAB R2013b (version 8.2.0.701) was used to create the proposed fuzzy resin model of shrinkage and bursting strength. The construction of a fuzzy resin modelling for shrinkage and bursting strength is depicted in Figure 4.

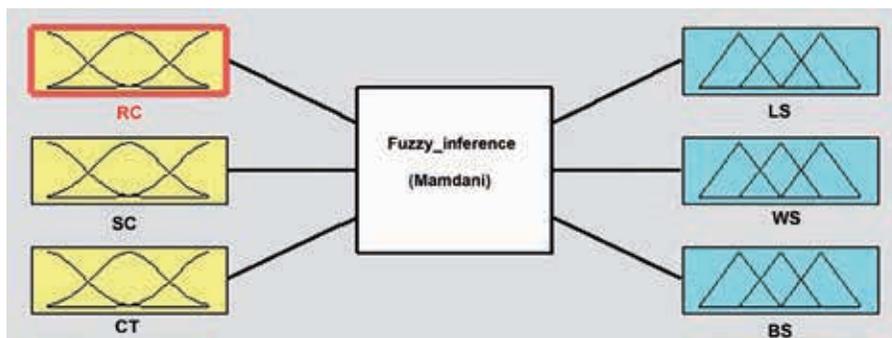


Figure 3: Fuzzy inference mechanism (Mamdani)

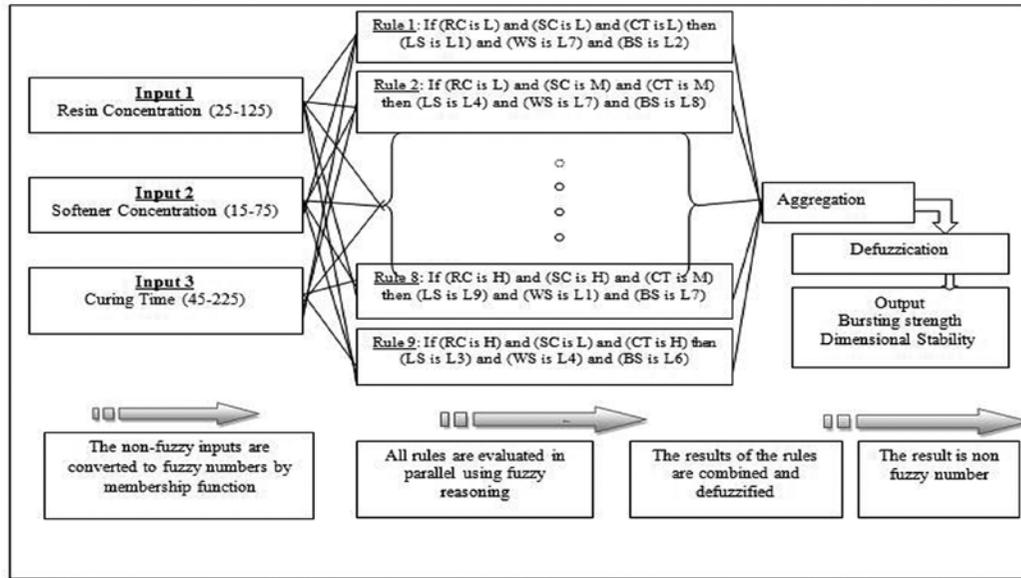


Figure 4: Schematic representation of a fuzzy expert system for the modelling of dimensional stability and bursting strength

For fuzzification, three possible linguistic fuzzy sets, namely low (L), medium (M), and high (H), were chosen for the input variables RC, SC and CT. Likewise, nine output linguistic fuzzy sets, i.e. from L1 to L9 (Level 1, Level 2 ... Level 9) were considered for LS, WS and BS. In the present study, triangular shaped membership functions were used for both input and output variables due to their accuracy [18, 20]. Moreover, a Mamdani max-min inference approach and the centre of gravity defuzzification method were applied in this work. The selection of the number of membership functions and their initial values was based on the system knowledge and experimental conditions [18, 20]. There is a level of membership for each linguistic word that is applied in each input variable. Equations 2-7 were used for fuzzification, as shown in Figures 5-10.

$$RC(i_1) = \begin{cases} i_1, & 25 \leq i_1 \leq 125 \\ 0, & otherwise \end{cases} \quad (2)$$

$$SC(i_2) = \begin{cases} i_2, & 15 \leq i_2 \leq 75 \\ 0, & otherwise \end{cases} \quad (3)$$

$$CT(i_3) = \begin{cases} i_3, & 45 \leq i_3 \leq 225 \\ 0, & otherwise \end{cases} \quad (4)$$

$$LS(o_1) = \begin{cases} o_1, & 1.6 \leq o_1 \leq 6.7 \\ 0, & otherwise \end{cases} \quad (5)$$

$$WS(o_2) = \begin{cases} o_2, & 0.2 \leq o_2 \leq 2 \\ 0, & otherwise \end{cases} \quad (6)$$

$$BS(o_3) = \begin{cases} o_3, & 270 \leq o_3 \leq 318 \\ 0, & otherwise \end{cases} \quad (7)$$

where  $i_1$ ,  $i_2$  and  $i_3$  represent the first (RC), second (SC) and third (CT) input variables respectively and  $o_1$ ,  $o_2$  and  $o_3$  represent the output variables (LS), (WS) and (BS), as shown in equations 2-7.

The triangular formed membership functions for the fuzzy variables RC, SC, CT, LS, WS and BS were developed using the MATLAB Fuzzy Toolbox as shown in Figures 5-10. The membership functions for input variable RC were constructed using the linguistic fuzzy sets low (L), medium (M), and high (H) with values of RC (25 g/l, 75 g/l and 125 g/l) as depicted in Figure 5. Likewise, the membership functions for input variables SC and CT were created by taking the fuzzy numbers low (L), medium (M) and high (H) with values of SC (15 g/l, 45 g/l and 75 g/l) and CT (45 s, 135 s, 225 s), respectively, as illustrated in Figures 6-7. In the same way, the membership functions for the output variables LS, WS and BS were formed using the fuzzy numbers Level 1, Level 2, Level 3, Level 4, Level 5, Level 6, Level 7, Level 8 and Level 9 with value ranges LS (1.6-6.7%), WS (0.2-2%) and BS (270-318 kPa), respectively, as shown in Figures 8-10. The values were given in such a way that they were equally spaced and covered the whole input and output space. The values of input and output variables were selected based on the expert system knowledge, previous experience and arbitrary choice.

From a textile point of view and previous experience, it is known that output variable shrinkage is related to input variable resin concentration. This is because shrinkage is efficiently decreased due to the formation of a cross-link between cellulose and resin, while the output variable bursting strength is influenced by the input variable softener concentration. The reason lies

in the fact that softener compensates for the loss of bursting strength during resin application. Moreover, the input variables resin concentration and softener concentration have a synergetic effect with curing time. After fuzzification, a total of 9 fuzzy rules were created based on expert knowledge and previous experience, as presented in Table 1.

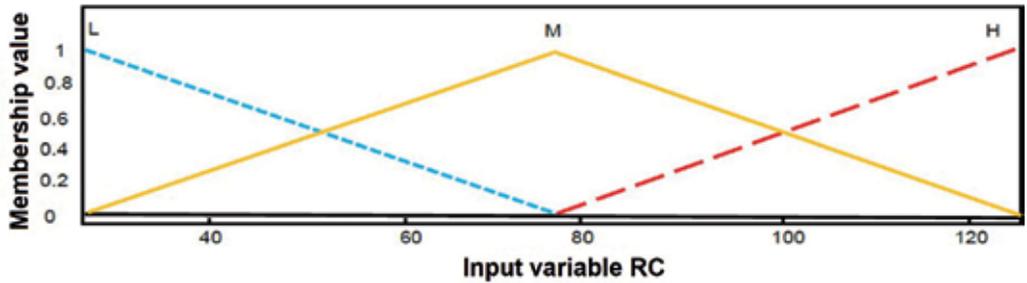


Figure 5: Membership function of RC

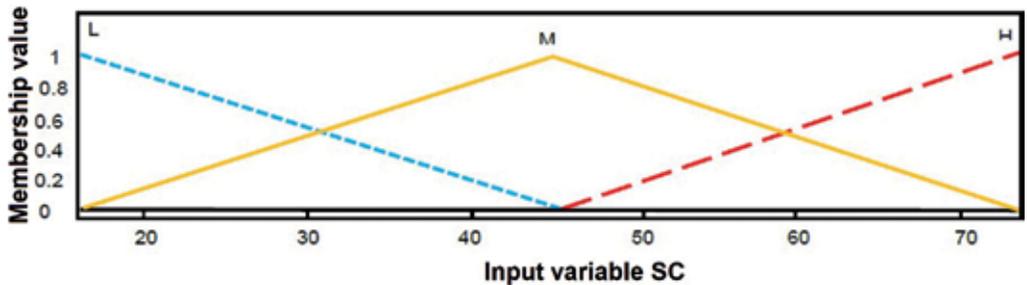


Figure 6: Membership function of SC

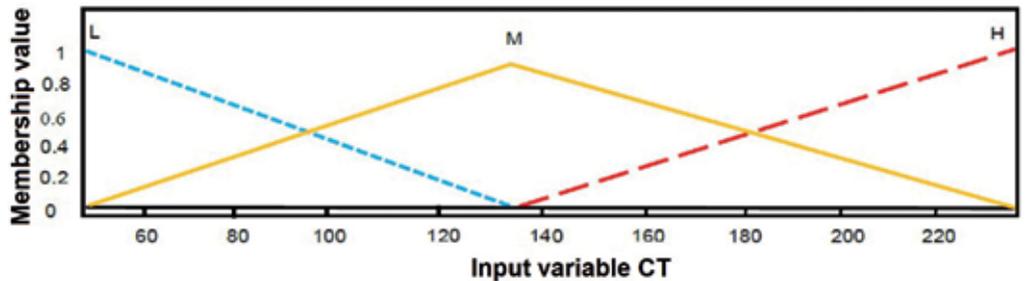


Figure 7: Membership function of CT

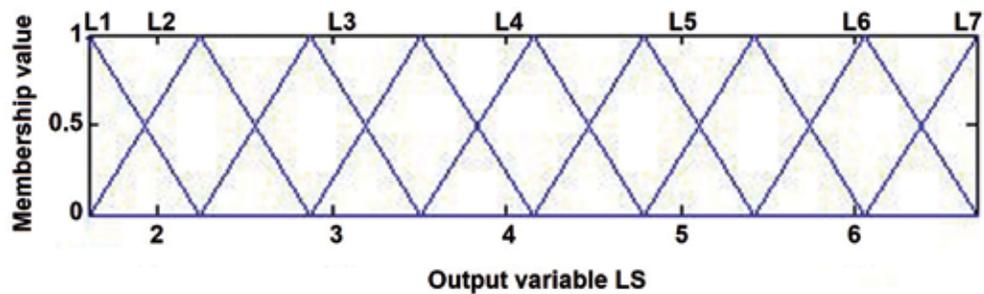


Figure 8: Membership function of LS

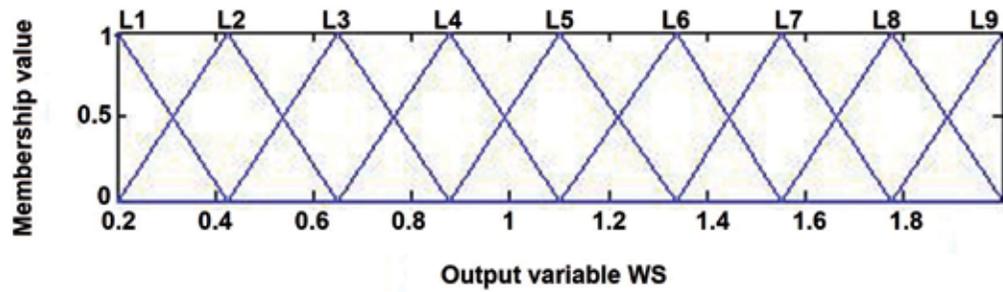


Figure 9: Membership function of WS

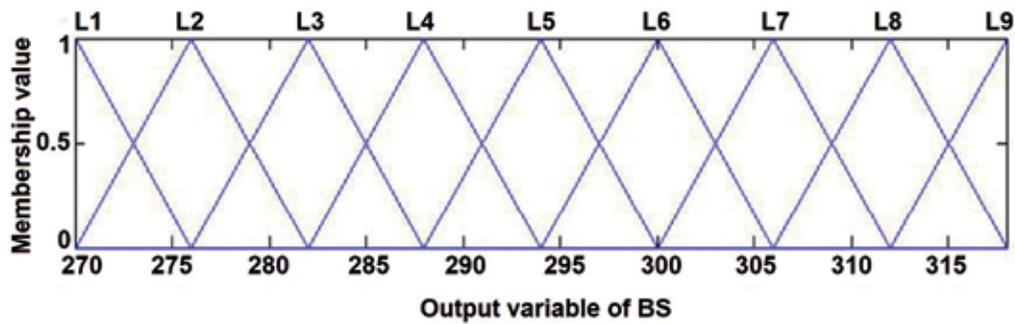


Figure 10: Membership function of BS

Table 1: Fuzzy inference rules

Rules no.	Input variables			Output variables		
	RC	SC	CT	LS	WS	BS
1	L	L	L	L1	L7	L2
2	L	M	M	L4	L7	L8
3	L	H	H	L7	L8	L3
4	M	M	M	L5	L5	L5
5	M	L	M	L1	L9	L9
6	M	M	H	L8	L1	L8
7	H	M	L	L9	L1	L4
8	H	H	M	L9	L1	L7
9	H	L	H	L3	L4	L6

The calculation of the membership values for the developed membership functions of RC, SC and CT based on the Figures 5-10, created rules and the equations presented above (2-7) is presented as follows:

$$\mu_L(RC) = \begin{cases} \frac{75 - i_1}{75 - 25}; & 25 \leq i_1 \leq 75 \\ 0; & i_1 \geq 75 \end{cases} \quad (8)$$

From equation 8:

If we put  $i_1=25$  then,

$$\mu_L(RC) = \left( \frac{75 - 25}{75 - 25} \right) = \frac{50}{50} = 1 \quad (8a)$$

If we put  $i_1=50$  then,

$$\mu_L(RC) = \left( \frac{75 - 50}{75 - 25} \right) = \frac{25}{50} = 0.5 \quad (8b)$$

If we put  $i_1=75$  then,

$$\mu_L(\text{RC}) = \left( \frac{75 - 75}{75 - 25} \right) = \frac{0}{50} = 0$$

$$\mu_M(\text{RC}) = \begin{cases} \frac{i_1 - 25}{75 - 25}; & 25 \leq i_1 \leq 75 \\ \frac{125 - i_1}{125 - 75}; & 75 \leq i_1 \leq 125 \\ 0; & i_1 \geq 125 \end{cases}$$

From equation 9:

If we put  $i_1=25$  then,

$$\mu_M(\text{RC}) = \left( \frac{25 - 25}{75 - 25} \right) = \frac{0}{50} = 0$$

If we put  $i_1=75$  then,

$$\mu_M(\text{RC}) = \left( \frac{75 - 25}{75 - 25} \right) = \frac{50}{50} = 1$$

If we put  $i_1=125$  then,

$$\mu_M(\text{RC}) = \left( \frac{125 - 125}{125 - 75} \right) = \frac{0}{50} = 0$$

$$\mu_H(\text{RC}) = \begin{cases} \frac{i_1 - 75}{125 - 75}; & 75 \leq i_1 \leq 125 \\ 0; & i_1 \leq 75 \end{cases}$$

From equation10:

If we put  $i_1=75$  then,

$$\mu_H(\text{RC}) = \left( \frac{75 - 75}{125 - 75} \right) = \frac{0}{50} = 0$$

If we put  $i_1=100$  then,

$$\mu_H(\text{RC}) = \left( \frac{100 - 75}{125 - 75} \right) = \frac{25}{50} = 0.5$$

If we put  $i_1=125$  then,

$$\mu_H(\text{RC}) = \left( \frac{125 - 75}{125 - 75} \right) = \frac{50}{50} = 1$$

$$\mu_L(\text{SC}) = \begin{cases} \frac{45 - i_2}{45 - 15}; & 15 \leq i_2 \leq 45 \\ 0; & i_2 \geq 45 \end{cases}$$

From equation 11:

If we put  $i_2=15$  then,

$$\mu_L(\text{SC}) = \left( \frac{45 - 15}{45 - 15} \right) = \frac{30}{30} = 1$$

If we put  $i_2=30$  then,

$$\mu_L(\text{SC}) = \left( \frac{45 - 30}{45 - 15} \right) = \frac{15}{30} = 0.5$$

If we put  $i_2=45$  then,

$$\mu_L(\text{SC}) = \left( \frac{45 - 45}{45 - 15} \right) = \frac{0}{30} = 0$$

$$\mu_M(\text{SC}) = \begin{cases} \frac{i_2 - 15}{45 - 15}; & 15 \leq i_2 \leq 45 \\ \frac{75 - i_2}{75 - 45}; & 45 \leq i_2 \leq 75 \\ 0; & i_2 \geq 75 \end{cases}$$

From equation12:

If we put  $i_2=15$  then,

$$\mu_M(\text{SC}) = \left( \frac{15 - 15}{45 - 15} \right) = \frac{0}{30} = 0$$

If we put  $i_2=45$  then,

$$\mu_M(\text{SC}) = \left( \frac{45 - 15}{45 - 15} \right) = \frac{30}{30} = 1$$

If we put  $i_2=75$  then,

$$\mu_M(\text{SC}) = \left( \frac{75 - 75}{75 - 45} \right) = \frac{0}{30} = 0$$

$$\mu_H(\text{SC}) = \begin{cases} \frac{i_2 - 45}{75 - 45}; & 45 \leq i_2 \leq 75 \\ 0; & i_2 \leq 45 \end{cases}$$

From equation13:

If we put  $i_2=45$  then,

$$\mu_H(\text{SC}) = \left( \frac{45 - 45}{75 - 45} \right) = \frac{0}{30} = 0$$

If we put  $i_2=60$  then,

$$\mu_H(\text{SC}) = \left( \frac{60 - 45}{75 - 45} \right) = \frac{15}{30} = 0.5$$

If we put  $i_2=75$  then,

$$\mu_H(\text{SC}) = \left( \frac{75 - 45}{75 - 45} \right) = \frac{30}{30} = 1$$

$$\mu_L(\text{CT}) = \begin{cases} \frac{135 - i_3}{135 - 45}; & 45 \leq i_3 \leq 135 \\ 0; & i_3 \geq 135 \end{cases}$$

From equation 14:

If we put  $i_3=45$  then,

$$\mu_L(CT) = \left( \frac{135 - 45}{135 - 45} \right) = \frac{90}{90} = 1 \quad (14a)$$

If we put  $i_3=90$  then,

$$\mu_L(CT) = \left( \frac{135 - 90}{135 - 45} \right) = \frac{45}{90} = 0.5 \quad (14b)$$

If we put  $i_3= 135$  then,

$$\mu_L(CT) = \left( \frac{135 - 135}{135 - 45} \right) = \frac{0}{90} = 0 \quad (14c)$$

$$\mu_M(CT) = \begin{cases} \frac{i_3 - 45}{135 - 45}; & 45 \leq i_3 \leq 135 \\ \frac{225 - i_3}{225 - 135}; & 135 \leq i_3 \leq 225 \\ 0; & i_3 \geq 225 \end{cases} \quad (15)$$

From equation15:

If we put  $i_3=45$  then,

$$\mu_M(CT) = \left( \frac{45 - 45}{135 - 45} \right) = \frac{0}{90} = 0 \quad (15a)$$

If we put  $i_3= 135$  then,

$$\mu_M(CT) = \left( \frac{135 - 45}{135 - 45} \right) = \frac{90}{90} = 1 \quad (15b)$$

If we put  $i_3=225$  then,

$$\mu_M(CT) = \left( \frac{225 - 225}{225 - 135} \right) = \frac{0}{90} = 0 \quad (15c)$$

$$\mu_H(CT) = \begin{cases} \frac{i_3 - 135}{225 - 135}; & 135 \leq i_3 \leq 225 \\ 0; & i_3 \leq 135 \end{cases} \quad (16)$$

From equation16:

If we put  $i_3=135$  then,

$$\mu_H(CT) = \left( \frac{135 - 135}{225 - 135} \right) = \frac{0}{90} = 0 \quad (16a)$$

If we put  $i_3=180$  then,

$$\mu_H(CT) = \left( \frac{180 - 135}{225 - 135} \right) = \frac{15}{90} = 0.5 \quad (16b)$$

If we put  $i_3= 225$  then,

$$\mu_H(CT) = \left( \frac{225 - 135}{225 - 135} \right) = \frac{90}{90} = 1 \quad (16c)$$

Similarly, the membership value of other variables can be calculated. To demonstrate how the membership values of the developed membership functions from Figures 5-7 are determined, the following equations were explained.

**Equation 11:** If we put RC = 75 g/l, then one membership function  $\mu_M(RC)$  is mapped and  $\mu_M(RC)$  is found to be 1 from the equation (9b).

**Equation 17:** If we put SC = 45 g/l, then one membership function  $\mu_M(SC)$  is mapped and  $\mu_M(SC)$  is determined as 1 from the equation (12b).

**Equation 23:** If we put CT = 135 s, then one membership function  $\mu_M(CT)$  is mapped and  $\mu_M(CT)$  is calculated as 1 from the equation (15b).

From the above equations (9b), (12b) (15b), it evident that if RC is M, SC is M and CT is M, then rule 4 is to be fired. In the defuzzification stage, the truth degrees ( $\mu$ ) of each rule are counted with the help of the min and by taking the max between the active rules [21]. The firing strength ( $\mu$ ) of input variables for rule 4 is calculated as follows:

$$\mu_4 = \min\{\mu_M(RC), \mu_M(SC), \mu_M(CT)\} = \min(1, 1, 1) = 1 \quad (17)$$

The crisp output was subsequently counted. Haghghat et al. stated that in many circumstances, for a system whose output is a fuzzy set, it is essential to aggregate several fuzzy sets into a single fuzzy set using an aggregation method [24]. Finally, by using equations (1) and (17) with Figure 8, the crisp output of lengthwise shrinkage (LS) is calculated as shown below:

$$LS^{crisp} = \frac{1 \times 4.15}{1} = 4.15 \quad (18)$$

#### Prediction performance measure

The prediction accuracy of the developed model was investigated using a global prediction error, such as mean absolute error (MAE) and coefficient of determination ( $R^2$ ). The formulations of those accuracy measures are given below.

$$MAE = \frac{1}{N} \sum_{i=1}^{i=N} \left( \frac{|E_a - E_p|}{E_a} \times 100 \right) \quad (19)$$

$$R^2 = 1 - \left( \frac{\sum_{i=1}^{i=N} (E_a - E_p)^2}{\sum_{i=1}^{i=N} (E_a - E_M)^2} \right) \quad (20)$$

where  $E_a$  represents the actual result,  $E_p$  represents the predicted result,  $E_M$  represents the mean value and  $N$  represents the number of observations.

The coefficient of determinations ( $R^2$ ) compares the accuracy of the model to the accuracy of a standard model. The mean absolute error (MAE) gives the deviation between the predicted and experimental values and is required to reach zero [18].

### 2.3 Experimental work for the validation of the fuzzy resin model

#### Fabrics

The fabric used in this study was a single jersey viscose knit structure with a mass per unit area of 190 g/m<sup>2</sup>. The fabric was knitted on a Pailung single jersey circular knitting machine, with a 30-inch (76.2 cm) diameter, and E20 and 90 yarn feeders.

#### Chemicals

The chemicals used in this study include: Felosan NOF as a wetting agent (CHT, Bangladesh), Kappavon CL as an anti-creasing agent (Kapp-Chemie, Bangladesh), Sirrix 2UD as a sequestering agent (Clariant, Bangladesh), Kappazon H53 as a peroxide stabilizer (Kapp-Chemie, Bangladesh), Reaknit FF(DMDHEU) as a cellulose cross-linking resin (CHT, Bangladesh), Polysiligen as silicon softener (CHT, Bangladesh), MgCl<sub>2</sub> as a catalyst, sodium carbonate, H<sub>2</sub>O<sub>2</sub> and acetic acid, all of commercial grade.

#### Machinery and equipment

The following machinery and equipment were used: a Slavos sample winch dyeing machine (Greece), an Ehwa suntex platinum pin stenter with padding mangle (Korea), a Lafer compactor (Italy), a Wascator washing machine (SDL, England) and a Pneumatic Bursting tester (SDL, England).

#### Fabric pre-treatment

Viscose is a regenerated cellulose fibre that is free from natural impurities, such as fat, oil and wax.

However, it contains residual chemicals, such as sulfur and spinning lubricant that were used in the viscose manufacturing stage. Hence, mild pre-treatment is performed for viscose fibre to remove the aforementioned residual chemicals and added impurities. The fabric samples were subjected to pre-treatment in an industrial-scale winch dyeing machine at 90 °C for 30 minutes using an anti-creasing agent (Kappavon CL 1.0 g/l), sequestering agent (Kappquest FE, 0.5 g/l), wetting agent (Felosan NOF 1.0 g/l), soda ash (2.5 g/l), Hydrogen peroxide 50% (1.0 g/l) and stabilizing agent (Kappazon H53 0.3 g/l). Finally, the pre-treated fabric was hot washed, rinsed and neutralized using 1.0 g/l acetic acid and dried.

#### Resin finishing

The industrial-scale pre-treated fabrics were divided into nine samples (each 10 kg) that were subjected to resin finishing treatments on an open stenter padder at 75% pick-up according to a set of values for resin concentration (25 g/l, 75 g/l and 125 g/l), softener concentration (15 g/l, 45 g/l and 75 g/l), and curing time (45 s, 135 s and 225 s) under the experimental conditions shown in Table 2, followed by drying at 120 °C for 2.5 minutes and curing at 170 °C for the times specified in the experimental conditions, and finally compacted perfectly. The recipes were prepared with the specified amount of resin, softener and MgCl<sub>2</sub> catalyst (20% of the amount of resin used as recommended by the resin manufacturer).

#### Measurement of shrinkage and bursting strength

After resin treatment, a total of 18 (eighteen) samples were prepared from all fabrics, nine of which were for shrinkage testing and nine for bursting strength testing. All 18 samples were then subjected to conditioning on a flat surface for at least 24 hours before testing under standard atmospheric conditions at a relative humidity of 65% ± 2% and a temperature of 20 ± 2°C. Firstly, the lengthwise and widthwise shrinkage of the samples was calculated using equations (21) and (22) after washing the samples according to AATCC TM-135. The test sample with lengthwise and widthwise marking is shown in Figure 11.

Table 2: Experimental conditions for resin treatment

Parameters	Unit	Level		
Resin concentration	g/l	25	75	125
Softener concentration	g/l	15	45	75
Curing time	s	45	135	225

$$\text{Lengthwise shrinkage} = \frac{L_b - L_a}{L_b} \times 100 \quad (21)$$

$$\text{Widthwise shrinkage} = \frac{W_b - W_a}{W_b} \times 100 \quad (22)$$

where  $L_b$  represents length before washing,  $L_a$  represents length after washing (Figure 11),  $W_b$  represents width before washing and  $W_a$  represents width after washing (Figure 11).

Subsequently, the bursting strength (kPa) of each resin treated sample was measured using a pneumatic bursting tester with a specimen of 30 mm in diameter according to the ISO-139388-1 test method.

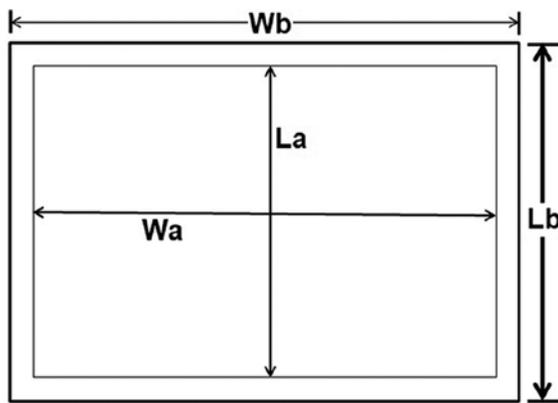


Figure 11: Test sample with lengthwise and widthwise marking

### 3 Results and discussion

#### 3.1 Analysis of model performance

The graphical operations of developed fuzzy resin finishing models are depicted using the two examples in Figures 12 and 13. For a simple demonstration, out of nine rules only two fuzzy rules are explained here. According to Rule 4, if RC (resin concentration) is M, SC (softener concentration) is M and CT (curing time) is M, then outputs LS (lengthwise shrinkage) is L5 (Level 5), WS (widthwise shrinkage) is L5 (Level 5) and BS (bursting strength) is L5 (Level 5). Moreover, according to Rule 8, if RC is H, SC is H, and CT is M, then the outputs are as follows: LS is L9, WS is L1 and BS is L7. An example is given for Rule 4, where if input RC is 75 g/l, SC is 45 g/l and CT is 135 s, then then all nine fuzzy rules will be evaluated simultaneously to determine the fuzzy output of shrinkage and bursting strength. However, some of the rules will remain defunct as ‘fuzzy and’ function has been used in the antecedent part of the fuzzy rules and no output fuzzy set will be produced. The outputs of active fuzzy rules are then aggregated to arrive at a final output fuzzy set. Lastly, the Fuzzy-predicted outputs from the MATLAB’ fuzzy rule viewer were found to be LS is 4.15%, WS is 1.11% and BS is 294 kPa, as shown in Figure 12. Likewise, in the case of Rule 8, if input RC is 125 g/l, SC is 75 g/l and CT is 135 s, then predicted values are found to be LS is 6.5%, WS is 0.269% and BS is 306 kPa, as presented in Figure 13.

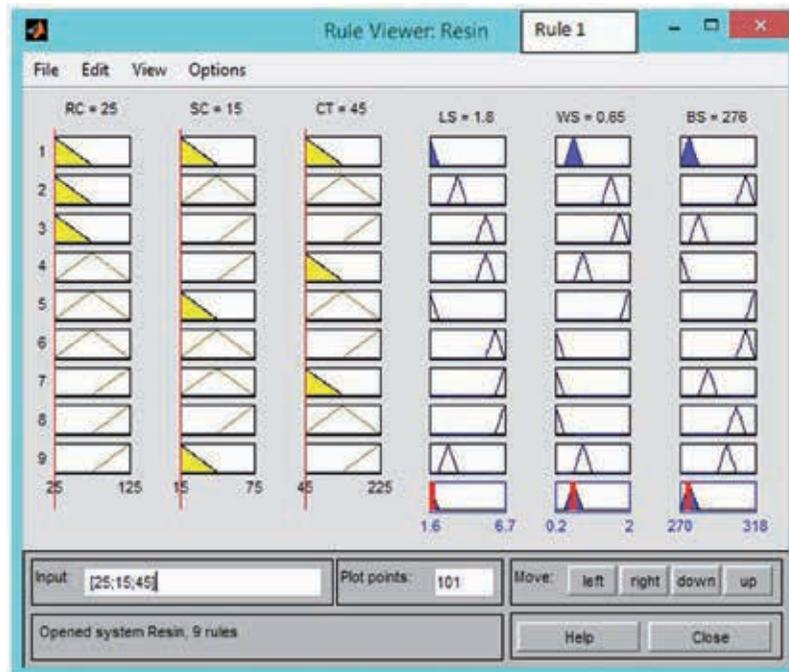


Figure 12: Graphical operation of the fuzzy expert system model (Rule 4)

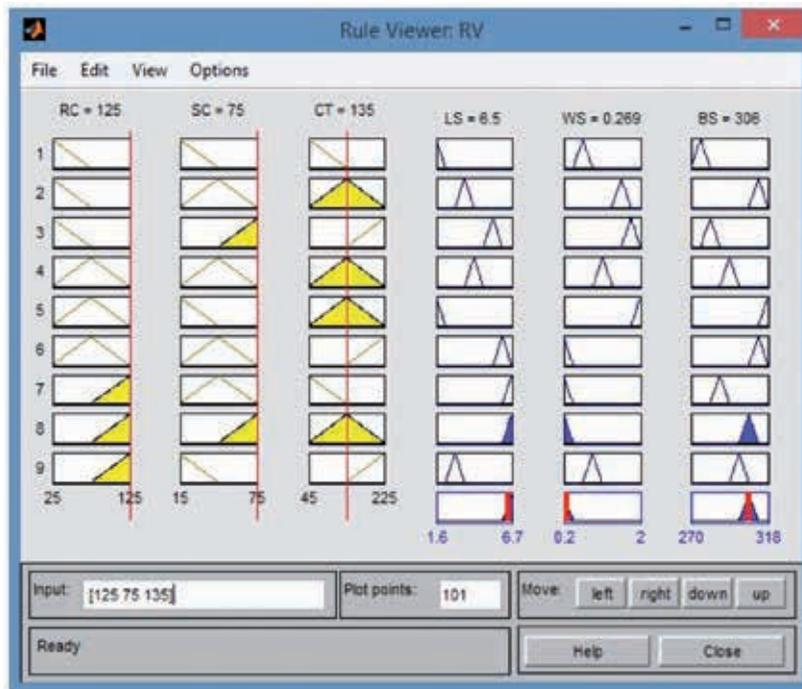


Figure 13: Diagrammatic action of the fuzzy expert system (Rule 8)

### 3.2 Analysis of experimental results

#### 3.2.1 Effect of resin concentration, softener concentration and curing time on fabric shrinkage

Figures 14 and 15 depict the effect of resin concentration, softener concentration and curing time on fabric shrinkage. It is clear from the figures that the slope of shrinkage (%) is non-linear with the effect of resin concentration, softener concentration and curing time. With an initial increase in resin concentration, the number of cross-links between the resin and free hydroxyl groups in the viscose cellulose chains quickly increases, resulting in a drastic decrease in fabric shrinkage. The shrinkage of viscose fabrics is mainly due to their ability to absorb more moisture because of the existence of hydroxyl groups in the cellulose. As the result of water absorption, the movement of cellulose polymer chains is also enabled in the amorphous regions by disrupting the internal hydrogen bonds between the cellulose chains. When the fabric is dried after wetting, the hydrogen bonds between the cellulose chains are reformed in new relaxed positions. With an increase in resin concentration, the hydroxyl groups of adjacent cellulose chains are cross-linked, thus making the fibres less inclined to water absorption and chain disturbances, resulting in a decrease in

fabric shrinkage. Nevertheless, further increases in resin concentration result in a gradual decrease in shrinkage. This is because the number of available free hydroxyl groups decreases in the viscose cellulosic fabric with initial cross-linking.

Moreover, it is evident from Figures 14 and 15 that the effect of softener concentration on fabric lengthwise and widthwise shrinkage control is non-linear, as well as less significant. Furthermore, at a lower resin concentration, the effect of increasing time is considerably significant in reducing fabric shrinkage due to effective resin cross-linking. However, a higher resin concentration slightly compensates for the reduced time, while shrinkage is reduced effectively, even when curing time is reduced.

#### 3.2.2 Effect of resin concentration, softener concentration and curing time on fabric bursting strength

The effect of resin concentration, softener concentration and curing time on the fabric bursting strength is illustrated in Figure 16 (a–b). It is clear from Figure 16 (a) that an increase in resin concentration results in a decrease in fabric bursting strength. This may be attributed to several factors, including an increase in fibre brittleness, a decrease in yarn elongation and slippage properties, fabric stiffening or some cellulosic degradation during

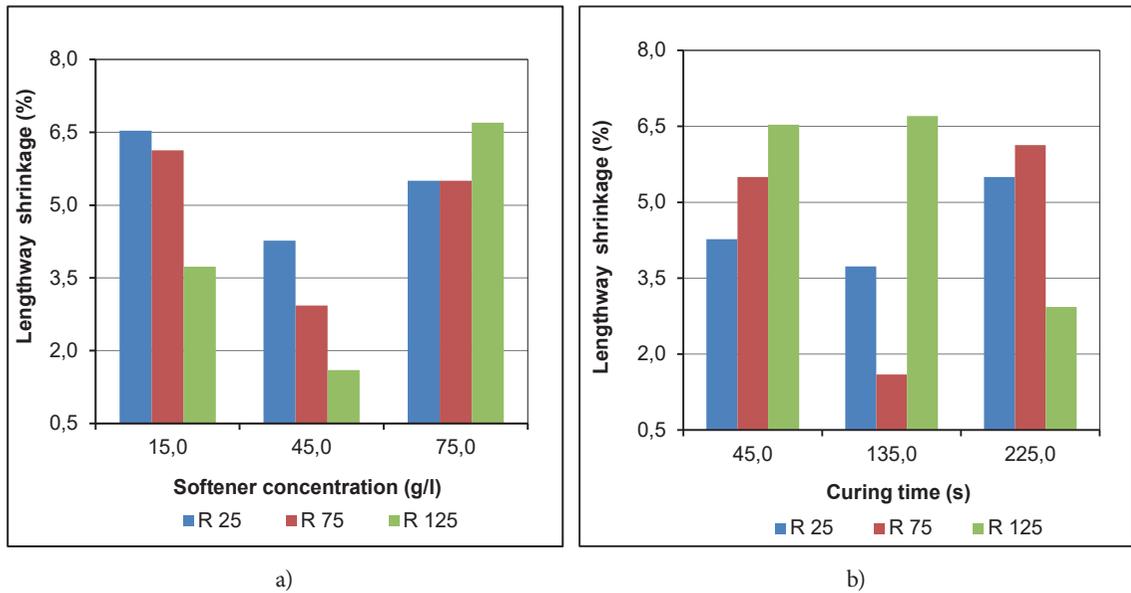


Figure 14: Effects on lengthwise shrinkage: a) resin and softener, and b) and resin and curing time

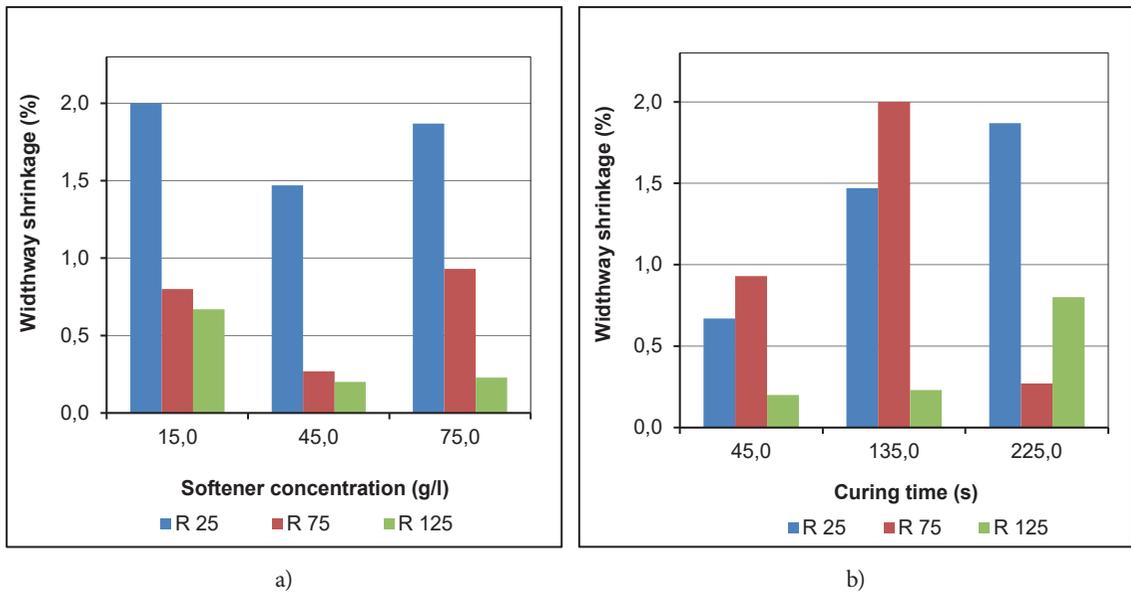


Figure 15: The effects on widthwise shrinkage: a) resin and softener, and b) resin and curing time

acidic resin finishing conditions. Likewise, the effect of resin concentration and curing time on the fabric bursting strength is shown in Figure 16 (b). It is evident from Figure 16 (b) that the effect of increasing curing time is more prominent at a lower resin concentration. However, at a higher resin concentration, there is a significant bursting strength loss, even at a reduced curing time. The addition of a softener results in an improvement in fabric bursting strength. This may be attributed to a decrease in fibre and yarn brittleness and stiffening,

and an increase in yarn slippage properties due to the use of a softener.

The effectiveness of increasing softener concentration to improve fabric bursting strength is better at a lower resin concentration but poor at a higher resin concentration. This is because any loss of fabric bursting strength due to stiffening and brittleness induced by the resin may be compensated for or recovered by the softener. However, any loss of fabric bursting strength that occurs due to cellulose degradation can not be recovered through the application of softeners.

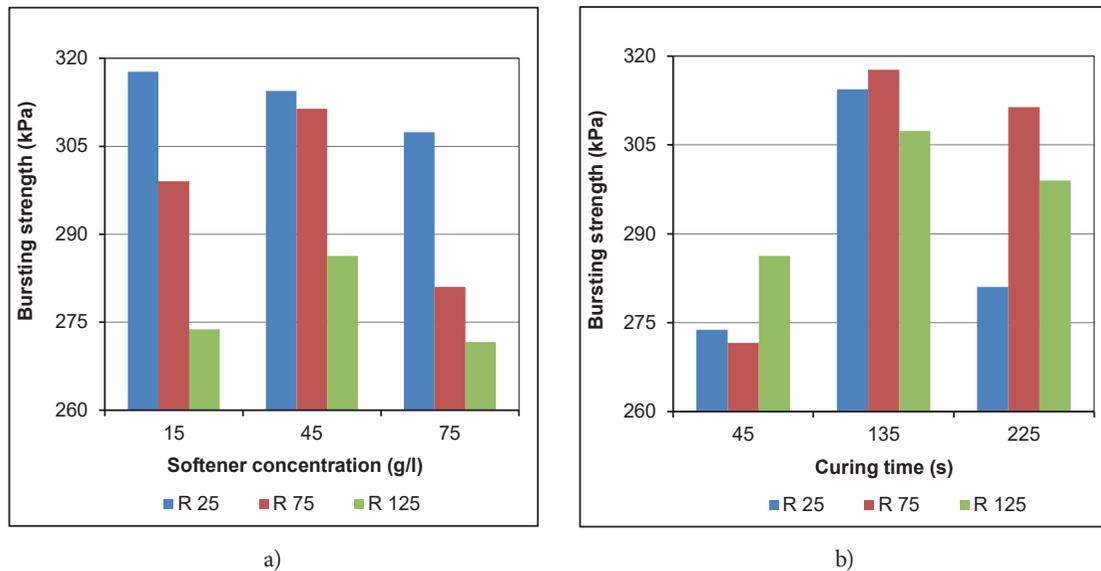


Figure 16: Effects on bursting strength: a) resin and softener, and b) resin and curing time

### 3.3 Validation of fuzzy resin model

The developed resin finishing model was validated using 9 (nine) sets of experimental data that were not used for the development of the proposed model. In fact, the fuzzy resin model was built based on the fuzzy expert knowledge and previous experience of the corresponding author. The corresponding author has more than 15 years of experience in the areas of production and R&D in the textile dyeing-finishing industry as a dye house general manager. The prediction was performed using the MATLAB® fuzzy rule viewer. The results from the developed fuzzy resin prediction model were then compared with

the experimental results. A comparison of predicted and experimental values of shrinkage and bursting strength of viscose plain knitted fabrics are shown in Table 3.

Further, the correlations between the predicted and experimental values of shrinkage and bursting under divergent resin finishing conditions are illustrated in Figures 17a, 17b, 17c. The mean absolute errors (MAE) between the predicted and experimental (actual) values of lengthwise shrinkage (LS), widthwise shrinkage (WS) and bursting strength (BS) were found to be 3.74%, 5.60% and 0.45%, respectively. In addition, the correlation coefficients ( $R$ ) from

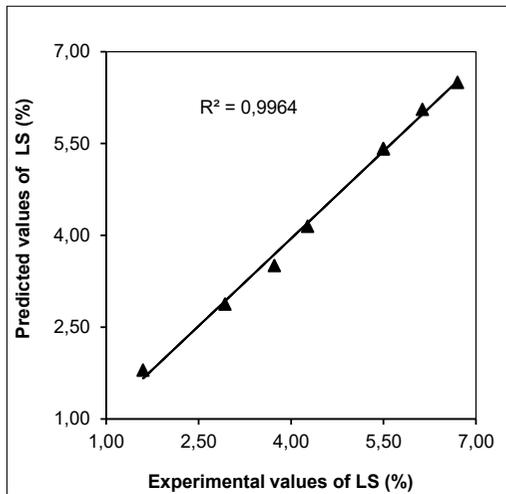
Table 3: Comparisons of predicted and experimental shrinkage and bursting strength

SL #.	Resin (g/l)	Softener (g/l)	Curing time (s)	Lengthwise shrinkage			Widthwise shrinkage			Bursting strength		
				Ev	Pv	AE (%)	E <sub>v</sub>	P <sub>v</sub>	AE (%)	E <sub>v</sub>	P <sub>v</sub>	AE (%)
1	25	15	45	-1.90	1.8	2.81	0.67	0.65	2.99	273.80	276	0.80
2	25	45	135	-3.73	3.51	5.9	1.47	1.55	5.44	314.40	312	0.76
3	25	75	225	-5.50	5.42	1.45	1.87	1.78	4.81	281.00	282	0.36
4	75	75	45	-5.50	5.42	1.45	0.93	0.88	5.91	271.60	272	0.15
5	75	15	135	-1.60	1.8	12.5	2.00	1.93	3.50	317.70	316	0.54
6	75	45	225	-6.13	6.06	1.14	0.27	0.269	0.37	311.40	312	0.19
8	125	75	135	-6.70	6.5	2.99	0.23	0.26	13.04	307.40	306	0.46
9	125	15	225	-2.93	2.88	1.71	0.80	0.87	8.75	299.00	300	0.33
Mean Absolute Error (%)				3.74			5.60			0.45		
Co-efficient of determination ( $R^2$ )				0.996			0.992			0.996		

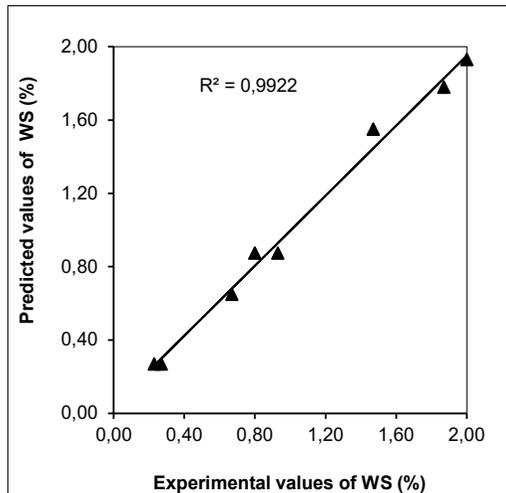
E<sub>v</sub> = experimental value; P<sub>v</sub> = predicted value; AE = absolute error

the predicted and experimental values of LS, WS and BS were found to be 0.998 ( $R^2 = 0.996$ ), 0.992 ( $R^2 = 0.992$ ) and 0.998 ( $R^2 = 0.996$ ), respectively, and elucidated the good agreement by the developed FES resin model.

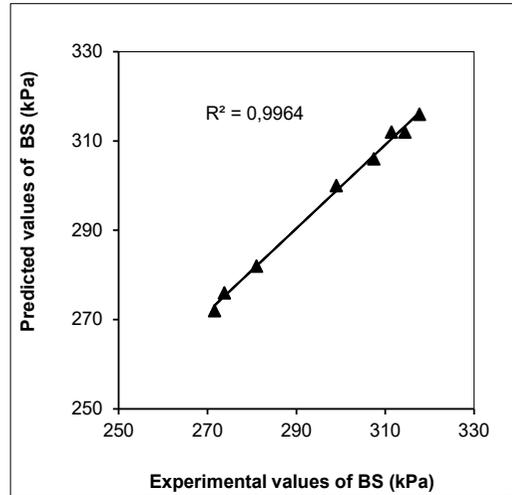
Additionally, it is evident from Table 3 that all the results of  $R^2$  and MAE (%) are very close to each other, which indicates the ability and accuracy of the fuzzy resin model to predict the shrinkage control and bursting strength of viscose plain knitted fabrics. It can be decisively stated that the model developed in this study can perform effectively with good prediction accuracy in a non-linear complex field. From Table 3, the optimal parameters in the resin-finishing process were found to be a resin concentration of 75 g/l, a softener concentration of 45 g/l and a curing time of 225 seconds, as the best shrinkage control with the desired fabric bursting strength.



a)



b)



c)

Figure 17: Correlation between a) experimental and predicted values of lengthwise shrinkage, b) experimental and predicted values of widthwise shrinkage, and c) experimental and predicted values of fabrics bursting strength

#### 4 Conclusion

It was found from our experimental study that the shrinkage of fabric is significantly reduced by increasing the resin concentration and curing time, accompanied by a severe loss in the fabric bursting strength. However, such loss of fabric bursting strength can be improved by increasing softener concentration with some loss in shrinkage control. Moreover, there was a significant interaction between the resin concentration and curing time, and between the resin and softener concentrations. It is obvious that the effects of resin concentration, softener concentration and curing time on shrinkage control are not linear. The FES resin model in this regard is found to be extremely effective in a non-linear domain for determining the optimal resin finishing conditions for best shrinkage control with a minimum loss in fabric bursting strength. The optimal parameters in the resin-finishing process were identified as a resin concentration of 75 g/l, a softener concentration 45 g/l and a curing time 225 seconds. In the current study, the FES resin model was developed by taking resin concentration, softener concentration and curing time as input variables to predict the shrinkage and bursting strength of viscose plain knitted fabric. The fuzzy resin model derived in this research was confirmed by experiment data. The mean absolute

errors between the experimental values of lengthwise shrinkage ( $LS$ ), widthwise shrinkage ( $WS$ ) and bursting strength ( $BS$ ) and those predicted by the FES resin model were found to be 3.74%, 5.60% and 0.45%, respectively. Likewise, the coefficients of determination ( $R^2$ ) from the experimental and predicted values of  $LS$ ,  $WS$  and  $BS$  were found to be 0.996 and 0.992 and 0.996, respectively. The results indicate the brilliant prediction performance of the developed fuzzy resin model. It can thus be decisively concluded that the fuzzy model built in this study can be applied in the textile and dyeing industries for selecting significant process parameters and their required levels to achieve a targeted level of product quality. Conversely, without such a model, a production engineer must conduct numerous trials based on assumptions to achieve the target product quality.

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## Definition of the Main Features of Material Assemblies for Thermal Protective Clothing During External High-temperature Effect Modelling

*Opredelitev glavnih značilnosti modeliranja kompletov materialov za zaščitna oblačila pri visokih okoljskih temperaturah*

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### Abstract

A computational-experimental method of material selection for thermal protective clothing design is proposed in this article. The intended operating temperature of the garment lies within the range of 40–170 °C. The prerequisite for the research was the lack of information regarding changes in the physical-mechanical and ergonomic characteristics of material assemblies during their use under high-temperature conditions. During the initial stage of research, there was a problem associated with the selection of the most important and the exclusion of the least significant indicators, in order to further reduce the number of experimental tests in laboratory and industrial conditions. The authors used the method of expert evaluations to solve the problems related to the selection of the most significant indicators for material assemblies. Material assemblies were formed by varying the combinations of heat-resistant, heat-insulation and lining layers of materials. Initial information for the proposed method was obtained from the experimental tests of sixteen material assemblies. According to the results of the ranking, the main parameters of material assemblies were identified as follows: the temperature range for which the use of clothing is intended, thickness, mass per unit density, rupture resistance, relative tearing elongation, change in linear dimensions during mechanical loads, air permeability and change in assembly thickness during cyclic loads. It was established that the assembly that includes heat-resistant material of the Nomex comfort N.307 220 top, Nomex Serie 100 heat-insulation lining and Nomex TER 135 lining provides the necessary level of protection, reliability and ergonomics, and meets cost requirements.

Keywords: laboratory testing, personal protective equipment, uptime, the level of reliability, industrial hazardous, multi-layered material assemblies, metallurgy, thermal aging

## Izvleček

V članku je predlagana računsko-eksperimentalna metoda izbire materialov za oblikovanje toplotnozaščitnih oblačil v predvidenem temperaturnem okolju 40–170 °C. Informacije o spremembi fizikalno-mehanskih in ergonomskih lastnosti kompletov materialov pod vplivom visokotemperaturnih učinkov, ki so bile potrebne za raziskavo, niso bile poznane. V začetni fazi raziskave je bilo treba določiti najpomembnejše in izključiti najmanj vplivne kazalnike, kar je omogočilo nadaljnje zmanjševanje števila eksperimentalnih testov v laboratorijskih in industrijskih razmerah. Avtorji so z metodo ekspertnih evalvacij odpravili probleme, povezane z izbiro najpomembnejših kazalnikov kompletov materialov. Kompleti materialov so bili oblikovani s kombiniranjem toplotnoodpornih in toplotnoizolativnih materialov in materialov za podloge. Vhodne informacije za predlagano metodo so pridobili z eksperimentalnimi meritvami šestnajstih kompletov materialov. Glede na rezultate razvrstitve so bili opredeljeni bistveni parametri priprave kompletov materialov, in sicer temperaturno območje, za katero je namenjena raba oblačila, debelina, ploščinska masa, odpornost proti trganju, relativno raztezanje pri trganju, sprememba dimenzij med mehanskim obremenjevanjem, zračna prepustnost in sprememba debeline kompleta pri cikličnih obremenitvah. Ugotovljeno je bilo, da potrebno raven zaščite, zanesljivosti, ergonomije in stroškov zagotavlja komplet, ki je vseboval toplotno odporen material Nomex comfort N.307 220, toplotno izolativno plast Nomex Serie 100 in podlogo Nizdelan TER 135.

*Ključne besede:* osebna varovalna oprema, dejavniki tveganja v industriji, večplastni kompleti materialov, metalurgija, toplotno staranje

## 1 Introduction

The creation of personal protective equipment new types and models of protective clothing designed for work under high temperatures does not lose its relevance due to the emergence of new heat-resistant and fire-resistant materials. Modern thermal protective materials have the original raw material structure of fibres, which are subjected to fire-resistant and anti-static treatment [1, 2]. Designers of thermal protective clothing (TPC) have the ability to create ergonomic kits with the necessary protective properties.

According to [3], personal protective equipment must clearly comply with the list and levels of possible industrial hazardous factors. At the same time, they must meet the requirements of ergonomics and not create any additional risks and other adverse factors under the intended conditions of use. The main purpose of personal protective equipment is to guarantee the safety of the worker without hindering the performance of their basic functions. Such requirements can be met through the selection of appropriate materials, which further determine the basic protective functions, the level of reliability and ergonomics of protective clothing and personal protective equipment of common use.

The aim of TPC is to limit the risks associated with work under elevated ambient temperatures [4]. The need for the use of TPC arises at metallurgical plants, at mining, foundry and forging shops, at manufacturing enterprises involved in construction and glass products, and in the engine rooms of power

plants etc. At such enterprises, the temperature in the production facilities may fluctuate in the range of 40–170 °C, while work is performed under heavy physical loads. An analysis of the problem of creating effective TPC reveals that its existing varieties partially prevent the access of thermal energy from an external source to the body of the worker, but also limit the process of human heat exchange. Mandatory requirements regarding protective, physical-mechanical and thermophysical characteristics are considered in TPC materials.

The latest heat-resistant materials are distinguished by their raw material composition. Natural fabrics are predominantly used, but the proportion of synthetic and mixed fabrics is increasing over time [5, 6]. The right selection of materials in assemblies plays a leading role in designing TPC that meets the intended requirements. The properties of TPC are determined primarily by the characteristics of material assemblies, which can be divided into four main groups:

- thermophysical characteristics, which determine the range of temperatures at which the material does not lose its protective properties, and the level of fire resistance;
- physical-mechanical characteristics, which determine resistance to mechanical load (tear, bending, puncture, shrinkage, etc.), which in turn as a significant effect on the service life, maintainability and possibility of multiple cleanings;
- ergonomic characteristics, which affect the level of comfort in use (weight and thickness) and mitigate additional risks during movement and heat

load (air permeability, thermal conductivity, water vapor permeability, hygroscopicity and biological inertness); and

- the index of cost-effectiveness, which determines the competitiveness of a product. The cost of the material assembly dictates 60–80% of the cost of a garment.

Appropriate thermo-physical indices make it possible to provide the desired level of protection against prolonged exposure to high temperatures. Physical-mechanical parameters determine the weight of the product and the level of mechanical strength of the structure. Ergonomic indices ensure compatibility with the user (size), taking into account performed movements and working positions in the context of the workload, and with other personal protective equipment that is intended to simultaneously protect individual parts of a worker's body (head, arms and legs). The desire to solve complex problems during the creation of TPC leads to the use of multilayer material assemblies with the consistent arrangement of heat-resistant, heat-insulation and lining layers [8]. Developing a material assembly structure is a challenging task because the selection of materials and their integration into a single system is influenced by a number of requirements, some of which are controversial. In the course of designing, the goal should be to agree on the physical-mechanical and thermophysical parameters, and the requirements for ergonomics and reliability. For example, increasing the number of layers and additional elements makes it possible to improve the protective properties, and increase the level of reliability and uptime. On the one hand, the greater the number of elements, the easier it is to realize a high level of protection, including the possibility of reinforcing the design through the principle of the differentiation of protection (reinforced zonal arrangement of assemblies with one or more thermal insulation layers in the assemblies) in separate areas of the human body [9]. On the other hand, a large number of individual elements increases the likelihood of damage to the structure and leads to an increase in the mass of a product. Obviously, the choice of elements and their degree of detail may vary depending on indicators of the conditions of use and the resolution of certain problems regarding the level of protection and length of use.

A number of design requirements have been established for TPC. Several standardized (mandatory) indicators apply to materials that can be used for the production of TPC: protective properties are deter-

mined by the heat resistance of the surface layer; the weight of clothes is determined by the mass per unit area of materials that are part of the assembly and by the number of layers; and the guaranteed length of use under increased mechanical loads is determined by the physical-mechanical characteristics of materials. Such requirements and restrictions necessitate the careful selection of the appropriate materials.

It is known that material assemblies have indicators that are different from those of individual materials, and vary depending on the impact of an aggressive production environment.

The aim of this article was to experimentally determine the physical-mechanical and ergonomic characteristics of material assemblies under external effect of elevated temperatures between 40–170 °C. The material assemblies are intended for the creation of thermal protective clothing for the employees of metallurgical enterprises. The prerequisite for the research was the lack of information regarding changes in the physical-mechanical and ergonomic characteristics of material assemblies during their use under high-temperature conditions. This effect leads to the thermal aging of textile materials and, as a consequence, to changes in their declared properties and indices [7]. The choice of effective material assemblies presupposes the availability of reliable information regarding the main characteristics that meet the conditions of their use.

## 2 Materials and methods

The study was performed for certain samples of heat-resistant top materials (nine samples of heat-resistant top materials) (Table 1), the insulation pad (three samples) (Table 2) and lining (two samples) (Table 3). Manufacturers provided statements regarding raw material composition, structure, mass per unit area, thickness, change in linear dimensions after wet treatments and breathability.

For the heat-resistant layer, samples of fabrics that meet the thermophysical requirements for TPC, with fire-resistant and oil-protecting properties, and an antistatic effect, were selected. The selected samples of the Proban KS-52, FlameStat Lite, Tecasafe HA 9001 top materials were treated with a special impregnation for protection against high temperatures, while the Nomex BV-107, Nomex ADV 240 and Nomex comfort N307 fabrics contain heat-resistant fibre in their composition [10].

The main purpose of the heat-insulation layer is to provide protection against heat stress and create a comfortable clothing microclimate of TPC.

The lining layer was used to ensure hygienic requirements for water and vapor absorption, and biological inertia [13].

Table 1: Characteristics of heat-resistant materials

Material	Coded marking	Producer	Raw material composition (%)	Mass per unit area (g/m <sup>2</sup> )	Thickness (mm)
Nomex BV-120	R1	TenCate Protect, Netherland	Nomex (100%)	265	0.43
Proban XB 9340	R2	TenCate Protect, Netherland	Cotton (75%); Kevlar (25%); impregnation Proban	340	0.50
FlameStat Lite	R3	Carrington, Great Britain	Cotton (100%); impregnation Proban, antistatic	250	0.44
Tecasafe XA 9001	R4	Carrington, Great Britain	PAN (54%); cotton (45%); static-control (1%)	360	0.45
Nomex BV-107	R5	TenCate Protect, Netherland	Nomex (94%); Kevlar (5%); static-control (1%)	230	0.45
Proban KS -52	R6	TenCate Protect, Netherland	Cotton (100%); fire-resistant treatment	343	0.58
Nomex comfort N.307 180	R7	Estambril, Spain	Nomex (93%); Kevlar (5%); static-control (2%)	180	0.41
Nomex comfort N.307 220	R8	Estambril, Spain	Nomex (93%); Kevlar (5%); static-control (2%)	220	0.45
Nomex ADV 240 GRI RS	R9	Estambril, Spain	Para-aramid (60%); Nomex (40%)	240	0.60

Table 2: Characteristics of heat-insulation materials

Material	Coded marking	Producer	Raw material composition (%)	Mass per unit area (g/m <sup>2</sup> )	Thickness (mm)
Needled fabric RigChief	I1	Daletec, Norway	Cotton (100%); impregnation Pyrovatex, antistatic	100	2.8
Needled batt fabric	I2	Netkam, Ukraine	Wool (55%); cotton (20%); viscose (25%)	110	3.5
Needled fabric Nomex Serie 100	I3	Dupont, USA	Viscose ignifuga (50%); Nomex (43%); Kevlar (5%); static-control (2%)	100	3.0

Table 3: Characteristics of lining materials

Material	Coded marking	Producer	Raw material composition (%)	Mass per unit area (g/m <sup>2</sup> )	Thickness (mm)
Cotton sheeting uniformly dyed, art. 1667/25381	L1	Cherkaschkyi Schovkovyi Kombinat (Cherkaschkyi Silk Plant), Ukraine	Cotton (100%)	195	0.41
Nomex TER 135	L2	Dupont, USA	Nomex III N.302 (50%), Viscose FR (50%)	135	0.38

Table 4: Composition material assemblies

Number of assemblies	Coded composition of assemblies	Heat-resistant material	Heat-insulation material	Lining material
1	R1 I1 L1	Nomex BV-120	Needled fabric RigChief	Cotton sheeting uniformly dyed, art. 1667/25381
2	R2 I1 L1	Proban XB 9340	Needled fabric RigChief	Cotton sheeting uniformly dyed, art. 1667/25381
3	R3 I1 L2	FlameStat Lite	Needled fabric RigChief	Nomex TER 135
4	R4 I2 L1	Tecasafe XA 9001	Needled batt fabric	Cotton sheeting uniformly dyed, art. 1667/25381
5	R5 I2 L1	Nomex BV-107	Needled batt fabric	Cotton sheeting uniformly dyed, art. 1667/25381
6	R6 I2 L1	Proban KS -52	Needled batt fabric	Cotton sheeting uniformly dyed, art.1667/25381
7	R7 I3 L2	Nomex comfort N.307 180	Needled fabric Nomex Serie 100	Nomex TER 135
8	R8 I3 L2	Nomex comfort N.307 220	Needled fabric Nomex Serie 100	Nomex TER 135
9	R9 I3 L2	Nomex ADV 240 GR I RS	Needled fabric Nomex Serie 100	Nomex TER 135

Assemblies of selected materials were developed for further experimental studies (Table 4). Samples of assemblies were developed in order to simulate indicators of individual elements of TPC under production

conditions by varying combinations of heat-resistant, heat-insulation and lining material layers.

During the initial stage of the research, there was a problem associated with the selection of the most important and the exclusion of the least significant indicators in order to further reduce the number of experimental tests in laboratory and industrial conditions. The generally accepted procedure of screening factors due to the large number of indicators is quite cumbersome and time consuming, and requires a great deal of time to implement the necessary measures [12]. Such difficulties can be overcome through the use of expert opinions, and by taking into account the ability of the designer to make rational decisions when it is impossible to fully formalize them. The authors used the method of expert evaluations to solve the problems related to the selection of the most significant indicators for material assemblies.

Expert evaluation to determine the main indicators of the material assemblies involved the formation of a group of experts, the surveying of that group, the processing of the expert evaluation, an analysis of the results and an assessment of the reliability of the expert evaluation performed by the group. During the first stage, twelve respondents were selected, including specialists from TPC manufacturing companies, user representatives, and specialists from research and testing centres and laboratories in the textile industry. They were given the task of ranking the group

indicators of material assemblies in accordance with their impact on the predicted properties of TPC. The method of direct evaluation of the ranking of the initial requirements was difficult to use due to the large number of characteristics and their indicators. The indicators that were analysed are different in terms of both quantitative estimates and qualitative characteristics. Therefore, the use of standard methods of expert evaluation is inefficient and requires adaptation to the specifics of the tasks. A method for determining the rank of a group of indicators on the basis of a normalized significant coefficient was proposed [13]. Experts identified four groups that determine the requirements for the physical-mechanical, thermo-physical, ergonomic and economic characteristics of the material assembly. For each group, the normalized indicators are defined in the following sequence:

1. during ranking  $m$ , experts were asked to place  $n$  requirements in the order they consider most reasonable and assign to each requirement  $X$  rank from 1 to  $N$ . According to this, rank 1 received the most preferred indicator, and rank  $N$  – the least important; and
2. the normalized coefficient  $K_q$  for each of the  $q$  group of requirements was determined using the formula:

$$K_q = \frac{\sum_{j=1}^m X_q^j}{\sum_{q=1}^n \sum_{j=1}^m X_q^j} \quad (1),$$

where  $X_q^j$  represents the rank of the  $q$ -th group of requirements, given by  $j$ -th expert;  $q = 1 \dots n$ ;  $j = 1 \dots m$ . In the calculations,  $n = 4$  and  $m = 12$ .

After ranking, the following results were obtained (Table 5).

In the second stage, the experts were offered a ranking of indicators in each group of characteristics. According to the algorithm, the normalized coefficients  $K_i$  for 20 materials was determined. The total normalized ranking factor  $K$  of alternative solutions was calculated using the formula:

$$K = K_q \cdot K_i \tag{2}$$

where  $K_i$  represents the normalized coefficient for each  $i$ -th group of indicators.

According to the results of ranking, the most significant 10 indicators with respect to material assemblies were singled out (see Table 6).

The reliability of the evaluation of the expert group depends on the level of agreement of the opinions of the experts participating in the survey. The consistency of experts' opinions is assessed using the generalized coefficient  $W$ , which is defined as a generalized rank correlation coefficient for a group comprising  $m$  experts [14]. The generalized coefficient is calculated with the help of "Concurrer" program. The values of the calculated generalized coefficients were

Table 5: Ranking of requirements with respect to material assembly

Characteristics	Physical mechanical	Thermophysical	Ergonomic	Economic
Normalized coefficient, $K_q$	0.24	0.17	0.28	0.31
Rank	2	1	3	4

Table 6: Ranking of indicators of materials

Characteristics	Indicator	Normalized coefficients			Rank
		$K_i$	$K_q$	$K$	
Thermophysical	Temperature range for the use of clothing	0.013	0.17	0.002	1
	Antistatic (ohmic resistance)	0.088	0.17	0.015	12
	Fire resistance	0.092	0.17	0.016	14
Physical mechanical	Thickness	0.028	0.24	0.006	6
	Mass per unit area	0.022	0.24	0.005	5
	Rupture resistance (warp)	0.015	0.24	0.003	2
	Rupture resistance (weft)	0.016	0.24	0.004	3
	Relative tearing elongation (warp)	0.058	0.24	0.014	9
	Relative tearing elongation (weft)	0.059	0.24	0.014	10
	Fabric stiffness	0.083	0.24	0.019	20
	Change of assembly thickness during cyclic loads	0.053	0.24	0.012	7
	Change of linear dimensions during mechanical loads	0.055	0.24	0.013	8
Ergonomic	Air permeability	0.015	0.28	0.004	4
	Thermal conductivity	0.053	0.28	0.015	11
	Vapor permeability	0.055	0.28	0.015	13
	Water-resistance	0.06	0.28	0.017	17
	Water absorbency	0.057	0.28	0.016	15
	Biological hazard	0.058	0.28	0.016	16
Economic	Unit costs	0.059	0.31	0.018	18
	Materials output ratio	0.061	0.31	0.019	19

in the range  $W = 0.79–0.88$  for the ranking of the tasks of four groups of characteristics. Such values indicate a high level of consensus between the experts. Based on the results of the rating assessment by specialists in various fields, the main indicators that received the highest ranks (from 1 to 10) were selected for further research: the intended temperature range for the use of clothing, thickness, mass per unit area, rupture resistance, relative tearing elongation, change in linear dimensions during mechanical loads, air permeability and change in assembly thickness during cyclic loads.

The practice of determining the quantitative parameters of material assemblies involves experimental tests. In laboratory conditions, the characteristics of materials are determined under normal conditions (temperature ranging from 20–22 °C and relative humidity ranging between 40–60%).

The results of experimental tests are given as a deterministic quantity, which is represented by the mathematical expectation (average value) of a random variable. The obtained parameters were interpolated under the assumption of linearity and the invariance of the initial conditions, which does not correspond to real operating conditions. It is known that the structure and properties of textile materials change during operation, and high temperature has destructive effect on textile materials [15]. This effect leads to the thermal aging of materials and thus to the gradual failure of TPC. It is therefore expedient to study experimental changes in the quantitative parameters of the main indicators during the simulation of high-temperature effects.

To determine the mathematical expectation of the corresponding characteristic  $Z_i$  ( $i = 1, 2, \dots$ ) with a given accuracy,  $n$  of independent tests should be performed.

We give the mathematical expectation of each of the characteristics as

$$\hat{Z}_i = \hat{Z}_i \quad (3),$$

where  $\hat{Z}_i$  represents the estimate of the mathematical expectation of the desired random parameter.

The value of  $\hat{Z}_i$  will be in the interval

$$\underline{Z}_i - \Delta \leq \hat{Z}_i \leq \underline{Z}_i + \Delta \quad (4),$$

where  $\Delta$  represents the upper bound of error of calculation results, which is determined by the known

value of route-mean-square deviation  $\sigma(Z)$  and the given accuracy  $K_r$ ,

$$\Delta = \frac{K_r \cdot \sigma(Z)}{\sqrt{n}} \quad (5).$$

It follows from formula (5) that the total number of tests  $n$  in laboratory conditions for each indicator will be:

$$n = \left( \frac{K_r \cdot \sigma(Z)}{\Delta} \right)^2 \quad (6).$$

As previous tests have shown, at a given accuracy of 0.95, the required number of measurements is in the range from 5 to 12.

Changes in the performance of assemblies during the tests were characterized by the magnitude of deviations from estimated mathematical expectation, which is calculated using the formula:

$$\delta = \frac{A_1 - A_2}{(A_1 + A_2)/2} \cdot 100\% \quad (7),$$

where  $A_1$  is the estimate of the mathematical expectation the parameters that have been measured before thermal exposure;  $A_2$  is the same parameter after thermal exposure.

For assemblies that are planned for use in the production of TPC, the tests were performed in the laboratory according to standardized methods [16, 17]. Prepared samples of material assemblies were pre-conditioned in a heat chamber at an air temperature of 22–24 °C and relative air humidity of 50–60%. The assemblies were formed in layers and threaded along one side in the direction of the base. Tests to determine air permeability were performed under a pressure of 50 Pa with the help of a FF-12 device (Hungary). The assemblies' thickness change test was performed by applying a variable load of  $P_1 = 50–1100$  kPa for 30 seconds and unloading  $P_{unl} = 1100–50$  kPa for five minutes after each of the ten cycles.

To determine the change in the linear dimensions of the assemblies, samples with a size of 300 mm x 300 mm were formed. Control lines 200 mm long and 200 mm wide were applied on the upper and inner layers of the materials. The simulation of high-temperature effects was carried out in the mode of preliminary thermal exposure on material assemblies in a "Rohs" heat chamber (SPC Technology, Ukraine) at temperature 170 °C  $\pm$  5 °C, for 15 minutes.

The research was performed in the accredited “Textile - TEST” analytical and research testing laboratory at the Kyiv National University of Technology and Design.

### 3 Results

#### 3.1 Indices of the physical-mechanical characteristics of assemblies and changes thereto under the effects of temperature

Prior to testing, the selected material assemblies were kept in a heat chamber to prevent exposure to environmental moisture (1 day). Mass per unit area measurements were performed before and after thermal exposure (Table 7).

As follows from Table 7, the assemblies nos. 2 and 6 have the highest mass per unit area values. The fif-

teen-minute temperature effect caused the mass per unit area to change due to the thermal aging of the materials. Therefore, it is possible to assume further changes in other physical-mechanical characteristics. Based on the results of studies, it was determined that assemblies nos. 2 and 3 can be excluded from further testing. The mass per unit area of assemblies nos. 2 and 3 increased, which will lead to an increase in the mass of TPC during use.

Experimental tests to determine the rupture resistance and relative tearing elongation of the material assemblies were performed taking into account the conditions of wearing ready-made clothing (Table 8). The size of the lining layer was assumed to be 10% larger than the surface layer in terms of structural features. Due to such features, the tearing of the heat-resistant, heat-insulation and lining layers of the assemblies occurred simultaneously.

Table 7: Mass per unit area indices of material assemblies

Assembly number	Coded composition of assemblies	Mathematical expectation and confidence interval of mass per unit area ( $\text{g}/\text{m}^2$ )		Deviation of mass per unit area (%)
		Before thermal exposure	After thermal exposure	
1	R1 I1 L1	$541 \pm 4$	$534 \pm 5$	-1.30
2	R2 I1 L1	$754 \pm 9$	$771 \pm 11$	2.23
3	R3 I1 L2	$543 \pm 6$	$553 \pm 8$	1.82
4	R4 I2 L1	$731 \pm 9$	$728 \pm 11$	-0.41
5	R5 I2 L1	$662 \pm 6$	$661 \pm 7$	-0.15
6	R6 I2 L1	$778 \pm 12$	$790 \pm 16$	1.53
7	R7 I3 L2	$463 \pm 2$	$455 \pm 4$	-1.74
8	R8 I3 L2	$518 \pm 9$	$514 \pm 3$	-0.78
9	R9 I3 L2	$534 \pm 2$	$524 \pm 3$	-1.89

Table 8: Indices of rupture resistance

Assembly number	Coded composition of assemblies	External force direction	Mathematical expectation and confidence interval of rupture resistance (N)		Deviation of rupture resistance (%)
			Before thermal exposure	After thermal exposure	
1	R1 I1 L1	warp	$224 \pm 3$	$218 \pm 5$	-2.71
		weft	$175 \pm 8$	$163 \pm 6$	-7.10
4	R4 I2 L1	warp	$173 \pm 4$	$183 \pm 5$	5.62
		weft	$81 \pm 5$	$79 \pm 3$	-2.5
5	R5 I2 L1	warp	$197 \pm 4$	$199 \pm 5$	1.01
		weft	$116 \pm 6$	$126 \pm 5$	8.26
6	R6 I2 L1	warp	$153 \pm 5$	$143 \pm 6$	-6.76
		weft	$93 \pm 3$	$92 \pm 5$	-1.08

Assembly number	Coded composition of assemblies	External force direction	Mathematical expectation and confidence interval of rupture resistance (N)		Deviation of rupture resistance (%)
			Before thermal exposure	After thermal exposure	
7	R7 I3 L2	warp	156 ± 6	147 ± 6	-5.94
		weft	133 ± 4	146 ± 3	9.32
8	R8 I3 L2	warp	163 ± 6	173 ± 6	5.95
		weft	156 ± 1	167 ± 4	6.81
9	R9 I3 L2	warp	245 ± 3	242 ± 5	-1.23
		weft	200 ± 7	183 ± 6	-8.88

As follows from Table 8, the assembly no. 9 (R9 I3 L2) had the largest breaking force, while assemblies nos. 1, 6 and 9 demonstrated a decrease in value for up to 9% after thermal exposure.

The results of the determination of the relative tearing elongation of the thermal protection assemblies before and after thermal exposure are shown in Table 9.

According to the test results, the values of relative elongation decreased by 13% and 11% respectively in material assemblies nos. 1 and 9 with the coded composition R1 I1 L1 and R9 I3 L2. The largest increase in elongation was recorded for assembly no. 4 (R4 I2 L1) – up to 40%.

### 3.2 Ergonomic performance of the assemblies

*and changes thereto under the effect of temperature*

In the design of protective clothing, preference is given to materials that will subsequently ensure the stable linear dimensions of clothing parts and individual elements during operation [18]. Tests were performed to determine the change in linear dimensions of the assemblies before and after thermal exposure (Table 10). According to the results of the tests, it can be stated that all assemblies have stable dimensions after thermal exposure, as the change in linear dimensions does not exceed 0.4%.

TPC should be used continuously for the work shift (up to four hours). Work is performed under a high mechanical load. During their activity, a worker performs work, converting mechanical energy into

Table 9: Value of relative tearing elongation

Assembly number	Coded composition of assemblies	External force direction	Mathematical expectation and confidence interval of relative tearing elongation (%)		Deviation of elongation (%)
			Before thermal exposure	After thermal exposure	
1	R1 I1 L1	warp	16 ± 1	14 ± 1	-13.33
		weft	6 ± 1	5 ± 1	-18.18
4	R4 I2 L1	warp	16 ± 2	24 ± 2	40.00
		weft	16 ± 1	19 ± 2	17.14
5	R5 I2 L1	warp	16 ± 2	20 ± 1	22.22
		weft	17 ± 2	20 ± 1	16.21
6	R6 I2 L1	warp	10 ± 2	12 ± 1	18.18
		weft	16 ± 1	18 ± 2	11.76
7	R7 I3 L2	warp	39 ± 1	50 ± 1	24.72
		weft	38 ± 2	40 ± 1	5.13
8	R8 I3 L2	warp	37 ± 1	41 ± 2	10.26
		weft	41 ± 2	48 ± 1	15.73
9	R9 I3 L2	warp	19 ± 1	17 ± 1	-11.11
		weft	7 ± 1	6 ± 1	-15.38

Table 10: Values of change in the linear dimensions of thermal protection assemblies

Assembly number	Coded composition of assemblies	External force direction	Mathematical expectation and confidence interval of linear dimensions (mm)		Change in linear dimensions (%)
			Before thermal exposure	After thermal exposure	
1	R1 I1 L1	warp	197.8 ± 3.8	197.8 ± 4.9	0
		weft	197.6 ± 1.0	197.4 ± 1.4	-0.10
4	R4 I2 L1	warp	199.3 ± 1.3	198.4 ± 1.5	-0.45
		weft	199.7 ± 1.9	199.2 ± 2.0	-0.25
5	R5 I2 L1	warp	201.5 ± 2.5	201.7 ± 2.2	0.10
		weft	195.8 ± 1.8	195.4 ± 1.7	-0.20
6	R6 I2 L1	warp	199.6 ± 1.4	199.5 ± 1.3	-0.05
		weft	200.3 ± 1.9	199.6 ± 3.6	-0.35
7	R7 I3 L2	warp	198.0 ± 2.6	198.0 ± 2.6	0
		weft	198.0 ± 2.0	198.0 ± 2.0	0
8	R8 I3 L2	warp	199.8 ± 1.5	198.8 ± 2.1	0.05
		weft	199.8 ± 1.5	199.7 ± 1.5	-0.05
9	R9 I3 L2	warp	199.6 ± 1.0	197.9 ± 4.9	0
		weft	199.6 ± 1.0	199.4 ± 1.4	-0.10

thermal energy. Under conditions of high ambient temperatures, this can lead to the disruption of heat transfer processes in the body of a worker. Changes in the functional state of the body are caused by the muscular work (static and dynamic) of the torso, and upper and lower extremities [19]. The intensity of heat generation depends on physical activity and the duration thereof, the human and health conditions, the environmental (ambient) temperature, pressure, humidity and air velocity. In different levels of activity, a worker emits from 250 W to 450 W of heat, compared with 100–150 W at rest, meaning that the comfort

zone of a worker at rest does not coincide with the comfort zone under a work load [20, 21]. Under such conditions, the air permeability of the assemblies is an important indicator [22] (Table 11).

Based on the results of studies, it was determined that assemblies nos. 1 and 9 had the highest levels of breathability. The results of the experiment showed an increase in breathability of up to 4% after thermal exposure, which is due to the uneven structure of such material assemblies.

Under the effect of an external high-temperature environment on a worker in TPC, an increase in the dura-

Table 11: Values of air permeability

Assembly number	Coded composition of assemblies	Mathematical expectation and confidence interval of air permeability ( $\text{dm}^3\text{m}^{-2}\text{s}^{-1}$ )		Deviation in breathability (%)
		Before thermal exposure	After thermal exposure	
1	R1 I1 L1	54.6 ± 2.1	56.8 ± 2.3	3.95
4	R4 I2 L1	11.1 ± 1.4	11.9 ± 1.4	6.96
5	R5 I2 L1	22.6 ± 1.2	22.3 ± 1.8	-1.34
6	R6 I2 L1	22.8 ± 1.3	22.5 ± 1.1	-1.33
7	R7 I3 L2	49.8 ± 2.2	51.6 ± 2.9	3.55
8	R8 I3 L2	27.7 ± 1.2	28.6 ± 1.7	3.19
9	R9 I3 L2	90.04 ± 4.2	92.75 ± 4.2	3.06

tion of comfort for the human body temperature in the under-clothing space can be achieved by increasing the thickness of the insulating layer [23]. Improving the protective properties of TPC from mechanical stress in terms of design is possible by increasing the thickness of the assemblies in the areas of shoulders and chest, knees and elbows. Such a prerequisite has led to the performance of multi-cycle loading and

unloading tests for multilayer assemblies before and after thermal exposure. Such tests were performed for assemblies containing one heat-insulation layer (Table 12) and two heat-insulation layers (Table 13).

Table 12: Value of changing the thickness of assemblies with a single heat-insulation layer

Assembly number	Coded composition of assemblies	Dynamic deforming force (kPa)	Mathematical expectation and confidence interval of material assembly thickness (mm)		Deviation in thickness (%)
			Before thermal exposure	After thermal exposure	
1	R1 I1 L1	100	4.12 ± 0.16	4.14 ± 0.06	0.48
		1000	2.83 ± 0.18	2.88 ± 0.24	1.75
4	R4 I2 L1	100	4.56 ± 0.06	5.14 ± 0.13	11.96
		1000	2.51 ± 0.13	3.09 ± 0.20	20.71
5	R5 I2 L1	100	4.87 ± 0.15	5.48 ± 0.19	11.79
		1000	2.45 ± 0.16	3.10 ± 0.21	23.42
6	R6 I2 L1	100	5.57 ± 0.26	6.03 ± 0.28	7.93
		1000	3.75 ± 0.15	4.17 ± 0.09	10.61
7	R7 I3 L2	100	3.60 ± 0.09	3.60 ± 0.09	0
		1000	2.35 ± 0.06	2.38 ± 0.11	1.27
8	R8 I3 L2	100	4.00 ± 0.07	4.13 ± 0.15	3.2
		1000	2.70 ± 0.04	2.88 ± 0.23	6.45
9	R9 I3 L2	100	4.08 ± 0.27	4.15 ± 0.08	1.21
		1000	2.72 ± 0.15	2.76 ± 0.20	1.46

Table 13: Value of changing the thickness of assemblies with two heat-insulation layers

Assembly number	Coded composition of assemblies	Dynamic deforming force (kPa)	Mathematical expectation and confidence interval of material assembly thickness, mm		Deviation in thickness (%)
			Before thermal exposure	After thermal exposure	
10	R1 2I1 L1	100	7.63 ± 0.22	7.64 ± 0.32	0.13
		1000	5.14 ± 0.27	5.20 ± 0.29	1.16
11	R4 2I2 L1	100	8.10 ± 0.46	8.02 ± 0.20	-0.99
		1000	5.13 ± 0.28	5.16 ± 0.45	0.58
12	R5 2I2 L1	100	7.93 ± 0.29	7.6 ± 0.74	-4.25
		1000	4.91 ± 0.38	4.92 ± 0.44	0.20
13	R6 2I2 L1	100	9.01 ± 0.65	8.85 ± 0.90	-1.79
		1000	5.45 ± 0.24	5.56 ± 0.45	1.2
14	R7 2I3 L2	100	6.8 ± 0.13	6.55 ± 0.10	-3.75
		1000	4.26 ± 0.15	4.30 ± 0.10	0.93
15	R8 2I3 L2	100	7.28 ± 0.17	7.11 ± 0.30	-2.36
		1000	4.90 ± 0.10	4.98 ± 0.38	1.62
16	R9 2I3 L2	100	7.44 ± 0.32	7.41 ± 0.55	-0.40
		1000	5.0 ± 0.34	5.12 ± 0.30	2.37

As follows from Tables 12 and 13, when using one thermal insulation layer, the mechanical loading and thermal treatment of the assemblies increase the thickness of assemblies nos. 4, 5, 6, and 8 (up to 20%), and

has practically no effect on the thickness of assemblies nos. 1, 7 and 9. Assemblies with two heat-insulation layers maintained a stable size after multi-cycle loading.

Determining the calculated parameters based on the results of experimental tests involved estimating the mathematical expectation of each indicator with a given accuracy of 0.95. Under such conditions, the number of experimental tests included 5–12 measurements.

According to the results of tests and calculations, it was determined that it is advisable to develop TPC using assembly no. 8 or 9. Comparing the cost of the materials in these two assemblies led to giving preference to assembly no 8.

The results of experimental tests and theoretical research were used during the development of a set of protective clothing for the workers of metallurgical enterprises. The set is designed to perform work under the external effect of elevated temperatures within the range 40–170 °C. The clothes are made of an assembly consisting of heat-resistant material of the Nomex comfort N.307 220 top, Nomex Serie 100 heat-insulation lining, and Nomex TER 135 lining. New design and technological solutions for clothes that consist of a jacket and overalls or trousers with overlays are offered. The total weight of TPC does not exceed 4.5 kg.

The thermal protective layer of the material consumption for an assembly is 6.8–8.1 m<sup>2</sup>. During the manufacture of the products of the set seams of different purpose were used.

## 4 Conclusion

Protective clothing is designed to ensure that, under the foreseeable conditions of its use, a worker can perform the risk-related work normally with the highest possible degree of protection.

The performed studies made it possible to formulate an improved sequence of execution of works on the design of TPC, which is intended for protection against the high temperatures of the external production environment. The required properties of TPC can be achieved by applying available information regarding the indicators of relevant materials. During the first stage, requirements relevant to the indices of the materials that are of significant importance during the creation of TPC were defined: protective, physical-mechanical, ergonomic and economic. It

was shown that the achievement of all requirements is possible when multi-layered material assemblies are used. The tasks related to the selection of the most significant indicators for material assemblies were solved using the method of expert evaluations through the determination of the ranks of the indicators on the basis of standardized coefficients. The consistency of experts' opinions was assessed using the concordance coefficient. During the second stage, nine variants of three-layer material assemblies were developed. Changes in mass per unit area, rupture resistance, percentage tearing elongation, linear dimensions, thickness during mechanical loads, air permeability under normal conditions and after simulation of thermal effects were determined for each assembly. It was established that the assembly that comprises the Nomex comfort N.307 220 top, Nomex Serie 100 heat-insulation lining and Nomex TER 135 lining provides the necessary level of protection, reliability and ergonomics, and meets cost requirements. The obtained research results were implemented during the creation of thermal protective clothing for the workers of metallurgical enterprises. New design and technological solutions for clothing that consist of a jacket and overalls or trousers with overlays were offered. The total weight of TPC does not exceed 4.5 kg.

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## Effect of Parameters on the Runoff Erosion Control Performance of Structurally Modified Jute and Coir Geomeshes over Loamy Sand

*Vpliv parametrov na površinsko spiranje ilovnatopeščene površine s pomočjo strukturno modificiranih geomrež iz jutnih in kokosovih vlaken*

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### Abstract

Soil erosion is a serious environmental problem that can be controlled using bioengineering techniques. In using a bioengineering technique, temporary reinforcement is performed with geomeshes until vegetation takes root. In this study, structurally modified jute and coir geomeshes were tested for runoff erosion control and runoff volume over loamy sand at different slope angles. The laboratory results revealed that all parameters (slope angle, type of weave and type of material) had a significant effect on the erosion control performance of geomeshes. The slope angle contributed most (52.34%) to runoff erosion control, followed by weave type (25.79%) and type of material (12.28%). At lower and medium slope angles (of 15° and 30°, respectively) the twill-woven structure of coir geomeshes provided better erosion control than plain- and satin-woven structures, while plain-woven jute geomeshes demonstrated better erosion control at all slope angles. To understand the overall impact, a germination test was also conducted. According to the germination test results, the twill weave of jute geomeshes provided the highest rooting length. In general, plain-woven jute geomeshes are preferred for better erosion control on a high slope angle, while plain and twill can be used on a low slope angle.

Keywords: soil erosion, runoff erosion, geomeshes, natural fibre, loamy sand, jute, coir, germination

### Izvleček

Erozija tal je resen okoljski problem, ki ga je mogoče nadzorovati z bioinženirskimi tehnikami. Pri tem z geomrežami prehodno ojačijo tla, dokler se ne vzpostavi vegetacija. V raziskavi so bile proučevane strukturno modificirane geomreže iz jutnih in kokosovih vlaken, namenjene zaščiti tal pred erozijo zaradi spiranja ilovnatnega peska. Laboratorijski rezultati so pokazali, da vsi proučevani parametri, tj. kot naklona tal, tkalska vezava geomrež in njihova surovinska sestava, pomembno vplivajo na učinkovitost nadzora erozije. Na erozijo najbolj vpliva kot naklona (52,34 %), sledita vrsta tkalske vezave (25,79 %) in vrsta vlaken (12,28 %). Pri najmanjšem in srednjem kotu naklona (15°, 30°) je geomreža iz kokosovih vlaken v vezavi keper zagotovila boljši nadzor nad erozijo od geomrež v vezavah platno in atlas. Geomreža iz jutnih vlaken v vezavi platno pa je pokazala najboljši nadzor nad erozijo pri vseh kotih naklona. Za poznavanje celovitega učinka je bil izveden test kalitve semen. Ugotovljeno je bilo, da geomreže iz jutnih vlaken omogočajo največjo dolžino

korenin. *Geomreža iz jutnih vlaken v vezavi platno zagotavlja boljši nadzor nad erozijo pri velikih naklonih terena, za majhen naklon terena pa so primernejše geomreže v vezavah platno in keper.*

*Ključne besede: erozija tal, erozija odtokov, geomreža, naravno vlakno, ilovnat pesek, juta, kokos, kalitev*

## 1 Introduction

Soil is the unconsolidated material sediments and solid particle deposits of disintegrate rocks. The process of the transportation and detachment of top soil along with other organic materials by wind, water and human activity is known as 'soil erosion' [1-3]. This has been the most prevailing problem since the 1930s and, after the 90 years of research, it is still a major concern of researchers. In the last few decades, the global rate of erosion has been exceeding new soil formation by 10 to 20 times. It is the second most pressing environmental problem throughout the world's terrestrial ecosystem, after population growth. According to a report by the World Health Organization, more than 3.7 billion people are malnourished in the world due to the loss of cropland as the result of soil erosion [4]. At present, around 80% of the world's agricultural land suffers moderate to severe erosion, while 10% of that land experiences slight erosion. As the result of soil erosion, about 30% of the world's plowable land has become unproductive and has been abandoned for agricultural use during the last 40 years [4-5]. The United States is losing soil 10 times faster, while China and India are losing soil 30 to 40 times faster than the natural replenishment rate. About 3,280,000 km<sup>2</sup> of land (i.e. 53% of the total land area of the country) is prone to the soil erosion in India. About 29% of total eroded soil is permanently lost to the sea, 10% is deposited in reservoirs resulting in the loss of storage capacity by 1-2% annually, and 61% of eroded soil is being transported from one place to another. Given the seriousness of the soil erosion problem, integrated soil erosion control measures are needed. It has been said that the economics of the situation strongly suggest that soil erosion control is a better policy than sediment removal, as controlling erosion is only 1/15 as expensive as sediment removal. The cost-benefit ratio between erosion control removal is 5.2:1 [4, 7]. The main types of erosion are runoff erosion, interrill, rill, gully and streambank erosion. Among the various types of erosion, runoff erosion induced by runoff rainwater is the most common and results in various minor to major problems. It leads to the loss of soil structure and other organic matter in the soil, along with other devastating problems, such as landslides,

floods and desertification [8]. Moreover, it reduces soil stability, which is an important factor for the construction of roads, embankments, underlays at hill slopes and seashores. The control of soil erosion is important from environmental and engineering aspects. This can be achieved through a proper soil management system or bioengineering techniques [9-10]. Bioengineering techniques use natural vegetation and geotextile materials/Rolled Erosion Control Products (RECPs) to address geotechnical problems. RECPs used in erosion control applications provide temporary support to the soil until permanent vegetation takes root. Hence, open weave RECPs made of natural fibres, such as like coir and jute, are highly preferred in runoff erosion control applications on hill slopes and disturbed lands [11-12]. The biodegradability of natural RECPs nourishes rooted vegetation with nutrients and offers a sustainable solution to control soil erosion and land degradation [13]. According to previous findings, plain-woven coir geomeshes demonstrate 40-60% erosion control, while jute geomeshes demonstrate 70-80% erosion control [13, 14]. It is well known that commercially available, plain-woven RECPs effectively mitigate runoff erosion. However, limited studies are available regarding the performance of structurally modified jute and coir geomeshes, at lower, medium and higher slope angles.

## 2 Materials and methods

### 2.1 Materials

For this study, different jute and coir geomeshes were taken, as shown in Table 1. Jute and coir geomeshes were prepared with an average mesh opening size of 19 x 19 mm and 10 x 12 mm according to commercially available geomeshes. The structure of the geomeshes was changed taking into account a previous study [14].

Full factorial design was used to identify the effect of three independent factors, i.e. type of material, slope angle and type of weave, on erosion control percentages, as shown in Table 2.

The performance of jute and coir geomeshes was studied for soil erosion control using soil from Hoshiarpur (foothills of the Himalayas). Hoshiarpur

Table 1: Properties of jute and coir geomeshes

Material	Type of weave	Linear density (tex)	Diameter (mm)	Mesh opening size (mm)	Weight per unit area (g/m <sup>2</sup> )	Flexural rigidity ( $\mu\text{Nm}$ )			
						Dry		Wet	
						Warp	Weft	Warp	Weft
Jute	Plain	4724	3.9	10 x 12	541.7	1289	989	540	313
	Twill	4724	3.9	10 x 12	541.7	828	634	289	125
	Satin	4724	3.9	10 x 12	541.7	324	227	162	65
Coir	Plain	5090	4.5	19 x 21	580	5753	2877	2034	1423
	Twill	5090	4.5	19 x 21	580	4763	2316	1578	1137
	Satin	5090	4.5	19 x 21	580	3862	1673	732	345

Table 2: Full factorial design

Sr no.	Type of material	Slope angle ( $^{\circ}$ )	Type of weave
1	Jute	15	Plain
2	Coir	30	Twill
3		45	4 end satin

lies at an altitude of 296m and a latitude between 31.51° N and 75.91°. The soil texture was observed through sieve analysis based on IS:2720, and contains 85% sand, 10% silt and 5% clay [15].

## 2.2 Runoff erosion test

A runoff erosion control test was carried out according to the ASTM D 7101 standard, with some modifications in ramp size (Figure 1 (a)) [16]. According to previous

studies, a ramp size of 50 cm length and 25 cm width was used. V jet nozzles were used to simulate rainfall of 100 mm/h for three minutes during each trial. Runoff erosion tests were carried out with soil (soil-infiltration conditions) and without soil (zero-infiltration conditions) to better understand the interaction between soil and geomesh (Figure 1(b) and Figure 1(c)). Under soil-infiltration conditions, soil from the Hoshiarpur region was placed in a soil tray measuring

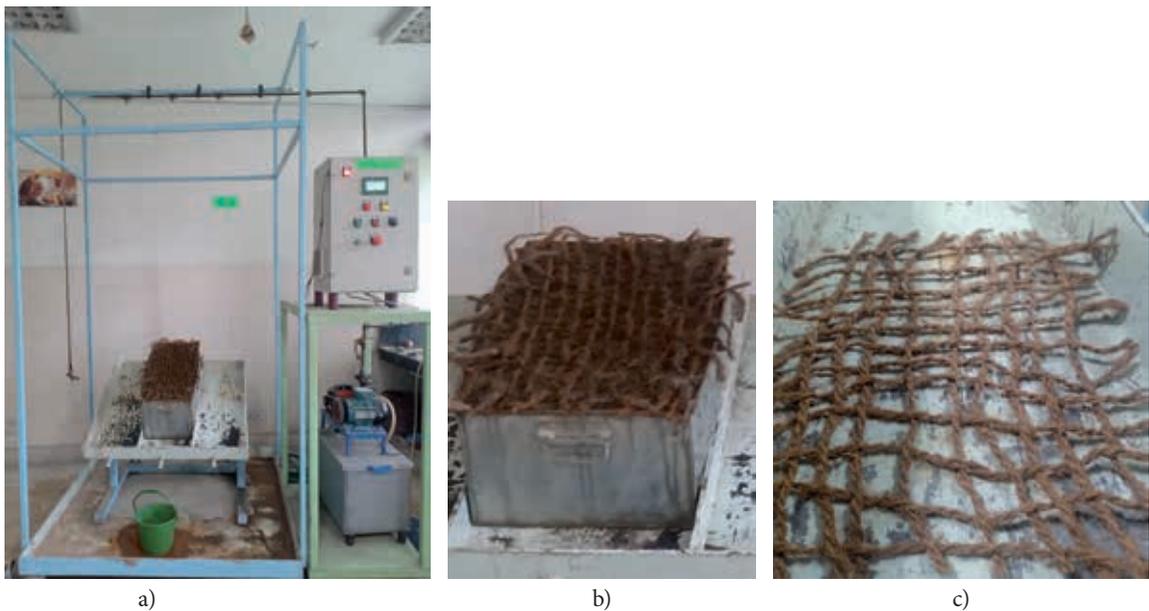


Figure 1: Runoff erosion test at different infiltration conditions: a) runoff erosion set-up, b) soil-infiltration conditions, c) zero-infiltration conditions

50 cm in length, 25 cm in width and 25 cm in depth. Based on the ASTM D 698 standard, soils were compacted in the test tray at 16% moisture content by dropping a 5.50lb rammer from a height of 30 cm. The prepared soil tray was covered with geomesh and placed at different slope angles on the instrument and subjected to rainfall. Rainfall was simulated for three minutes for each sample, while runoff water with eroded soil was collected using the sedimentation method. The first three tests were not considered in the calculation in order to avoid errors that may occur due to the initial absorption of the water in the soil and geomeshes. Similarly, testing was also performed for uncovered soil trays (without geomeshes) to evaluate control test performance. Erosion control percentages can be calculated using the equation 1:

$$\text{Erosion control (\%)} = \frac{E - C}{E} \quad (1)$$

where,  $E$  represents eroded soil without geomesh (g) and  $C$  represent eroded soil with geomesh (g).

Under soil-infiltration conditions, water was absorbed by the soil, which hinders the intended role of geomeshes. To understand the overall effect of geomeshes, a runoff erosion test was carried out under zero-infiltration conditions (without soil). Under zero-infiltration conditions, geomeshes were laid on a smooth surface and subjected to rainfall for three minutes to collect runoff water. Rainfall was then halted for three minutes to collect culmination discharge. Due to the moisture-retaining capacity and storage effect of geomeshes, water continued to drain even after rainfall was stopped. This culmination volume provided the overall runoff erosion performance of geomeshes at different slope angles.

### 2.3 Germination test

A germination test was performed in accordance with the ASTM D 7322 standard, according to which, earthen pots were filled with different soil and sown with an equal number of wheat seeds (60 seeds/pot). Each soil was covered with the different structures of geomeshes, while one was left uncovered. These pots were kept at a uniform temperature, lighting conditions and 30–35% humidity for 21 days. Two trials of germination tests were conducted to ensure the reliability of test results. At the end of 21 days, the percentage of vegetation was calculated using equation 2:

$$\text{Vegetation (\%)} = \frac{\text{Number of plants germinated in the pot with geomesh}}{\text{Number of plants germinated in the pot without geomesh}} \quad (2)$$

The number of roots, length of roots and total rooting were the important factors that determine the soil stability and germination of plants. After 21 days, 10 plants were randomly uprooted, and the average primary root length and average number of roots per plant were measured in each pot [14]. Total rooting length after 21 days was calculated using equation 3.

$$\text{Total rooting length} = N \times n \times L \quad (3)$$

where,  $N$  represents the total number of plants after 21 days,  $n$  represents the average number of roots per plant and  $L$  represents the average primary root length (cm).

## 3 Result and discussion

Structurally modified (plain, twill and satin) jute and coir geomeshes were placed at different slope angles (15°, 30° and 45°) to identify runoff erosion control percentages and runoff volume under soil-infiltration conditions (with soil). The performance of different jute and coir geomeshes is presented in Table 3.

### 3.1 Runoff erosion control performance of jute and coir geomeshes under soil-infiltration conditions

Figure 2 shows the effect of structurally modified jute and coir geomeshes at a 15° slope angle. Twill-woven coir geomeshes demonstrated the highest erosion control percentage (59.1%), while plain and satin demonstrated erosion control percentages of 47.5% and 39.9%, respectively.

It is evident from Figure 2 that erosion control percentage increases when the plain-woven structure of the geomeshes was altered to twill-woven geomeshes and decreases when altered to a satin-woven structure. Coir fibre contains 35–45% lignin, which leads to higher flexural rigidity in the plain-woven structure (Table 1) [17–18]. For this reason, coir geomeshes demonstrated less contact with the soil, resulting in lower erosion control. As the structure of the geomesh is changed from plain to twill, float length at the back side of geomesh increases, which results in lower flexural rigidity. The flexural rigidity of the geomeshes was decreased from 2877  $\mu\text{Nm}$  to 2316  $\mu\text{Nm}$

Table 3: Runoff erosion control performance of jute and coir geomeshes at different slope angle

Type of material	Type of weave	Runoff volume (ml)			Soil erosion control (%)		
		Slope angle (°)					
		15	30	45	15	30	45
Coir	Plain	483	492	501	47.5	39.8	32.1
	Twill	442	456	519	59.1	52.8	24.2
	4 end satin	499	534	561	39.9	23.5	11.9
Jute	Plain	289	328	349	73.1	64.9	45.2
	Twill	299	362	372	62.1	55.9	29.5
	4 end satin	315	381	394	52.2	44.2	14.8

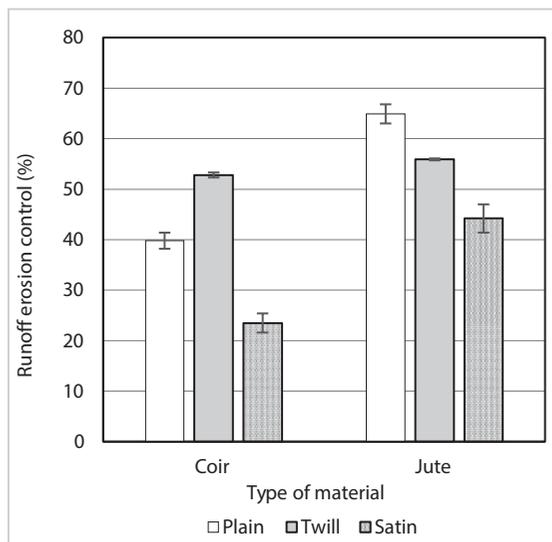


Figure 2: Runoff erosion control performance of structurally modified jute and coir geomeshes at a 15° slope angle

by changing the structure. The satin-woven structure demonstrated the lowest erosion control due to the lowest flexural rigidity because its structure becomes disturbed and increases the area for the direct impact of rain. Another reason for the lower erosion control is the higher erodibility of the soil [14-15]. Loamy sand contains 85% sand, 10% silt and 5% clay, which starts to erode in a short span of time and exerts pressure on the weft. For this reason, structure the of the coir geomeshes becomes disturbed and a greater area of soil is exposed to the rainfall. Error bars confirm that there is a significant difference in erosion control performance of the different woven structures of the jute and coir geomeshes. In contrast to coir geomeshes, plain-woven jute geomeshes

demonstrated better erosion control on loamy sand. Plain-woven geomeshes demonstrated the highest erosion control percentage (73.1%), followed by twill and satin at 62.1% and 52.2%, respectively. This is because jute has a lower amount of lignin, which results in lower flexural rigidity [19-20] (Table 1). Moreover, with change in its structure, its flexural rigidity significantly decreases up to 227  $\mu\text{Nm}$ . For this reason, twill and satin-woven structures of geomeshes cannot withstand the water flowing velocity, resulting in lower erosion control. Analysis of variance shows that the type of weave has a significant effect on the erosion control performance of geomeshes. The type of weave contributes 24.85% to the erosion control performance of geomeshes (Table 4).

Coir and jute geomeshes demonstrated a similar trend at a 30° slope angle (Figure 3). At a 45° slope angle, the plain-woven structure of jute and coir geomeshes demonstrated better erosion control. At a higher slope angle, the velocity of the flowing water increased, which decreased the cohesion between the soil particles and led to the easy transportation of the soil particles. Under similar conditions, lower erosion control was observed at a higher slope angle (Figure 4) [21-22].

ANOVA also confirmed that the slope angle influenced the erosion control performance of jute and coir geomeshes, and contributed the most (52.41%) to the total (Table 4).

In general, jute geomeshes demonstrated better erosion control than coir geomeshes. This is due to their higher moisture absorption and lower flexural rigidity. For this reason, jute geomeshes demonstrated better drape with soil and thus followed the contours over the soil surface for better erosion control [23]. In

Table 4: ANOVA of the runoff erosion control performance of jute and coir geomeshes

Effect	Contribution (%)	SS	Degree of freedom	MS	F-ratio	p-value
Weave	24.85	1292.70	2	646.35	24.209	0.000
Slope angle	52.41	2725.66	2	1362.83	51.044	0.000
Type of material	13.18	685.73	1	685.73	25.684	0.000
Weave * Type of material	4.40	229.13	2	114.57	4.291	0.045
Error	5.13	266.99	10			
Total	100	5200.21				

contrast to jute geomeshes, coir geomeshes demonstrated high flexural rigidity, which made it difficult for them to follow the contours of the soil and control erosion. The type of material also had a significant influence on the erosion control performance of geomeshes, and contributed 13.18 % to runoff erosion control. The type of material and weave had a significant interaction effect and contributed 5.13% to the total (Table 4).

### 3.2 Runoff erosion control performance of jute and coir geomeshes under zero-infiltration conditions

Table 5 shows the performance of modified jute and coir geomeshes under zero-infiltration conditions (without soil). Coir geomeshes demonstrated higher runoff than jute geomeshes due to the stiffer protruding fibres on its surface. These protruding

fibres reduced the contact between coir geomesh and metallic ramp, as shown in figure 5(a), while jute geomeshes draped well with the metallic surface (Figure 5 b). Hence, a lower runoff was observed in jute geomeshes.

Similarly, jute geomeshes also outperformed coir geomeshes in culmination discharge. Culmination discharge depends upon the water absorption and storage effect of the geomeshes. Jute geomeshes have a higher water absorption capacity than coir [24]. Due to this quality, culmination discharge is higher in jute than coir geomeshes. It can also be inferred from Table 5 that the slope angle also affects the runoff volume and culmination discharge. It was observed that runoff volume decreased as the weave was changed in both type of geomeshes. This is because the weaves reduced the flexural rigidity of the geomeshes. The runoff volume and culmination discharge of jute and

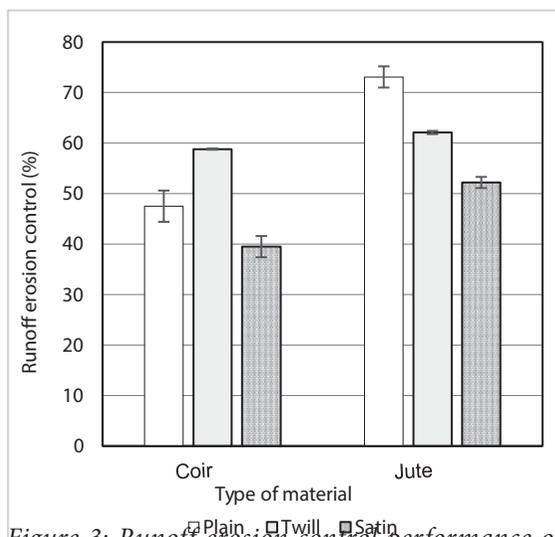


Figure 3: Runoff erosion control performance of structurally modified jute and coir geomeshes at a 30° slope angle

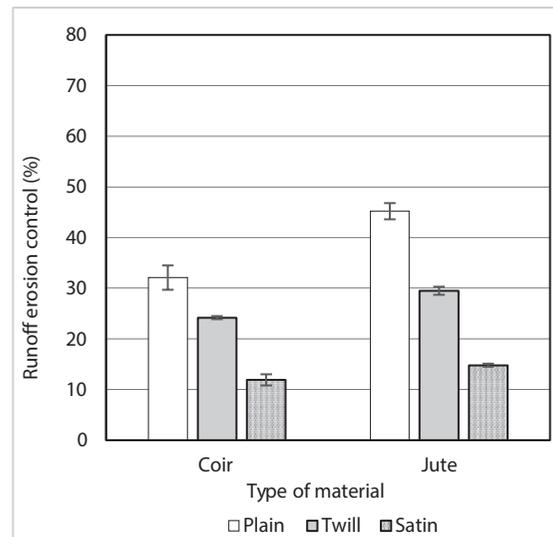
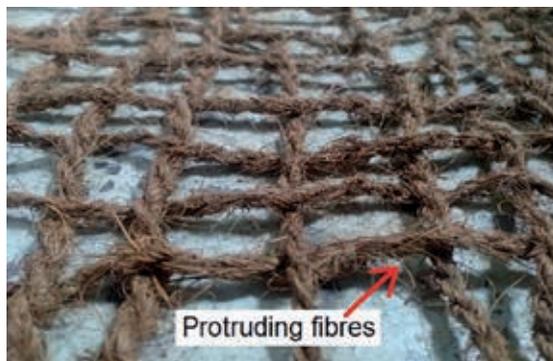


Figure 4: Runoff erosion control performance of structurally modified jute and coir geomeshes at a 45° slope angle

Table 5: Runoff volume and culmination discharge under zero-infiltration conditions

Type of geomeshes	Slope angle (°)	Runoff volume (ml)			Culmination discharge (ml)		
		Plain	Twill	Four end satin	Plain	Twill	Four end satin
Jute	15°	385	322	304	162	168	174
	30°	396	360	345	171	180	206
	45°	415	376	365	184	196	s10
Coir	15°	571	555	543	42	43	44
	30°	584	560	549	48	53	55
	45°	602	595	578	49	56	59



a)



b)

Figure 5: Geomeshes under zero-infiltration conditions: a) coir geomeshes b) Jute geomeshes

coir geomeshes under zero-infiltration conditions at different slope angles followed the same trends as observed under soil-infiltration conditions, while the culmination discharge of coir geomeshes at different angles and different weaves increased. However, no significant differences were observed.

### 3.3 Germination test

Figure 6 shows germination results after 21 days for jute and coir geomeshes. Table 6 shows the germination pattern of different woven jute and coir structures over loamy sand soil. At the end of third day, 19, 24 and 28 plants were germinated in plain, twill and satin respectively. The satin-woven structure demonstrated the highest initial vegetation growth because of its higher float length than the other structures. A higher float length decreased the flexural rigidity of the geomeshes. Thus, once plants emerged from the surface, the satin-woven structure facilitated the disorientation of the structure and the growth of plants, while the plain-woven structure had less contact with the soil surface because of its higher flexural rigidity than the satin-woven structure. This led to the lower



Figure 6: Germination results after 21 days

initial vegetation growth during the initial days. The twill-woven structure offered moderate flexural rigidity and a float length that restricted plant growth compared with the satin structure. Thus, in the satin weave, all the 60 seeds germinated within nine days, while 12 days were required for germination in the twill weave. In the plain weave, however, only 55 seeds had germinated by the end of 21 days. In

Table 6: Germination performance of jute and coir geomeshes

Type of material	Type of weave	Numbers of plants germinated							Total vegetation (%)	Average number of roots	Average length of roots (cm)	Total rooting length (cm)
		Day 3	Day 6	Day 9	Day 12	Day 15	Day 18	Day 21				
Coir	Untreated	48	52	56	60	60	60	60	100	4.5	24	6480
	Plain	0	15	38	40	45	48	49	81.66	5	28	6860
	Twill	0	32	35	44	48	50	52	86.67	5	33	8580
	4 end satin	0	42	43	48	52	54	54	90	4	16	3456
Jute	Untreated	48	60	60	60	60	60	60	100	5	24	7200
	Plain	19	42	44	51	55	55	55	91.67	8	35	15400
	Twill	24	54	58	60	60	60	60	100	6	60	21600
	4 end satin	28	58	60	60	60	60	60	100	4	20	4800

coir geomeshes, germination occurred on the sixth day and started after two days in jute geomeshes. This reason for the difference in the start of germination lies in the fact that jute geomeshes facilitate the faster initial growth of vegetation due to their finer and softer yarns than the coir geomeshes [18].

In jute geomeshes, 100% vegetation was observed at the end of 21 days. When the plain geomesh was applied to the bare surface, the vegetation percentage decreased to 91.67%. However, twill and satin-woven jute geomeshes resulted in the same vegetation percentage as the bare surface at the end of 21 days (Table 6). In coir geomeshes, the vegetation percentage also decreased with a change in the geomesh structure. Plain, twill and satin weaves demonstrated percentages of 81.66%, 86.67% and 90%, respectively. The bare surface demonstrated 100% vegetation because there was no external obstruction for the plants. Amongst all the geomeshes, plain-woven geomeshes demonstrated the highest flexural rigidity, which led to a lower vegetation percentage (Table 1). Jute geomeshes demonstrated a higher vegetation percentage than coir geomeshes in all types of structures. This is because germinating plants were not able to pass through the coir yarns due to the rigid fibres, which offered higher resistance to the plants, while in jute geomeshes, the plants passed through the geomeshes due to its finer and softer yarns. Moreover, different types of weave also affected the number of roots and root length in loamy sand. On the bare surface, an average root length of 24 cm

was observed, while root lengths of 35 cm, 60 cm and 20 cm were observed in plain, twill and satin weaves, respectively. It is evident from Table 6 that root length increased when the plain and twill structure of geomesh was used in place of the bare surface, but decreased when a satin-woven structure was used. The satin-woven structure had a longer float length, which increased its contact with the soil. For this reason, the movement of air through the soil was restricted, resulting in a lower root length in the satin-woven structure [14, 25]. The highest number of roots per plant were observed when plain geomesh was applied to the bare surface: five roots per plant were observed on the bare surface while, eight, six and four roots per plant were observed in plain, twill and satin weaves, respectively (Table 5). Coir geomeshes also demonstrated similar trends to jute geomeshes.

Total rooting length was the main criterion that determined the overall germination performance of the geomeshes. In general, in jute geomeshes, the number of plants germinated at the end of 21 days was highest in the satin-woven structure. However, the total rooting length was lowest because of lower number of roots per plant and lower root length. The twill-woven structure demonstrated the longest root length, while the moderate number of roots per plant and moderate vegetation percentage led to a higher total rooting length. For similar reasons, the twill-woven structure demonstrated the highest rooting length.

## 4 Conclusion

In this study, the performance of structurally modified jute and coir geomeshes was evaluated at different slope angles. It was observed that all factors (slope angle, weave type and type of material) had a significantly effect on runoff erosion control performance. The slope angle contributed most (52.34%) to erosion control, while the type of weave and material type contributed 25.79% and 12.28%, respectively. It is evident from this study that as the slope angle was increased, runoff erosion control decreased. At lower and medium slope angles, the twill-woven coir geomeshes demonstrated better performance, while plain-woven jute geomeshes demonstrated better erosion control at all slope angles. With a change in structure, the flexural rigidity of coir geomeshes decreased, which helped improve erosion control. The twill-woven structure of jute and coir demonstrated the highest vegetation rate at the end of 21 days. The highest root length was also observed in the twill-woven structure. For this reason, the highest rooting length was observed in the twill-woven structure of jute and coir geomeshes. In general, at a lower slope angle, plain- or twill-woven geomeshes can be used, while at a higher slope angle, plain-woven jute geomeshes should be used for better erosion control.

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## Consumers' Attitude Towards Eco Friendly Textile Products

*Stališča potrošnikov do okolju prijaznih  
tekstilnih izdelkov*

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### Abstract

Care for the environment is directly connected to consumer behaviour. The authors summarize in their research the findings of different authors who have examined various aspects of socially responsible consumption, green consumption and ethical consumer behaviour. Their results have shown that consumers contribute to active environmental problem solving by selecting and purchasing environment-friendly textile products. Today's consumers have started to act more ethically and responsibly when purchasing and considering the products they need. The goal of this paper is to explore Slovenian consumers' perception of eco-friendly textile products, to investigate underlying attitudes and perceptions, and consumers' willingness to buy eco-friendly textile products in the future. Research questions focus on knowledge about eco-friendly textile products, attitudes, perception and purchasing behaviour as it relates to eco-friendly textile products. Slovenian consumers express a positive attitude towards eco-friendly textile products. They perceive eco-friendly textile products as products with a certificate, as produced in an eco-friendly manner, as products with a higher price than conventional products and as sustainable products. The most important factor when purchasing eco-friendly textile products is the label "product with certificate" or "certified product", as consumers buy eco-friendly textile products labelled as such. Respondents intend to purchase eco-friendly textile products in the future. The results of this research could be used in planning the further development of the eco-friendly textile products market. Overall positive attitudes toward eco-friendly textile products should be reinforced, while consumers can be influenced through targeted advertising. The results of this research can be used to plan further marketing activities.

Keywords: consumer behaviour, attitudes, perception, consumers, eco-friendly textile products, Slovenia

### Izvleček

*Skrb za okolje je neposredno povezana z vedenjem potrošnikov. V članku so povzete ugotovitve različnih raziskav, v katerih so avtorji raziskali vidike družbeno odgovorne porabe, okolju prijazne porabe in etičnega vedenja potrošnikov. Njihovi rezultati kažejo, da potrošniki z izbiro in nakupom okolju prijaznih tekstilnih izdelkov pripomorejo k aktivnemu premagovanju okoljskih problemov. Današnji potrošniki so ob nakupu in razmišljanju o izdelku, ki ga potrebujejo, začeli delovati bolj etično in odgovorneje. Namen te študije je raziskati, kako slovenski potrošniki dojemajo okolju prijazne tekstilne izdelke, proučiti njihova stališča, dožemanje in njihovo pripravljenost za nakup okolju prijaznih tekstilnih izdelkov*

v prihodnosti. Slovenski potrošniki izražajo pozitiven odnos do okolju prijaznih tekstilnih izdelkov. Okolju prijazne tekstilne izdelke dojemajo kot izdelke s certifikatom in okolju prijazne izdelave, kot izdelke z višjo ceno od konvencionalnih in kot trajnostne izdelke. Najpomembnejši dejavnik nakupa okolju prijaznega tekstilnega izdelka je oznaka »izdelek s certifikatom« oz. »certificirani izdelek«. Potrošniki nameravajo tudi v prihodnosti kupovati okolju prijazne tekstilne izdelke. Rezultate te raziskave bi lahko uporabili za načrtovanje nadaljnjega razvoja trga okolju prijaznih tekstilnih izdelkov. Splošen pozitiven odnos do okolju prijaznih tekstilnih izdelkov bi morali okrepiti, na potrošnike pa lahko vplivamo s ciljnim oglaševanjem. Rezultate raziskave lahko uporabimo za načrtovanje nadaljnjih trženjskih aktivnosti. Ključne besede: vedenje potrošnikov, stališča, dojemanje, potrošniki, okolju prijazni tekstilni izdelki, Slovenija

## 1 Introduction

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Understanding consumer behaviour extends to various branches of science and is a key to successful marketing. Consumer behaviour is defined as the behaviour consumers demonstrate while searching for, purchasing, using, disposing of and evaluating products, services and ideas that are expected to satisfy their needs [1]. The purchase decision-making process is defined as the comprehensive process of consumer decision making that occurs mostly at five, and occasionally, at seven consecutive levels: problem/need identification, data and information collecting, evaluation of alternatives, consumer choice and post-purchase behaviour/result. In the seven-level model, the use of purchased alternative (satisfaction or dissatisfaction with a purchased product) and disposal of unused products and their parts are added [2, 3]. Ethical or environmentally responsible purchasing is an extended purchasing process, where the consumer takes an in-depth approach to considering a purchase and collects the needed information due to a strong affiliation with ethical issues and dilemmas. Most models of ethical decision making and consumption are built on the cognitive process: (1) beliefs determine attitude, (2) attitude leads to intention and (3) intention results in behaviour. Social norms and behavioural control additionally influence intention and behaviour [4]. The most important reasons to buy ethical clothing are a consumer's feeling that by purchasing ethical clothing they are helping to make the world a better place and solving the problems of the fashion industry. Both are based on altruistic values [5]. Consumer behaviour is, during the purchase decision making process, affected by various factors. These can be divided into several factor (influence) groups: psychological (motivation, attitude, learning and memory), social (reference groups, family, individual's role and position, and status), personal (age and level of a family's life cycle, occupation and financial sit-

uation, lifestyle, personality and self-image, values and beliefs), cultural (culture and social class) [6–8], economic (price-monetary and non-monetary aspect, income and quality) [9], individual differences and environmental impacts [10–14]. In the research of authors [7] and [15], we observed a classification into internal and external influence factors on purchase decision making. Among other important factors that influence the purchase decision making of environmentally conscious consumers, the following are worth mentioning: care for one's own health, which means self-protection for such consumers, the strong identity of an environmentally conscious consumer, their self-confidence, sense of happiness and one's own satisfaction, family, friends and peers [5].

Today, the common goal of a company or organization is to encourage consumers to buy through different forms of marketing communications. In order to raise consumer awareness about the environment-friendliness of products, companies evaluate their products through certificates. Ecologists observed the first signs of consumer impact on the environment sixty years ago [16]. The textile industry has a significant environmental impact in the form of water pollution, high energy consumption and high greenhouse gasses emissions. [17]. In selecting an environment-friendly product, consumers pay more attention to certificates when choosing food than a textile product [18]. Some authors [17] point out that the textile industry, which is one of the greatest threats to clean air and water, consumes an excessive amount of energy. The production of polyester and cotton, in particular, is deemed the largest pollutant of oceans, and as one of the causes of global warming and biodiversity loss. In the past few years, textile brands have been appearing that aspire to adopt sustainable behaviour, use ethical and environment-friendly product materials and have been introducing the 5R approach (*Reduce, Reuse, Recycle, Redesign, Reimage*) [19]. To quote one author: [20] "Consumer awareness

of the negative influences of production on the environment is on the rise. An increasing number of consumers are aware that they themselves can contribute to environmental protection with carefully thought through purchase decisions and are thus oriented to products whose impact on the environment is lesser than others. Even companies are adapting to this changed and modified way of consumer thinking. In order to influence consumer purchase preferences, they label their products with environmental certificates. The primary aim of environmental certificates should be informing consumers about the environmental impact of a product”.

Certificates represent one of the constituent parts of the traceability and provability of the environmental and social responsibility of a product. The traceability of raw materials used in a product in the textile industry has not reached the level of importance as in, for example, the food industry [12]. According to researches [9, 21], certificates are an influencing factor in willingness to pay more for environment-friendly textile products. Companies, non-governmental, government and international organizations, as well as industrial sectors have been developing an increasing number of different certificates [15].

### 1.1 Eco-friendly textile products

The protection of the environment and the sustainability of consumer behaviour are the two most important reasons for the introduction of eco-labelling schemes. Eco-labelling aims to promote products with reduced environmental impacts throughout their life cycle, and functions by encouraging consumers to alter their consumption patterns and to make wiser use of resources and energy in the drive for sustainable development. Examples of eco-labels on the EU market include the “EU Flower”, the OEKO-TEX® Standard 100, The Blue Angel (Der Blaue Engel), the Mobius loop (recycling symbol), GOTS (Global Organic Textile Standard) and others [22]. Since the textile industry is a very good example of the fastest growing and most ecologically harmful industries in the world, various innovations have been implemented in order to safeguard our planet. In order to protect our environment, we must take some preventive measures and introduce technologies that can maintain the balance of our eco-system and make final products free from toxic effects. Eco-friendly clothing can be termed as clothing made of natural fibres, such as organic cotton and hemp, clothing that has been organically dyed with vegeta-

bles or any fabrics that use small amounts of water, energy and chemicals that affect the environment. Natural fibres have intrinsic properties, such as mechanical strength, low weight and are healthier to the wearer, which makes them particularly attractive [23–25]. The word “eco” is short for ecology. Ecology is the study of interactions between organisms and their environment. Therefore, “eco” friendly (or “ecology friendly”) is a term used to refer to goods and services deemed to inflict minimal damage on the environment. Environmentally (some authors also use “eco”) friendly products are market-oriented products that cause minimal environmental degradation, while their production is linked to a product development process that is structured in a way that considers the impacts on the environment throughout the product life cycle [26]. “Think globally, act locally” is the slogan of the future for the global textile industry [23, 27]. Any textile product that is produced in an eco-friendly manner and processed under eco-friendly requirements is known as an eco-friendly textile product. The terms sustainable fashion, eco-fashion and eco-tech are also used. When purchasing products bearing the sign “eco-friendly”, consumers may be sure that at least 95% of the materials used are of eco-friendly origin, that the product complies with all inspection regulations, that it is packed in biodegradable packaging, and that it includes a code and specific data regarding the inspection body. Therefore, any certified organic product should be marked with the label “eco-friendly product”.

Materials can be considered “eco-friendly” on the basis of the most various factors [23]:

- Renewability of the product
- Ecological footprint of resources – area of fertile land needed for the full growth of a product
- Determining the eco-friendliness of a product – amount of chemicals required for the production of products.

Numerous surveys show that consumers are concerned about the environment, but this does not always lead to actions, such as the purchase of environmentally responsible products [28, 29]. Previous studies have indicated that the largest proportion of participants consider clothing composition first and foremost, while only a small percentage consider eco-labels and the environmental impact. Consumers are willing to pay no more than 10% extra for a textile product with an ecological label attached. The largest proportion of respondents identified themselves as

average eco-conscious, although they didn't show any knowledge of eco-labels. It is necessary to increase the level of awareness about sustainable materials, as well as trust in eco-labelling systems through transparent standardisation and certification systems. The popularisation of eco-labels, more transparent and coherent labelling systems and the regulation of words such as "green" and "bio" may increase consumers' willingness to choose more sustainable alternatives and consequently pay more for sustainable products [30].

In academic literature regarding consumer preference for responsibly labelled goods, authors have paid little attention to the relevance of considering a label "certified product". While surveying literature and previously research, it was observed that the leading countries in the textile industry are India, other Asian countries, the USA and Columbia. The most relevant research and data regarding the proposed subject have been published in these geographic regions.

The aim of this paper is to explore Slovenian consumers' perception of eco-friendly textile products, to investigate underlying attitudes and perception, and consumers' willingness to buy eco-friendly textile products in the future. An important aspect of the research was to determine how the label "product with certificate" or "certified product" affects consumer decisions to purchase eco-friendly textile products in the decision-making process. On the market, a "product with certificate" or "certified product" is denoted by a legally defined sign. This paper gives the latest account of consumers' attitude towards eco-friendly textile products in Slovenia.

## 2 Methodology

### 2.1 Method

In order to gain knowledge about consumers' perceptions of eco-friendly textile products, to understand the influencing factors in the development of the eco-friendly textile market and to determine how the label "product with certificate" or "certified product" affects consumer decisions to purchase eco-friendly textile products, a quantitative research study was carried out. Before placing the questionnaire in an online environment, we conducted a pilot test of the questionnaire with five consumers in order to identify potential ambiguities in answering the survey questions. There were no missing data in the anal-

ysis, as only completed questionnaires were used for that purpose. Since data were collected as a field intercept survey, respondents who did not complete the questionnaire were never included. Respondents were selected based on a snow ball sampling method. The snowball sampling method is based on the use of networks: a few study subjects are selected to join the group and these recruit other subjects to participate in the study sample. These new subjects recruit other subjects to join the sample until the estimated number of sample subjects has been reached. The same method has also been used in other research [5, 31]. Data were collected via an online questionnaire. These individuals were given a short summary of the purpose of the study before completing the questionnaire. The final usable sample consisted of 141 consumers in Slovenia. A request for the submission of demographic information was included at the end of the questionnaire.

### 2.2 Questionnaire and hypotheses

The questionnaire was divided into the following thematic sections for research purposes:

- level of recognition of the term "eco-friendly textile products",
- assessment of the purchasing behaviour of consumers with respect to "eco-friendly textile products",
- perception of "eco-friendly textile products",
- tendency to purchase eco-friendly textile products due to a "product with certificate" or "certified product" label,
- willingness to pay a higher price and future purchase intentions, and
- demographic data at the end of the questionnaire.

The introduction letter and draft questionnaire were developed for the purpose of the research. Instructions were provided so that the respondents would do well completing the questionnaire, and to ensure that they could progress as quickly as possible through it. Also, the definition of an "eco-friendly textile product" was presented to the respondents in the introductory part of the questionnaire. The questionnaire examined various factors and determinants that influence the attitudes, perceptions, knowledge and purchase of eco-friendly textile products in Slovenia. The data were collected in April 2020 using a structured questionnaire administered in an online survey using closed (questions with multiple choice answers) and open questions (question without al-

ternative answers). The questionnaire developed for the study had questions representing three different components of the study, namely, the socio-economic profile of consumers, their purchasing behaviour with regard to textile products, and their perception of various product attributes. The first component included questions related to the socio-economic information of respondents, such as gender, age, education level and region of residence. The second component related to the purchasing behaviour of consumers and consisted of questions related to consumers' purchase frequency and their perception of eco-friendly textile products. To understand product attributes (the third component of the study), questions were asked related to various product attributes. In order to analyse the relative importance of these product attributes, consumers' perception of attributes was graded using the Likert scale (1 = not at all important, 2 = somewhat important, 3 = important, 4 = very important and 5 = extremely important). An important element of the research focused on determining how the label "product with certificate" or "certified product" affects consumer decisions to purchase eco-friendly textile products.

The specific hypotheses tested in this study were as follows:

Previous studies have indicated that the largest proportion of participants consider clothing composition first and foremost, while only a small percentage consider eco-labels and the environmental impact. The largest proportion of respondents identified themselves as average eco-conscious, although they didn't show any knowledge of eco-labels. Hence, we present the following hypothesis:

H1: The definition of "eco-friendly textile products" has not been clearly integrated in the perception of young consumers.

Consumers are concerned about the environment, but this does not always lead to actions, such as the purchase of environmentally responsible products. Hence, we present the following hypothesis:

H2: Young consumers rarely purchase eco-friendly textile products (less than once a month).

Product attributes play a major role in the purchase choices of consumers and are crucial determinants of product success/failure and the associated marketing strategies. A product can be defined in terms of its different attributes (factors), such as social, personal, economic and environmental. Purchasing decisions for different products mainly depend on a combination of these attributes. By raising consumer aware-

ness regarding the negative influences of conventional production, the importance of the environmental friendliness of products is increasing.

H3: The most important factor for purchasing eco-friendly textile products is the label "product with certificate" or "certified product".

Some socio-demographic characteristics and the purchasing behaviours of consumers influence their perception of eco-friendly textile products. Hence, we present the following hypothesis:

H4: Young women consider eco-friendly textile products to be of higher quality than young men do.

Certificates represent one of the constituent parts of the traceability and provability of the environmental and social responsibility of a product. Certificates are an influencing factor in willingness to pay more for environment-friendly textile product. Hence, we present the following hypothesis:

H5: There is a high tendency for purchasing eco-friendly textile products due to a "product with certificate" or "certified product" label.

Previous studies have indicated that consumers are willing to pay more for clothing made of organic cotton and have reached the conclusion that consumers are prepared to pay prices that are 25% higher. Also, previous studies have indicated consumers that would be willing to pay more for environmentally friendly products, provided they were certain the products are truly environmentally friendly. Hence, we present the following hypothesis:

H6: Young consumers are willing to pay a higher price for eco-friendly textile products than for conventional textile products.

Consumers have started to act more ethically and responsibly when purchasing and considering the products they need. The most important reasons for purchasing ethical clothing are the consumer's feeling that by purchasing ethical clothing they are helping to make the world a better place and solving the problems of the fashion industry. Hence, we present the following hypothesis:

H7: Young consumers intend to purchase eco-friendly textile products in the future.

### 2.3 Sample description

The sample consisted of 141 respondents, 72% of who were women and 28% of whom were men. At 70%, the age group from 18 to 35 years accounted for the the highest proportion of respondents, followed by the age group from 36 to 55 years at 28% and the age group above 55 at 2%. Most respondents completed

secondary school (38%), 35% had a higher education (1st level), 23% had a master's degree (2nd level), 4% completed primary school and 4% had a doctorate. Most of respondents (34%) came from Ljubljana, 33% came from the Maribor-Murska Sobota region, 13% came from the Celje and Trbovlje region, 9% came from the Nova Gorica and Koper region, 6% came from Novo Mesto and 6% came from Kranj (Figure 1).

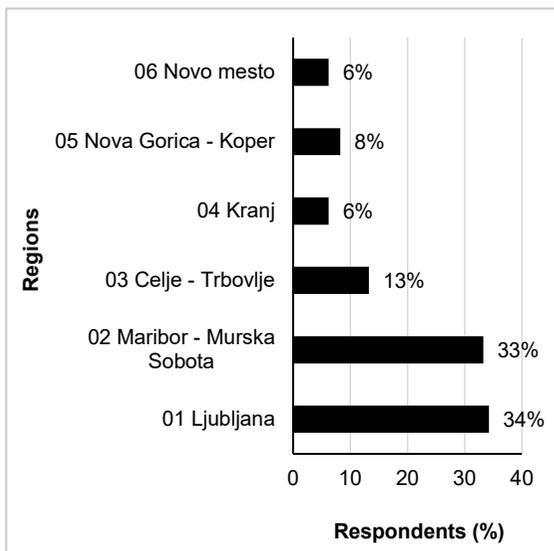


Figure 1: Region of residence of respondents

In methodological terms, the main research is defined as quantitative research based on a large statistical population and a large number of statistical subjects included in a statistical sample. In this way, the method of data processing was justified by the employment of statistical methods providing exact conclusions. Research validity was justified by the fact that the research was carried out in a stable economic environment. Research implementation was limited in time, which mainly eliminated the influence of unforeseen events on the research results. Research reliability was justified by the fact that identical results would be obtained by repeating the research under the same conditions and in the same economic environment. The reliability of the measurement tool used for quantitative research was tested using the Cronbach Alpha test. Generalisation: results are used within the field of textile products for comparability purposes, while the specificities of textile products and the textile industry must be taken into account.

#### 2.4 Data analyses

The answers were processed, graphically presented and interpreted by using the Statistical Package for Social Sciences (SPSS). The data obtained from the questionnaire were analysed using univariate analysis in order to verify distributions of frequencies and to detect possible errors that occurred during the research and/or data entering. The results and individual variables were compared and displayed. The level of comparison was set at,  $\alpha = 0.05$ . The data collected from the questionnaire were put through a validity assessment (Kaiser-Meyer-Olkin measure - KMO = 0.892; Bartlett's test sign = 0.000). The reliability of the measurement tool relates primarily to the accuracy of results. Reliability indicates the accuracy, trust ability, stability and repeatability of test results [31–33]. Cronbach's alpha coefficient of reliability was used in this study. Cronbach's alpha coefficient method is a method used to determine the reliability of a test based on internal consistency. The reliability analysis of the research yielded a Cronbach's Alpha value of 0.85 and a significance level of 0.000, which means that the reliability of the questionnaire is acceptable. The hypotheses are presented and tested in next chapter.

### 3 Results and discussion

#### 3.1 Level of recognition of the term "eco-friendly textile products"

Eco-friendly is a word interpreted by individuals in a variety of ways and contexts. The term has many different meanings and interpretations and is often associated and sometimes confused with terms such as "green", "ecological", "environmental", "natural" and "sustainable" [34]. For example, what is eco-friendly to one consumer may not be eco-friendly to another. Moreover, the interpretations of the term of producers and regulators may differ from those of the consumers [34, 35].

The definition of "eco-friendly textile products" is not yet clearly established on the textile market and in the perception of consumers. Data from quantitative research have shown that knowledge and recognition of eco-friendly textile products is not high. Some 57% of respondents have clearly integrated the definition of eco-friendly products into their thinking. Respondents defined eco-friendly products as textile products with little negative influence on the environment and people's health. The term is broad and includes not only ecologically produced raw ma-

terials, but also production methods with little negative effect on the environment. Environment-friendly products are also those that are biodegradable and can be recycled. They are environmentally friendly, i.e. their production consumes less input material and energy per production/service unit, do not contain hazardous substances and do not produce hazardous waste. They consume less energy during their operation/use, enable reuse or are recycled. They are safe to use and are the least harmful to the environment over their lifetime.

Based on the obtained results, hypothesis 1 that "the definition of eco-friendly textile products has not been clearly integrated in the perception of young consumers" was confirmed. The conclusion that can be drawn from these facts is that better knowledge could be achieved through constant the education of young consumers. This means that there is a need in Slovenia to continuously inform young consumers about the meaning of eco-friendly products and the meaning of product labelling. Institutions of public importance and manufacturers should consider this obligation. It is in the best interest of all subjects involved in the production, research and consumption of textile products to raise quality to a higher level in Slovenia. According to [36], information regarding eco-friendly products is an important factor, representing the only instrument that consumers have to differentiate between the attributes of eco-friendly products and those of conventional products, and to form positive attitudes and high-quality perceptions of these products. Knowledge about eco-friend-

ly products is not only determined by socio-demographic variables (education level, income, values and lifestyle), but is also affected by information provided by public administration, the mass media, environmental associations and shopping sites.

### 3.2 Consumers' perception of eco-friendly textile products

Prepared attributes of eco-friendly textile products were included in the questionnaire in order to help consumers express their points of view regarding such products. Characteristics of eco-friendly textile products were prepared based on the results of existing research by [10, 34, 37] and others. Respondents expressed their opinions by marking the appropriate answer (1 = strongly disagree and 5 = strongly agree). Average evaluations of recounted characteristics were calculated based on obtained evaluations, and the overall view of Slovenian consumers regarding eco-friendly textile products was determined accordingly (Figure 2).

Research shows that consumers perceive eco-friendly textile products as products labelled with certificates, such as "certified products" (AM = 4.1; SD = 1.1), as products that have been produced in an environmentally friendly manner (AM = 3.8; SD = 1.3), as products with a higher price than conventional products (AM = 3.5; SD = 1.3), as sustainable (AM = 3.2; SD = 1.3), as products that manifest a modern design (AM = 3.0; SD = 1.3), as aesthetically friendly (AM = 3.0; SD = 1.1), as of high quality (AM = 3.0; SD = 1.1), as of high quality



Figure 2: Average mean values (AM) consumers' perception of eco-friendly textile products

(AM = 3.0; SD = 1.1) and products with pleasant texture (AM = 3.0; SD = 1.1) (Figure 2).

Some socio-demographic characteristics and purchasing behaviours of consumers influence their perception of eco-friendly textile products. Our research showed that female consumers perceive eco-friendly textile products to be of higher quality (AM = 3.23; SD = 0.91) than men do (AM = 2.30; SD = 1.09). T-test and Levene's test results indicated equal variances between both male and female consumer groups ( $F = 1.057$ ;  $p = 0.000$ ). Significant differences could be observed. Women considered eco-friendly textile products to be of higher quality than men did. Based on the obtained results, hypothesis 4 that "young women consider eco-friendly textile products to be of higher than young men do" was confirmed.

### 3.3 Consumers' purchasing behaviour with respect to eco-friendly textile products

The purchasing behaviour of consumers was assessed on the basis of purchasing frequency and preferred market place (Table 1).

Table 1: Consumers' purchasing behaviour with respect to eco-friendly textile products

Frequency of purchase	Percent of respondents (%)
Weekly	3
Twice a month	6
Monthly	21
Rarely	72

Source: Author's own source.

The results indicate that about 72% of respondents rarely purchase eco-friendly textile products (less than once a month). Based on the presented results, hypothesis 2 that "young consumers rarely purchase eco-friendly textile products (less than once a month)" was confirmed.

Based on the Likert scale, we examined the importance of various product attributes to respondents in purchasing decisions. Using the Likert scale of 1 = not at all important, 2 = somewhat important, 3 = important, 4 = very important and 5 = extremely important, they defined their affinity for given attributes.

The results showed (Figure 3) that the most important attributes for a consumer's selection are a "product with certificate" or "certified product" label (AM = 3.7; SD = 1.4) and price (AM = 3.7; SD = 1.2). Ranking third and fourth in terms of importance were design (AM = 3.5; SD = 1.4) and style (AM = 3.5; SD = 1.4). They were followed by: aesthetically friendly (AM = 3.4; SD = 1.4), colour (AM = 3.4; SD = 1.4), texture value (AM = 3.3; SD = 1.3), appearance of high quality (AM = 3.3; SD = 1.3), brand (AM = 3.0; SD = 1.3) and environmentally friendly production (AM = 2.8; SD = 1.2). Based on the presented results, hypothesis 3 that "the most important factor for purchasing eco-friendly textile products is the label "product with certificate" or "certified product" was confirmed. Fibre content has an important impact on how the texture feels to the skin.

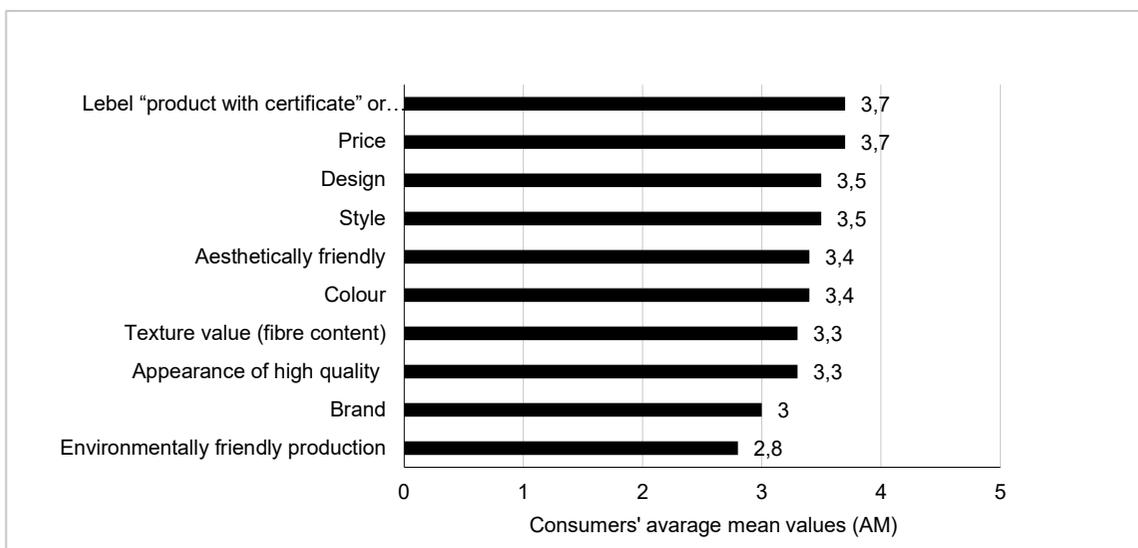


Figure 3: Importance of various product attributes in purchasing decisions (average mean values (AM))

### 3.4 Attitude towards a label "product with certificate" or "certified product" label

An important element of this research was dedicated to determining the importance of the "product with certificate" or "certified product" label in the decision-making process. Respondents expressed their opinions by marking the appropriate answer (1 = strongly disagree and 5 = strongly agree). The results show that consumers interviewed decided that the label "product with certificate" or "certified product" is the most important factor in decisions to purchase eco-friendly textile products (AM = 3.5, SD = 1.4). Consumers always buy eco-friendly textile products with a "product with certificate" or "certified product" label (AM = 3.3; SD = 1.4) (Table 2). We can thus confirm there is a significant tendency to purchase eco-friendly textile products with a "product with certificate" or "certified product" label. Based on the obtained results, hypothesis 5 that "there is a high tendency for purchasing eco-friendly textile products due to a "product with certificate" or "certified product" label was confirmed.

Special attention will be paid to comparing the influence of the "certified product" labelling/feature with the influences of other researched factors on consumers' decisions to purchase environmentally friendly textile products. Should it be demonstrated that a "certified product" label bears at least as strong a statistically typical influence on consumers' purchasing decisions as other influential factors, the research results will contribute positively to the more responsible focus of corporate management on the environment, and to the more efficient management of the online marketing elements of environmentally friendly products in marketing departments. An additional challenge in the process of forming and implementing a marketing communications strategy, together with raising consumer awareness, will be emphasizing the importance of the quality or integrity of products, since the "certified product" label enters the purchase decision-making process during the phase of collecting information regarding an

environmentally friendly textile product, while the consumer considers such information when evaluating alternatives, and even makes a decision about the final purchase on the basis of that information.

### 3.5 Willingness to pay a higher prices and future buying intentions

Respondents were asked to indicate if they were willing to pay a higher price for eco-friendly textile products than for conventional textile products, and how much extra they were willing to pay. A total of 4% of respondents would definitely be reluctant to pay more money and 16% did not know whether they would pay premium prices for eco-friendly textile products. Most consumers (53%) were willing to pay a premium price, paying 11-20% more, 24% would pay 21-30% more, 6% would pay 31-50% more, and 1% of respondents were willing to pay 51-100% more for eco-friendly textile products (Figure 4). Based on the presented results, hypothesis 6 that "young consumers' are willing to pay a higher price for eco-friendly textile products than for conventional textile products" was confirmed.

Authors [10] have investigated whether consumers are willing to pay more for clothing made of organic cotton, and have come to the conclusion that consumers are prepared to pay a 25% higher price. Research regarding the attitude of Europeans to the formation of a single market for organic products has shown that more than three-thirds of respondents would be willing to pay more for environmentally friendly products, provided that they were certain the products are truly environmentally friendly (77%). On the other hand, just over a half of Europeans (55%) feel they are adequately informed about the environmental impact of purchased and used products [38]. Author [11] points out that consumers perceive the price for ethical and environmentally responsible textile as too high and disproportionate to conventional products. Authors [12] have researched consumer decisions to purchase environmentally friendly fashion accessories.

Table 2: Attitude towards a "product with certificate" or "certified product" label

Eco-friendly textile products	N	AM	SD
A "product with certificate" or "certified product" label is the most important factor in purchasing decisions.	141	3.5	1.4
I always buy eco-friendly textile products with a "product with certificate" or "certified product" label.	141	3.3	1.4

N = Number, AM = Average Mean, SD = Standard deviation

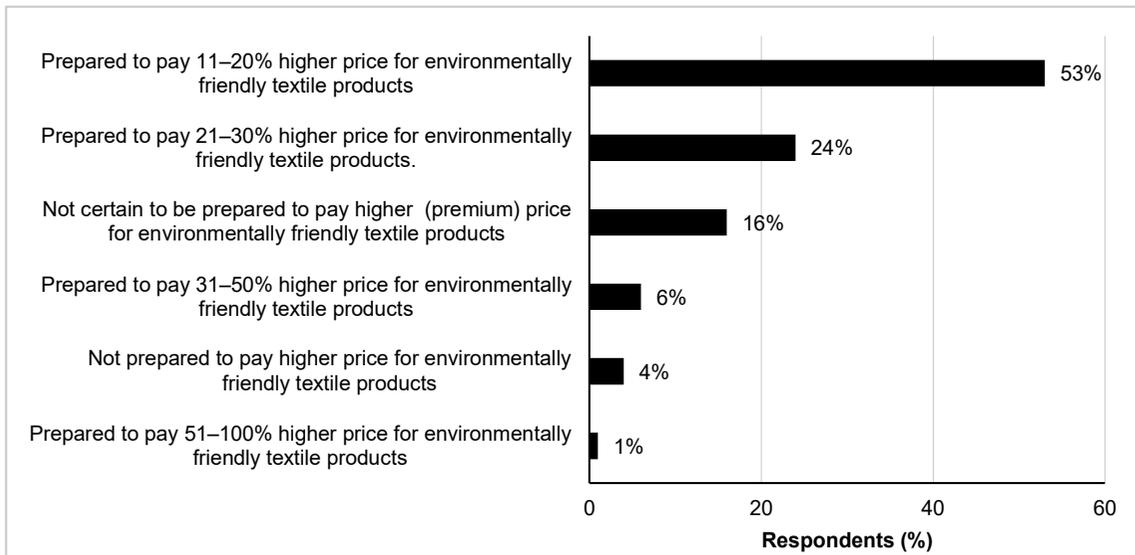


Figure 4: Willingness to pay a higher price for eco-friendly textile products

They argue that consumers are willing to pay more for an environmentally friendly fashion accessory. Authors [39] concluded that mothers were willing to purchase eco-friendly clothing for their children, but were reluctant to pay more for it. Author [40] found a weak correlation between educational level and willingness to pay a price premium for ethically produced clothing ( $p = 0.004$ ), and concluded that demographics were poor predictors of consumers' willingness to pay a price premium. Authors [41] and [42] found no significant effect of gender, age, education level, income, or ethnicity on willingness to pay a price premium for eco-friendly cotton.

Future purchasing intentions were measured using the five-point Likert scale, with 1 meaning "strongly disagree" with eco-friendly textile products and 5 meaning "strongly agree" with the intention of future purchases. About 60% of the respondents ( $AM = 3.7$ ;  $SD = 1.1$ ) intend to purchase eco-friendly textile products in the future. Based on the obtained results, hypothesis 7 that "consumers intend to purchase eco-friendly textile products in the future" was confirmed.

In a period of saturated markets, companies are becoming increasingly aware of the importance of familiarity with the characteristics of consumers, their motives for the purchase of a certain product and their opinions regarding products. Companies pay more attention to market and consumer research, which is considered to be one of most important factors in determining market success [43].

## 4 Conclusion

Today's consumers are increasingly aware of environmental and social issues, and are looking for responsible products [44] that are more durable, fairer and produced from recycled materials [45]. We often talk about "socially and environmentally responsible consumption" defined as a consumption pattern that takes into account the needs of current generations without compromising those of future generations [46, 47]. The aim of this paper was to provide a better understanding of consumers' awareness, attitude and perception of eco-friendly textile products, i.e. to gain insight into consumers' preferences, motives, attitudes and interest to buy eco-friendly textile products in Slovenia.

Based on the research results, the following conclusions can be drawn:

- The definition of "eco-friendly" has not been clearly established on the textile market and in the perception of consumers. A total of 57% of respondents have clearly integrated the definition of eco-friendly. This means that there is a need in Slovenia to continuously inform consumers about the importance of eco-friendly products and product labelling.
- Factors that explain consumers' perception of eco-friendly textile products include products that bear a label such as "certified product", products produced in an environmentally friendly manner, products with a higher price than conventional products, sustainable products, products

that manifest a modern design, products that are aesthetically friendly, high-quality products and products with a pleasant texture.

- About 72% of respondents rarely purchase eco-friendly textile products (less than once a month).
- The most important factor in purchasing eco-friendly textile products is a “product with certificate” or “certified product” label (56%; AM = 3.7).
- The results show that there is a significant tendency to purchase eco-friendly textile products with a “product with certificate” or “certified product” label. Consumers always buy eco-friendly textile products with a “product with certificate” or “certified product” label.
- Consumers are willing to pay a higher price for eco-friendly textile products than for conventional textile products.
- More than 60% of respondents intend to purchase eco-friendly textile products in the future.

This paper gives the latest insight into purchasing behaviour and attitudes towards eco-friendly textile products in Slovenia. According to the research results, an important task for producers will be to increase consumers' knowledge of what an eco-friendly product is and how to differentiate it in the marketplace. Therefore, knowing how consumers perceive eco-friendly textile products by understanding the reasons for buying them would probably help the marketers of eco-friendly textile products create an appropriate communication message. The enhancement of consumers' knowledge can be achieved through constant, consistent, meaningful, planned and proactive communication, which always includes both information and education [48], which in turn can influence consumer decisions in the purchasing process. The results of the research could be used for planning further marketing activities. These results are particularly useful for product marketing and future product development in the eco-friendly textile sector, as they provide guidelines on how to address and satisfy the needs of existing eco-friendly consumers more successfully and, if possible, adapt to eco-friendly consumers in order to attract new segments and a new generation of consumers on the textile market.

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# Influence of Needle-punching Parameters for the Preparation of Polypyrrole-coated Non-woven Composites for Heat Generation

*Vpliv parametrov iglanja na izdelavo s polipirolom prevlečenih kompozitnih vlaknovin za generiranje toplote*

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## Abstract

This work deals with the preparation and characterization of electrically conductive needle-punched non-woven composites for heat generation. Electro-conductive non-woven composites were prepared through the in situ chemical polymerization of pyrrole with  $\text{FeCl}_3$  (oxidant) and p-toluene sulfonic acid (dopant). A two-stage double-bath process was adopted for the in situ chemical polymerization of pyrrole. The effect of parameters such as fibre fineness, needle-punching density and depth of needle punching on a polypyrrole add-on, and surface resistivity were studied by employing the Box-Behnken response surface design. It was observed that fibre fineness was the most influential parameter of the polypyrrole add-on. The lowest surface resistivity of the polypyrrole coated sample ( $200 \text{ g/m}^2$ , prepared with a punch density of  $200 \text{ punch/cm}^2$ , a punching depth of 6 mm and fibre fineness of 2.78 dtex) was found to be  $9.32 \text{ k}\Omega/\square$  with a polypyrrole add-on of 47.93%. This non-woven composite demonstrated good electrical conductivity and exhibited Joule's effect of heat generation. Due to the application of a 30 V DC power supply, the surface temperature of the non-woven composite rose to  $55 \text{ }^\circ\text{C}$  from a room temperature of  $37 \text{ }^\circ\text{C}$ . Optical and electron microscopy images of the non-woven composites showed that PPy molecules formed a uniform coating on the non-woven surface. FTIR studies evidenced the coating of PPy on a polyester surface. These coated non-woven composites were highly electrically conductive and practically useful for the fabrication of heating pads for therapeutic use.

Keywords: non-woven, composite, surface resistivity, electroconductive textile, needle-punching, fibrous material, coated fabric

## Izvleček

V delu je predstavljena priprava in karakterizacija elektroprevodnih iglanih kompozitnih vlaknovin za generiranje toplote, ki so bile izdelane in situ s kemično polimerizacijo pirola z oksidantom  $\text{FeCl}_3$  in dopantom p-toluensulfonsko kislino. Uporabljen je bil dvostopenjski dvokopelni postopek. Vplivi parametrov, kot so finoča vlaken in gostota ter globina iglanja, na dodani polipirol in površinsko upornost so bili proučevani glede na odzivno površino, zasnovani z metodo Box-Behnken. Ugotovljeno je bilo, da na dodatek polipirola najbolj vpliva finoča vlaken. Najnižja površinska upornost  $9,32 \text{ k}\Omega/\square$  je bila dosežena na vzorcu s 47,93-odstotnim nanosom polipirola na vlaknovini površinske mase

200 g/m<sup>2</sup>, gostote iglanja 200 vbodov/cm<sup>2</sup>, globine iglanja 6 mm in finoče vlaken 2,78 dtex. Ta kompozitna vlaknovina je imela dobro električno prevodnost in je izkazovala Joulov učinek generiranja toplote. Zaradi uporabe enosmerne, 30-voltne, napajanja se je temperatura površine kompozitne vlaknovine zvišala s sobne temperature, tj. 37 °C, na 55 °C. Na posnetkih kompozitnih vlaknovin s pomočjo optičnega in elektronskega mikroskopa je vidno, da so molekule polipirola tvorile enakomerno prevleko na površini vlaknovine. Študije FTIR so potrdile prisotnost polipirola na površini poliestrskih vlaken. Te prevlečene kompozitne vlaknovine so bile električno visokopredvodne in primerne za izdelavo grelnih blazinic za terapevtsko uporabo.

*Ključne besede:* netkane tekstilije, kompozit, površinska upornost, elektroprevodna tekstilija, iglanje, vlaknati material, premazana tekstilija

## 1 Introduction

The use of metal wires or metal coating is a common practice for imparting electrical conductivity to textiles [1-3]. Also, metal fibres were used during staple spinning to manufacture electro-conductive yarns [4-5]. Those yarns were used in weaving or knitting to produce electro-conductive fabrics. However, the processing of those yarns was difficult and they lose their textile properties [6]. Many such limitations associated with processability, low mechanical strength and poor flexibility could be successfully overcome by coating/applying conducting polymers on textile substrates [7-9]. PPy has been used primarily as a conductive polymer due to its high conductivity, low toxicity [10], commercial availability and high stability in the air relative to other conducting polymers [11-13].

The heating effect of PPy-coated polyethylene terephthalate-lycra woven fabrics was studied by Kaynak & Håkansson [14]. Those coated fabrics exhibited reasonable electrical conductivity and effective heat generation. At an applied voltage of 24 V, the maximum temperature achieved was 40.55 °C. In another study, PPy was incorporated into cotton woven fabrics and various properties such as anti-static, anti-microbial, heat generation were investigated [15]. A conducting textile composite was prepared by embedding PPy in natural and manmade cellulosic fibres, such as cotton, viscose, cupro and lyocell, using in situ vapor phase polymerization [16]. Their various electrical properties such as voltage-current characteristics, voltage-temperature characteristics, etc., were also studied [17]. It was suggested by Sparavigna et al. and Macasaquit & Binag that 100% polyester fabrics could easily be made electrically conductive using a PPy coating, while they were practically useful for many applications, including flexible, portable surface-heating elements for medical or other applications [17-18]. An electrical resistor was formed within

a fabric by sewing a highly conductive silver-coated yarn into less conductive PPy-treated knitted fabric [19]. This was found to be a useful method for enabling electrical connection to a fabric for the characterization of a fabric's resistivity and the design of a fabric resistor. In another study, it was reported that fabrics knitted with silver yarn, together with elastomeric yarn, could generate sufficient heat to warm the body. Those fabrics could be used to manufacture personal heating garments that could generate heat in relation to applied voltage [20]. In another study, when a constant voltage of 9 V was applied to cotton/PPy fabric for 10 minutes, its surface temperature rose to 90 °C, while the performance of the fabric did not deteriorate after numerous repetitive cycles [15]. Similar observations were also made by Maity et al. in the case of PPy-coated polyester-woven, spunlace and needle-punched non-woven composite fabrics prepared using in situ chemical polymerization [8]. PPy-coated polyester-woven, spunlaced and needle-punched non-woven composite fabrics demonstrated an exponential rise in surface temperature when voltage was applied, while the rise in temperature was found to be related to the duration of voltage application [8]. Among these three types of fabric structures, the needle-punched non-woven composite performed better in terms of good electrical conduction and heat generation. This was attributed to its porous and bulky structure. In another study, various polyester needle-punched non-woven composites were prepared and PPy applied to them using in situ chemical polymerization. It was shown that a porous and bulky non-woven structure facilitated the better penetration of a reagent liquor between fibre interstices with a more extensive polymerization yielding a higher polymer add-on [12, 21]. The highest PPy add-on of 16.28% was achieved for a polyester needle-punched non-woven composite (250 g/m<sup>2</sup>, 200 punches/cm<sup>2</sup>, 8 mm depth of penetration and 6.66 dtex fibre) [12]. These PPy-coated non-woven

composites were evaluated for electro-magnetic shielding application, but heat generation behaviour was not studied.

Most of the studies available in literature concern woven and knitted fabric composites that are relatively thin compared with needle-punched non-woven composites. There are limited works available regarding PPy-coated needle-punched structures and the evaluation of the same in terms of Joule's effect of heat generation. Fibre fineness and the process parameters of needle-punching affect the structural features of the manufactured non-woven composites. Thus the aim of the present work was to investigate the effect of fibre fineness, needle-punching intensity and needle punching depth on a PPy add-on, and the surface resistivity of manufactured non-woven composites by employing a Box-Behnken response surface design and the characterization of the same

in terms of Joule's effect of heat generation. Polyester needle-punched non-woven composites are currently used in many applications, such as home furnishing, upholstery, filtration, filler materials for winter garments, geotextiles, etc., while this functional modification of the same may open up new dimensions of its application.

## 2 Materials and method

### 2.1 Preparation of polyester needle-punched non-woven composites using a $3^3$ Box-Behnken design

Polyester fibres were used as the raw material in the preparation of needle-punched non-woven composites on a "Dilo" needle-punching machine. The virgin polyester fibres of three different finenesses (3.33 dtex,

Table 1: Box-Behnken design parameters of the experiment and the levels thereof

Level	Punch density (punch/cm <sup>2</sup> )	Punch depth (mm)	Fibre fineness (dtex)
	A	B	C
-1	150	6	3.33
0	200	9	6.66
1	250	12	9.99

Table 2: Scheme of experiments according to the  $3^3$  Box-Behnken Design with five central repeat runs

Run	A: punch density (punch/cm <sup>2</sup> )	B: punch depth (mm)	C: fibre fineness (dtex)
1	0	0	0
2	1	0	1
3	0	0	0
4	0	0	0
5	0	1	-1
6	-1	0	1
7	-1	1	0
8	1	0	-1
9	0	1	1
10	1	-1	0
11	0	0	0
12	-1	0	-1
13	0	-1	1
14	0	-1	-1
15	-1	-1	0
16	1	1	0
17	0	0	0

6.66 dtex and 9.99 dtex) and a cut length of 51 mm were procured from Reliance Industries Ltd., India. A three-factor, three-level ( $3^3$ ) Box-Behnken response surface design was employed for an experimental plan to prepare needle-punched non-woven samples. The parameters were fibre fineness, punch density and punch depth, the value of which are shown in Table 1. The  $3^3$  Box-Behnken design has only 17 experimental runs in comparison to 27 runs for the full factorial design. In this Box-Behnken design, the treatment combinations are at the midpoints of the edges of the process space and at the centre, and thus do not consider extreme treatment conditions that result in the increased consumption of energy and chemicals, and may also have a harsh effect on the substrate of non-woven composites. Thus, according to the design, 17 varieties of needle-punched non-woven samples, each of the same mass per unit area ( $200 \text{ g/m}^2$ ), were prepared, as shown in Table 2. All these samples were coated with PPy and tested for thickness, porosity, PPy add-on, and surface resistivity.

## 2.2 Chemicals

The chemicals used were ferric chloride ( $\text{FeCl}_3$ ) as the oxidant, pyrrole as the monomer and p-Toluenesulfonic acid (PTSA) as the dopant. All of the chemicals used were of laboratory grade and purchased from Sigma Aldrich, India.

## 2.3 Method of preparation of PPy-coated non-woven composites

Polyester needle-punched, non-woven samples were hydrolysed for the better fixation of PPy on a polyester surface. It was reported that the hydrolysis of polyester fabrics with 10% sodium hydroxide (NaOH) solution for 20 minutes at boiling ( $95^\circ\text{C}$ ) at a 1:40 material to liquor ratio was the optimum condition of hydrolysis for achieving maximum add-on without significant loss of fabric strength [8]. Therefore, all polyester non-woven composites were hydrolysed employing the same process conditions.

After hydrolysis, the non-woven composites were neutralized with 0.1 N acetic acid solutions. A two-stage double-bath process was then employed for the in situ chemical polymerization of pyrrole. In the first stage, the non-woven fabric was soaked with the monomer in a monomer bath. In the second stage, the in situ polymerization of pyrrole was conducted in an oxidant bath. A monomer bath was prepared by dissolving pyrrole in 0.5 M concentration in de-ionized water. Material to liquor ratio of monomer bath was 1:40. An oxidant bath was prepared by dissolving  $\text{FeCl}_3$  and PTSA in de-ionized water. The amount of  $\text{FeCl}_3$  and PTSA used were 0.5 M and 0.05 M, respectively. Material to liquor ratio was 1:40. Hydrolysed samples were first allowed to soak in a monomer bath for 15 minutes at room temperature. The oxidant bath was cooled to  $5^\circ\text{C}$  in a cryostat and pyrrole soaked non-woven samples were taken from monomer bath and dipped into the oxidant bath for in situ polymerization. The time of the in situ polymerization was 1 hour. Polymerized samples were then taken out from the oxidant bath, thoroughly rinsed with cold water and dried in an oven at  $80^\circ\text{C}$  for 4 hours before measurement.

## 2.4 Measurement of surface resistivity

The surface resistivity of the PPy-coated non-woven composites was measured inside a control chamber at 0% relative humidity using a concentric ring probe electrode according to the test procedure described in ASTM D-257 standard [22]. The concentric ring probe electrode was fabricated according to the aforementioned standard with 12.5 mm outer radius of the inner ring electrode and 32 mm inner radius of the outer ring electrode, as shown in Figure 2. Surface resistivity measured from such an electrode probe can be expressed using equation 1.

$$\rho = R_s \times \frac{2\pi}{\ln\left(\frac{R_2}{R_1}\right)} = R_s \times K \quad (1)$$

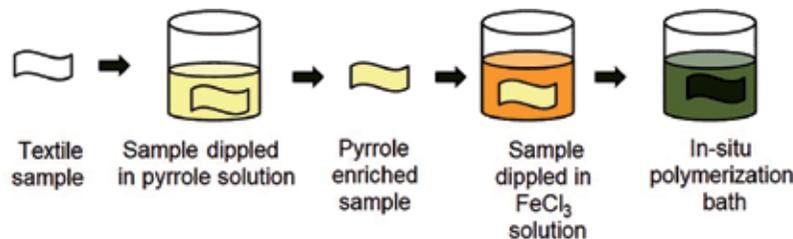


Figure 1: In situ chemical polymerization of pyrrole onto polyester non-woven composites using the double-bath process [13]

where,  $\rho$  represents surface resistivity ( $\Omega$ ),  $R_s$  represents the resistance of the sample ( $\Omega$ ),  $R_1$  represents the outer radius of the inner ring (mm),  $R_2$  represents the inner radius of the outer ring (mm) and  $K$  represents a geometric constant.

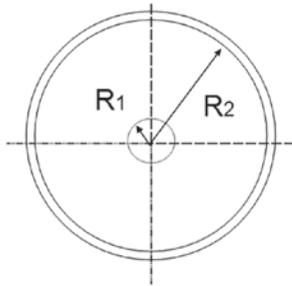


Figure 2: Schematic diagram of concentric ring electrode probe

### 2.5 Measurement of electrical characteristics

The higher the electrical conductivity of non-woven composites, the higher the Joule's effect of heat generation will be because generated heat  $H \propto I^2$ , where  $I$  represents the current flowing through the conductor. Thus, the measurement of voltage-current (V-I) characteristics will help to predict the heat generation behaviour of the non-woven composites. For this measurement, composite specimens were cut into 15 cm  $\times$  12.5 cm pieces. Two copper plates measuring 12.5 cm  $\times$  2.5 cm were placed on the two opposite edges of the specimen as electrodes. The length of the electrodes ( $D$ ) was 12.5 cm, as shown in Figure 2. The distance between the two electrodes ( $L$ ) was maintained at 10 cm. Electrode plates were then connected with a variable DC power supply ( $U$ ), as shown in Figure 3. Due to the applied DC voltage across the samples, current flowed in the circuit that was measured by connecting an ammeter, while the surface temperature was measured by a non-contact type infra-red thermometer.

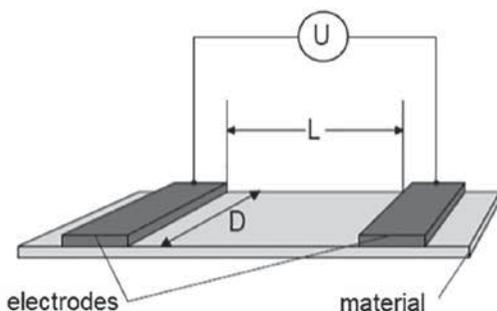


Figure 3: Parallel plate method to measure electrical characteristics of non-woven composites

### 2.6 Optical microscope analysis

Morphological analysis of uncoated and PPy-coated composites was carried out using an optical microscope (Nikon Eclipse E200) at a magnification of 10 $\times$ .

### 2.7 Analysis of samples using FTIR

FTIR analysis of untreated uncoated and PPy-coated composites was carried out using a Bruker 10074570 spectrometer. Spectra were recorded in the range of 4,000  $\text{cm}^{-1}$  to 600  $\text{cm}^{-1}$  by accumulating 24 scans at a resolution of 4  $\text{cm}^{-1}$ .

## 3 Result and discussion

### 3.1 PPy add-on and surface resistivity of electro-conductive composites

According to the Box-Behnken design, 17 experimental runs were performed to prepare 17 varieties of PPy-coated non-woven composite samples. Thirty observations were made for all these PPy-coated composite samples for the purpose of measuring PPy add-on and surface resistivity. Average values are shown in Table 3. It is evident that the highest PPy add-on of about 47.93% was achieved in the case of sample no. 14. The surface resistivity of this sample was found to be the lowest among all samples (9.32  $\text{k}\Omega/\square$ ). In a previously reported study, PPy add-on of 16.28% was achieved for polyester needle-punched non-woven composite (250  $\text{g}/\text{m}^2$ , 200 punches/ $\text{cm}^2$ , punching depth of 8mm and 6.66 dtex fibre) [12]. Better PPy add-on may be achieved due to alkali hydrolysis and the use of finer fibres for needle-punching [7].

### 3.2 Effect of the process parameters on surface resistivity of PPy-coated composites

The effect of the process parameters of needle punching, such as punch density, punching depth and fibre fineness, on the surface resistivity of coated composites was investigated by employing the Box-Behnken design and using a response surface methodology. The main effects and interaction effects of these parameters were analysed by employing analysis of variance (ANOVA), as shown in Table 4. The "Model F-value" of 4.15 implies model significance relative to noise. There is only a 3.00 % chance that a "Model F-value" this large could occur due to noise. Values of "Prob > F" of less than 0.05 indicate model terms are significant. The lowest p-value (0.0812) is observed for punch

Table 3: Surface resistivity and PPy add-on of PPy-coated needle-punched non-woven composites

Run	Factor A: punch density (punch/cm <sup>2</sup> )	Factor B: punching depth (mm)	Factor C: fibre fineness (dtex)	Average surface resistivity (kΩ/□)	Average PPy add-on (%)
1	0	0	0	406	14.2
2	1	0	1	384	13.3
3	0	0	0	368	14.2
4	0	0	0	286	14.2
5	0	1	-1	38	15.2
6	-1	0	1	479	18.0
7	-1	1	0	702	15.7
8	1	0	-1	155	17.4
9	0	1	1	364	8.9
10	1	-1	0	713	19.2
11	0	0	0	358	14.2
12	-1	0	-1	998	16.2
13	0	-1	1	171	13.7
14	0	-1	-1	9	47.9
15	-1	-1	0	269	7.3
16	1	1	0	316	8.0
17	0	0	0	296	13.0

density(A), which is the most influential parameter of all. Among interaction effects, AB interaction was significant in that it indicated their combined effect during the needle-punching process. The “lack of fit F-value” of 17.80 implies that lack of fit is significant. There is only a 0.82% chance that a “lack of fit F-value” this large could occur due to noise. The coefficient of determination ( $R^2$ ) is found to be 0.8060, which implies that the fit of the model is

sufficient for predicting the response. The model equation in the coded unit is shown in equation 2.

$$\rho = 342.91 - 109.70A + 19.70B + 12.16C + 270.27A^2 - 113.17B^2 - 109.18C^2 - 207.50AB + 187.08AC \quad (2)$$

This equation is good at predicting the theoretical surface resistivity of PPy-coated composites with known process parameters.

Table 4: Significance of PPy-coated needle-punched composite samples for the surface resistivity

Source	Sum of squares	DF	Mean square	F-value	Prob > F	
Model	8.044×10 <sup>5</sup>	9	1.006×10 <sup>5</sup>	4.15	0.0300	Significant
A	96277.11	1	96277.11	3.98	0.0812	
B	3104.33	1	3104.33	0.13	0.7295	
C	1183.17	1	1183.17	0.05	0.8306	
A <sup>2</sup>	3.076×10 <sup>5</sup>	1	3.086×10 <sup>5</sup>	12.71	0.0074	Significant
B <sup>2</sup>	53925.39	1	53925.39	2.23	0.1739	
C <sup>2</sup>	50189.93	1	50189.93	2.03	0.1878	
AB	1.722×10 <sup>5</sup>	1	1.722×10 <sup>5</sup>	7.11	0.0285	Significant
AC	1.400×10 <sup>5</sup>	1	1.400×10 <sup>5</sup>	5.78	0.0429	Significant

Source	Sum of squares	DF	Mean square	F-value	Prob > F
Residual	1.937×10 <sup>5</sup>	8	24206.63		
Lack of fit	1.833×10 <sup>5</sup>	4	45837.45	17.80	0.0082
Pure error	10303.22	4	2575.81		
Core total	9.981×10 <sup>5</sup>	16			

### 3.3 Effect of process parameters on the surface resistivity of PPy-coated composites

The effect of process parameters on the surface resistivity of PPy-coated needle-punched non-woven composites were analysed using 3-D response surface plots. The combined effect of punch density and punching depth on surface resistivity of the PPy-coated composites is shown in Figure 4. It is evident that, as punch density increases, the surface resistivity of the composites initially decreases and then increases. As punch density increases, the non-woven structure became more compact with small pores. As a result, PPy molecules were trapped inside fibre interstices and could not be released after washing, resulting in higher a PPy add-on, as well as a decrease in electrical resistivity. However, after achieving the lowest value of surface resistivity, it increased with an increase of punch density. This is due to the formation of large holes and needle-marks in the non-woven structure due to rigorous punching that allows PPy molecules to wash out easily from fibre interstices. As the punching depth increases, surface resistivity increases. This is because a low punching

depth results in a gentle action, which in turn results in a uniform and bulky non-woven structure that is able to entrap PPy molecule in fibre interstices. This would be difficult to achieve in a compact structure prepared with a high punching depth.

The combined effect of punch density and fibre fineness on surface resistivity of the PPy-coated needle-punched non-woven composites is shown in Figure 5. It is evident that if fibre fineness increases at a high punch density, surface resistivity increases, which means that non-woven composites prepared with finer fibres have a lower electrical resistivity. Finer fibres have a higher specific surface area than that of coarser fibres. As a result, finer fibres can hold more PPy, resulting in lower surface resistivity. If the accessible area of the fibre surface is increased, PPy molecules can be deposited on the surface, which is believed to have happened here. At a low punch density, however, surface resistivity decreases slightly as fibre fineness increases because, at a low punch density, thicker fibres are difficult to entangle to form a uniform non-woven structure, which leads to poor PPy add-on and higher resistivity. Punch density has the same effect on surface resistivity as discussed above.

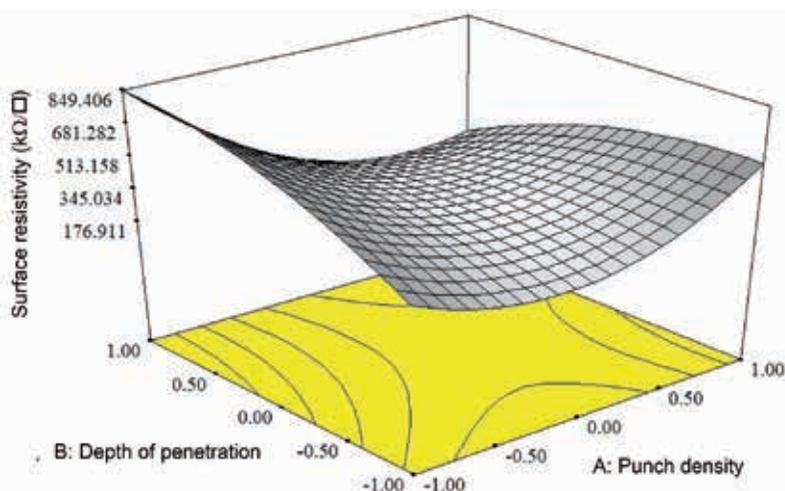


Figure 4: Effect of punch density and punching depth on the surface resistivity of PPy-coated needle-punched non-woven composites

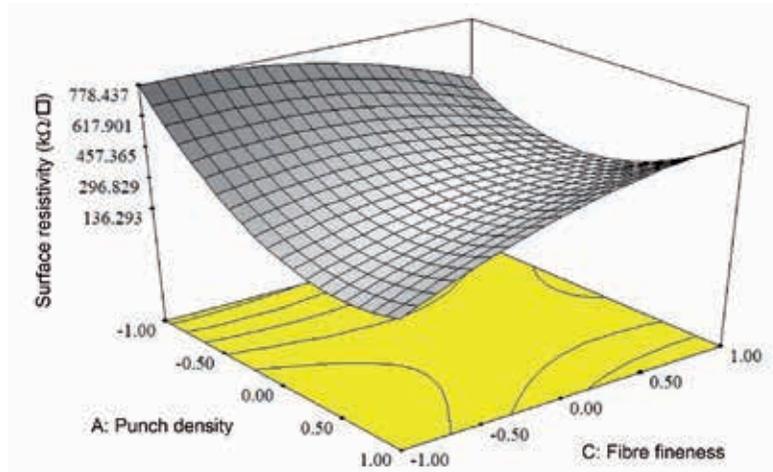


Figure 5: Effect of punch density and fibre fineness on the surface resistivity of PPy-coated needle-punched non-woven composites

### 3.4 Voltage-temperature (V-T) characteristics

The electro-conductive composites were tested for their voltage-temperature characteristics by applying a different range of direct current (DC). The surface temperature of the composites was measured using a non-contact type infra-red thermometer after an interval of three minutes of voltage application. The results obtained for sample no. 14 are shown in Figure 6. It is evident that as the applied voltage was gradually increased, the surface temperature increased due to Joule's effect. According to Joule's law, the heating effect produced by an electric current (I) through a conductor of resistance (R) for a time (t) is given using equation 3.

$$H = I^2 R t \quad (3)$$

Joule's law states that the amount of heat production in a conductor is directly proportionate to the square of electric current flowing through it, directly proportionate to the resistance of the conductor, and directly proportionate to the time for which electric current flows through the conductor. Thus, an exponential trend of rising temperature was observed due to the application of DC voltage to the samples. For this sample, the surface temperature was raised by 18 °C from room temperature by applying of 30 V. Similar exponential trends were also observed in the case of PPy-coated polyester needle-punched non-woven by Maity et al. [8].

### 3.5 Voltage-current characteristics of electro-conductive non-woven composites

Ohmic conductors show liner V-I characteristics. To verify the nature of the PPy-coated needle-punched non-woven composites, current flow through the

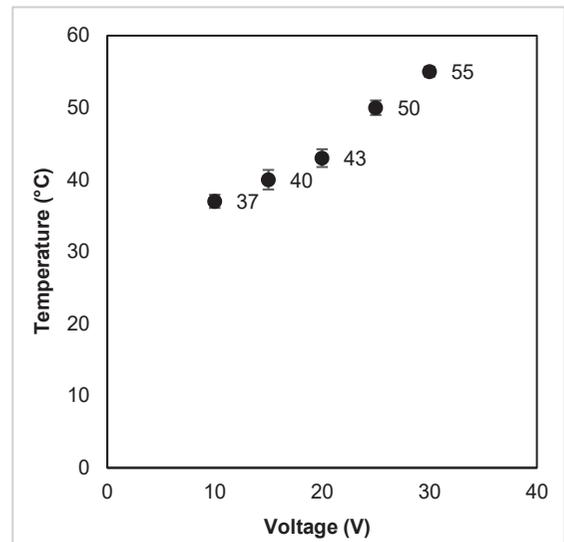


Figure 6: Voltage-temperature characteristics (V-T) of PPy-coated polyester needle-punched non-woven composite (200 g/m<sup>2</sup>, 200 punch/cm<sup>2</sup>, punching depth of 6mm, 3.33 dtex fibre)

samples due to the application of voltage was measured using an ammeter connected to the circuit. The results obtained for sample no. 14 are shown in Figure 7. V-I characteristics obtained as such follow a linear trend of  $I = 0.0383V$  with a coefficient of determination of  $R^2 = 0.998$ , which means current is directly proportionate to applied voltage such as an Ohmic conductor. The electrical resistance of the sample is  $1/0.0383 = 26 \Omega$ . Similar linear V-I characteristics were also observed in the case of PPy-coated composites up to the  $\text{FeCl}_3$  concentration of 9 g/l used during the in situ polymerization [8, 16]. However, an exponential trend was observed if a higher concentration of  $\text{FeCl}_3$  was used.

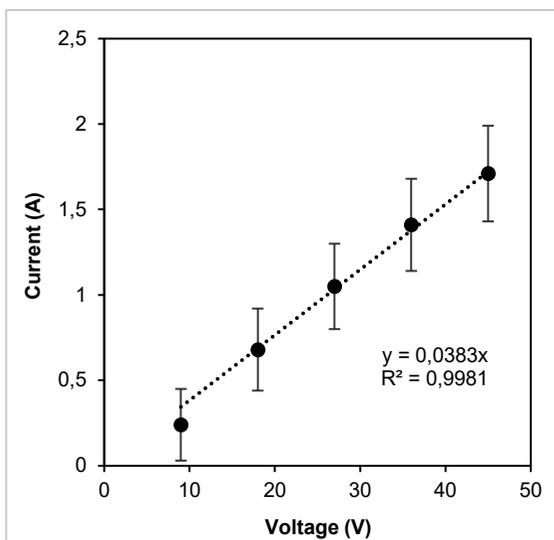


Figure 7: Voltage-current (V-I) characteristics of PPy-coated polyester needle-punched non-woven composite (200 g/m<sup>2</sup>, 200 punch/cm<sup>2</sup>, punching depth of 6 mm, 3.33 dtex fibre)

### 3.6 Time-temperature characteristics of the electro-conductive non-woven composites

The time duration of the application of voltage may affect the heat generation behaviour of the electro-conductive non-woven composites [8]. To investigate this, fixed DC voltage (20 V) was supplied to the samples and allowed to heat up for an extended period. The results for sample no. 14 are shown in Figure 8. It is evident that temperature increased with time up to six minutes and then levelled off at a temperature of 49 °C. It can thus be concluded that these conductive non-woven composites heated up quickly due to the application of a fixed voltage and

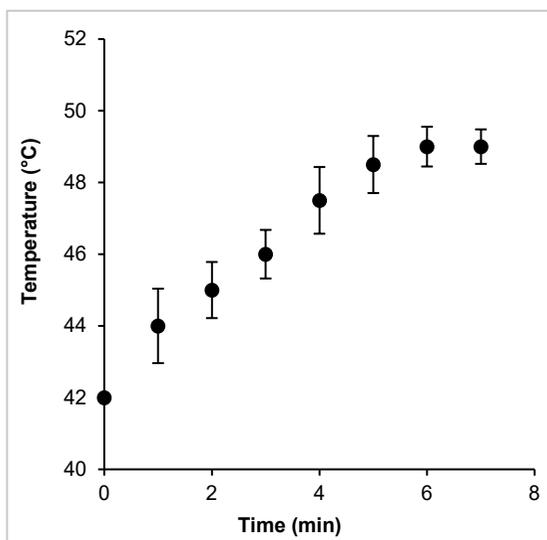
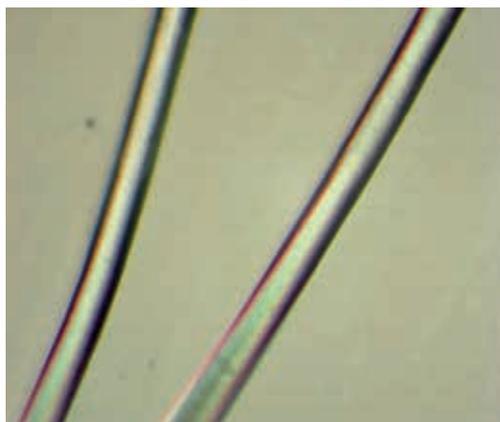


Figure 8: Effect of time on heat generation behaviour of PPy-coated polyester needle-punched non-woven composite (200 g/m<sup>2</sup>, 200 punch/cm<sup>2</sup>, punching depth of 6 mm, 3.33 dtex fibre)

then levelled off at a particular temperature. Such behaviour of the non-woven composites is suitable for the fabrication of a heating pad or garment.

### 3.7 Optical microscopy image analysis of PPy-coated polyester

Optical and electron microscopic images of uncoated and PPy-coated polyester fibres (taken from the coated composite) are shown in Figures 9 and 10, respectively. It is evident that all individual fibre surfaces were coated with PPy molecules. As a result, the fibres changed to greenish-black, similar to the colour of PPy. Uniform PPy coating was achieved on the surface of polyester fibres using granular morphology.



a)



b)

Figure 9: Optical microscopy image (10×) of uncoated polyester fibre (left) and PPy-coated polyester fibre (right)

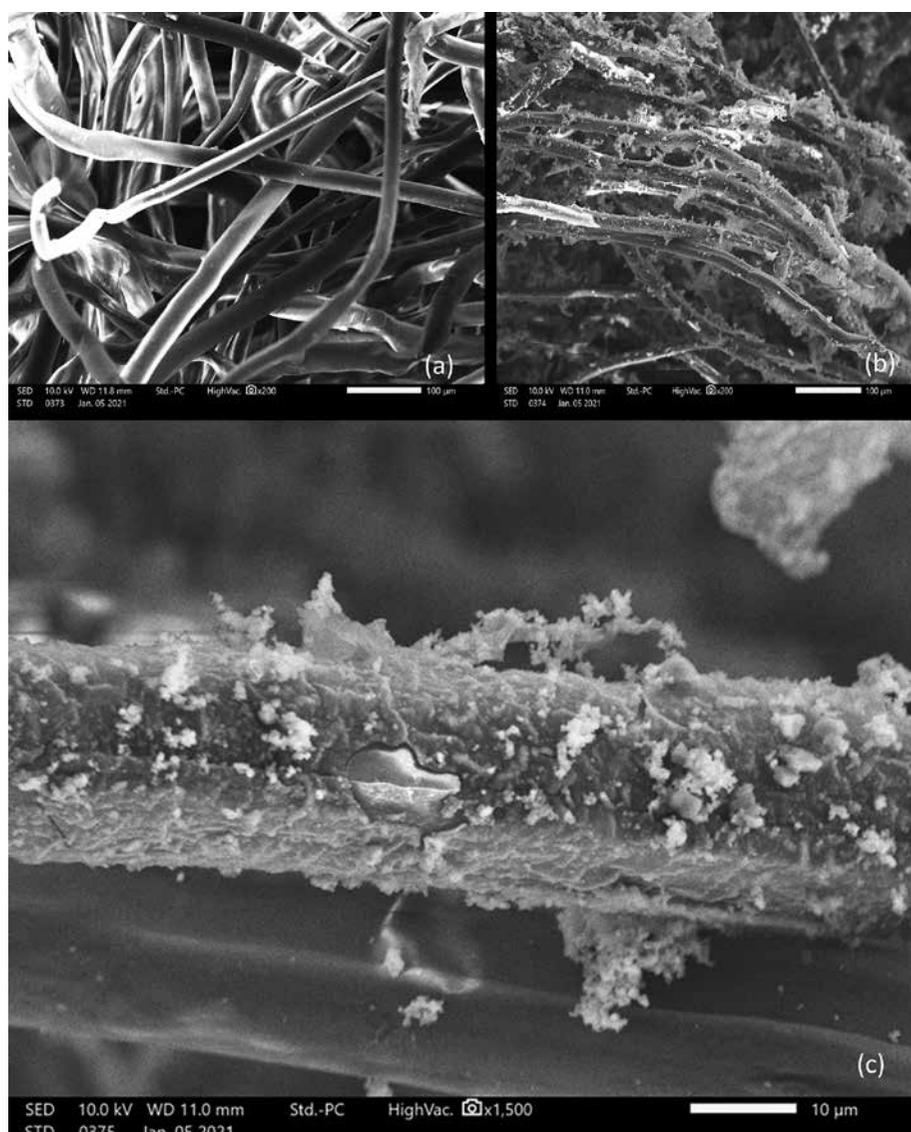


Figure 10: Electron Microscopy image of (a) uncoated polyester fiber 200 $\times$  (top left); (b) PPy coated polyester fiber 200 $\times$  (top right) and (c) PPy coated polyester fiber 1500 $\times$  (at bottom).

### 3.8 Fourier transform infrared (FTIR) spectroscopy analysis

The FTIR spectra of polyester fibre and PPy-coated polyester fibres are shown in Figure 11. The FTIR spectra reveal various functional groups and chemical bonds that are present in polyester fibre and PPy polymers to identify chemical modifications and intermolecular interaction between them. In the case of uncoated polyester, observed peaks at wavenumbers of 2,948  $\text{cm}^{-1}$  and 2,915  $\text{cm}^{-1}$  are attributed to aliphatic C-H stretching vibration, while the band at 1,455  $\text{cm}^{-1}$  is attributed to  $\text{CH}_2$  deformation and the band at 1,374  $\text{cm}^{-1}$  is attributed to the -COO-stretching of the ester group. In the case of PPy-

coated polyester, the band at 1,541  $\text{cm}^{-1}$  is attributed to the -C=C ring stretching vibration of PPy, while the band at 1,458  $\text{cm}^{-1}$  is attributed to  $\text{CH}_2$  deformation and the band at 1,304  $\text{cm}^{-1}$  is attributed to the -CN stretching of PPy. The bands at 1,168  $\text{cm}^{-1}$ , 1,028  $\text{cm}^{-1}$ , 900  $\text{cm}^{-1}$ , and 784  $\text{cm}^{-1}$  are attributed to the characteristics of bending (C-H) vibration of PPy [10]. The band at 1,374  $\text{cm}^{-1}$  due to the -COO-stretching of ester group in uncoated polyester vanished after PPy coating. These observations reveal significant intermolecular interaction between PPy and polyester fibres. This intermolecular interaction confirms the affinity of PPy molecules to polyester surface to achieve a durable coating.

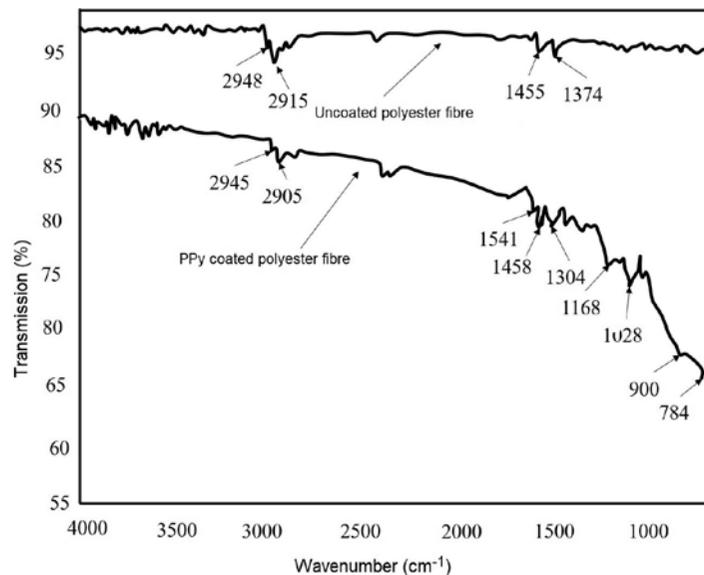


Figure 11: FTIR spectra of untreated and PPy-coated polyester fibre

## 4 Conclusion

The effects of parameters, such as needle-punching density, needle-punching depth and fibre fineness were investigated by employing the  $3^3$  Box-Behnken design and a response surface methodology. The results revealed that punch density was the most influential effect, while AB interaction (punch density-needle punching depth) was important for achieving the lowest surface resistivity. The highest PPy add-on of about 47.93% was achieved in the case of 200 g/m<sup>2</sup> non-woven fabric prepared with 200 punch/cm<sup>2</sup>, a punching depth of 6 mm and 3.33 dtex fibre. The surface resistivity of this composite sample was found to lowest among all samples (9.32 k $\Omega$ ). Voltage-temperature characteristic followed an exponential trend and the surface temperature of the PPy-coated composite was raised by 18 °C from room temperature by applying 30 V. Voltage-current characteristic followed a linear trend such as ohmic conductors with a linear resistance of 26  $\Omega$ . The composites heated up quickly within six minutes and reached a stable temperature of 49 °C when a contact supply of 20 V was applied. Optical and electron microscopy images revealed the uniform but granular morphology of PPy molecules over the polyester composite surface. FTIR studies revealed intermolecular interaction between PPy molecules and polyester fibres. Such PPy-coated needle-punched non-woven composites can be a potential material for the fabrication of a non-metallic, light-weight, flexible heating pad for therapeutic use and for heating garments for cold weather.

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