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Mikrokapsuliranje na področju tekstilstva

Pregledni znanstveni članek

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Izvleček

Prispevek opisuje proces, najpomembnejše tehnologije in uporabo mikrokapsuliranja v tekstilni industriji. Uporaba mikrokapsul nenehno narašča, še posebej v tekstilnih industrijah zahodne Evrope, na Japonskem in v Severni Ameriki. Kljub široki uporabi v industriji pa mikrokapsuliranje ostaja ljudstvu večinoma neznan proces. Je cenovno primerna in dolgotrajna metoda za shranjevanje hitro hlapljivih snovi. Mikrokapsule so primerne za mnoge izdelke. Ti proizvodi imajo različne lastnosti, ki so odvisne od narave kapsuliranih snovi. Nekateri so opisani v tem prispevku, na primer izdelki s PCM-materiali, dezodorantskimi apreturami, protimikrobnimi apreturami, ognjevarnimi apreturami, sistemom spreminjanja barve itd.

Ključne besede: mikrokapsuliranje, nadzorovano sproščanje, tekstil, tehnologije, apreture

1 Uvod

Potrebe, zahteve in pričakovanja potrošnikov za bolj zdravo in udobnejše življenje naraščajo iz dneva v dan tudi na področju tekstilij. Dandanes so lahko tekstilije obdelane tako, da nas ščitijo pred različnimi neugodnimi vplivi, a so kljub temu zelo udobne. Njihove lastnosti so raznolike. Lahko so vodoodporne, protimikrobne, negorljive itd. Takšne lastnosti se lahko doseže s posebnimi kemičnimi mešanicami, apreturami, ki se vežejo na površino vlaken. Na določen način pa lahko aktivne substance, na primer dišave, vnesemo tudi v matrico vlaken. Pri vsem tem je zelo pomembno, da so lastnosti, ki jih dosežemo z apreturami, obstojne pri pranju in dolgotrajne.

Microencapsulation in textiles

Review

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Abstract

This paper describes current processes, technologies and applications related to microencapsulation in the textile industry. The use of microencapsulation continues to grow especially in the textile industries of Western Europe, Japan, and North America. Although it is widely used in industry, microencapsulation remains relatively unknown to the public. It is a cost- effective method for storing volatile substances over a long period of time. Microcapsules are available for a wide range of products. These products are given various properties related to the nature of the encapsulated substances. Some of these are described in this paper, including products with phase-change properties, fragrance finishes, antimicrobial finishes, fire retardants, color-change properties and color formers.

Key words: microencapsulation; controlled release; textile; technologies; finishes

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1 Introduction

Every day, textile consumers are experiencing greater needs, expectations and demands in terms of a healthier and more comfortable life. Today, fabrics can be treated to protect the consumer from various adverse conditions while maintaining consumer comfort. The available textile properties are becoming increasingly diverse (e.g. water resistance, anti-microbial effects, etc). These kind of properties can be achieved by binding special chemical compounds to the fiber surfaces. Active substances (e.g. fragrances) can also be incorporated into the fiber matrix. In designing such techniques, it is important that the end product is resistant to washing and is long lasting.

Microencapsulation is a versatile process used to apply different properties to textiles. It is a cost effective and lasting method in storing various substances over a long period of time. This process has been widely used in many industrial and scientific fields for several decades, including the textile industry (different finishes), the chemical industry, cosmetics (shampoos, face creams, deodorants), electronics, the food industry (food aromas, packaging), photography and marketing (labels, posters, flyers).

In this paper we focus on the use of microencapsulation for textile applications. To date, much research has been focused on the application of microencapsulated substances with various fabrics. Cooperation with the COST865-Bioencapsulation Sciences to Applications program provided us with new knowledge used for writing this article.

2 Microencapsulation

Microcapsules are small particles of between one and several hundred micrometers, composed of a liquid, solid or gas core surrounded by one or more protective coatings.

Aside from protecting the core substances, microencapsulation is also used for:

- *separation of reactants,*
- *controlled release,*
- *reduction of toxicity and*
- *reduction of volatility.*

Using similar techniques, nanocapsules (<

Eden od postopkov za apliciranje različnih snovi in s tem lastnosti na tekstilije je mikrokapsuliranje, ki je cenovno primerna in dolgotrajna metoda za shranjevanje različnih substanc. Ta proces je v široki uporabi na mnogih industrijskih in znanstvenih področjih že nekaj desetletij. Uporablja se v tekstilni industriji (razne apreture), kemični industriji, kozmetiki (dezodoranti, šamponi, kreme), elektroniki, biotehnologiji, prehrambni industriji (arome v hrani, paketiranje), fotografiji, marketingu (etikete, plakati, letaki) itd.

V članku smo se osredotočili predvsem na uporabo mikrokapsul v tekstilstvu, saj je bilo že samo na tem področju veliko narejenega z apliciranjem raznih mikrokapsuliranih substanc na različne tekstilije. Pri pisanju tega prispevka nam je bilo v veliko pomoč tudi sodelovanje pri programu COST 865 – Bioencapsulation Sciences to Applications.

2 Mikrokapsuliranje

Mikrokapsule so majhni delci velikosti od enega do več sto mikrometrov, sestavljeni iz tekočega, plinastega ali trdnega jedra in ovojnice, ki ščiti jedrni material pred zunanjimi vplivi. Ovojnica je lahko samo ena ali pa jih je več.

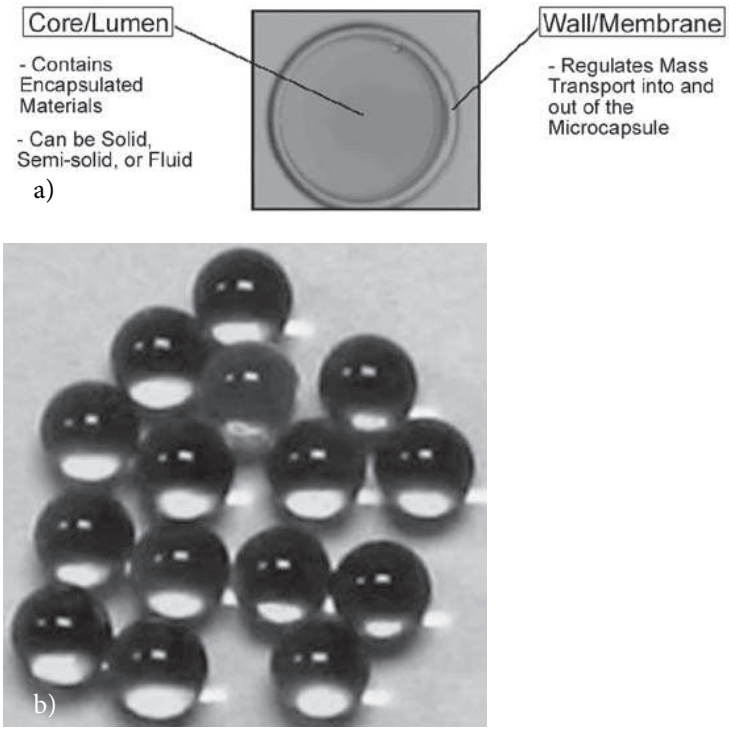

Figure 1: a) a structure of a microcapsule [1], b) microcapsules [2]

Cilji mikrokapsuliranja so poleg zaščite jedrne substance še:

- ločitev reaktantov,
- kontrolirano sproščanje,

1µm) and millicapsules (> 1mm) can also be produced.

Many substances can be encapsulated such as water, perfumes, dyes, antimicrobials, phase change materials, vitamins and drugs. Thus, encapsulated systems are amenable to simple small molecules, large complex molecules and combinations of both [3].

The polymer used for the coating can be natural (for instance starch, lipid, and gelatine), synthetic (polyamide, polypropylene, polyester) or semi-synthetic (cellulose acetate). The permeability of the microcapsule is dependent on the coating material [4] which can be chosen depending on the ultimate application.

The impermeable coat can release the microcapsule's active substance by:

- *outer force,*
- *high temperature,*
- *light,*
- *solvent or water.*

In other words, the coating can break, melt, burn, decompose, dry up or dissolve. For example, in medicine, impermeable capsules are used to contain the active medicament. These capsules are designed to dissolve at an appropriate pH to release the active substance inside.

Permeable microcapsule coatings continually release the core substances and the rate of release can be controlled [6].

There are many different methods of microencapsulation depending on the application and desired properties of the microcapsules [3].

The microencapsulation process can be divided into three steps:

1. *The active compound is incorporated into the matrix or microcapsule core. This can be in the form of a solution, emulsion or suspension, resulting in a liquid or a dispersed solid system. To accomplish this step, mixing, dispersing, drying, grinding and sieving processes may be used.*
2. *Mechanical operation:*
   - *A liquid matrix is prepared, either by making a liquid-in-air by dropping or spraying or by making a liquid dispersion by emulsification or micro-emulsification.*
   - *A solid matrix is prepared by spraying a solution on agitated particles via fluid bed, pan coating or agglomeration.*

– redukcija toksičnosti,
– redukcija hlapljivosti, itd.

Poleg mikrokapsul se lahko proizvajajo tudi nanokapsule (< 1µm) ali pa milikapsule (> 1mm).

Mikrokapsuliramo lahko mnogo snovi, na primer vodo, dišave, barvila, protimikrobne apreture, PCM- materiale (angl. phase change materials), vitamine, zdravila in še veliko drugih. To pomeni, da je mikrokapsuliran sistem lahko tak z majhnimi molekulami ali s precej kompleksnimi molekulami. Lahko pa je mešanica obeh [3].

Ovojnica je lahko iz naravnih (škrob, lipidi, želatina), sintetičnih (poliamid, polipropilen, poliester …) ali pa polsintetičnih (na primer celulozni acetat) polimerov in od nje je odvisna prepustnost mikrokapsule [4]. Glede na namen uporabe izberemo mikrokapsule z ustrezno prepustnostjo.

Sprostitev substance iz mikrokapsule z neprepustno ovojnico lahko povzročimo z:

– zunanjo silo,
– visoko temperaturo,
– svetlobo,
– topilom ali vodo.

Z drugimi besedami, mikrokapsule lahko počijo, se stalijo ali zgorijo, se razgradijo, posušijo ali raztopijo. V medicini se na primer uporabljajo neprepustne mikrokapsule z zdravilom v jedru. Pri ustreznem pH se ovojnica raztopi in tako se jedrna substanca sprosti.

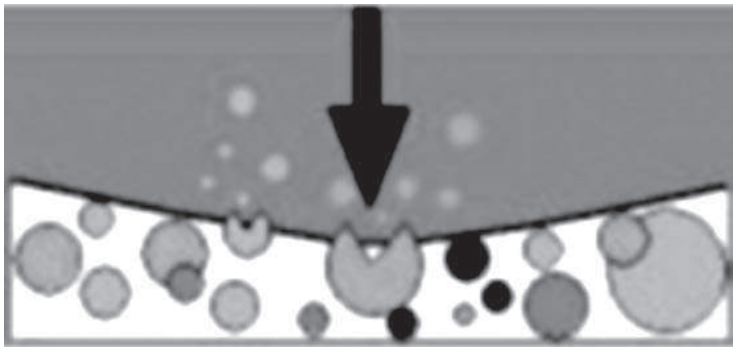

Figure 2: The release of active substances-impermeable microcapsules[5]

Prepustne ovojnice mikrokapsul nenehno sproščajo jedrno snov. Sproščanje je lahko počasno ali zadrževalno [6].

Obstaja mnogo metod mikrokapsuliranja, izbira pa je odvisna od namena uporabe in lastnosti mikrokapsul [3].

Proces mikrokapsuliranja lahko razdelimo na tri korake:

1. Vnos aktivne substance v matrico oziroma jedro mikrokapsule. Ta je lahko v obliki raztopine, emulzije ali suspenzije, končni rezultat pa je tekoč ali pa razpršen trdni sistem. Procesi, ki so za to potrebni, so lahko mešanje, pršenje, sušenje, mletje oziroma drobljenje (grinding) in presejanje.

3. *The coating solution is then stabilized either by a chemical process (polymerization), physicochemical process (gelation, coacervation) or physical process (drying, solidification).*

2.1 Methods of dispersion to form microcapsules

The dispersion of liquids as droplets is the first step for incorporation of the active ingredient inside of a liquid matrix. Dispersion can be accomplished via the following techniques:

- *prilling,*
- *spray technologies,*
- *emulsification,*
- *microemulsification.*

2.1.1 Prilling

The goal of prilling is to produce small droplets and thus microcapsules with low size dispersion. In most cases, energy input is required to reduce size of droplets. Some systems that are currently applied for prilling are as follows:

- *electrostatic generators [7] (application of an electric potential across a pending droplet);*
- *nozzle resonances technology [8] (application of a specific vibrational frequency);*
- *jet cutter [9] (cutting the liquid jet by a series of wires fixed on a turntable which spins at high speed);*
- *spinning disks [10] (liquid jets flow onto spinning disks, where they break into droplets).*

2.1.2 Spray technologies

A liquid can be separated into fine droplets by flowing through an air/liquid nozzle or over a fast rotating device. The productivity of these techniques is superior to prilling technologies. Spray technology is widely used in the food industry [11].

2.1.3 Emulsification

A liquid containing the ingredient to be encapsulated can be dispersed into another immiscible liquid to form an emulsion. For microencapsulation, systems that allow dispersion at low shear stress are preferred, to avoid denaturation of the active ingredients. The simplest and most common emulsification system consists of a reactor equipped with a turbine (Figure 5a). However,

2. Mehanski postopek:
   - tekoča matrica: pršenje v zrak (making a liquid-in-air) ali tekoča disperzija (emulgiranje ali mikro-emulgiranje);
   - trdna matrica: pršenje raztopine po delcih, za kar je potrebno tresenje (tekoča plast ali pan coating ali aglomeracija)
3. Stabilizacija kapljic oziroma raztopine za ovojnico s kemičnimi procesi (polimerizacija), fizikalno-kemičnimi procesi (želatiniranje, koacervacija) ali fizikalnimi procesi (sušenje, strjevanje).

2.1 Metode razpršitve za oblikovanje mikrokapsul

Disperzija tekočine v obliki kapljic je prvi korak za vnos aktivne substance v tekočo matrico. Ta postopek lahko razdelimo na:

- oblikovanje kapljic iz curka tekočine,
- tehnologije pršenja,
- emulgiranje,
- mikroemulgiranje.

2.1.1 Oblikovanje kapljic iz curka tekočine

Cilj tega postopka je proizvodnja majhnih kapljic – mikrokapsul iz curka tekočine, ki priteče skozi tanko iglo. V večini primerov je potrebna energija, da kapljice, ki nastanejo, niso prevelike. Sistemi, ki se uporabljajo, so:

- elektrostatični generatorji [7] (dovajanje električnega potenciala na nastajajočo kapljico);
- šobna resonančna tehnologija [8] (ustvarjanje vibracij z določeno frekvenco na kapljici, nastajajoči iz curka tekočine, ki priteče iz šobe);
- „rezilnik“ curka [9] („rezanje“ curka tekočine z več žicami, ki so pritrjene na hitro vrteči se plošči);
- rotirajoči diski [10] (curek tekočine teče na rotirajoči disk, na katerem se razprši v kapljice).

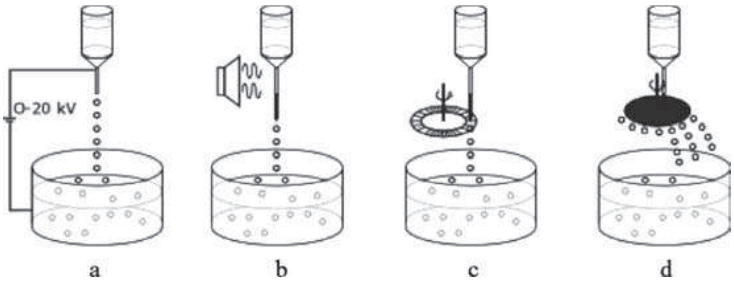

Figure 3: Different prilling methods: a) electrostatic generators, b) nozzle resonances technology, c) jet cutter, d) spinning disks

2.1.2 Tehnologije pršenja

Do razpršitve tekočine v majhne kapljice pride, ko ta teče skozi zračni/tekoči dulec oziroma lijak ali pa na hitro rotirajoči mehanizem. Produktivnost je boljša kot pri tehnologijah oblikovanja kapljice iz igle. Ta tehnologija se veliko uporablja v prehrambni industriji [11].

there has been increased interest in developing continuous systems, especially static mixers. This consists of a pipe where elements are inserted to promote fine division of the liquids in the static mixers (Figure 5b). Such systems allow emulsion preparation in a fraction of second at high flow rate (a few liters per square centimetre of section). In most of these systems, dispersion occurs in a turbulent regime which results in a large droplet size distribution [12].

2.1.4 Microemulsification

Through careful compositional selection of the two immiscible liquid systems, it is possible to reduce the surface tension to near zero. At such conditions, gentle agitation can promote formation of a very fine dispersion (less than micrometer- size droplets), and thus a very stable emulsion.

2.2 Formation of microcapsules from a liquid dispersion

The droplets produced via dispersion must then be transformed into solid- like particles by a stabilization process. Some of the most common encapsulation technologies, given various dispersion and stabilization methods, are shown in Table 1 [3].

- *Solidification. The liquid that forms droplets can be melted and then solidified by a reduction in temperature. In practice, solidification techniques are usually used in conjunction with prilling and spraying.*
- *Evaporation. Solvent is evaporated from liquid droplets formed from a polymer and volatile solvent. Solvent removal leads to active ingredients entrapped in polymeric beads. Emulsification of a polymer in a volatile organic solvent and water followed by solvent removal is referred to as "the solvent evaporation" method. Spray drying is also considered an evaporation process consisting of spraying a polymer solution (aqueous), followed by droplet drying.*
- *Gelation. Droplets of a gel forming solution are dropped into a gelation bath, forming hydrogel beads. Gelation can occur due to ionic bonding between polymer chains or simply by cooling. Gelation can also occur by spraying a thermogel (spray chilling) or trough emulsification, followed by cooling or pH change.*

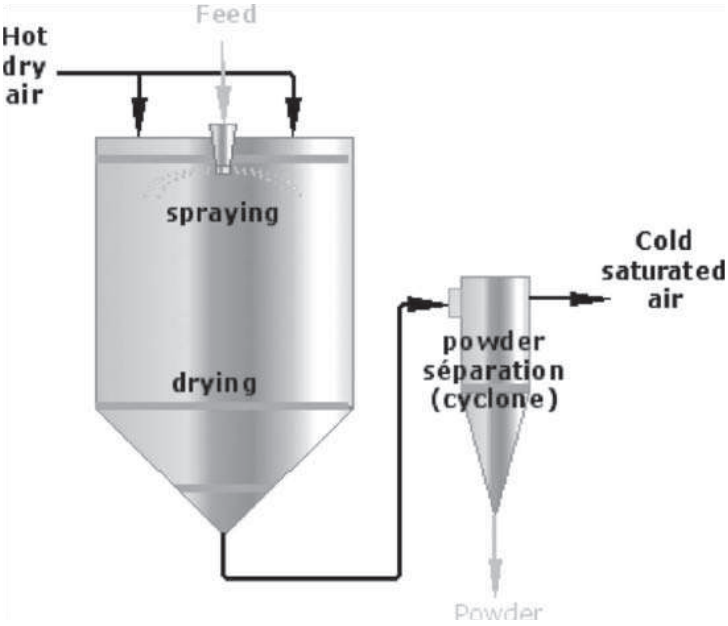

Figure 4: Spray systems

2.1.3 Emulgiranje

Tekočino, ki vsebuje sestavine, ki jih želimo kapsulirati, lahko razpršimo v drugo tekočino, s katero se prva ne meša, tako da nastane emulzija. Pri tem so favorizirani sistemi, pri katerih poteka disperginanje ob majhni strižni sili, da ne pride do denaturacije učinkovine. Najbolj preprosta metoda je uporaba reaktorja, ki je opremljen s turbino (slika 5a). Vseeno pa je povečan interes za kontinuirane sisteme, še posebno so priljubljeni statični mešalci. Ti sestojijo iz cevi, v katero so vstavljeni elementi, ki pospešujejo ločitev tekočin v statičnem mešalniku (slika 5b). Takšni sistemi omogočajo izdelovanje emulzije v manj kot sekundi pri visoki hitrosti dotoka. V večini primerov disperzija poteka v turbulentnem sistemu, zato so nastale kapljice velike [12].

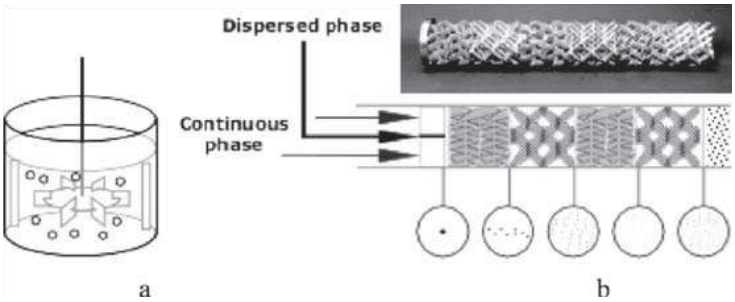

Figure 5: Liquid/liquid emulsification systems: (a) mechanical dispersion with a turbine; (b) a static mixer

2.1.4 Mikroemulgiranje

Če skrbno izberemo zmes dveh tekočih sistemov, ki se med seboj ne mešata, lahko dosežemo zmanjšanje površinske napetosti

– *Polymerization. Emulsified droplets containing a monomer react with a second monomer which is soluble in the continuous phase to form a membrane at the interface. This is referred to as interfacial polymerization. There are many derivatives of this method.*
– *Coacervation. An oil phase is emulsified in a polymer- water solution and the polymer is precipitated (e.g. by changing the pH). This polymer precipitate (coacervate) has a tendency to accumulate at the interface. Simple coacervation involves one polymer and complex coacervation involves two polymers. Coacervation achieved by combining polymeric solutions of opposite charge, is referred to as interfacial coacervation, or "polyelectrolyte complex formation".*
– *Micellar. A surfactant and polymer are dispersed to obtain small self-assembled structures. The most common structure is a liposome, generally represented by a bi-layer cell- like structure. Structures range from stable emulsions to complex multi-layer vesicles.*

All capsules described may need to be separated, washed and dried. The capsules can take form as solid microspheres, liquid core capsules, or hydrogel beads. They may be coated or treated further to obtain the final desired properties.

2.3 Methods of encapsulation by coating, agglomeration and layering

To avoid agglomeration, coating solid particles requires agitation during the spray coating skoraj na vrednost nič. V takšnih pogojih lahko z rahlim stresanjem ustvarimo fino disperzijo (kapljice, manjše od mikrometra), kar je zelo stabilen sistem za emulzijo.

2.2 Tvorba mikrokapsul iz tekoče disperzije

Kapljice, nastale z disperzijo, je treba pretvoriti v trdne delce s procesom stabiliziranja. Tabela 1 prikazuje najpogostejše, a ne vse, tehnologije kapsuliranja z metodami zamreževanja disperzije in metodami stabiliziranja [3].

Table 1: Usual terminology in microencapsulation technology

	Prilling	Spraying	Emulsification	Micro-emulsification
Solidification	hot-melt prilling	spray cooling		
Evaporation		spray-drying	solvent evaporation	solvent evaporation
Gelation	gelation	spray chilling	thermal gelation	
Polymerisation			interfacial polymerisation	in situ polymerisation
Coacervation	interfacial coacervation		coacervation	
Micellar formation				liposomes

– Strditev. Znižanje temperature tekočine, ki tvori kapljico povzroči njeno strditev. V praksi je strjevanje po navadi združeno s postopkom oblikovanja kapljice iz curka tekočine in pršenjem.
– Izhlapevanje. Ko je kapljica oblikovana iz hlapljivega topila in polimera, izhlapevanje topila vodi do nastanka polimernih biserov oziroma kapljic z aktivnimi sestavinami v notranjosti. Pršilno sušenje pomeni pršenje vodne polimerne raztopine in sušenje kapljic. Emulgiranje polimernega hlapljivega organskega topila v vodi, ki mu sledi odstranjevanje topila, se imenuje „metoda izhlapevanja topila".
– Želatiniranje. S kapljanjem kapljic raztopine, ki tvori gel, v kopel za želatiniranje, se oblikujejo hidrogelne kapljice. Želatiniranje je lahko posledica ionskih vezi, ki nastanejo med polimernimi verigami, ali pa hlajenja. Želatiniranje se lahko izvaja tudi kot pršenje termogela (spray chilling) ali z emulgiranjem, ki mu sledi hlajenje ali sprememba pH.
– Polimerizacija. Emulgirane kapljice, ki vsebujejo monomer, lahko reagirajo z drugim monomerom, topnim v osnovnem mediju, da oblikujejo membrano na mejni oziroma vmesni ploskvi. To imenujemo medpovršinska polimerizacija. Iz te metode lahko izpeljemo še mnogo drugih, sekundarnih metod.
– Koacervacija. Če je oljna faza emulgirana v polimerni vodni raztopini in pride do obarjanja polimera, na primer kot posledica spreminjanja pH, ima polimerna oborina težnjo po

process. These coatings must be stabilized or solidified by cooling or drying [13].

Spraying must be slow enough to avoid agglomeration. If well controlled, agglomeration may allow the formation of large particles from fine powders. Small particles can be sequentially sprayed with active ingredients to form layers. This process is referred to as layering.

Coating, agglomeration and layering can be combined to obtain diverse structures with various properties required for many applications.

Three conditions are necessary to establish a coating [13]:

- *Agitation. Agitation is required to avoid unwanted agglomeration, to rotate particles for uniform coating, and circulate particles to ensure a homogenous coating throughout each batch. Agitation is typically achieved by flowing gas upward through the particles to create a suspended particle bed, referred to as a fluid bed.*
- *Spraying. The coating can then be sprayed from the top, bottom, or inside of the particle bed.*
- *Solidification. Solvent evaporation is achieved by injecting hot air (fluid bed) or heating the reactor (pan coating) which stabilizes the coatings. There is also increased interest in hot melt coating in which a melted solution is sprayed directly onto the particles. Alternatively, a new powder dry technology is based on spraying a very fine powder conjunction with a plasticizer. The powder then coalesces at the surface of the particles at room temperature.*

There are many methods and variants dealing with microencapsulation and there is a lot of technological and scientific knowledge, especially in chemistry that engineers and scientists must refer to.

3 Application to textiles

Microcapsules can be physically applied to textiles from solution, dispersion or emulsion by padding, coating, spraying or immersion. These methods all require a binder such as acrylic, polyurethane, silicone or starch. The binder serves to fix capsules onto the fabric and to hold them in place during washing and wear. Micro-

akumuliranju na mejni ploskvi. To je proces koacervacije: preprosti, če gre le za en polimer, in kompleksni, če sta vključena dva polimera. Če je koacervacija dosežena s kapljanjem polimerne raztopine v polimerno raztopino nasprotnega naboja, se proces imenuje medpovršinska koacervacija ali „polielektrolitska kompleksna tvorba".

- Miceliranje. Z dispergiranjem tenzida in nekega polimera lahko nastanejo majhni skupki z različnimi strukturami. Najbolj znan je liposom, ki ga poznamo kot dvoslojno celici podobno strukturo, čeprav lahko struktura variira od stabilne emulzije do kompleksnega mnogoslojnega mehurčka.

Vse te kapsule je treba ločiti med seboj, jih oprati in včasih tudi posušiti. Lahko so v obliki trdnih mikrokroglic, kapsule s tekočim jedrom ali pa v obliki hidrogelnih kapljic. Lahko jih obdelamo še naprej ali oplaščimo, da dosežemo želene končne lastnosti.

2.3 Metode kapsuliranja z oplaščenjem, aglomeracijo in plastenjem

Da bi se izognili aglomeraciji, je pri nanosu zunanje plasti, plašča, na trdne delce potrebno stresanje le-teh. Pri tem na delce pršimo raztopino. Tako nastane ovojnica, ki jo je treba stabilizirati oziroma utrditi, na primer s hlajenjem ali sušenjem [13].

Pršenje ne sme biti prehitro zaradi potencialne aglomeracije. Če je postopek dobro nadziran, lahko z aglomeracijo nastanejo večji delci iz finih praškov. Ti lahko nastanejo tudi tako, da na majhne delce pršimo aktivne sestavine in tako oblikujemo plasti (plastenje).

Oplaščenje, aglomeracijo in plastenje lahko združujemo oziroma kombiniramo med seboj, da dosežemo raznolike strukture in s tem lastnosti, ki jih zahtevajo različne aplikacije.

Za osnovanje ovojnice oziroma plašča so potrebna tri dejanja [13]:

- Tresenje. Tresenje je potrebno: a) da bi se izognili neželeni aglomeraciji; b) da bi povzročili vrtenje delcev za nastanek enakomerne ovojnice; c) da bi povzročili kroženje delcev in s tem zagotovili homogenost ovojnice v celotni seriji. Tresenje lahko dosežemo z zračnim tokom, ki vodi v suspendirano plast delcev, imenovano tekoča plast oziroma tekoča postelja, ali pa z mehanskim procesom, kot je tresenje posode z delci.
- Pršenje. Prši se lahko od zgoraj, spodaj ali pa samo po sebi v plasti delcev.
- Utrjevanje. Z dovajanjem vročega zraka (tekoča plast) ali segrevanjem reaktorja dosežemo izhlapevanje topila (stabilizacija ovojnice). Veliko se uporablja metoda oplaščenja z vročo talino, pri čemer staljeno zmes pršimo po delcih. Nova tehnologija pa temelji na pršenju zelo finega praška in plastifikatorja. Drobci praška se povežejo oziroma „zlijejo" med seboj na površini delcev pri sobni temperaturi.

Področje mikrokapsuliranja ponuja veliko metod in možnosti. Inženirji in znanstveniki, ki se ukvarjajo z mikrokapsulami, si morajo pridobiti tudi veliko tehnološkega in strokovnega znanja, še posebno na področju kemije.

capsules can be applied to textiles including silk, cotton and synthetic fibers. With the right choice of coat, these textiles incorporated with microcapsules are harmless to the human body [14].
Microcapsules can be also incorporated into fibers, which can provide certain advantages [4]:
- *permanent incorporation of microcapsules into fibers,*
- *fibers maintain their properties,*
- *fibers can be multifunctional,*
- *incorporation of microcapsules directly with fibres does not affect subsequent dyeing, spinning and weaving processes.*

4 The use of microcapsules in textiles

4.1 Phase-change materials (PCM)

Microencapsulation of phase-change materials can reduce the influence of extreme variations in temperatures. Phase-change materials change their aggregation from solid to liquid within certain temperature ranges. In this way, thermoregulation of clothing can be achieved, providing constant temperature to the consumer. In other words, these materials are used to protect from cold and heat [17]. PCM capsules were first applied by NASA in the early 1980s for use in space suits [18]. Today these kinds of microcapsules are applied in various products including vests, parkas, snowsuits, blankets, mattresses and duvets [19].
Outlast Technologies manufacturers microencapsulated PCMs called Thermocules™. Thermocules™ can be applied as a finish on fabrics or infused into fibers during the manufacturing process.
At the Textiles Department of Faculty of Natural Sciences and Engineering in Ljubljana microcapsules with PCM core materials were incorporated into polypropylene fibers. These microcapsules were produced in Aero, d.d. Fibres were produced on the laboratory machine for spinning and extending (Extrusions Systems Limited) [4].

4.2 Fragrance finishes

Numerous attempts have been made to add fragrances directly to fibers and fabrics; however in these attempts the intended aroma was re-

3 Aplikacija mikrokapsul na tekstilije

Mikrokapsule so lahko aplicirane na tekstilije v obliki raztopine, disperzije ali emulzije z impregniranjem, premazovanjem, škropljenjem oziroma pršenjem ali izčrpanjem. Za vse te postopke je potrebno vezivo, ki je lahko akril, poliuretan, silikon, škrob itd. Njegova naloga je fiksirati mikrokapsule na blago tako, da ostanejo na svojem mestu tudi med pranjem in nošenjem. Mikrokapsule se lahko aplicira na svilo, bombaž, sintetična vlakna itd. S pravo izbiro materiala za ovojnico so človeku in koži neškodljive [14].

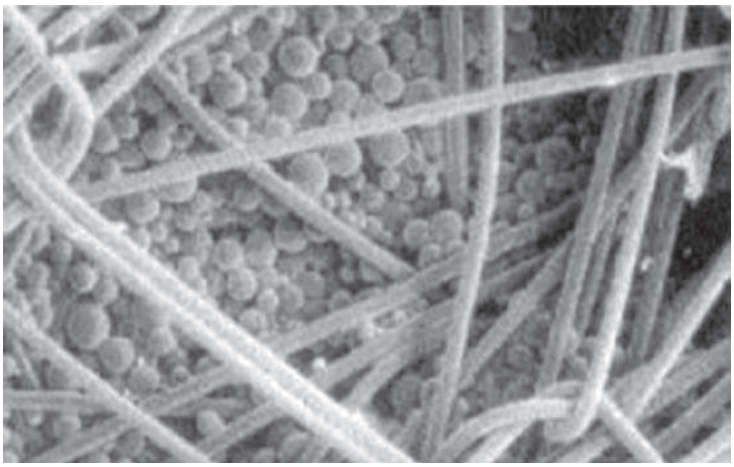

Figure 6: Microcapsules coated on the surface of fabric[15]

Mikrokapsule so lahko tudi vgrajene v vlakna. Ta način ima nekatere prednosti [4]:
- trajna vgradnja mikrokapsul v vlakna,
- vlakna ohranijo svoje lastnosti,
- multifunkcionalnost vlaken,
- vlakna z mikrokapsulami ne zahtevajo spreminjanja procesov barvanja, predenja ali tkanja.

Figure 7: Incorporation of microcapsules into fibres (Outlast®) [15]

4 Uporaba mikrokapsul v tekstilstvu

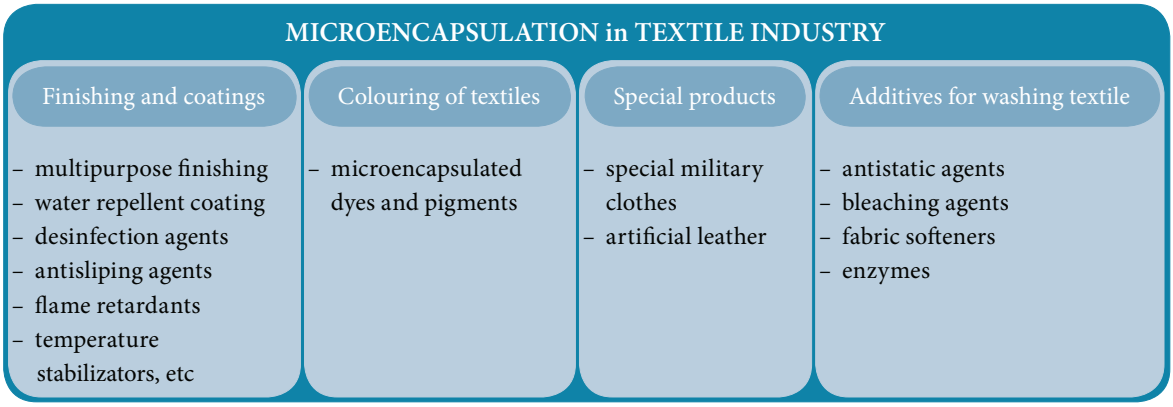

Figure 8: Application of microcapsules on textile [16]

moved after one or two wash cycles. By utilizing microencapsulated fragrances, aroma has been maintained on fabrics for a longer period of time. Microcapsules can contain essential oil flavors such as lavender, rosemary and pine which can be applied for aromatherapy. This is used mainly to help with insomnia, headaches, and to prevent bad odor. For example, the Slovenian producer Aero Celje has developed microcapsules with essential oils that were applied to shoe insoles [5].

Vitamins and moisturizers are commonly applied to all types of textile substrates, including hosiery and underwear.

Microencapsulated lemongrass- based substances exhibit good protection against mosquitoes. Microcapsules are activated during wear to release the specific aromas [20].

4.3 Fire retardants

Application of fire retardants to textile products often decreases softness and adversely affects the colors. To mitigate these problems, microcapsules with fire retardant cores were developed. They have been applied to fabrics used in various military applications (e.g. tentage) [21]. Microcapsules with fire retardant active substance are often applied to cotton and other cellulose fabrics, due to their high inherent flammability. Additionally, these fabrics are some of the most flammable material used for protective clothing, accounting for more than 60% of the world annual consumption. Because of their desirable properties, pure cotton and cotton blends are the most

4.1 PCM- materiali

Namen mikrokapsuliranja PCM- materialov (angl. phase- change materials) je zmanjšati učinek neugodnih zunanjih vplivov, to je ekstremnih temperaturnih variacij. PCM- materiali so materiali, ki spremenijo agregatno stanje iz trdnega v tekoče v določenem temperaturnem območju. Na ta način je zagotovljena konstantna temperatura in s tem dosežena termoregulacija oblačil. Z drugimi besedami, takšne materiale nanašajo na tekstil za zaščito pred mrazom in vročino [17]. PCM- kapsule so najprej uporabili pri NASI v zgodnjih osemdesetih letih, in sicer za vesoljske obleke [18]. Danes pa so takšne mikrokapsule aplicirane na različne materiale, telovnike, vetrovke, obleke za smučanje, odeje, vzmetnice itd. [19].

Outlast Technologies proizvaja mikrokapsulirane PCM- materiale, Thermocules™. Ti so lahko naneseni na blago v obliki apreture ali pa jih vgradijo v vlakna med izdelovanjem le-teh.

Na Oddelku za tekstilstvo Naravoslovnotehniške fakultete v Ljubljani so vgradili mikrokapsule z jedrnim materialom PCM v polipropilenska vlakna. Mikrokapsule so izdelali v podjetju Aero, d. d. Vlakna so bila narejena na predilno-raztezalni napravi Extrusion Systems Limited [4].

4.2 Dezodorantske tekstilije

Že mnogokrat so poizkušali aplicirati razne dišave neposredno na vlakna in tkanine, vendar je bila slabost v tem, da je aroma izgi-

Figure 10: Deodorizing shoe insoles with long-lasting effect, made in Aero d.d.

commonly used textile material in the design and production of protective clothing [22].

4.4 Thermochromic and photocromic microcapsules (color-changing technology)

The application of photochromic and thermochromic microcapsules can be found in textiles such as product labeling as well as medical and security applications. Systems that change color in response to temperature are referred to as thermochromatic. These systems undergo a change in molecular structure when heat is input. This change is related to the absorption spectrum changes of the thermochromic molecules. Similarly, systems that change color in response to UV light are referred to as photochromatic [23]. In both systems, color change is reversible. Today, microencapsulated thermochromatic dyes are produced that change color at specific temperature, in response to human contact [18].

4.5 Antimicrobials

Accumulation of microbes is often the cause for the loss of useful properties, implying microbiological decay of the underlying fibers. At appropriate temperature, humidity and dirt level, multiplication of microorganisms (bacteria, mould, mites, virus, etc.) can be uncontrollable [25]. In response to this problem, demand has grown for antimicrobial finishes for textiles used in medical and technological applications. Nanotechnology offers many opportunities to realize these functionalities [26]. Antimicrobial finishes can also be applied to textiles by microencapsulation [27].

Brace GmbH produces microcapsules with antiallergy active ingredients [28] that are applied to mattresses. These microcapsules are resistant to high temperatures and effective for up to two years.

4.6 Counterfeiting

Microencapsulation can provide a hidden marking system that can be used to prevent counterfeiting of high value textiles, including branded and designer goods. In these applications, microcapsules are applied to labels containing a color activator. Detection is then achieved by exposure to solvent or UV light that

nila že po prvem ali drugem pranju. Z mikrokapsuliranjem pa so dosegli, da dišave ostanejo na blagu dlje časa. Mikrokapsule lahko vsebujejo razna eterična olja, na primer sivke, rožmarina, bora in druge. Tekstilije z nanosom takšnih dišav se uporabljajo v aromaterapiji za lajšanje raznih težav, na primer pri nespečnosti, glavobolu in za odpravljanje neprijetnega vonja. Slovenski proizvajalec Aero Celje je izdelal mikrokapsule z eteričnim oljem in jih nanesel na obutveni vložek [5].

Na razne tekstilije, vključno s spodnjim perilom in nogavicami, so lahko naneseni tudi vitamini in vlažilni preparati.

Mikrokapsulirana substanca na osnovi limonine trave je obdelava, ki zagotavlja učinkovito zaščito pred komarji. Mikrokapsule se aktivirajo med nošenjem in pri tem sproščajo snov, ki odganja komarje [20].

4.3 Zaviralci ognja

Nanos zaviralcev ognja na tekstilne proizvode pogosto povzroči njihovo togost in ima negativne učinke na obarvanost. Da bi se izognili takšnim neprijetnostim, so bile izdelane mikrokapsule z aktivno jedrno substanco, ki zavira ogenj. Te se med drugim nanašajo na tkanine, ki se uporabljajo za vojaške namene, na primer za šotorsko opremo [21]. Mikrokapsule z zaviralci ognja pogosto aplicirajo tudi na bombažne in druge celulozne tekstilije, saj so najbolj gorljiv material, ki se veliko (več kot 60 odstotkov svetovne letne uporabe) uporablja za zaščitna oblačila. Zaradi uporabnosti bombaža in njegovih mešanic je namreč to najpogosteje uporabljen material za oblikovanje in proizvodnjo zaščitnih oblačil [22].

4.4 Mikrokapsule s termokromnimi in fotokromnimi barvili (tehnologija spreminjanja barv)

Nanos fotokromnih in termokromnih mikrokapsul (tehnologija spreminjanja barve) se uporablja tudi na področju etiketiranja izdelkov ter medicinskih in varnostnih aplikacij. Pri enem od sistemov se barva spreminja oziroma reagira na temperaturo, to je termokromatski sistem. Termokromizem razložimo kot spremembo molekulske ali nadmolekulske strukture pod vplivom toplote, ki je

Figure 11: Colour changing mouse pad [24]

can break the microcapsules open to release and develop the inner content [18].

Nocopi Technologies Inc. patented a covert security ink that can be applied to various products. It is invisible and cannot be detected by any light. The secure Copi Mark ink remains invisible until it comes in contact with a specially formulated ink applied by pen. The resulting image can be made visible to the naked eye or under UV light [29].

4.7 Additives for washing textiles-fragrances, enzymes

Microencapsulation is an elegant way of improving the performance of aromas in washing powders and conditioners. This can also help to protect unstable or non-substantive biodegradable fragrances from aggressive detergent compounds [30].

The enzymes in liquid detergents have to be stabilized at relatively high water content. Additionally, the protease that is normally present, needs to be inactive to avoid unwanted proteolysis of the enzymes. One of Novozymes' latest innovations has been the development of microcapsules for these enzymes. Release from the capsules is triggered by a change in ion strength experienced when liquid detergents are added to the wash water [31].

4.8 Dyeing and printing

Microencapsulation is also used as a way of delivering dyes and pigments to textiles, for dyeing and printing. One example application is tridimensional printing. This technique is mostly used for printing T-shirts. The printing paste is composed of small spheres (10–20

povezana s spremembami v absorbcijskem spektru termokromne molekule. Sprememba barve je reverzibilna. Pri drugem, tj. fotokromatskem sistemu, pa snov reagira na UV- svetlobo oziroma pod vplivom svetlobe spremeni kemično strukturo in s tem tudi spekter elektromagnetnega valovanja [23]. Posledica tega je sprememba barve, ki je prav tako povratna. Danes se proizvajajo tudi mikrokapsulirana termokromatska barvila, ki spreminjajo barvo pri določeni temperaturi, kot reakcija na človeški dotik [18].

4.5 Protimikrobne apreture

Mikrobi so pogosto povezani s pojavom izgube uporabnih lastnosti tkanine, ki je posledica mikrobiološkega razpada vlaken. Mikroorganizmi se pri ustrezni vlagi, temperaturi in umazaniji zelo hitro razmnožijo tako na naravnih kot sintetičnih vlaknih [25]. Med mikroorganizme štejemo bakterije, plesni, glivice, viruse, pršice itd. Da bi preprečili tovrstne probleme, pomembnost in zahteve protimikrobnih apretur neprestano naraščajo, še posebej v primeru medicinskih in tehničnih tekstilij. Na tem področju funkcionalizacije tekstilij nanotehnologija ponuja mnogo novih možnosti [26]. Eden od načinov apliciranja protimikrobnih apretur na tekstilije je njihovo mikrokapsuliranje in vgraditev v vlakensko matrico tekstilnega materiala [27].

Podjetje Brace GmbH proizvaja mikrokapsule, ki vsebujejo antialergijsko snov [28]. Nanašajo jih na vzmetnice. Mikrokapsule so odporne proti visoki temperaturi in učinkujejo do dve leti.

4.6 Ponarejanje

Mikrokapsuliranje se uporablja tudi pri problemih ilegalnega kopiranja oziroma ponarejanja tekstilij z visoko dodano vrednostjo, zaščitno blagovno znamko itd., saj zagotavlja skrit, vendar jasen označevalni sistem. Mikrokapsule, nanesene na etiketo, vsebujejo aktivator barve. Z uporabo UV- svetlobe ali topila se mikrokapsule zlomijo, vsebina se sprosti in s tem se barva razvije. Na ta način lahko preverimo originalnost izdelka [18].

V Nocopi Technologies Inc. so patentirali prikrito varnostno črnilo, ki je lahko naneseno na različne proizvode. Je nevidno in se ga

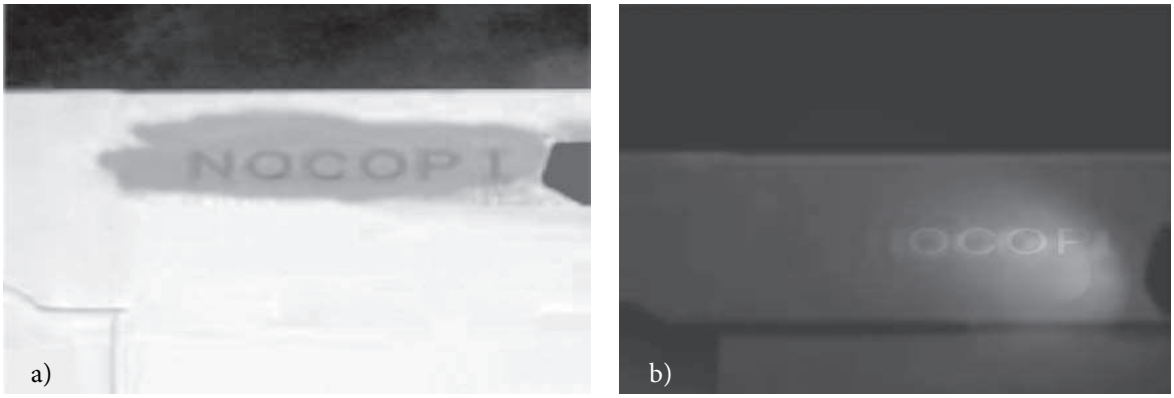

Figure 12: Revealing the image using special Nocopi pens a) view with a naked eye or b) view under UV light

μm) with an active core. This core substance contains products that decay and release gas at certain temperature (90–140 °C), thus expanding into a layer, a few millimetres thick. The capsule coat is made of acrylonitrile and vinyldenchloride [32].

Various dyes can also be encapsulated, including cationic, reactive, acid and disperse [33]. Solid dye particles are coated with a hydrophilic coat (e.g. gelatine, agar-agar and methyl-cellulose). The size of these capsules is 10–200 μm. In this way we can combine dyes with reactive substances without inducing reaction until the microcapsules break open under desired conditions (steam, etc).

4.9 Well-being

Skintex® [34] from Cognis (the holding company of Pulcra Chemicals) is an innovative technology that adds extra value to clothes and tolerates several washes. Textiles employing this technology can have various effects, including cooling, moisturizing, relaxing, energizing and aromatherapy.

- *Textiles with a cooling effect. High-tech microcapsules are built directly into the fabric and release natural ingredients like Myritol® and menthol. The fresh Skintex®* effect is ideal for clothing worn close to the skin.*
- *Textiles with a moisturizing effect. Microcapsules filled with natural, high-grade ingredients such as Vitamin E, passion fruit oil and olive oil derivates are released gradually onto the skin, thus supplying the desired effects for many hours. These textiles are ideal for underwear and shirts.*

One of the most innovative products of Skinkiss Ltd. is caffeine slimming tights. These revolutionary tights use caffeine as an active ingredient. The caffeine is introduced into the fiber using micro-capsules that are activated at body temperature, causing them to act directly onto the skin [35].

5 Conclusion

The wide range of microcapsule applications to textiles described in this paper are only some of the most interesting applications. Today, microcapsules have been presented in nearly every

ne da odkriti z žarki vsake svetlobe. Varno črnilo Copi Mark ostane nevidno, dokler ne pride v stik s specialnim črnilom, ki se ga nanese s posebnim peresom. Nato lahko skriti napis na proizvodu odkrijemo na dva načina: lahko postane viden s prostim očesom ali pa ostane neviden in ga zaznamo z uporabo UV- svetilke [29].

4.7 Dodatki v pralna sredstva – dišave, encimi

Mikrokapsuliranje je eleganten način za izboljšanje obstojnosti, odpornosti oziroma vzdržljivosti arom tudi v pralnih praških in mehčalcih za perilo. Njegova prednost je tudi v tem, da ščiti nestabilne dišave pred agresivnimi komponentami detergentov [30].

Kapsuliramo lahko tudi encime. Encimi v tekočih detergentih morajo biti stabilizirani oziroma morajo imeti zadostno stabilnost pri relativno veliki količini vode; v primerih, v katerih je prisoten tudi encim proteaza, mora biti ta v neaktivni obliki, da ne pride do neželene proteolize drugih encimov. Ena od novosti v podjetju Novozymes je proizvodnja mikrokapsul, ki vsebujejo encime. Sproščanje mikrokapsul je zavirano kot posledica spremembe ionske moči, ko tekoči detergent dodamo v pralno tekočino [31].

4.8 Barvanje in tiskanje

Mikrokapsuliranje se uporablja tudi pri barvanju in tiskanju tekstilij. Eden od primerov tiskanja je tridimenzionalni tisk. Največkrat se uporablja za tiskanje majic (T-shirt) z raznimi reklamnimi napisi. Pasta je sestavljena iz majhnih kapsul (10–20 μm), te imajo v jedru določeno snov, pri visoki temperaturi (90–140 °C) pa razpadejo in sproščajo plin oziroma se same uplinijo. Pasta ekspandira v nekaj milimetrov visoko plast. Ovojnico mikrokapsul sestavljata akrilonitril in vinilidenklorid [32].

Mikrokapsuliramo lahko tudi različna barvila (kationska, kisla, reaktivna, disperzna, redukcijska). Trdni delci barvila so prevlečeni z visokomolekularno hidrofilno ovojnico, na primer iz želatine, pektina, agar agarja, metilceluloze ali poliakrilne kisline. Premer mikrokapsul je 10–200 μm. V tej obliki lahko barvila kombiniramo z reaktivnimi snovmi, pri čemer ne pride do reakcije, dokler se ovojnice mikrokapsul ne odprejo, kar je posledica vpliva različnih dejavnikov, na primer pare [33].

4.9 Izboljšanje počutja

V podružnici podjetja Pulcra Chemicals, Cognisu, so razvili inovativno tehnologijo Skintex® [34], ki tekstilijam doda vrednost in je obstojna proti več pranjem. Tekstilije s takšno tehnologijo imajo lahko hladilni ali vlažilni učinek, učinek aromaterapije, sproščanja itd.

- Tekstilije s hladilnim učinkom: proizvodnja tekstilij z mikrokapsulami, ki imajo v jedru substance na osnovi naravnih sestavin, kot sta na primer Myritol® in mentol. Takšne tekstilije so najprimernejše za oblačila, ki so v neposrednem stiku s kožo.
- Tekstilije z vlažilnim učinkom: proizvodnja tekstilij z mikrokapsulami, ki imajo v jedru vlažilne substance, na primer

field. Encapsulation has become a very powerful tool, because it is invisible and can be active via slightest stimulus. The application of microcapsules to textiles is not technically difficult, and is advantageous because it does not affect the underlying textile properties.

vitamin E, derivate olivnega olja, olje pasijonke. Te tekstilije so najprimernejše za spodnje perilo in majice.

Najnovejši proizvod podjetja SkinKiss Ltd. so hlačne nogavice z mikrokapsulami, ki vsebujejo kofein [35]. Med nošenjem začnejo mikrokapsule pod vplivom telesne temperature sproščati aktivno snov – kofein, ki povzroči zmanjšanje obsega stegen in celulita.

5 Zaključki

V tem prispevku so opisane samo nekatere od mnogih zanimivih možnosti apliciranja mikrokapsul na tekstilije. Danes skoraj ne obstaja področje, na katerem mikrokapsule ne bi bile prisotne. Postale so zelo uporabno sredstvo, saj so nevidne in se lahko aktivirajo ob najrahlejšem dotiku, prav tako pa njihov nanos na tekstilije ni zahteven in ne vpliva na karakteristike oziroma ne poslabša lastnosti tekstilij.

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