

## Hydrolysis of reactive dyes in flax dyeing, depending on the type of process

Original Scientific Paper

Received July 2007 • Accepted September 2007

### Abstract

Flax fibres (*Linum ussitatissimum*) belong to the group of natural cellulose fibres, and their qualities depend on the flax species, fibre/steam parts' separation methods, pre-treatment, dyeing and finishing. When flax garments need to have top-colour fastness, reactive dyes are used for dyeing. Reactive dyes are simple to use and produce brilliant hues, with good wet-colour fastness due to the covalent bonds between the fibres and the dye. Pre-treatment and bleaching of flax fibres influences their hydrophilicity, physical-chemical, and dyeing properties. The kinetic and thermodynamic nature of a dyeing process also depends on the dye's chemical structure, various technological procedures, and the dyeing parameters.

A common problem resulting from reactive dyeing is highly-colourated waste dye-baths that result from dye hydrolysis during the dyeing procedure. Therefore, the present research focused on the influences of two dyeing processes (exhaustion and impregnation) and two reactive dye systems on dye hydrolysis during flax fabric dyeing. The obtained results indicated that the amount of hydrolysed and unbound dyes

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## Hidroliza reaktivnih barvil pri barvanju lanu v odvisnosti od vrste procesa

Izvirni znanstveni članek

Poslano julij 2007 • Sprejeto september 2007

### Izvleček

Lanena vlakna (*Linum ussitatissimum*) spadajo v skupino naravnih celuloznih vlaken, njihova kakovost pa je odvisna od sorte lanu, postopkov ločevanja vlaken od drugih delov stebela, predobdelave, barvanja in postopkov plemenitenja. Kadar laneni izdelki zahtevajo vrhunske obstojnosti, uporabljamo za barvanje reaktivna barvila, ki jih odlikujejo razmeroma enostaven postopek barvanja, briljantni barvni toni ter zaradi nastanka kovalentnih vezi med vlaknom in barvilom tudi dobre mokre obstojnosti. Postopki predobdelave in beljenja lanenih vlaken vplivajo zaradi razgradnje primesi na hidrofilito, hkrati pa tudi na fizikalno-kemijske in barvalne lastnosti vlaken. Kinetične in termodinamične zakonitosti procesa barvanja le-teh so odvisne tudi od kemijske strukture barvil, različnih tehnoloških postopkov in parametrov barvanja.

Pogosto težavo pri barvanju z reaktivnimi barvili predstavlja močna obarvanost odpadnih barvalnih kopeli, kar je predvsem posledica hidrolize reaktivnih barvil med procesom barvanja. Zato smo se v raziskavi osredotočili na vpliv postopka barvanja (izčrpavanje in impregnacija) in reaktivnega sistema barvil na hidrolizo reaktivnih barvil pri barvanju lanene tkanine. Iz rezultatov dela izhaja, da je količina hidroliziranih in nevezanih barvil odvisna od vrste barvila, njegove kemijske strukture, velik pomen pa ima tudi primeren izbor postopka barvanja.

Ključne besede: lan, barvanje, vinilsulfonska reaktivna barvila, bireaktivna barvila, hidroliza, spektroskopija, barvna metrika

depends primarily on the type of dye and its chemical constitution, and also on selecting an appropriate dyeing process.

*Key words:* flax, dyeing, vinylsulphone reactive dyes, bi-reactive dyes, hydrolysis, spectroscopy, colourimetry.

## 1 Introduction

Many different reactive dyes are present in the market today, including the predominant mono-functional dyes with one reactive system in a structure. Disadvantages of these dyes from a technological and ecological point of view is the current usage of large amounts of electrolytes during dyeing and a poor exhaustion rate that is a consequence of dye hydrolysis. Coloured effluents represent a major ecological problem [1, 2].

Reactive dyes are capable of forming covalent bonds between a carbon atom of the dye ion or molecule and an oxygen atom of a cellulose fibre hydroxyl group. Chemical reactivity of the applied dye, affinity of the reactive dye to the flax fibre and the diffusion rate of reactive dye into the flax fibre have the largest effect on the bonding of the reactive dye with the fibre. The principal structure of the dye combines the chromophore linked with the organic compound, which is an unsaturated donor group and has an effect on light absorption, i.e. colour. The most important chromophores are: azo ( $-N=N-$ ), nitro ( $-NO-O-$ ), nitroso ( $-N=O$ ), carbonyl ( $>C=O$ ), ethylene ( $>C=C<$ ) and 1,4 benzoquinone groups. Chromogen is also a carrier of auxochromic groups that have an effect on the affinity of dye to the fibre. Amongst them, the most important are: hydroxyl ( $-OH$ ), amino ( $-NH_2$ ) and the substituted amino group ( $-N(CH_3)_2$ ).

Reactive dyes are classified into mono-functional or bi-functional, in regard to the number of reactive groups, their similarity or diversity, and the position of the reactive system in the dye molecule. The first class contains mono- or bi-reactive dyes, while the second class contains homo-bifunctional or hetero-bifunctional including two equal or two different reactive systems, bonded separately or directly. Reactive

## 1 Uvod

Na tržišču so danes prisotna različna reaktivna barvila, med katerimi še vedno prevladujejo monofunkcionalna, ki imajo v svoji strukturi le en reaktivni sistem. Njihova pomanjkljivost s tehnološkega in ekološkega vidika je sočasna uporaba velikih količin elektrolita pri barvanju in ob tem nizka stopnja izčrpavanja, ki je posledica hidrolize barvil. Obarvanost odpadnih vod pa pomeni njihovo ekološko obremenitev [1, 2].

Reaktivna barvila so sposobna tvoriti kovalentne vezi med ogljikovim atomom molekule ali iona barvila in kisikovim atomom hidrosilne skupine celuloznega vlakna. Na vezavo reaktivnega barvila z vlaknom imajo največji vpliv kemična reaktivnost barvila, afiniteta reaktivnega barvila do lanenega vlakna in sposobnost difuzije reaktivnega barvila v laneno vlakno. Osnovno barvilo sestavlja kromofor, vezan na organsko spojino, ki je nenasičena donorska spojina in vpliva na absorpcijo svetlobe oz. na barvo. Najpomembnejši kromoforji so: azo ( $-N=N-$ ), nitro ( $-NO-O-$ ), nitroso ( $-N=O$ ), karbonilna ( $>C=O$ ), etilenska ( $>C=C<$ ) in 1,4-benzokinonska skupina. Kromogen je tudi nosilec avksokromnih skupin, ki vplivajo na afiniteto barvila do vlakna. Med temi so najpomembnejše: hidroksilna ( $-OH$ ), amino ( $-NH_2$ ) in substituirana amino skupina ( $-N(CH_3)_2$ ).

Glede na število reaktivnih skupin, njihovo enakost ali različnost ter položaj reaktivnega sistema v molekuli barvila razvrščamo reaktivna barvila v monofunkcionalna in bifunkcionalna. V prvi skupini so barvila lahko mono- ali bireaktivna, v drugi skupini pa najdemo homobifunkcionalna in heterobifunkcionalna, kar pomeni, da ta barvila vsebujejo dva enaka ali dva različna reaktivna sis-

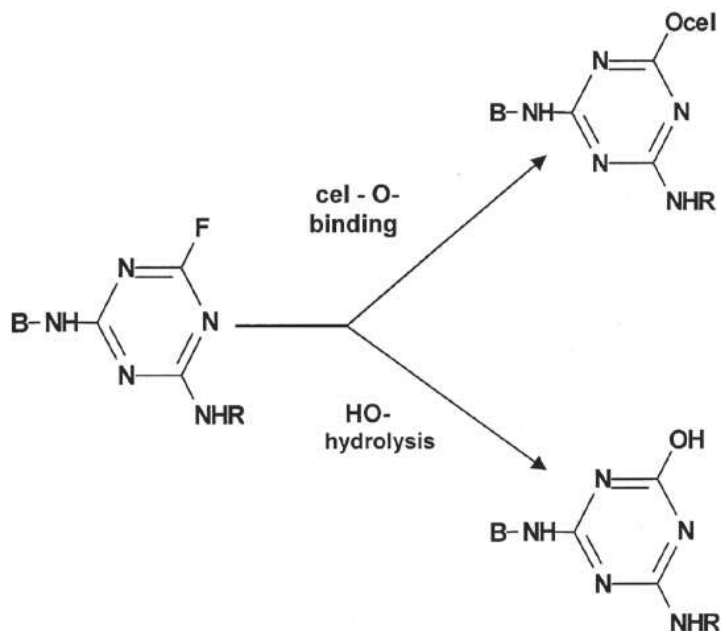


Figure 1: Binding and hydrolysis of reactive dyestuff

dyes can be also be classified as poly-functional. A primary amino group ( $-NH_2$ ), secondary amino/imino group ( $>NH$ ) or acrylamino group ( $-N(CH_3)-$ ) is often the bridge link or group that links the reactive system to the chromophore. The bridge-link also influences the reactivity, substantivity, bond-stability and affinity of the dye to the cellulose.

A solubilizing hydrophilic group is also attached to the dye molecule, resulting in its solubility in water. Therefore, the dyestuffs for cellulose fibre dyeing must have one to four sulphonic groups. Reactive dyes are azo (monoazo, diazo), anthraquinone, phthalocyanine and threphenilmethine dyes in regard to chromogen. Approximately 80% of all reactive dyes are based on the azo chromogen [2, 3, 4, 5].

Dyes are covalent bonded with  $-OH$  groups of cellulose fibres, and could also react with  $-OH$  groups of water, causing dye hydrolysis. The reaction that occurs between dyes and fibres is an addition or nucleophilic substitution, where the carbon atoms of reactive dyes attract free pairs of electrons on the fibres' nucleophilus. Figure 1 shows the bonding reaction and reactive dye hydrolysis [6, 7, 8, 9].

The dyeing procedures are complex physical-chemical processes that play an active part during the dye's transition from the dyeing medium onto the fibre. The dyeing procedure depends on the physical-chemical properties of fibres, chemical structures of dyestuffs, and the technological parameters of dyeing [10].

Here, we studied the dyeing characteristics of three bi-reactive dyes with two reactive groups: chlorotriazine and vinylsulphonic, and three mono-reactive dyes with one vinylsulphonic reactive group in the dyeing procedure of flax fabric that was alkaline pre-treated and bleached. Dyeing of flax is the same or similar to the dyeing procedures of other cellulose fibres, although flax has a more compact structure and a larger amount of alloys. Comparative dyeing was performed according to the exhaust and padding processes, using different concentrations of selected dyes. Analysis of the dye-bath was accomplished by spectrophotometric determination of dye-bath exhaustion rate, measurement of dye residual after soaping and extraction, and calculation of the fixed dye amount on the fibre.

tema, ki sta lahko ločena ali direktno vezana. Reaktivna barvila pa so lahko tudi polifunkcionalna.

Kot vezni most ali skupina, ki povezuje reaktivni sistem s kromoforjem, je pogosto primarna amino skupina ( $-NH_2$ ), sekundarna amino/imino skupina ( $>NH$ ) ali akrilamino skupina ( $-N(CH_3)-$ ). Vezni most tudi vpliva na reaktivnost, substantivnost oz. stopnjo vezave, stabilnost vezi in tudi na afiniteto barvila do celuloze.

Na molekulo barvila je vezana tudi vodotopna hidrofilna skupina, ki omogoča topnost barvila v vodi. Barvila, namenjena barvanju celuloznih vlaken, morajo zaradi vodotopnosti vsebovati od ene do štiri sulfonske skupine. Glede na kromogen so reaktivna barvila azo (monoazo, diazo), antrakinonska, ftalocianinska in trifenilmetsinska barvila. Približno 80 % vseh reaktivnih barvil temelji na azo kromogenu [2, 3, 4, 5].

Barvila se z  $-OH$  skupinami celuloznih vlaken vežejo kovalentno, reagirajo pa tudi z  $-OH$  skupinami vode, kar povzroča hidrolizo barvil. Vrsta reakcije, ki poteka med barvili in vlakni, je adicija ali nukleofilna substitucija, pri kateri ogljikovi atomi reaktivne skupine privlačijo proste pare elektronov na nukleofilu vlakna. Reakcijo vezanja in hidrolize barvil prikazuje slika 1 [6, 7, 8, 9].

Barvalni proces je zbir fizikalno-kemijskih procesov, ki sodelujejo pri prehodu barvil iz barvalnega medija na vlakno. Proces barvanja je odvisen od fizikalno-kemijskih lastnosti vlaken, kemijske strukture barvil in tehnoloških parametrov barvanja [10].

V raziskavi smo proučevali barvalne lastnosti treh bireaktivnih barvil z dvema reaktivnima skupinama, klorotriazinsko in vinilsulfonsko, ter treh monoreaktivnih barvil z eno vinilsulfonsko reaktivno skupino v procesu barvanja lanene tkanine, ki smo jo alkalno predobdelali in belili. Barvanje lanu običajno poteka po enakih ali podobnih postopkih kot barvanje drugih celuloznih vlaken, za lan pa je značilna gostejša in kompaknejša struktura ter večje količine inkrustov. Barvanje je bilo primerjalno izvedeno tako po postopku izčrpavanja kot impregnacije z različnimi koncentracijami izbranih barvil. Analiza barvalnih kopeli je vključevala spektrofotometrično določevanje izčrpanja barvalne kopeli, ostanke barvil po barvanju, miljenju in ekstrakciji ter iz tega izračunano količino barvila, vezanega na vlakno.

## 2 Metode dela

### 2.1 Lanena tkanina

Uporabili smo tkanino iz 100 % lanu (Hungary LEN – Madžarska), v vezavi platno in s finostjo preje 50 tex.

### 2.2 Postopki predobdelave

Postopek alkalne predobdelave in beljenja je potekal na laboratorijskem barvalnem aparatu LABOMAT (podjetja Mathis), pri kopelnem razmerju 1 : 10 v destilirani vodi s pomožnimi sredstvi in kemikalijami, ki jih prikazuje preglednica 1.

## 2 Working methods

### 2.1 Flax fabric

We used 100% flax fabric (Hungary LEN) in a plain weave, with a yarn fineness of 50 tex.

### 2.2 Pre-treatment procedures

Alkaline pre-treatment and bleaching were performed using a laboratory dyeing apparatus LABOMAT (Mathis) at a liquor ratio of 1:10 in distilled water with various auxiliaries and chemicals, as summarized in Table 1.

TANEX PS (Sybron/Tanatex) is a sequestering agent for heavy metal ions' removal in highly alkaline baths. TANAWET BC (Sybron/Tanatex) is a highly effective wetting and washing agent, used for alkaline pre-treatment. TANEX RENA liq. (Sybron/Tanatex) is a stabilizer suitable for all peroxide bleaching processes, and is a turbid water-soluble liquid with good stability at pH 7–13. TANASPERSE OH (Sybron/Tanatex) is a sequestering agent during cellulose fibre bleaching. It is a colourless, alkaline and water-soluble liquid, stable on alkaline and oxidation agents. PLEXSEN CA (Sybron/Tanatex) is a washing agent used after bleaching. PLEXSEN APR (Sybron/Tanatex) is an acid sequestering agent.

### 2.3 Dyes and auxiliaries

Dyeing was conducted according to the exhaust and padding procedures using two different groups of dyestuffs, Bezaktiv S are combined bi-reactive dyes and Bezaktiv V are mono-reactive vinylsulphone dyes. Both groups of dyes are produced by Bezema (Swiss), and are suitable either for padding processes or for exhaust dyeing. Table 2 summarizes the applied dyestuffs, their maximal absorption wavelengths, and dye abbreviation.

### Exhaustion process

Dyeing was performed by means of the LABOMAT (Mathis) laboratory dyeing apparatus at a temperature of 60 °C (Bezaktiv S dyestuffs) or 40 °C (Bezaktiv V dyestuffs). Applied auxiliaries and chemicals, and dyeing conditions were also described. Dyeing with selected dyes was performed at three concentrations: 0.1%, 1%, and 3%.

Table 1: Pre-treatment and bleaching procedures at liquor ratio 1 : 10

Procedure	Additions	Conditions
Alkaline pre-treatment	2 mL/L TANEX PS 3 mL/L TANAWET BC 9 g/L Na <sub>2</sub> CO <sub>3</sub> 4 g/L NaOH 100 %	T = 950 °C t = 30 min
Rinsing	without auxiliaries	T = 800 °C t = 15 min
Bleaching	2 mL/L TANEX RENA liq 1 mL/L TANASPERSE OH 2 g/L NaOH 100 % 13 mL/L H <sub>2</sub> O <sub>2</sub> 35 %	T = 950 °C t = 70 min
Washing 1	2 mL/L PLEXSEN CA	T = 800 °C t = 10 min
Washing 2	2.5 mL/L PLEXSEN APR	T = 500 °C t = 10 min

Posamezna tekstilna pomožna sredstva imajo naslednji pomen: TANEX PS (Sybron/Tanatex) je pomožno sredstvo za sekvestriranje ionov težkih kovin v močno alkalnih kopelih. TANAWET BC (Sybron/Tanatex) je visoko učinkovito omakalno in pralno sredstvo, uporabno za alkalno predobdelavo. TANEX RENA liq. (Sybron/Tanatex) je ekološki stabilizator izjemne kakovosti, ki je primeren za vse procese peroksidnega beljenja. Je motna v vodi topna tekočina, njena pH-stabilnost je dobra v področju 7–13. TANASPERSE OH (Sybron/Tanatex) je sekvestrirno sredstvo za beljenje celuloznih vlaken. Je prozorna alkalna tekočina, dobro topna v vodi, odporna proti alkalijam in oksidacijskim sredstvom. PLEXSEN CA (Sybron/Tanatex) je pralno sredstvo po beljenju. PLEXSEN APR (Sybron/Tanatex) je kislo sekvestrirno sredstvo.

### 2.3 Barvila in tekstilna pomožna sredstva

Barvanje smo izvajali po postopku izčrpavanja in postopku impregnacije z dvema skupinama barvil. Barvila Bezaktiv S so kombinirana bireaktivna barvila in Bezaktiv V so monoreaktivna vinilsulfonska barvila. Obe skupini barvil sta primerni za impregnacijske in izčrpalne postopke barvanja, prodaja pa jih podjetje Bezema iz Švice. V preglednici 2 so navedeni uporabljena barvila, njihove valovne dolžine maksimalne absorpcije in skrajšana imena barvil, uporabljena pri prikazovanju rezultatov.

### Postopek barvanja z izčrpavanjem

Barvanje vzorcev smo izvajali na laboratorijskem barvalnem aparatu LABOMAT (podjetja Mathis) pri temperaturi 60 °C (barvila Bezaktiv S) in 40 °C (barvila Bezaktiv V). Dodatki posameznih po-

Figures 2 and 3 represent dyeing diagrams when dyeing with Bezaktiv S or Bezaktiv V dyestuffs.

Recipe and dyeing conditions for Bezaktiv S dyestuffs:

1.5 g/L SARABID LDR  
 1.5 g/L MEROPAN DPE  
 $x$  g/L  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$   
 $y$  % Bezaktiv S  
 5 g/L  $\text{Na}_2\text{CO}_3$   
 $y$  mL/L  $\text{NaOH}$ ,  $w(\text{NaOH}) = 32.5\%$   
 $T = 600$  °C  
 $t = 110$  mins  
 Liquor ratio: 1 : 20  
 Mass of sample: 8 g

Recipe and dyeing conditions for Bezaktiv V dyestuffs:

$x$  g/L  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$   
 $y$  % Bezaktiv V  
 5 g/L  $\text{Na}_2\text{CO}_3$   
 $z$  mL/L  $\text{NaOH}$ ,  $w(\text{NaOH}) = 32.5\%$   
 $T = 400$  °C  
 $t = 100$  mins  
 Liquor ratio: 1 : 20  
 Mass of sample: 8 g

#### Padding process (Pad-batch)

Impregnation with a dye-bath was performed using a laboratory pad mangle (Mathis) with applied dyes, auxiliaries and chemicals, and under dyeing conditions that were also described.

Recipe and dyeing conditions for Bezaktiv S dyestuffs:

$x$  g/L Bezaktiv S  
 80 g/L urea  
 4 g/L SUBITOL RNC  
 50 mL/L water glass  
 19 mL/L  $\text{NaOH}$ ,  $w(\text{NaOH}) = 32.5\%$   
 $T_{\text{impregnation}} = 20 - 250$  °C  
 Wet pick-up (Ou) = 100%  
 $T_{\text{fixation}} = \text{room}$   
 $t_{\text{fixation}} = 18$  hours

Recipe and dyeing conditions for Bezaktiv V dyestuffs:

$x$  g/L Bezaktiv V

Table 2: Applied dyestuffs, wavelength of absorption maximum ( $\lambda_{\text{max}}$ ) and dyes abbreviation

Dyestuff	$\lambda_{\text{max}}$ (nm)	Abbreviation
BAZAKTIV ROT S – 3B 150	560	RD S
BEZAKTIV GELB S – 3R 150	440	RU S
BEZAKTIV BLAU S – GLD 150	612	MO S
BEZAKTIV ROT V – BN	520	RD V
BEZAKTIV GELB V – GR	410	RU V
BEZAKTIV SCHWARZ V – B 150	600	MO V

možnih sredstev in kemikalij ter pogoji barvanja so podani v nadaljevanju. Barvanje je potekalo v treh koncentracijah (0,1 %, 1 % in 3 %) z izbranimi barvili.

Sliki 2 in 3 prikazujeta diagrama barvanja z barvili Bezaktiv S oz. Bezaktiv V.

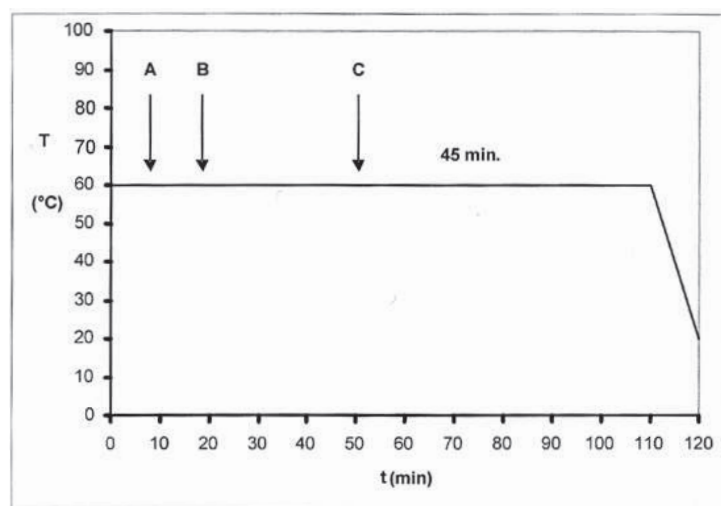


Figure 2: Dyeing diagram for Bezaktiv S dyestuffs

Receptura in pogoji barvanja za barvila Bezaktiv S:

1,5 g/L SARABID LDR  
 1,5 g/L MEROPAN DPE  
 $x$  g/L  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$   
 $y$  % Bezaktiv S  
 5 g/L  $\text{Na}_2\text{CO}_3$   
 $y$  mL/L  $\text{NaOH}$ ,  $w(\text{NaOH}) = 32,5$  %  
 $T = 60$  °C  
 $t = 110$  min  
 kopelno razmerje (KR): 1 : 20  
 masa vzorca: 8 g

Receptura in pogoji barvanja za barvila Bezaktiv V:

$x$  g/L  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$

100 g/L urea  
 4 g/L SUBITOL RNC  
 50 mL/L water glass  
 0.46 mL/L NaOH, w(NaOH) = 32.5%  
 $T_{\text{impregnation}} = 20 - 250 \text{ }^{\circ}\text{C}$   
 Wet pick-up (Ou) = 100%  
 $T_{\text{fixation}} = \text{room}$   
 $t_{\text{fixation}} = 9 \text{ hours}$

After dyeing with both groups of dyes, the samples were squeezed and dried. The dried samples were first soaped and then extracted. Soaping was performed at boiling temperature for 20 mins with 0.5 g/L COTOBLANC NSR.

MEROPAN DPE (Bezema) is a foam-free protective colloid with sequestering properties on Ca and Mg ions, and dispersing properties on water-soluble flax accompanying substances. SARABID LDR (Bezema) is a levelling agent for cellulose dyeing with reactive dyestuffs. SUBITOL RNC (Bezema) is a wetting agent with good washing capacity, with little foaming. COTOBLANC NSR (Bezema) is a product for fast soaping after reactive dyeing, which removes unfixed and hydrolysed dyestuff from the fibre.

#### 2.4 Determination of dye residual in a dye-bath

Determination of residual dye in a dye-bath was performed using a UV-VIS CARY 50 spectrophotometer (Varian). First, the absorbances of known dye concentrations were measured, the absorption coefficients calculated from the results, and the wavelengths of maximal absorption ( $\lambda_{\text{max}}$ ) were determined for each dye.

Next, the dye residues in various dye-baths were spectrophotometrically determined by measuring the absorbance at a maximal absorption wavelength for each individual dye. Thereafter, the amount of dye residues were calculated according to the Lambert-Beer law (1) [6, 11]:

$$A = k \times l \times c \quad (1)$$

Where:

A – absorbance,

k – absorption coefficients of dye in L/(g·cm),

l – optical length in cm,

c – concentration of dye in dye-bath in mL/L.

y % Bezaktiv V  
 5 g/L  $\text{Na}_2\text{CO}_3$   
 z mL/L NaOH, w(NaOH) = 32,5 %  
 $T = 40 \text{ }^{\circ}\text{C}$   
 $t = 100 \text{ min}$   
 kopelno razmerje (KR): 1 : 20  
 masa vzorca: 8g

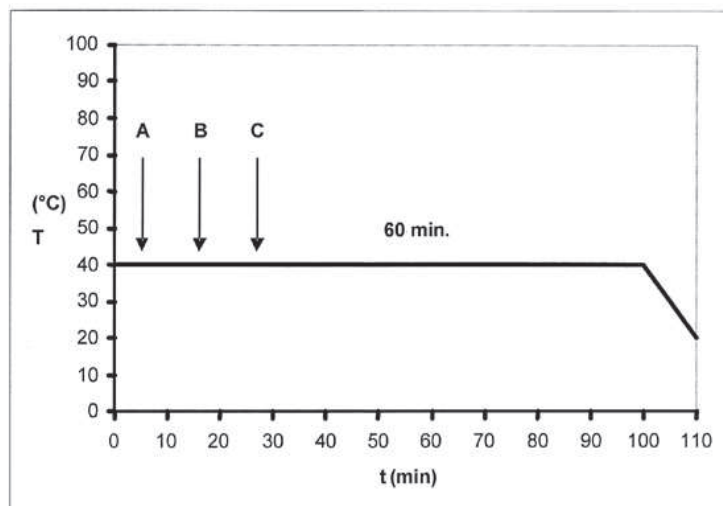


Figure 3: Dyeing diagram for Bezaktiv V dyestuffs

#### Postopek barvanja z impregnacijo (pad-batch)

Impregniranje vzorcev z barvalno kopeljo je potekalo na laboratorijskem fularju (Mathis), z dodanimi barvili, kemikalijami in tekstilnimi pomožnimi sredstvi ter pri pogojih, ki so podani v nadaljevanju.

Receptura in pogoji barvanja za barvila Bezaktiv S:

x g/L Bezaktiv S  
 80 g/L sečnina  
 4 g/L SUBITOL RNC  
 50 mL/L vodno steklo  
 19 mL/L NaOH, w(NaOH) = 32,5 %  
 $T_{\text{impregnacije}} = 20 - 25 \text{ }^{\circ}\text{C}$   
 ožemalni učinek (OU) = 100 %  
 $T_{\text{fiksiranja}} = \text{sobna}$   
 $t_{\text{fiksiranja}} = 18 \text{ ur}$

Receptura in pogoji barvanja za barvila Bezaktiv V:

x g/L Bezaktiv V  
 100 g/L sečnina  
 4 g/L SUBITOL RNC  
 50 mL/L vodno steklo  
 0,46 mL/L NaOH, w(NaOH) = 32,5 %  
 $T_{\text{impregnacije}} = 20 - 25 \text{ }^{\circ}\text{C}$   
 ožemalni učinek (OU) = 100 %  
 $T_{\text{fiksiranja}} = \text{sobna}$   
 $t_{\text{fiksiranja}} = 9 \text{ ur}$

## 2.5 Determining the amount of unfixed dye

Unfixed and hydrolysed reactive dye was extracted from the dyed and soaped samples by a combined solution of 0.02 mol/L of sodium hydroxide and 0.06 mol/L of sodium dihydrogenphosphate. The extraction was accomplished in boiling temperatures up to the point of colourless extract. Baths from the individual cycles were collected in a volumetric flask with a known volume. The absorbances were then measured and the concentration of the dye in the dye-bath was calculated according to the Lambert-Beer law and the preliminarily determined absorption coefficient. The amount of dyestuffs on the fibres and in dye-bath effluents was established, with regard to the obtained results.

## 2.6 The assessment of colour and colour differences

Dyed fabrics were colourimetrically evaluated in a CIELAB colour space by means of a system for colour measuring in a spectral area of 400–700 nm wavelengths using a two-ray SF 600+ spectrophotometer (Datacolor) with an Ulbricht sphere and measuring geometry of  $d/8^\circ$ . The source of light was a halogen lamp with xenon lightning that gives standardized daylight D65. All samples in the two layers were measured three times. On the basis of the obtained results, total colour differences ( $\Delta E^*$ ) (2), lightness differences ( $\Delta L^*$ ), and the samples positioned at  $a^*$  (red/green) and  $b^*$  (yellow/blue) of colour space axes were calculated according to the equation (2) [11].

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \quad (2)$$

$\Delta a^*$  – differences at red/green axis

$$(\Delta a^* = a^* \text{ sample} - a^* \text{ standard})$$

$\Delta a^* > 0$  means more red or less green

$\Delta a^* < 0$  means more green or less red

$\Delta b^*$  – differences at yellow/blue axis

$$(\Delta b^* = b^* \text{ sample} - b^* \text{ standard})$$

$\Delta b^* > 0$  means more yellow or less blue

$\Delta b^* < 0$  means more blue or less yellow

$\Delta L^*$  – lightness differences

$$(\Delta L^* = L^* \text{ sample} - L^* \text{ standard})$$

$\Delta L^* > 0$  means lighter

$\Delta L^* < 0$  means darker

Po barvanju z obema skupinama barvil smo vzorce samo oželi in jih posušili. Posušene vzorce smo najprej milili in nato še ekstrahirali. Miljenje je potekalo pri temperaturi vrenja s sredstvom COT-OBLANC NSR (0,5 g/L) v času 20 minut.

MEROPAN DPE (Bezema) je zaščitni koloid s sekvestrirnimi lastnostmi za Ca- in Mg-ione in dispergirnimi lastnostmi za vodotopne primesi na lanu. SARABID LDR (Bezema) je egalizirno sredstvo za barvanje celuloze z reaktivnimi barvili. SUBITOL RNC (Bezema) je omakalno in pralno sredstvo, ki se ne peni. CO-TOBLANC NSR (Bezema) je sredstvo za miljenje po reaktivnem barvanju, pri čemer se z vlaken odstrani nevezano in hidrolizirano barvilo.

## 2.4 Določanje ostanka barvila v barvalni kopeli

Določevanje ostanka barvila v barvalni kopeli smo izvajali na dvožarkovnem spektrofotometru UV-VIS Cary 50 podjetja Varian. Najprej smo opravili meritve absorbance znanih koncentracij barvila in izračunali absorpcijske koeficiente ter določili valovno dolžino maksimalne absorpcije ( $\lambda_{\max}$ ) za posamezno barvilo. Ostanek barvila v različnih kopelih smo določali spektrofotometrično, tj. z merjenjem absorbance pri valovni dolžini maksimalne absorpcije posameznega barvila. Nato smo s pomočjo Lambert-Beerovega zakona (1) izračunali koncentracije ostankov barvil [6, 11].

$$A = k \times l \times c \quad (1)$$

A – absorbanca,

k – absorpcijski koeficient posameznega barvila v L/(g·cm),

l – optična pot v cm,

c – koncentracija barvila v kopeli v mL/L.

## 2.5 Določevanje količine nevezanega barvila

Nevezano in hidrolizirano reaktivno barvilo smo iz pobarvanih in miljenih vzorcev ekstrahirali z raztopino, sestavljeno iz 0,02 mol/L natrijevega hidroksida in 0,06 mol/L natrijevega dihidrogenfosfata. Ekstrakcija je potekala pri temperaturi vrenja do brezbarvnosti ekstrakta. Kopeli posameznih ciklusov smo zbirali v merilno bučko znanega volumna. Nato smo izmerili absorbanco tej kopeli in na podlagi znanega absorpcijskega koeficienta za posamezno barvilo po Lambert-Beerovem zakonu izračunali koncentracijo barvila v kopeli. Iz dobljenih podatkov pa smo določili količino barvila na vlaknu in v odpadnih kopelih.

## 2.6 Vrednotenje barv in barvnih razlik

Obarvane tkanine smo vrednotili v barvnem prostoru CIELAB s pomočjo sistema za merjenje barv v spektralnem območju z valovno dolžino 400–700 nm na spektrofotometru SF 600+ (proizvajalca Datacolor), ki je dvožarkovni spektrofotometer z Ulbrichtovo kroglo in geometrijo merjenja  $d/8^\circ$ . Kot vir svetlobe smo uporabili halogensko žarnico in ksenonsko bliskavico, ki v kombinaciji s filtrom

*Fabric dyed according to the padding procedure was chosen as the standard, and fabric dyed by the exhaustion technique was considered to be the sample.*

### 3 Results and discussion

*Dyeing was performed according to the exhaust and padding procedure. Figure 4 shows the amount of residual dye in the dye-bath effluents after dyeing with regard to the absorbance measured at a wavelength of individual dye absorption maximum, and to the differences between the residual amounts of two chemically different groups of reactive dyes, Bezaktiv S and Bezaktiv V. The dyestuffs' residuals in the dye-baths were significantly distinguished, with regard to the type of dye and hue, and were higher for Bezaktiv S dyestuffs, on average. From these results, it is evident that the chemical structure of applied dye had a notable influence on dye residual in a dye-bath. Hence, dye residue was maximal at RU S dye, followed by RU V dye, which remained greater than 25% of a bi-functional or 22% of a vinylsulphone dye at a 0.1% concentration. The higher the concentration, the lower the percent of dye residual in the dye-baths, although this does not necessarily correspond to a lower amount of residual. The lowest amount of dye (~ 5% at all concentrations) remained after dyeing with RD V dye, while values for RD S dye lay between 7 and 10%. The MO V dye residual in the dye-bath was also a little lower, and for other dyes, lay between 10 and 14%.*

*Dye residual in a dye-bath effluent is a significant indicator of successful dyeing, although it is insufficient to clearly state the amount of fixed dye on the fibre. Therefore, the measurement of dye residual after soaping was performed, and the extraction was performed eliminating the rest of the unfixed and hydrolysed dye from the fibres. Only then can the final evaluation of dye amount fixed on the fibre and the dye residual in dye-bath effluents be given.*

*Figure 5 represents the analysis of Bezaktiv S dyestuff. We can see that the amount of dye fixed on the fibre does not show the same results as those obtained by analysis of dye residuals in dye-baths after dyeing. The differences between*

dajeta standardizirano dnevno svetlobo D65. Meritve so potekale z izključenim leskom. Za vsak obarvan vzorec smo izvedli tri meritve, pri čemer je bila tkanina v dveh plasteh. Na podlagi meritev smo izračunali celotno barvno razliko ( $\Delta E^*$ ) (2) in razlike v svetlosti ( $\Delta L^*$ ) ter položaj vzorcev na  $a^*$  (rdeče-zeleni) in  $b^*$  (rumeno-modri) osi v barvnem prostoru po naslednjih zvezah [11]:

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \quad (2)$$

pri čemer je:

$\Delta a^*$  – razlika na rdeče-zeleni osi ( $\Delta a^* = a^*$  vzorca –  $a^*$  standarda),  $\Delta a^* > 0$  pomeni bolj rdeče oz. manj zeleno,  $\Delta a^* < 0$  pomeni bolj zeleno oz. manj rdeče;

$\Delta b^*$  – razlika na rumeno-modri osi ( $\Delta b^* = b^*$  vzorca –  $b^*$  standarda),  $\Delta b^* > 0$  pomeni bolj rumeno oz. manj modro,  $\Delta b^* < 0$  pomeni bolj modro oz. manj rumeno;

$\Delta L^*$  – razlike v svetlosti ( $\Delta L^* = L^*$  vzorca –  $L^*$  standarda),  $\Delta L^* > 0$  pomeni svetleje,  $\Delta L^* < 0$  pomeni temneje.

Pri vrednotenju barvnih razlik smo kot standard izbrali tkanino, pobarvano po impregnacijskem postopku z reaktivnimi barvili, za vzorec pa tkanino, pobarvano prav tako z reaktivnimi barvili po izčrpalnem postopku.

### 3 Rezultati z razpravo

Proces barvanja je potekal po postopku izčrpanja in po postopku impregnacije. Na sliki 4 so na podlagi merjenja absorbance pri valovni dolžini maksimalne absorpcije posameznega barvila prikazane količine ostanka barvila v kopeli po postopku barvanja in posamezne razlike med količino ostanka dveh kemijsko različnih skupin reaktivnih barvil Bezaktiv S in Bezaktiv V. Ostanke barvil v kopeli se glede na vrsto in barvni ton barvil med seboj pomembno razlikujejo in so v povprečju višji pri barvilih Bezaktiv S. Iz rezultatov je razvidno, da na ostanek barvila v kopeli močno vpliva struktura posameznega barvila, saj so ostanke najvišji pri RU S-barvilu, sledi mu RU V-barvilo, ki ga pri koncentraciji 0,1 % ostane preko 25 % pri bifunkcionalnem oziroma 22 % pri vinylsulfonskem barvilu. Ostanek barvila v barvalnih kopelih se z višanjem koncentracije v odstotkih sicer znižuje, kar pa seveda ne pomeni količinsko nižjih ostankov. Najmanj barvila ostane po procesu barvanja v kopeli z RD V-barvilom, pri katerem se vrednosti ostankov pri vseh koncentracijah gibljejo okrog 5 %, medtem ko so te vrednosti za RD S-barvilo med 7 in 10 %. Tudi pri modrem barvilu so ostanke nekoliko nižji pri MO V-barvilu, sicer pa so ostanke barvil v kopeli med 10 in 14 %.

Ostanek barvila v kopeli predstavlja pomemben kazalnik uspešnosti barvanja, vendar ne zadošča za definiranje količine vezanega barvila na vlaknu. Zato smo opravili meritve ostanka barvila tudi v kopeli po procesu miljenja in naknadno izvedli še ekstrakcijo, s katero smo odstranili preostanek nevezanega in hidroliziranega bar-



diverse dyes are low RD S dyestuff, the dye residual in a dye-bath effluent is the lowest, the portion of eliminated dye after soaping and extraction is the highest, and the total amount of fixed dye on the fibres is the lowest – under 70% at a 0.1% concentration. In contrast, the residual of RU S dyestuff in a dye-bath effluent is the highest, and the amount of dye removed during soaping and extraction is minimal. The total amount of fixed dye on the fibres is almost the same as for the red dye. The same statement is also valid for blue MO S dyestuff with intermediate results.

Figure 6 shows the results for Bezaktiv V dyestuffs with the similar conclusions mentioned above for Bezaktiv S dyestuffs. Moreover, the amount of fixed dye on the fibres is not in relation to the amount of dye residual in the dye-baths after dyeing, especially considering that the amount of eliminated dyes after soaping and extraction must also be taken into consideration. Generally, the amount of fixed dye is slightly higher, but still within 2%.

The presented research has established the impact of dyeing procedures on the amount of fixed and/or hydrolysed dye. Therefore, the same fabric was dyed with identical dyestuffs by the pad-batch procedure. Concurrently, the residual dye after soaping and extraction was monitored, and superior differences were noticed in different dyes depending on the dyeing procedure. It is obvious from Figure 7 that major differences originated at RU S dyestuff with the highest dye residual in the dye-bath effluent after exhaust dyeing, indicating its superior applicability for padding procedures due to its sensitivity and hydrolysis in diluted dye-baths. However, the hydrolysis is slower and proceeds to a lower extent in a more concentrated impregnation of dye-baths. Differences in the amount of unfixed dye as a consequence of dyeing procedure were also observed at MO S and at RD S dyestuffs, but only to a minor extent (within two percent).

The pad-batch process using Bezaktiv V dyestuffs had similar results to those mentioned above (Figure 8). Better results were achieved during padding, especially for the RU V dye, followed by the MO V dye. Meanwhile, the fixation of the RD V dye was lower.

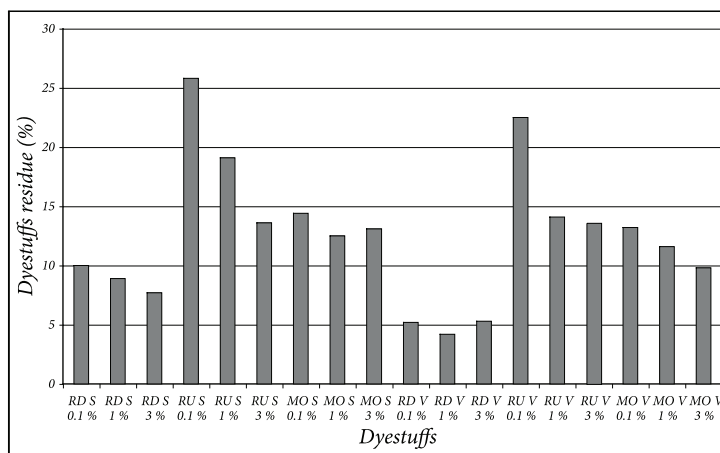


Figure 4: Dyestuff residue in dye-bath after exhaust dyeing using Bezaktiv S and Bezaktiv V dyestuffs

vila z vlaken. Šele na ta način lahko podamo končno oceno o količini vezanega barvila in količini barvila, ki je ostalo v kopeli in na vlaknu kot ostanek ali v hidrolizirani obliki.

Slika 5 prikazuje analizo reaktivnega barvila Bezaktiv S; opazimo lahko, da količine barvila, vezanega na vlakno, ne kažejo enakih rezultatov, kot jih lahko dobimo iz analize ostankov barvil v kopeli po barvanju. Razlike med posameznimi barvili so majhne, saj opazimo pri RD S-barvilu, pri katerem je ostanek barvila v kopeli najnižji, najvišji delež barvila, odstranjenega s postopkom miljenja in pri ekstrakciji, kar nam daje skupno najnižjo količino barvila, vezanega na vlakna, ki je pri koncentraciji 0,1 % pod 70 %. Obratno pa je pri RU S-barvilu, pri katerem je bil ostanek barvila v kopeli najvišji. Pri tem barvilu opazimo, da je količina barvila, ki ga odstranimo s postopkom miljenja in ekstrakcijo, minimalna. Skupno pa se količina barvila, vezanega na vlakna, bistveno ne razlikuje od rdečega barvila. Te ugotovitve prav tako veljajo za MO S-barvilo, ki tudi pri tej analizi kaže vmesne rezultate.

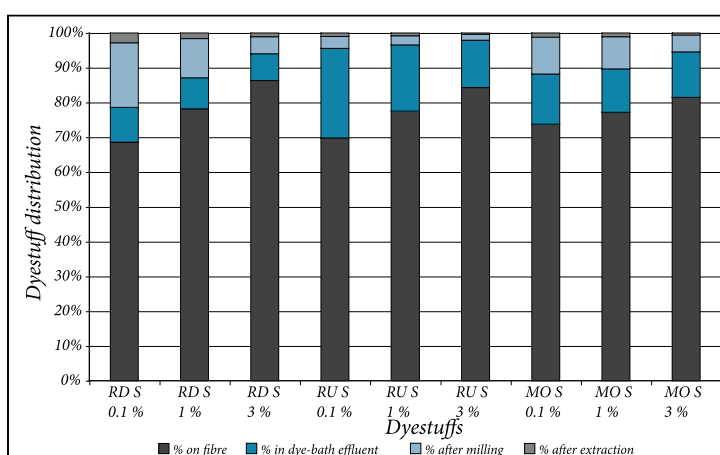


Figure 5: Amount of dye on the fibre, in dye-bath effluent, after milling, and after extraction at exhaust dyeing using Bezaktiv S dyestuffs

Dyed samples were also colourimetrically evaluated. The differences between flax fabrics dyed with exhaust dyeing and the padding process were determined. The resulting data are very important, as different technology was employed, as well as differences in the transfer of the dyeing recipes between individual procedures. The results also served to establish the effect of the amount of fixed dyes on the colour.

Table 3 summarizes colour differences for Bezaktiv S dyestuffs with regard to the dyeing procedure. Fabric dyed according to the padding process was used as a standard and compared with the exhaust dyed samples. Measurements were accomplished on the samples after dyeing and soaping. It is obvious from the results that the colour differences are above the tolerated limit value ( $\Delta E^* = 1$ ), although the samples were lighter after soaping, as expected. When the dye concentration was increased, the displayed dyeing differences also increased. However, the influence of the dye concentration on the total colour difference is insignificant. The results of colour differences, especially the lightness differences, i.e. positive differences in lightness or lighter samples after padding, are in agreement with the measurements of dye residuals in the dye-baths.

Similar results to those discussed above were obtained when samples dyed with Bezaktiv V dyestuffs were measured (Table 4). Total colour differences were slightly higher, implying a greater sensitivity of mono-reactive dyes to the dyeing procedures. Enormous differences could be noticed using the RD V dyestuff. The major sensitivities of these dyes led to a greater difference at the  $a^*$  and  $b^*$  axes, affecting the position of a colour sample in a colour space.

#### 4 Conclusions

Dyeing of flax fabric with reactive dyes represents an important dyeing procedure often employed in industrial production. Here, we wanted to review the expertise of chemically different reactive dyes' behaviour with various dyeing procedures by considering specific parameters for the final evaluation of the effectiveness of individual processes.

Slika 6 nam prikazuje rezultate za barvila Bezaktiv V, za katera so ugotovitve podobne kot pri barvilu Bezaktiv S. Tudi v tem primeru količina barvila, vezanega na vlaknih, ni odvisna od količine ostanke barvila v kopeli po procesu barvanja, saj je treba upoštevati tudi količine barvil, odstranjenih pri miljenju in ekstrakciji. Na splošno pa lahko opazimo, da je količina vezanega barvila nekoliko višja, vendar so razlike minimalne in znotraj dveh odstotkov.

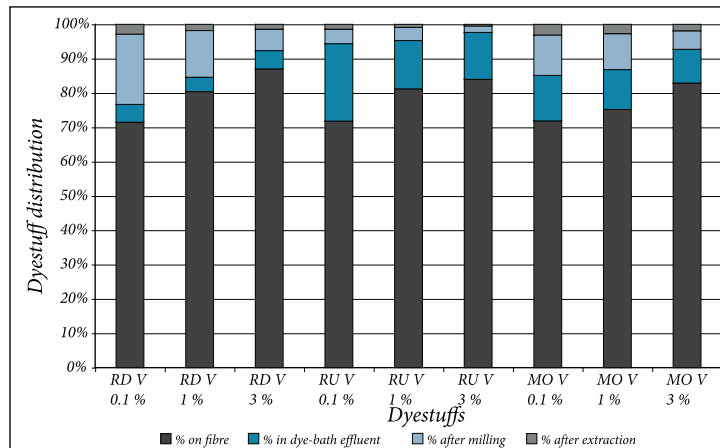


Figure 6: Amount of dye on the fibre, in dye-bath effluent, after milling, and after extraction at exhaust dyeing using Bezaktiv V dyestuffs

Z raziskavo smo želeli ugotoviti, v kolikšni meri je količina vezanega in/ali hidroliziranega barvila odvisna od postopka barvanja. Zato smo z enakimi barvili obarvali enako tkanino še po impregnacijskem postopku. Pri tem smo spremljali ostanke barvil po miljenju in ekstrakciji ter pri določenih barvilih opazili pomembne razlike, ki so povezane s postopkom barvanja. S slike 7 je razvidno, da nastajajo največje razlike pri RU S-barvilu, ki ima pri postopku barvanja z izčrpavanjem najvišje ostanke barvila v kopeli, iz česar lahko ugotovimo, da je to barvilo primerno predvsem za postopke barvanja z impregnacijo, saj je zelo občutljivo in hitro hidrolizira v razredčeni barvalni kopeli, medtem ko v bolj koncentriranih impregnacijskih kopelih hidroliza poteka počasneje in v manjši meri. Razlike v količini nevezanega barvila, ki so posledica postopka barvanja, opazimo tudi pri MO S-barvilu in v manjši meri tudi pri RD S-barvilu, vendar so pri slednjem razlike znotraj dveh odstotkov.

Podobne ugotovitve lahko podamo tudi pri barvanju z barvili Bezaktiv V po impregnacijskem postopku (slika 8). Boljše rezultate dosežemo s postopkom impregnacije zlasti pri RU V-barvilu, sledi MO V-barvilo, medtem ko je izkoristek RD V-barvila pri tem postopku barvanja slabši.

Obarvane vzorce smo vrednotili tudi s pomočjo sistema za barvno metriko. Rezultate smo uporabili zlasti za določanje razlik v obarvanosti lanene tkanine, pobarvane po postopku izčrpavanja ali po postopku impregnacije. To je zlasti pomembno s stališča uporabe posamezne tehnologije in prenosa receptur barvanja med posameznimi tehnologijami.

The obtained results of dye residual measurements in a dye-bath effluent after exhaust dyeing were inaccurate, due to the the diverse dyes' chemical structures and reactive groups, which have different sensitivities to hydrolysis, i.e. the dyes could be found in dye-baths or on the fibres in the hydrolysed form. Typical examples of such dyes applied in the present study are both red dyes with high exhaustion rates (between 90% and 95.8%, depending on the initial dye-bath concentration), followed by determination of a high amount of unfixd dye (between 6% and 23.3%) after soaping and extraction and, therefore, the lowest dyeing rate. In contrast, the results gained by both yellow dyes, which have the lowest exhaustion rate after dyeing (between 86.4% and 74.2%) and a minor dye residual after soaping and extraction (between 2.1% and 5.5%) showed a better dyeing rate in comparison to the red dyes.

Variable dye sensitivity to hydrolysis is revealed when comparing different procedures and dyeing conditions. Thus, both yellow dyes are particularly sensitive to the diluted dye-baths, and the dyeing rate is elevated during the padding process (between 91 and 95.1%, depending on concentration), i.e. the amount of hydrolysed dye is reduced. Better results during padding are also obtained when dyeing with both groups of blue dyes, while the results obtained for padding using red dyes are similar to those for exhaustion.

On the basis of the extended results, we conclude that the choice of dyeing procedure is significant, as the selection of an appropriate dyeing process controls the environmental pollution of dye residual in dye-bath effluents, and economically increases production efficiency.

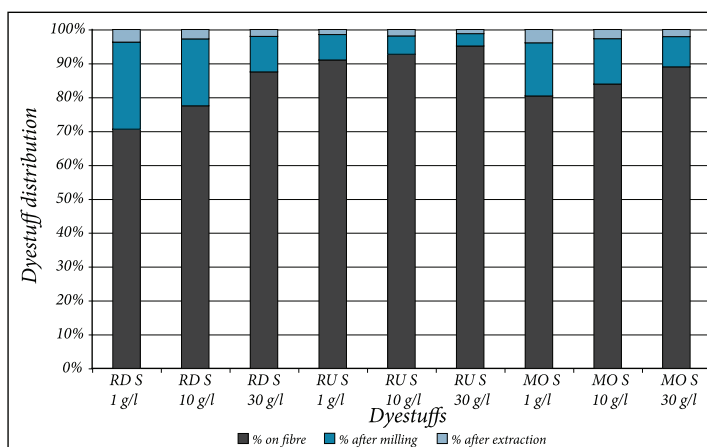


Figure 7: Amount of dye on the fibre, after milling, and after extraction at padding dyeing using Bezaktiv S dyestuffs

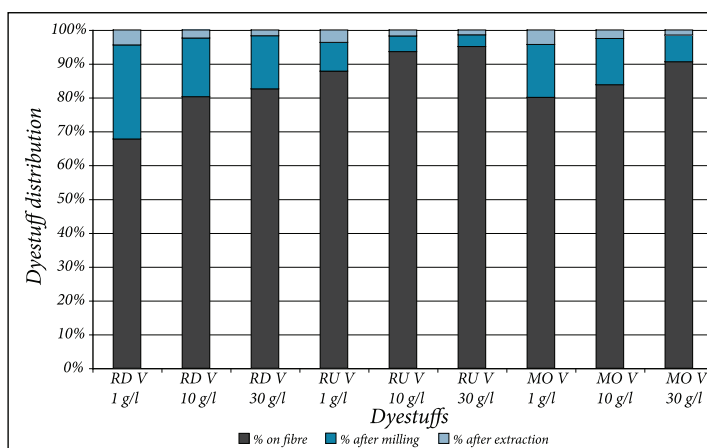


Figure 8: Amount of dye on the fibre, after milling, and after extraction at padding dyeing using Bezaktiv V dyestuffs

meznimi postopki. Istočasno smo s temi meritvami želeli ugotoviti tudi vpliv količine vezanih barvil na barvo.

Preglednica 3 nam podaja barvne razlike za barvila Bezaktiv S v odvisnosti od postopka barvanja. Pri tem smo uporabili tkanino, obarvano po postopku impregnacije, kot standard, s katerim smo primerjali vzorce, obarvane po postopku izčrpanja. Za primerjavo smo izvedli meritve na vzorcih po barvanju in po miljenju. Iz rezultatov vidimo, da so barvne razlike sicer nad dopustno mejo odstopanja ( $\Delta E^* = 1$ ), vendar se po postopku miljenja zmanjšajo, vzorci pa so po miljenju, kot smo pričakovali, tudi svetlejši. Barvne razlike se z višanjem koncentracije sicer povečujejo, vendar je vpliv koncentracije na celotno barvno razliko majhen. Rezultati barvnih razlik, predvsem razlike v svetlosti, nam potrjujejo tudi meritve ostankov barvil v barvalnih kopelih, saj so razlike v svetlosti pozitivne, kar pomeni, da so vzorci, obarvani po postopku izčrpanja, svetlejši.

Table 3: Colour differences of samples dyed with Bezaktiv S dyestuffs (standard-padding process)

Dyestuff	$\Delta E^*$		$\Delta L^*$		$\Delta a^*$		$\Delta b^*$	
	B	M	B	M	B	M	B	M
RD S 0.1%	3.146	2.683	1.041	1.219	2.356	1.845	1.806	1.604
RD S 1%	3.229	2.837	0.617	0.938	2.286	1.649	2.521	1.983
RD S 3%	3.447	2.936	0.548	0.728	1.115	0.942	2.642	1.996
RU S 0.1%	2.407	1.206	2.095	1.749	0.709	0.592	0.372	0.147
RU S 1%	2.387	1.739	2.216	1.943	0.716	0.618	0.523	0.389
RU S 3%	2.243	1.964	2.175	2.179	0.833	0.728	0.610	0.417
MO S 0.1%	3.147	2.674	1.487	1.168	-0.705	-0.803	2.434	1.849
MO S 1%	3.223	2.854	1.590	1.193	-0.436	-0.662	2.418	1.632
MO S 3%	3.303	2.927	1.620	1.279	-0.178	-0.390	2.736	1.481

B-after dyeing, M-after milling

Table 4: Colour differences of samples dyed with Bezaktiv V dyestuffs (standard-padding process)

Dyestuff	$\Delta E^*$		$\Delta L^*$		$\Delta a^*$		$\Delta b^*$	
	B	M	B	M	B	M	B	M
RD V 0.1%	6.147	4.839	3.294	2.693	6.457	4.593	1.046	0.923
RD V 1%	6.311	5.182	4.170	3.593	6.789	4.927	2.250	1.592
RD V 3%	6.548	6.184	4.398	3.931	5.879	5.183	2.516	1.834
RU V 0.1%	3.501	2.783	0.437	0.284	3.988	3.183	5.099	3.842
RU V 1%	3.897	2.845	0.625	0.472	4.258	3.583	5.608	4.183
RU V 3%	3.961	3.173	0.503	0.492	4.988	3.937	5.940	4.923
MO V 0.1%	3.235	2.743	0.287	0.480	2.276	2.194	2.934	2.173
MO V 1%	3.289	2.846	0.217	0.328	2.360	2.281	2.657	2.283
MO V 3%	3.345	2.958	0.022	0.173	2.457	2.318	2.567	2.381

B-after dyeing, M-after milling

Podobne rezultate opažamo tudi pri meritvah barvnih vrednosti vzorcev, obarvanih z barvili Bezaktiv V (preglednica 4). Celotne barvne razlike so sicer nekoliko večje, kar kaže na to, da so mono-reaktivna barvila bolj občutljiva za postopek barvanja. Velike razlike nastajajo zlasti pri RD V-barvilu. Večjo občutljivost teh barvil za postopek barvanja kažejo tudi večje razlike na barvnih oseh  $a^*$  in  $b^*$ , kar pomeni, da postopek vpliva tudi na položaj obarvanega vzorca v barvnem prostoru.

## 4 Sklepi

Barvanje lanene tkanine z reaktivnimi barvili predstavlja pomemben barvalni proces, ki se pogosto uporablja v proizvodni

praksi. V tem prispevku smo želeli prikazati pomen poznavanja obnašanja kemijsko različnih reaktivnih barvil pri različnih procesih barvanja in opozoriti na to, katere parametre moramo upoštevati, da lahko podamo končno oceno uspešnosti posameznega postopka.

Iz rezultatov barvanja po postopku izčrpavanja izhajajo, da nam ostanki barvil v kopeli po končanem postopku barvanja dajejo napačne predstave, saj so barvila glede na kemijsko konstitucijo in vrsto reaktivne skupine različno občutljiva za hidrolizo ter lahko ostanejo v kopeli ali pa ostaja pomemben delež barvil na samih vlaknih v hidrolizirani obliki. Tipičen primer takšnih barvil sta v raziskavi uporabljeni rdeči barvili, ki kažeta najvišji delež izčrpanja (med 90 % in 95,8 %, odvisno od začetne koncentracije v barvalni kopeli), česar z nadaljnjo analizo ne potrdimo, saj se s postopkom miljenja in ekstrakcije dodatno odstrani visok delež nevezanega barvila (med 6,0 % in 23,3 %) in tako imata ti dve barvili skupno najnižji izkoristek. Ravno nasprotni pa so rezultati pri obeh rumenih barvilih, ki kažeta najnižji delež izčrpanja barvila po končanem postopku barvanja (med 86,4 % in 74,2 %), vendar pri miljenju in ekstrakciji dodatno odstranimo zelo nizke deleže (med 2,1 % in 5,5 %), tako da ti dve barvili skupno kažeta celo nekoliko boljše rezultate izkoristka barvila.

Različna občutljivost barvil za hidrolizo se pokaže pri primerjavi različnih postopkov in pogojev barvanja. Tako sta rumeni barvili zlasti občutljivi za razredčene barvalne kopeli, medtem ko pri postopku impregnacije omogočata visok izkoristek (med 91 % in 95,1 %, odvisno od koncentracije) oziroma nizek delež hidroliziranega barvila. Boljše rezultate pri postopku barvanja z impregnacijo dobimo tudi pri obeh vrstah modrega barvila, medtem ko nam daje rdeče barvilo podobne rezultate kot pri postopku izčrpavanja.

Na podlagi podanih rezultatov ugotavljamo, da je izbira postopka barvanja pomembna, saj lahko s premišljeno izbiro obvladujemo tudi onesnaženje okolja z ostanki barvil v odpadnih kopelih in s tem tudi povečujemo ekonomsko učinkovitost proizvodnje.

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