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ECONOMICAL DYEING OF COTTON FIBRE

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Název tématu:

Barvení bavlněných tkanin

za snížených teplot

Zásady pro vypracování:

- Experimentálně sledujte vliv snižování teplot barvení na egálnost a stálosti vybarvení substantivními barvivy.
- 2. Snížené vytažení lázně při nižších teplotách se pokuste eliminovat zvýšenou dosací neutrálního elektrolytu.
- 3. Případné zhoršení egálnosti se pokuste zamezit přísadami vhodných TPP případně i jejich kombinacemi.

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I. INTRODUCTION

Textile finishing is one of the final improvements of textile to give them better quality and behaviour. One of the finishing technologies is dyeing of textile fibres.

Substantive dyes are relatively very easy to apply and, within the scape of their use, give generally good wet fastness which can be promotted by aftertreating the dyed material either with a cationic agent Syntefix pwd. or with its combination with copper sulphate. Apart from that another advantage of saturn dyes is their good light fastness.

The aim of this diploma work is to obtain information on how to get the best hue and saturation of the colour by decreasing the dyeing temperature and the dyeing time.

Another task is to test the colour fastness.

A respective dyeing temperature, dyeing time and some combinations of dye auxiliaries, which are the best to get a good hue and dark shade are indicated in the conclusion.

II. THEORETICAL PART

2.1. THE CELLULOSIC FIBRES

Textile fibres composed of pure cellulose are :

- a) Natural cellulosic fibres: abaca, coir, cotton, flax, jute, kenaf, sisal, etc.
- b) Man-made cellulosic fibres : cuprammonium, polynosic and viscose
- Of the very cellulosic textile fibres, only the commonly used one cotton will be considered here.

2.1.1. Cotton

2.1.1.1. Textile clasification

The word cotton is derived from Arabic. Depending upon the arabian dialect, it is pronounced "kutan", "gutn", "gutun", etc. As the cotton fibre is obtained from a plant it is classified as a natural, cellulose, seed, mono-cellular, staple fibre. The density of fibre is $1.52 \, \mathrm{g/cm^3}$, which makes cotton a rather heavy fibre.

2.1.1.2. Fibre morphology

2.1.1.2.1. The macro-structure of cotton

Under a microscope a cotton fibre appears as a very fine, regular fibre. It ranges in length from about 10 mm to 65 mm, depending upon the quality of a fibre.

Cotton fibres are amongest the finest in common use.

They range in fibre diometer from about 11 μm to 22 μm . Such very fine fibres permit the manufacture of fine, light-weight cotton fabrics and garments, despite the fact that it is a dense fibre.

2.1.1.2.2. The microscopic appearance of cotton

Under the microscope, the cotton fibre looks like a twisted ribbon or a collapsed and twisted tube. These twists or convolutions identify the cotton fibre under the microscope.

The convolutions and kidnely-shaped cross-section of the cotton fibre enable it to make only random contact with the skin. This type of contact is more compatible with human skin psychology and therefore more confortable. The countless minute air spaces which exist because of the convolutions and kidnely-shaped cross-section of cotton fibres increase the moisture absorbency of cotton textile materials thus making them more comfortable to wear. It is for these reasons that manufacturers crimp and texture man-made filaments and staple fibres in order to increase fibre moisture absorbency and minimise fibre contact with the skin.

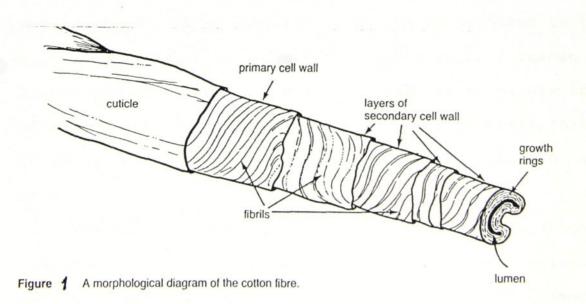
2.1.1.2.3. The micro-structure of the cotton fibre

The cotton fibre is a single plant cell. Its cross-section is oval, compared with the normal hexagonal plant cell. However like all plant cells, cotton has a distinct cuticle, well developed primary and secondary

walls, and lumen (see Fig 1).

The cuticule is the "very outside" or "skin" of the cotton fibre. It is composed of a waxy layer (cotton wax) only a few molecules thick. The waxy nature of the cuticle enables it to adhese tenaciously to the primary wall of the fibre. The inert nature of this cotton wax protects the rest of the fibre against chemical and other degrading agents. Kier boiling and bleaching during cotton finishing removes much of the cuticle or wax.

This enables cotton to absorb moisture more quickly. Subsequent laundering will gradually remove most of the remaining cuticle. As the extent of the cuticle is decreased further, deterioration of the cotton textile material increases.



The primaly cell wall, which is immediately underneath the cuticle, is about 200 nm thick. It is composed of very finethreads of cellulose, called fibrils These fibrils are about 20 nm thick, but their length is as yet unknown. The fibrils spiral at about 70° to the fibre axis. This spiralling imports strength to the primary cell wall and, hence, to the fibre. The primary cell wall can be visualised as a sheath of spiralling fibrils.

wall, which forms the bulk of the fibre. Concentric layers of spiralling, cellulosic fibrils, not unlike the growth rings of trees, make up the secondary wall. Its fibrils are about 10 nm thick, but of undefined length. Near the primary cell wall, the fibrils of the secondary wall spiral at about 20° to 30° to the fibre axis. This spiral angle widens to about 20° to 45° for the fibrillar layers nearer the lumen. Much of the strength and stability of the cotton fibre and, hence, of the yarns and fabrics may be attributed to these spiralling fibrils. Whenever the fibrils change the direction of their spirals, a weak area exists in the secondary wall structure. It is at these weak areas that the convolutions of the fibre also alter the direction of their twist.

The hollow canal, running the length of the fibre, is called the lumen. Its walls are the innermost, concentric layers of spirals of the secondary cell wall. The lumen was once the central vacuole of the growing cotton fibre. It was full of cell sap, which was composed of a dilute, aqueous solution of proteins, sugar, minerals and cell-waste products. When the sap eraporated, its constituents remained

Figure 2 The chemical formula of the cellulose polymer. It is thought that the cotton cellulose polymer is composed of 5000 cellobiose units, while the flax cellulose polymer is thought to contain 18 000 cellobiose units.

behind to contribute the colour of the cotton fibre. Further, as the sap evaporated, the pressure inside the fibre become less than the atmospheric pressure on the outside. This caused the fibre to collapse inward resulting in the characteristic kidnely-shaped cross-section of the cotton fibre.

2.1.1.3. The cotton polymer

The cotton polymer is a linear, cellulose polymer. The repeating unit in the cotton polymer is cellobiose which consists of two glucose unit (see Fig 2). It is not fully understood how cellulose is formed or polymerised by plants. It is therefore better to refer to cellobiose as the repeating unit of the cotton polymer rather than as its monomer.

The cotton polymer consits of about 5000 cellobiose units, that is, its degree of polymerisation is about 5000.

It is a very long, linear polmer, about 5000 nm in length and about 0.8 nm thick.

The most important chemical grouping on cotton polymer are hydroxyl groups or -OH groups. These are also present as methylol groups or -CH2OH. Their polarity gives rise to hydrogen bonds between the -OH groups of adjacent cotton polymers. Van der Waals' forces also occur but compared with the hydrogen bonds, the Van der Waals' forces are the little significance.

Cotton is a crystalline fibre. Its polymer system is about 65 to 70 per cent crysralline and, correspondingly, about 35 to 30 per cent amorphous. Therefore the cotton polymers are, in the main, well oriented and probably no further apart than 0.5 nm, in the crystalline regions. This is the maximum distance across which hydrogen bonds are the dominant and most important forces of attraction present in the polymer system of cotton. For this reason Van der Waals' forces which are also present have little relevance. Because the appearance of the cellulose polymer is not unlike a chain of hexagonal units, the polymer system of cotton can be imagined as a roll of wire nettings. The crystalline are therefore the well ordered lines and rows of hexagonal holes of the wire netting. The amorphous regions are a disarrangement of these orderly lines and rows of hexagons.

2.1.1.4.1. Effect of acids

Cotton fibres are weakened and destroyed by acids. Acidic conditions hydrolyse the cotton polymer at the glucoside oxyden atom, which links the two glucose units to form the cellobise unit (see Fig 2). Mineral or organic acids, being stronger than organic acids, will hydrolyse the cotton polymer more rapidly.

2.1.1.4.2. Effect of alkalis

Cotton fibres are resistant to alkalis and relatively unaffected by normal laundering. The resistance is attributed to the lack of attraction between the cotton polymers and alkalis.

Mercering without tension, or a slack mercerising, causes the cotton fibres to swell, that is, increase in thickness and contract the length. The swelling is thought to be due to alkali molecules or radicals, entering the amorphous region of the fibers' polymer system. In so doing, they force the cotton polymers further apart, which can be seen as swelling of the fibres. This result in somewhat greater inter-polymer spaces, permitting poorly aligned polymers to orients themselves more satisfactorily, as well as forming additional hydrogen bonds. This explains the increase in fibre strength on mercerising.

with mercering under tension, which can only be carried out of cotton yarn or fabric, little swelling or fibre contraction occurs. The fibre emerges with increased tenacity and with a distinct, though subdued lustre. Tensioning the cotton yarn or fabric in the aqueous, alkaline liquor assist the fibre polymers to align themselves further, leading to an increase in hydrogen bond formation. This explains the increase in tenacity achieved during mercerising under tension. Mercerising under tension also causes the fibre surface to become smooth and more regular, enabling it to reflect incident light more evently. This is responsible for the subdued lustre that is associated with tension mercerised cotton textile materials.

Either type of mercerising swells the fibres sufficiently to alter their normal kidney-shaped cross-section to a more circular one. In addition mercerising improves the dye-uptake of the cotton. This means that mercerised cotton fibres dye and print a deeper hue, that is a hue with more chroma, than equivalent unmercerised fibres, when using the same quality of dye.

2.1.1.4.3. Effect of bleaches

The most common bleaches used on cotton textile materials are sodium hypochlorite and sodium perborate.

Sodium hypochlorite (NaOCl) is a yellowish liquid, smelling of chlorine. Sodium perborate (NaBO2H2O2,3H2O) is a white powder, contained in most commonly available domestic laundry detergents. Sodium hypochlorite bleaches cotton

textile materials at prevailing room tempreatures. However, bleaching with sodium perborate is more effective when the laundry solution exceeds 50°C in temperature.

These two bleaches are examples of oxidising bleaches, which in the class of bleaches used most frequently on cotton textile materials. They bleach most effectively in alkaline conditions to which cotton textile materials are resistant.

Oxidising bleaches are so called because they liberate oxygen which does the actual bleaching. The reactions between the oxygen liberated by the bleach and the molecules on the fibre surface responsible for the cotton's discolouration are not known in any detail. In general it is considered that the oxygen forms water-soluble compouds with the molecules of the fibre surface contaminants. These water-soluble products are rinsed off the fibre during bleaching.

Careful bleaching leaves the fibre polymer system largely intact. Careful bleaching will restrict the chemical attack of bleaches to the fibre surface polymers of cotton.

2.1.1.4.4. Effect of sunlight and weather

The ultraviolet rays of sunlight provide photochemical energy while the infrared rays provide heat energy necessary to degrade the cotton polymers in the presence of atmospheric oxygen, moisture and air pollutants.

Atmospheric moisture (humidity) significantly contributes to the breakdown of the polymers on the surface of the cotton fibres through various hydrolytic reactions.

Initially, such degradation, or polymer hydrolysis, is noticed as a slight fibre discolouration. In due course, the reactive nature of the hydrolytic compounds formed accelerates the breakdown of the fibre. The weaking of the fibre and breakdown of the cotton textile material is, of course, a clear indication that polymer system of the fibre has broken down completely.

2.1.1.4.5. Colour-fastness

Cotton is considered to be a relatively easy fibre to dye and print. The classes of dye which may be used to colour cotton are azoic, direct, reactive, sulphur and vat dyes.

The ease with which cotton takes up dyes, and other colouring matter, is due to the polarity of its polymer and polymer system. This polarity will readily attract any polar dye molecules into the polymer system. In fact, dye molecules which can be dispersed in water will ne absorbed by the polymer system of cotton.

However, as with all other textile fibres, the dye molecules can only enter the amorphous region of the polymer system of cotton. The small inter-polymer spaces in the crystalline regions of the polymer system prevent the entry of the relatively large and bulky dye molecules.

2.2 PRE-TREATMENT

Prior to dyeing, the material should be well prepared, which means free from all impurities, especially greases, waxes, spinning oils, auxiliary agents, etc. The remains of auxiliaries used in kiering, washing and desizing must be throughly removed from the material together with the remains of cleaning liquors, since they could impair the process of dyeing.

The well prepared material, the perfectly dissolved dye and adequate process water, i.e., soft water free from mechanical impurities, these are the conditions for a successful application. If soft water is not available, softening agents, either on polyphosphate basis or the Syntron type, must be added to the dye bath. The choice of these agents, manufactured under the trade name "Syntron" in Czechoslovakia, depending on their type and on the dyestuff employed.

2.3. DYEING

The molecules of the organic compounds called dyes are responsible for the colour of dyed textile fibre materials.

In white light, the colour of a textile material depends upon which incident light waves are absorbes and which are reflected from the dye molecules within the polymer system of its fibres.

Why the dye molecules are coloured. Dye molecules are coloured because they are selectively able to absorb and reflect incident light.

Organic molecules become coloured, and thus useful dye molecules, if they contain at least one of each of the radicals called chromophores and auxochromes (see Table 1 and 2 and Fig 3).

In general, the chromophores give the dye molecule its particular colour, while the auxoxhromes identify the hue of the dye molecule's colour, makes the dye molecule more water soluble, and improves the colour-fastness properties of the dyed or printed fibre.

2.3.1.1. Chromophores

The name is derived from the Greek chroma-colour and phore-to bear.

Chromophores are unsaturated organic radicals as shown in Table 1 and Fig 3.

Their specific state of unsaturation enables them to absorb and reflect incident electromagnetic radiation within the very narrow band of visible light. More specifically: it is the loosely held electrons in the unsaturated bonds of the chromophores which cause in the adsorption of certain incident light waves.

Table 1 Chromophores, the radicals or chemical groups without which dye molecules would have no colour.

Name of chromophore	Basic formula of chromophore	How the chromophore exists in the dye molecule
Azo group	-N = N-	Azo-benzene radical N=N
Quinonoid group	H H	Anthraquinone radical
C	C = C $C = C$ $C = C$ $C = C$	
Tri-aryl methane group	=c(
Nitro group	-N_O	-NO ₂
Nitroso group	-N = O	-NO

Table 2 Auxochromes, the radicals or chemical groups which intensify the hues of the chromophores in dye molecules.

Name of auxochrome	Basic formula of auxochrome	How the auxochrome exists in the dye molecule
Acidic auxochromes	—СООН	Sodium carboxylate radical, —
Carboxyl group Sulphonic group	—SO₃H or	COONa Sodium sulphonate radical,
Sulphome group	O	O
	Ĭ	Ĭ
	-S-OH	$-\overset{\parallel}{\mathbb{S}} - \text{ONA or } -\text{SO}_3^- \text{Na}^+$
	Ö	Ö
Basic auxochromes		ч
Amino group	$-NH_2$	N
Substituted amino group;		Н
for example,	Н	Н
	-N	_N
methyl amino	CH ₃	CH ₃
or	or	or
dimethyl amino	CH ₃	CH ₃
	-N	-N
	CH ₃	CH ₃
Hydroxyl group	-OH	—ОН

auxochromes: hydroxy group amino group, H₂N OH benzene azo group NaO₃S SO3Na+ a chromophore sodium sulphonate an auxochrome amine group, an auxochrome b benzene ring 0 02N NH₂ nitro group, a chromophore quinoid group, a chromophore CH₃ - an auxochrome a chromophore -O3S -OCH₂CH₃ an auxochrome N-H-- an auxochrome

Figure 3 Structural formulae of various dye molecules. The shading indicates chromophores (dark) and auxochromes (light).

a C.I. Acid Blue 86, 44075 — an acid dye

b C.I. Disperse Violet 8, 62030 — a disperse dye
c C.I. Acid Violet 15, 43525 — an acid dye.

Note: The five-digit number for each dye is its Colour Index number.

So3Na+

an auxochrome

2.3.1.2. Auxochromes

The name is derived from the Greek auxien-to increase and chroma-colour.

Auxochromes are organic radicals as shown in Table 2 and Fig 3. Auxochroms fulfil the following important functions.

1. Their presence influences the orbits of the loosely held electrons of the unsaturated bonds of the chromophores. This causes these electrons to absorb and reflect incident light energy of specific wavelenghts only. This also intersifies and deepens the hue of the dye molecules colour.

A dye molecule without auxochromes would lack intensity of colour. This is because the incident light waves would not be absorbed and reflected as selectively as occurs in the presence of auxochromes.

- 2. Auxochromes tend to be polar. This increases the overall polarity of the dye molecule and makes it more readily suluble in water. This allows easier preparation of dye liquores, assists in more even dye uptake by the fibre, and results in more level dyeing.
- 3. The polarity of auxochromes enables the formation of forces of attraction between the dye molecule and the fibre. This improves the colour fastness properties of the dyed fibre.

As a result of the physical interaction between chromophores and auxochromes, textile dyes have some of the highest colour intensities of all colourants in common use. Dyeing is the process of colouring textile materials by immersing them in an aqueous solution of dye, called dye liquor. Normally the dye liquor consists of dye, water and auxiliary. To improve the effectiveness of dyeing, heat is usually applied to the dye liquor. The theory of aqueous dyeing, as explained bellow, is modified when an organic solvent is substitued for water.

The general theory of dyeing explains the interaction between dye, fibre, water and dye auxiliary. More specifically, it explains:

- 1. Forces of repulsion which are developed between the dye molecule and water.
- 2. Forces of attraction which are developed between the dye molecules and fibres.

These forces are responsible for the dye molecules leaving the aqueous dye liquid and entering and attaching themselves to the fibre.

The forces which anchor dyestuff molecules to textile fibres are complex. The process consists of three stages which are:

- 1. Migration of the dye from the solution to the interface accompanied by adsorption on the surface of the fibre.
- 2. Diffusion of the dye from the surface towards the centre of the fibre.
- 3. The anchoring of the dye molecules by covalent or hydrogen bonds, or other forces of a physical nature.

The assembly of dye molecules at the fibre surface (stage 1) is governed, in the main, by three influences. Firstly electro-potential forces, secondly temperature, and thirdly agitation.

All textile fibres, when immersed in water or aqueous solutions, acquire an electronic potential, often referred to as the zeta potential.

Most dyes, when in solution, are either in molecular and partially ionized state, or exist in the form of ionic micelles similar to those of soap. Increase of temperature tends to break down micelles into less aggregated units. In the case of the acid and the direct dyes the chromophore-containing ionts bear negative charges. These will be repelled by the zeta potential of the cellulosic fibres, but attracted by protein fibres when the aqueous phase is acidic.

The covalent and hydrogen bond forces which anchor the dye to the fibre are of short range, of the order of 0.1 to 0.5 nm. The electrostatic forces, however, have a range of about 10 nm. Where there is mutual repulsion between the

fibre and the dye ion, an extremely long time would be necessary before the inherent vibration of the molecules in the solution would have brought sufficient of them within the field of the short range forces for exhaustion to have proceeded to equilibrium. The problem, therefore, is to accelerate the migration to the surface of the fibre. This is achieved by increase of temperature which promotes vibrational activity, and also by the addition of electrolytes which counteract the effect of the negative zeta potential.

The effect of agitation is only apparent when the amount of dye in the solution is limited. Under such conditions the diffusion into the fibre (stage 2) could more rapid than the assembly at the surface, and agitation will then become a significant factor.

The second part of stage 2 is the diffusion of the dye molecules towards the centre of the fibre and this is governed by Fick's law which is expressed by the equation

$$\frac{dS}{dt} = -D \frac{dC}{dx} \dots [1]$$

in which:

dS - the amount of solute diffusing across unit area during the time interval dt

C - the concentration at a point x

D - the diffusion coefficient, namely the amount of solute diffusing in unit concentration gradient

D is therefore a measure of the ability of the dye to diffuse and also of the permeability of the fibre. The smaller the dye molecule the more readily will it diffuse, and the more porous the fine structure of the fibre the easier will be the passage of the molecules of the dye.

The rate of diffusion into the fibre is obviously dependent upon the concentration of dye molecules in the interfacial layer and, as explained above, this can under certain conditions be influenced by the degree of agitation.

With very few exceptions one or more of the following factors are responsible for the anchoring of the dye molecules to the fibre (stage 3):

- a) ionic (or salt) links
- b) hydrogen bonds
- c) covalent bonds
- d) physical forces
- e) entropy factors

Thermodynamic considerations

Initially the free energy of the solution is greater than that of the fibre phase. This difference in free energy is the driving force which will bring about transfer of the dye to the fibre till a stage of equilibrium reacted when the free energy in the liquid and solid phases are equal.

 μ_s = μ_f [2] where

 $\mu_{\text{\tiny S}}$ - chemical potential of the dye in the solution

μf - chemical potential of the dye in the fibre phase

where

μs°, μf° - is standard potentials

R - is the gas constant

T - is the absolute temperature

Cs(Cf) - is concentration of dye

in the solution (in the fibre phase)

2.3.2.1. Classification of dye molecule

Dye molecules are organic molecules which can be classified as:

- 1. anionic in which the colour is caused by the anionic part of the dye molecule
- 2. cationic in which the colour is caused by the cationic part of the dye molecule
- 3. disperse in which the colour is caused by the whole molecule

The first two dye molecule types are applied from an aqueous solution. The third is applied from an aqueous dispersion.

2.3.2.2. The behaviour of the fibre in the dye liquor

Textile fibres are organic compounds and develop a slight negative charge or potential when immersed in an aqueous solution. Since the dye molecule and textile fibre both become slightly negatively charged in aqueous solution, there is a tendency for the dye and the fibre to repel each other. Sufficient energy has to be built up in the dye

liquor to overcome this repulsive force and allow the dye and textile fibre to be attracted to one another, so that the dye molecules can enter the fibre.

2.3.2.3. The role of water

In addition to dissolving the dye, water acts as the medium through which the dye molecules are transferred into the fibre.

The polar groups in the dye molecules attract water molecules, and cause the dye to dissolve in water. This attraction between water and dye is on the whole undersirable, as the dye molecules resist leaving the water and entering the fibre. In some instances, however, it is desirable to reduce the rate at which dye leaves the water and enters the fibre so as to ensure a uniform colouration of the fibre.

In general heat is necessary to encourage the dye to leave the water and enter the fibre, as well as to ensure adequate penetration of the polymer system of the fibre. Heating the dye liquor causes water to dissociate somewhat more than pravailing room tamperatures and to become slightly more ionic. In this state, water tends to repel the organic dye molecule to a greater extent, ensuring readier uptake of the dye molecules by the fibre's polymer system.

Water, assisted by the heat, also swells fibers that are hydrophilic, making the polymers system more accessible to the relatively large dye molecules.

Heat is a form of energy. It is applied to increase the energy of the molecules of all constituents in the dye liquor and thus to increase the rate of the dyeing process. The application of heat not only results in an increases rate of dyeing but assists the dye molecules to penetrate the fibre. The application of heat to the dye liquor swells the fibre making it easier for the dye molecules to penetrate the fibre surface and enter deeper into amorphous regions. When the dye liquor has cooled and the fiber dried, the polymer system will close up again, that is return to its former dimension, trapping and entangling the dye molecules which entered it. In such circumstances, Van der Waals' forces may develop to assist the polymer system retain the dye molecules within the fibre polymer.

2.3.2.5. Dye auxiliaries

These chemical compounds include carriers or swelling agents, levelling agents, antifoaming agents, dispersing agents, detergents and wetting agents. The way in which these auxiliaries affect the dyeing process and their chemical constitution will be explained when this diploma work will define. However, the following provides some explanation for the way in which levelling agents are used in the dye liquor.

The addition of levelling agents to the dye liquor helps produce a more uniform colour in textile fibres. Levelling agents which tend to slow down the dye uptake of the fibres, are also termed retarding agents or retarders. The use of retarders is essential in situations in which dyes tend to "rush on to fibre" and result in an unevenly coloured textile material.

Levelling agents are surface active agents, and are chemically related to soaps, synthetics detergents and wetting agents. They may be anionic, cationic or non-ionic organic compounds.

2.3.2.5.1.1. Anionic levelling agents

The molecules of these compounds consist of a large negatively charged, or anionic, organic radical, with a cationic radical which is usually a sodium cation that assists the levelling agent's water solubility. When anionic agents are added to the dye liquor, their anions will be attracted to any positive site in the fibre. The anionic levelling agent tends to repel the similarly charged anionic dye molecule. As the dye bath is heated, the anionic dye molecules develop sufficient energy to overcome the repulsive forces between the retarder and the dye, both of which are anionic. The retarder slows the dye uptake of the fibre and results in a more uniformly dyed textile material.

2.3.2.5.1.2. Cationic levelling agents

The molecules of these compounds consist of a large positively charged, or cationic, organic radical, with an anionic radical which is usually a chloride ion and sometimes a bromide ion.

When cationic levelling agents are added to the dye liquor, their cations are attracted to the anionic dye molecules. This neutralises the electric charge on the dye molecules and minimises their substantivity for the fibres. The energy provided from heating the dye bath will cause the cationic radicals of the levelling agents to gradually dissociate from the anionic dye molecules. This gradual dissociation of the cation agents from the dye molecules slowly releases the dye and this ensures a more uniform absorbtion of the dye by the polymer system of the fibre.

2.3.2.5.1.3. Non-ionic levelling agents

Non-ionic levelling agents are mixture of alkylpolyglycolethers, which are prepared from alcohols and ethylen oxids. Non-ionic levelling agents are resistant to acid, alkalies and solution of all salts (to certain critical concentration).

Substantive dyes are mainly used for dyeing fibres of natural or regenerated cellulose in all stages of processing. Providing not very high wet fastness, fastness to boiling, bleaching and weather is demanded.

Substantive dyes may be divided into four technological classes:

- direct dyes (ordinary direct dyes)
- saturn dyes (lightfast direct dyes)
- rybantin dyes (special direct dyes)
- azogen dyes (diazotizing dyes)

2.4.1. Direct dyes

The attraction between fibre polymers and dye molecules is called substantivity. Direct dyes are also called substantive colours because of their excellent substantivity for cellulosic textile materials. Because of the very great substantivity direct dyes have for cotton, they have also been given the name of cotton colours.

The fibres most readily coloured with direct dyes are the man-made and natural cellulose fibres, that is, cotton and viscose fibres.

Classification of direct dyes by the society of dyers and colourists:

Class A (self-levelling)

Dyes which migrate well and therefore have high levelling power. They may dye unevenly at first but pursuer boiling will bring about even distribution.

Class B (salt controllable)

Dyes of poor levelling power where exhaustion must be brought about by controlled salt addition. If these dyes are not taken up uniformly in the initial stages it is extremely difficult to correct the unlevelness.

Class C (temperature controlable)

Dyes which are not self-levelling and highly sensitive to salt. The exhaustion of these dyes can not adequately be controlled by addition of salt alone they require additional control by temperature.

2.4.1.1. Dyeing with direct dyes

Direct dyes are applied to celluloise fibres from an aqueous liquor to which is added an electrolyte to the dye liquor sodium chloride. The addition of electrolyte to the dye liquor is essential to obtain adequate exhaustion of the dye molecules by the fibre.

When sodium chloride is added to the dye liquor it dissiciates completely into sodium ions (Na+) and chloride ions (Cl-). The cellulosic fibre in the dye liquor has a negative surface charge attracting to it the sodium cation. This neutralises the negative surface charge of the fibre, also referred to as the zeta potential enabling the dye anion of the direct dye to enter the fibre more readily. The presence of chloride ions in the dye liquor also assist the dye anion to leave the dye liquor and enter the fibre. This is result of repulsive forces between the dye anion and the chlorid anion.

The application of heat to the dye liquor increases the energy of the components of the dye liquor, swells the fibre and accelerates the rate at which dyeing occurs.

Graphic description of dyeing process

It is possible to draw the curves, from which we can read the process of dyeing. That means:

- a) how the exhaution of the dye is taken
- b) what influences of electrolytes are happen
- c) what influences of temperature can be happen and if is there a possibility to dye with low temperatures

The mains are exhaution curves (Figure X)

Graphic description of dyeing process under usual conditions in practice:

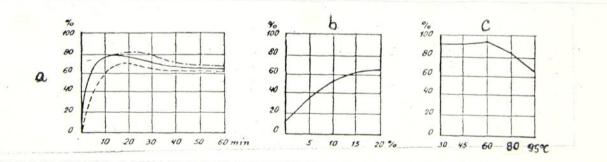


Figure X , Saturn Yellow LR extra

a - exhaution curve of rapid exhaustive dye
b - effect of electrolytes on exhaution of dye
c - effect of temperatures on exhaution of dye

The addition of an electrolyte to the dye liquor of anionic dye increases the uptake of the dye by the fibre. The electrolytes used in dyeing dissociate completely in the aqueous dye liquor. This increases the forces of repulsion between the dye molecules and water so the dye is attracted by the fibre.

The addition of electrolytes to the dye makes the dye liquor more ionic and thus increases the forces of repulsion between the electrolyte and the dye molecules. This attracts the dye to the fibre and increases the chances of the dye molecules entering the fibre. For the dye to enter the fibre, the surface charge of the fibre, which is negative, will have to be neutralised since both anionic dyes and textile fibres have the same charge. This is affected by the addition of cheap electrolytes such as sodium chloride or sodium sulphate. Both these electrolytes are extremly solubte and dissociate completely in an aqueous medium. The presence of these electrolytes makes the dye liquor more ionic which, together with the application of heat to the dye liquor, increases the energy of the molecules in the dye liquor and the forces of repulsion.

The use of electrolytes such as sodium chloride or sodium sulphate means that one of the dissociation products is sodium ions (Na+). Sodium ions are cationic, or positively charged, and in the dye liquor are attracred to the negatively charged textile fibres. Once on the surface of the fibre, the sodium cations neutralise the anionic surface charge of the fibre by forming a layer of Na+ about

10 nm thick on the surface of the fibres. The neutralised fibre can now attract the organic dye molecules which have a greater affinity for the fibre than the aqueous solution. The migration of the dye from the dye liquor to the fibres is accelerated by the application of heat to the dye liquor. See Figure Xb (PAGE 31)

2.4.1.1.2. Effect of liquor ratio

Percentage exhaustion continues to increase as the concentration of dye in the liquor becomes greater. Dyeing in a short liquor ratio, therefore, is an important factor in economizing in deystuff consuption. When producing heavy shades the amount of dye which is necessary can become very great if the machine is one which requires a high ratio of water to goods. With shades which are frequently repeated, such as blank, it is advisable to have a reserve tank in which the liquor can be stored for re-use after partification with a comparatively small amount of dye. Recipes with direct dyes will only give good repetition if it is possible to keep the liquor ratio constant.

2.4.1.1.3. Effect of pH

Direct dyes are almost invariably applied from a neutral solution. No advantage is gained by the addition of acid and there is a possibility that it may alter the shade. Mild alkali has a retarding effect upon the rate of adsorption, and up to 3 per cent of soda ash is sometimes added to counteract hardness in the water.

2.4.1.2. Molecular configuration and characteristics

$$N = N$$
 $N = N$
 $N =$

Figure 4 The structural formula for C.I. Direct Blue 71, 34140 — a direct dye

Figure 4 ilustrated the characteristic linear configuration of direct dye molecules. The auxochromes in the dye molecule are responsible for the good aqueous solubility of direct dyes. Many direct dyes are sodium salts of sulphonic acids. In this respect direct dyes are similar to acid dyes. In fact, some dyes which are applied as direct dyes and acid dyes may have the same formula. An examination of their molecular formula will not categorize the dye into one class or the other.

2.4.1.3. Properties of direct dyes

2.4.1.3.1. Light-fastness

Dyed direct colours have a moderate light-fastness, the light-fastness rating being about 3. This means that the direct dye anions seem to lack a stable electron arrangement, particularly in the chromophores. A relatively short exposure to direct sunlight is enough to initiate degradation of the dye molecule. The resultant breakdown of the direct dye anion is seen as fading of the dyed cellulosic textile material.

The wash-fastness rating of direct dyes is about 2-3. The comparatively poor wash-fastness of cellulosic textile materials dyed with direct dyes can be explained as follows.

The relatively large number of axochromes in the direct dye anion which contributes to the aqueous solubility of these dyes contributes to the poor wash-fastness of this class of dye.

Direct dyes, or more specifically the direct dye anions, are attached to the cellulose fibres by hydrogen bonds and Van der Waal's forces both of which are weak. Under aqueous consitions such as occur in laundering these weak bonds may be hydrolysed by the water molecules resulting in the removal of these dyes from the polymes system. The loss of dyes is seen as fanding of the cellulosic textile material.

2.4.1.3.3. Improving wash-fastness

Direct dyes are relatively easy to apply to cellulosic textiles. However, although easy to apply, they are also relatively easy to remove and this gives reason to say "easy on easy off". The advantage of direct dyes is their ease of application, comparatively low cost and wide range of available colours. It is for these reasons that direct dyes are still used on cellulosic textile materials.

The importance of direct dyes has resulted in the development of means to improve their wash-fastness. These aftre-treatments, as they are called, all aim to increase

the size of the direct dye molecule once it is located within the fibre. The larger dye molecule increases the forces of attraction between the dye molecule and the fibre. The increased molecular size makes it more difficult for the dye to be removed (washed out) from the fibre.

2.4.1.4. Scouring after dyeing

Dyeing always leaves some dye molecules on the surface of the fibres of the dyed textile materials. It is essential that these dye molecules be remove when dyeing is completed. If these dyes are not removed they may relust in two problems:

- Poor rub fastness which may result in the dye rubbing off onto adjecent materials
- 2. Poor wash fastness which may result in other fabrics being coloured by this excess dye during laundering.

2.4.1.5. Instrumental match prediction

Curve of reflectance - the development of instruments capable of recording the wavelengths of light reflected from dyed materials stimulated interest in the possibility of using such information to calculate the quantities of dyes required to match a given pattern. The calculations which were necessary, however, were too laborious until computers became available.

The basic information required is the relationship between reflectance of the dyed material and the concentration of the dyestuff. If a simple curve of

reflectance against concentration at a specified wavelength be plotted it is very far from a straight line but a linear relationship is desirable for instrumental match prediction.

Many efforts have been made to arrive at a mathematical expression which will give the desired linear relationship.

One which has often been used is the Kubelka-Munk equation :

$$\frac{K}{S} = \frac{(1 - R)^2}{2R}$$
 [5]

in which:

R - reflected light

K - coefficient of absorption

S - coefficient of scattering

Reflectance (to concrete limit) is directly proportional to the concentration of the dye on the fibre as is shown on the graph bellow. Red reflectance curve:

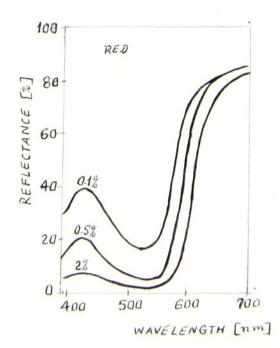


Figure 6.

The greater is the concentration of the dye on the fibre the low will be the degree of reflectance.

The modified Kubelka-Munk equation to conform the collimated incident light as would apply in a spectrophotometer or a colorimeter, and arrived at the equation:

$$\left(\begin{array}{c} 1 \\ \hline R_{\Lambda} \end{array}\right) = \left(\begin{array}{c} 1 \\ \hline R_{\Lambda} \end{array}\right)_{s} + a_{\Lambda}C \qquad \qquad [6]$$

in which :

 R_{λ} - the reflectance of the dyed sample at the wavelength

 $(R_{\lambda})_{s}$ - the reflectance of the substrate

a - the absorption coefficient of the dye

C - the concentration of the dye

This is taken into account in the C.I.E. System and, therefore, good matcg predication requires that the dyeing should have the same tristimulus values as the pattern when illuminated by the same light. The tristimulus values are defined by the following equations

$$X = \int_{\partial} E_{(\alpha)} R_{(\beta)} \bar{x}_{(\beta)} d\lambda \qquad \dots \qquad [7]$$

$$Y = \int_{\Lambda} E_{(\lambda)} R_{(\lambda)} \tilde{y}_{(\lambda)} d\lambda \qquad \dots \qquad [8]$$

$$Z = \int_{\Omega} E_{(\lambda)} R_{(\lambda)} \tilde{z}_{(\lambda)} d\lambda \qquad \dots \qquad [9]$$

where $\bar{x}_{(A)}$, $\bar{y}_{(A)}$, $\bar{z}_{(A)}$ are trichromaticity factors

The modified equation which is used to define the colour difference is called C.I.E. Lab, which is

DE =
$$[(DL)^2 + (Da)^2 + (Db)^2]^{\frac{1}{2}}$$
 [10] where

DE - total colour difference relative to the standard DL, Da, Db - are the differences in L, a and b relative to the L, a and b of the standard

L, a, b - are basic co-ordinates of C.I.E. Lab system, which are given by:

where

x, y, z - trichromaticity co-ordinates from the curve of reflectance for the given sources of light x_0 , y_0 , z_0 . Its graphic description is bellow:

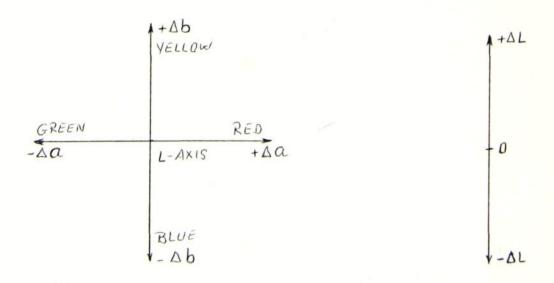


Figure 7.

The hue difference DH can be defined as a hue angle of the standard bases on the four basic shades (green, blue, red and yellow).

DH = $[(DE)^2 - (DL)^2 - (DC)^2]^{\frac{1}{2}}$ [14] where

L - lightness

The lightness difference DL is defined as bellow

DL = Lsample - Lstandared [15]

where

Lsample - is the lightness of the sample

Lstandared - is the lightness of the standared

for DL > 0 - the sample is lighter than the standared

for DL < 0 - the sample is dull than the standared

C is the metric chroma

The metric chroma difference DC is deffined as bellow

Csample - is the metric chroma of the samle

Cstandared - is the metric chroma of the standared

for DC > 0 - the sample is more saturated than the standared

for DC < 0 - the sample is less saturated than the standared

The metric chroma can be defined as bellow

$$C = (a^2 + b^2)^{\frac{1}{2}}$$
 [17]

for a and b see equations [12] and [13]

III. EXPERIMENTAL PART

- 3.1 TEXTILES, DYESTUFFS, CHEMICALS AND MACHINES WHICH ARE USED
- a) TEXTILE: unpre-treated cotton cloth
- b) DYESTUFFS: lightfast direct dyes (Czechoslovak products)

 Saturn blue LBRR (C.I. Direct blue 71)

 Saturn violet L4B (C.I. Direct violet 48)

 Saturn pure blue LB (C.I. Direct blue 106)

 Saturn brown L2G (C.I. Direct brown 115)

 Saturn brown LT

 Saturn khaki G

 Saturn red LG

c) CHEMICALS:

CaCl2

NaOH

Na2 S2 O4

NaCl

Na2 SO4

Na2CO3 (soda ash)

Na₂ HPO₄ 2H₂ O

Alfa-amylase: desizing agent on bath (bacterial alfa-amylase)

Syntron B: useful to water treatment, i.e. for water softening (for binding metals Fe⁺⁺ Fe⁺⁺⁺

Cu⁺⁺ Co⁺⁺ Ni⁺⁺ Mn⁺⁺ Zn⁺⁺ Cr⁺⁺⁺)

Syntapon LPG : is effective washing, wetting and

emulating anionic agent

- Alfonal K: wetting agent which contains alkanolamid of fattyacid, non-ionic agent, it gives a better and fast penetration of liquor to the material, it is useful for dyeing with all kinds of dyes with the exception of reactive dyes, it is possible to combine ALFONAL K with cationic, anionic and non-ionic levelling agents
- Spolion 8: is anionic wetting agent, it is an excellent and effective agent to all kinds of materials;

 because of this, it is possible to dye a material, which is not taken under kiering or washing process.

Non-ionic levelling agents: are mixture of alkylpolyglycolethers, which are of alcohols and ethylen oxids. These agents are resistant to acids, alkalis and solutions of all salts (to certain critical concentration).

- Slovapon N: Czechoslovak product, non-ionic levelling agent with great wetting and washing effect
- Slovasol O: Czechoslovak product, non-ionic levelling agent with wetting, washing and dispersing effect
- Slovasol 3520: Czechoslovak product, non-ionic levelling agent with wetting and washing effect

Cationic levelling agents: are good wetting agents, even in acid medium. They can be used like anti-static medium. They have no washing ability and with anionic agents will creat undissovable coagulant. So, we must not use these two agents together in one liquor.

Syntegal V20: Czechoslovak product, is a weak cationic levelling agent with great wetting effect

d) INSTRUMENTS (apparatus)

Scourotester (Hungary made) - dyeing machine with eight pressure containers on its rotary shaft, that gives motion to dye liquor.

Temperature of the dye liquor is regulated by termostat.

Data-Color - for objective colour measurment. Data color works with computer program DC-IRIS.

This program is capable to calculate and prefer automaticaly all important parameters, to work up a better variant of the results, to choose a recept out of two, three and more combinations of dyes, to test the colour difference, etc.

TexFlash (Swiss made Data-color) - texflash tests diffusion of lighting, observetion pass perpendicular to the sample. Then structure and surface treatment of the sample can be well tested.

3.2. PREPARATION OF SOLUTIONS

Depending on the depth of shade and liquor ratio, we prepared the solutions.

The formula, which we used is:

$$V = \frac{M_{m} \ V_{1} \ S}{C \ . \ 100} \ [ml] \ \ [18]$$

when

V - the volume of the dyestuff or chemical to be titrated

 M_{m} - the weight of the material

V1 - the volume of the liquor

S - the depth of shade

C - concentration of the solution to be prepared

3.3. PRE-TREATMENT OF COTTON

Cotton fibres contain oils, fats, and waxes together with other impurities. Garments, fabrics or yarns may contain oil and adventitious dirt collected during manufacturing.

Waxes are compounds formed by the esterification of fatty acids and complex monohydric alcohols with relatively high melting points.

Waxes are considerably more resistant to saponification than fats, and can only be hydrolysed with aqueous solution of alkalis under pressure, or at atmospheric pressure after prolonged boiling with alkoholic potash. Oils, fats, and waxes are all insoluble in water; most of them, however, dissolve in organic solvents such as chloroform, ether, petrol, benzene, or the chlorinated hydrocarbons. They also form emulsions of varying degrees of stability when shaken with water containing soap or other emulsifying agents. Fast and related substances can be removed from textiles by saponification when the fibre is not damaged by alkalis, as in the case of cotton.

For better results of dyeing the material should pass under this treatment.

In this experiment, desizing and kiering (alkalic scouring) are done.

1) desizing

the 4 l bath was prepared and it contains

5 g/l alfa-amylose

0.5 g/l CaCl2

0.1 g/l Spolion 8

The desizing was carried out at 60 - 70°C in the machine called "Jigger" for 2 hours. Then desizing throughly removed from the material with the remains of cleaning liquors, since they could impair the process of dyeing.

2) alkalic scouring

the 4 l bath was prepared and it contains

3 g/l Syntron B

5 g/l NaOH

1 g/l Alfonal K

1 g/l Na2 S2 O4

This process is also done in "Jigger". The scauring was carried out at 100°C for 2 hours. Then bath is cooled to 80°C, and the liquor was removed. Then the material was

washed with hot and cold water until the wash water becomes neutral.

3.4. DYEING WITH DIRECT DYES

3.4.1. Dyeing conditions

The weight of the material to be dyed is 2 g. The dye bath contains, according to the weight of the material:

- 3 % dyestuff
- 1 % soda ash
- 0 % , 1,2 % , 5 % wetting agents (Spolion 8, Alfonal K)
- 0 % , 1,2 % , 5 % levelling agents (Slovapon N, Slovasol O, Slovasol 3520)
- 20 % , 45 % , 75 % , 100 % , 125 % electrolyte (Nacl, Na2 SO4)

dyeing temperature Tb = 45°C, 60°C, 80°C, 98°C

Dyeing is started at 40°C and the temperature is raised (by 3°C per minute) to respective dyeing temperature and the dyeing is maintained at this temperature for respective dyeing time, i.e. 30 min, 45 min, 60 min and 90 min.

3.4.2. Dyeing procedures and their dyeing conditions

The aim of this diploma work was indicated in the introduction part. Experiment was taken by using different direct dyes. Samples are dyed in scourotester at different conditions and then washed in running tap water and dried in air at room temperature.

For some selected samples, colour hue and saturation

were evaluted.

Result of each dyeing procedure are shown in the tables bellow.

Procedure 1: The samples 1-52 was dyed by using 3% (according to the weight of the material to be dyed) of Saturn Blue LBRR and by using no dye auxiliary. Different amounts of electrolytes were used as it is seen in the tables 3 and 4. Liquor ratio 1:50 was used.

Table 3

No NaCl[%] Tb[°C] tb[min]

Table 4

No	Na2 SO4 [%]	Tb[°C]	tb[min]
27	20	98	30
28	45	98	30
29	75	98	30
30	100	98	30
31	20	98	60
32	45	98	60
33	75	98	60
34	100	98	60
35	20	80	30
36	45	80	30
37	75	80	30
38	100	80	30
39	20	80	60
40	45	80	60
41	75	80	60
42	100	80	60
43	20	60	30
44	45	60	30
45	75	60	30
46	100	60	30
47	20	60	60
48	45	60	60
49	75	60	60
50	100	60	60
51	125	60	30
52	125	60	60

Explanatory:

% according to the weight of material to be dyed

Tb - dyeing temperature

Procedure 2: The samples 53-104 was dyed by using 3% (according to the weight of the material to be dyed) of Saturn Violet L4B and by using no dye auxiliary. Different amounts of electrolytes were used as it is seen in the tables 5 and 6. Liquor ratio 1:50 was used.

Table 5

Table 6

No	NaCl[%]	Tb[°C]	tb[min]	No	Na2 SO4 [%]	Tb[°C]	tb[min]
53	20	98	30	79	20	98	30
54	45	98	30	80	45	98	30
55	75	98	30	81	75	98	30
56	100	98	30	82	100	98	30
57	20	98	60	83	20	98	60
58	45	98	60	84	45	98	60
59	75	98	60	85	75	98	60
60	100	98	60	86	100	98	60
61	20	80	30	87	20	80	30
62	45	80	30	88	45	80	30
63	75	80	30	89	75	80	30
64	100	80	30	90	100	80	30
65	20	80	60	91	20	80	60
66	45	80	60	92	45	80	60
67	75	80	60	93	75	80	60
68	100	80	60	94	100	80	60
69	20	60	30	95	20	60	30
70	45	60	30	96	45	60	30
71	75	60	30	97	75	60	30
72	100	60	30	98	100	60	30
73	20	60	60	99	20	60	60
74	45	60	60	100	45	60	60
75	75	60	60	101	75	60	60
76	100	60	60	102	100	60	60
77	125	60	30	103	125	60	30
78	125	60	60	104	125	60	60

Explanatory:

% according to the weight of material to be dyed

Tb - dyeing temperature

Procedure 3: The samples 105-112 was dyed by using 3% (according to the weight of the material to be dyed) of Saturn Blue LBRR and by using no dye auxiliary. Different amounts of electrolytes were used as it is seen in the tables 7 and 8. Liquor ratio 1:20 was used.

Table 7

No	NaCl[%]	Tb[°C]	tb[min]
105	20	60	60
106	45	60	60
107	75	60	60
108	100	60	60

Table 8

No	Na2 SO4 [%]	Tb[°C]	tb[min]
109	20	60	60
110	45	60	60
111	75	60	60
112	100	60	60

Procedure 4: The samples 113-120 was dyed by using 3% (according to the weight of the material to be dyed) of Saturn Violet L4B and by using no dye auxiliary. Different amounts of electrolytes were used as it is seen in the tables 9 and 10. Liquor ratio 1:20 was used.

Table 9

No	NaCl[%]	Tb[°C]	tb[min]
113	20	60	60
114	45	60	60
115	75	60	60
116	100	60	60

Table 10

No	Na2 SO4 [%]	Tb[°C]	tb[min]
117	20	60	60
118	45	60	60
119	75	60	60
120	100	60	60

Explanatory:

% according to the weight of material to be dyed

Tb - dyeing temperature

Procedure 5: The samples 121-172 were dyed by using different amounts of levelling, wetting agents, dye stuffs 3% Saturn Blue LBRR (Table 11 and 12) and 3% Saturn Violet L4B (Table 13 and 14). The hue and saturation of each dyed sample was evaluated visually. The results are shown in the Tables 11, 12, 13 and 14.

The following dyeing conditions were taken 40% NaCl, liquor ratio 1:50 was used and tb=30 minutes.

rable	= 11	7	rb =	60°(
No	13	[%]	Н	S
121		0	3-4	4-5
122 123	11	1.2	4 3-4	5 4-5
124 125	12	1.2	4-5 4	5 4-5
126 127	13	1.2	5 4-5	4-5 4-5
128 129	14	1.2	4-5 5	4-5 4
	Wi	[%]		
130 131	w1	1.2	4-5	5 4-5
132 133	w2	1.2	4 4-5	5 4-5

Table 12 Tb = 45° C

able	12		10 -	10
No	1 i	[%]	Н	S
134		0	3-4	4-5
135 136	11	1.2	4 3-4	4-5 4
137 138	12	1.2	4-5	4-5 4
139 140	13	1.2	5 4-5	4 4
141 142	14	1.2	4-5 5	3-4
	Wi	[%]		
143 144	w1	1.2	4-5	4 4
145 146	w2	1.2	4 4-5	5 4-5

Explanatory:

li - levelling agents

wi - wetting agents

11 - Slovasol 3520

12 - Slovasol O

13 - Slovapon N

14 - Syntegal V20

w1 - Spolion 8

w2 - Alfon@Y K

s - the depth of the shade (saturation)

H - the hue (uniformity in dyeing)

5 - excelent GRADES OF H and S

Tb - dyeing temperature

% according to the weight of material to be dyed

Table	13		Tb	=	60°	(
		Manager of the last	-	-		-

0

1.2

5

H

3 - 4

4

3-4

1.2 4-5 4-5

5

4-5

4-5

li[%]

No

147

148

149

150 12

11

r	[able	e 14	
	No	1:	i[%]
	160		0
	161 162	11	1.2
	163 164	12	1.2
	165 166	13	1.2
	167 168	14	1.2
		Wi	[%]
	169 170	w1	1.2
	171 172	w2	1.2

 $Tb = 45^{\circ}C$

S

4

4-5

4

4-5 5

4-5

4

3

4

4-5

4

H

3

4

3 - 4

4-5

4

5

4-5

5

4-5

4

4

4-5 4-5

4-5 4-5

1	51		5	4	5
	52	13	1.2	5 4	5 5
	54	14	1.2	4 4-5	4 3
		wi	[%]		
	56	w1	1.2	4-5	4 4
	58	w2	1.2	4 4-5	4 4-5

Explanatory :

li - levelling agents

wi - wetting agents

11 - Slovasol 3520

12 - Slovasol O

13 - Slovapon N 14 - Syntegal V20

w1 - Spolion 8

w2 - Alfonal K

s - the depth of the shade (saturation)

H - the hue (uniformity in dyeing)

1 - poor 5 - excelent 3 GRADE OF H and S

Tb - dyeing temperature

% according to the weight of material to be dyed

Procedure 6:

The samples were dyed under this conditions:

3 % Saturn blue LBRR (Table 15 and 16)

3% Saturn Violet L4B (Table 17 and 18)

40 % NaCl

tb = 30 min

1.2 % li

1.2 % wi

liquor ratio = 1:50

Table 15 Tb = 60° C Table 16 Tb = 45° C

No	li	wi	Н	S
173	11	w1	3	4
174	11	w2	2-3	4
175	12	w1	4	4
176	12	w2	3-4	4
177	13	w1	5	3-4
178	13	w2	3	4-5
179	14	w1	4	3
180	14	w2	2-3	5

No	li	wi	Н	S
181	11	w1	3-4	4
182	11	w2	3	4
183	12	w1	4-5	5
184	12	w2	5	2
185	13	w1	4-5	4
186	13	w2	4	3
187	14	w1	4-5	3
188	14	w2	5	2

No	li	wi	Н	S
189	11	w1	3	4-5
190	11	w2	2-3	4
191	12	w1	5	5
192	12	w2	3	4
193	13	w1	3-4	4
194	13	w2	4	3
195	14	w1	3-4	3-4
196	14	w2	4	3

Table 17 Tb = 60° C Table 18 Tb = 45° C

		1		
No	li	wi	Н	S
197	11	w1	3	5
198	11	w2	2-3	4
199	12	w1	5	3
200	12	w2	4-5	3-2
201	13	w1	5	3-2
202	13	w2	4	2
203	14	w1	4-5	3-2
204	14	w2	4	2

Explanatory:

li - levelling agents

11 - Slovasol 3520

12 - Slovasol O

13 - Slovapon N

14 - Syntegal V20

w1 - Spolion 8

w2 - Alfonel K

s - the depth of the shade (saturation)

H - the hue (uniformity in dyeing)

1 - poor 3 GRADES OF H and S

Tb - dyeing temperature

% according to the weight of material to be dyed

Procedure 7: The samples in Table 19 were dyed under these conditions: Tb = 60°C, tb = 30 minutes, 40% NaCl

Table 19

	Kh	R	Вр	12	13	w1		
	[%]	accor	ding	to th	e wei	ght		
No	of	the n	nateri	ial to	be d	yed	Н	S
214	3	_	-	_	neets.	_	4	5
215	3	-	-	1.2	_	-	4	4
216	3	_	_	5	_	_	4	5
217	3	-	_	_	1.2	-	4	4
218	3		-	-	5	-	4-5	3
219	3		-	1.2	_	1.2	4-5	3 5
220	3	_	_	_	1.2	1.2	4-5	5
221	-	3	_	-	-	_	4-5	3
222	-	3	_	1.2	_	-	4-5	4
223	-	3	_	5	_	-	4	3-4
224	-	3	-	_	1.2	-	3-4	5
225	-	3	-	-	5	_	4	3 - 4
226	-	3	-	1.2	_	1.2	5	3
227	_	3	-	_	1.2	1.2	5	3
228	-	-	3	-	-	-	3-4	3
229	-	-	3	1.2	-	-	4	3
230	-	-	3	5	-	_	4	3
231	-	-	3	-	1.2		4-5	3
232	-	-	3	-	5	_	5	2
233	-	-	3	1.2	_	1.2	5	2 4
234	-	-	3	-	1.2	1.2	4	5

Explanatory:

Kh - Saturn Khaki G

R - Saturn Red LG

Bp - Saturn pure Blue LB

12 - Slovasol O

13 - Slovapon N

w1 - Spolion 8

s - the depth of the shade (saturation)

H - the hue (uniformity in dyeing)

1 - poor 5 - excelent] G-RADES OF S and H

Tb - dyeing temperature

Procedure 8: The samples in Table 20 were dyed under these conditions: Tb = 45° C , tb = 30 minutes, 40% NaCl Table 20

	Kh	R	Вр	12	13	w1	12	
No				to th			Н	5
235	3	_	_	_	_	_	4	5
236	3	_	-	1.2	-	-	4-5	5
237	3	_	-	5	_	_	5	4
238	3	-	-	_	1.2	-	4	4
239	3	-	-		5	-	5	3
240	3	_	_	1.2	-	1.2	4	3 5
241	3	_	-	_	1.2	1.2	4	4
242	-	3	-	_	_	-	4-5	4
243	-	3	-	1.2	-	-	4	4
244	-	3	-	5	-	-	4	4
245	-	3	-	-	1.2		4	4
246	-	3	-	_	5	-	4-5	4
247	-	3	-	1.2	_	1.2	4	4
248	-	3	-	-	1.2	1.2	4	4
249	-	-	3	-	-	_	3	4
250	-	-	3	1.2	-	-	3	4
251	-	-	3	5	-	-	3	4
252	-	-	3	-	1.2	-	3	4
253	-	-	3	-	5	-	3	4
254	-	-	3	1.2	-	1.2	5	4
255	-	-	3	_	1.2	1.2	4	5

Explanatory:

Kh - Saturn Khaki G

R - Saturn Red LG

Bp - Saturn pure Blue LB

12 - Slovasol O

13 - Slovapon N

w1 - Spolion 8

s - the depth of the shade (saturation)

H - the hue (uniformity in dyeing)

1 - poor GRADES OF HandS

5 - excelent

Tb - dyeing temperature

Procedure 9: The samples in Table 21 were dyed under these conditions: Tb = 60°C, tb = 30 minutes, 40% NaCl

Table 21

	Br1	Br2	11	12	13	w1		
No		accor he ma					Н	S
256	3	_	_	_	_	_	3	4
257	3	-	-	1.2	_	_	3-4	4
258	3	-	_	5	_	-	3	5
259	3		-	_	11.2	_	4	4
260	3	-	-	-	5	-	3-4	5
261	3	-	-	1.2	-	1.2	4-5	4
262	3	-	-	-	1.2	1.2	5	4
263	-	3	-	-	-	-	4	4
264	-	3	-	1.2	-	_	3-4	5
265	-	3	-	5	-	-	3	4
266	-	3	-	-	1.2	-	3	4
267	-	3	-	-	5	-	4	4-5
268	-	3	1.2	-	-	1.2	5	2
269	-	3	-	1.2	-	1.2	4	3
270	-	3	-	_	1.2	1.2	4-5	3

Explanatory:

11 - Slovasol 3520

12 - Slovasol O

13 - Slovapon N

w1 - Spolion 8

s - the depth of the shade (saturation)

H - the hue (uniformity in dyeing)

5 - excelent GRADES OF S and H

Tb - dyeing temperature

Br1 - Saturn brown LT

Br2 - Saturn brown L2G

Procedure 10: The samples in Table 22 were dyed under these conditions: Tb = 45°C, tb = 30 minutes, 40% NaCl

Table 22

	Br1	Br2	11	12	13	w1		
No		accor he ma					Н	S
271	3	_	_	_	_	_	5	5
272	3	_	-	1.2	-	-	4	4
273	3	-	-	5	-	-	4	3-4
274	3	-	-	-	1.2	-	3	3
275	3	-	-	-	5	-	3	4
276	3	-	1.2	-	-	1.2	4-5	3
277	3	-	-	1.2	-	1.2	3-4	4
278	3	-	-	-	1.2	1.2	4-5	3 - 4
279	3	-	1.2	1.2	-	-	4-5	3-4
280	-	3	-	-	-	-	4	4
281	_	3	-	1.2	-	-	3	4
282	-	3	-	5	-	-	3	4
283	-	3	-	-	1.2	-	3	4
284		3	-	-	5	-	3	5
285	-	3	-	1.2	-	1.2	3	4
286	\ -	3	-	-	1.2	1.2	3	4

Explanatory:

11 - Slovasol 3520

12 - Slovasol O

13 - Slovapon N w1 - Spolion 8

s - the depth of the shade (saturation)

H - the hue (uniformity in dyeing)

5 - excelent] GRADES OF Hand S

Tb - dyeing temperature

Br1 - Saturn brown LT

Br2 - Saturn brown L2G

Procedure 11: Samples 205-213 were dyed under these

conditions :

3 % Saturn khaki G 40 % NaCl tb = 30 min different amount of 13 and w1 liquor ratio = 1:50 Tb = 60°C

Table 23

No	13[%]	w1[%]	Н	S
205	0	0	3-4	4
206	0.6	0.6	5	4
207	0.6	1.2	3-4	4
208	1.2	0.6	3-4	4
209	0.6	3	4	4
210	3	0.6	3 - 4	3-4
211	1.2	3	4-5	4-5
212	3	1.2	3-4	5
213	3	3	5	4-5

Explanatory:

13 - Slovapon N

w1 - Spolion 8

s - the depth of the shade (saturation)

H - the hue (uniformity in dyeing)

1 - poor 5 - excelent] GRADES OF Hand S

Tb - dyeing temperature

tb - dyeing time

% according to the weight of the material to be dyed

3.5. FASTNESS TESTS

A number of well dyed samples were selected to test their fastness to washing and perspiration.

Table 24: dyeing conditions of the selected samples

LE	Kh	В	Br1	11	12	13	w1	NaCl		
SAMPL	% 8		ding				f the		Tb [°C]	tb [min]
a	3	_	_	_	_		_	40	80	30
b	3	-	-	1.2	-	-	1.2	40	80	30
C	3	-	-	-	1.2	-	1.2	40	80	30
d	3	-		-	_	1.2	1.2	40	80	30
е	3	-	-	-	-	1.2	1.2	40	60	30
f	3	-	-	-	-	1.2	1.2	40	45	30
g	- 1	3	-	- 1	-	-	-	40	60	30
g h	-	3	-	-	1.2	-	1.2	40	60	30
i	-	3	_	-	_	1.2	1.2	40	60	30
i j k	-	3	-	- 1	-	1.2	-	40	60	30
	-	-	3	- 1	-	-	-	40	60	30
1	-	-	3	1.2	_	-	1.2	40	60	30
m	-	-	3	-	1.2	-	1.2	40	60	30
n	-	-	3	-	-	1.2	1.2	40	60	30
0	3	-	-	-	-	-	-	40	60	30
p	3	-	-	1.2	-	-	1.2	40	60	30
P	3	-	-	-	1.2	-	1.2	40	60	30
r	3	-	-	- 1	-	1.2	1.2	40	60	30

Explanatory:

Kh - Saturn khaki G

B - Saturn blue LBRR

Br1 - Saturn brown LT

11 - Slovasol 3520

12 - Slovasol O

13 - Slovapon N

w1 - Spolion 8

Tb - dyeing temperature

Materials which are used : undyed cotton and wool clothes (Standard types)

Apparatus which is used : Scourotester (see 3.1)

The specimem to be tested is placed between two pieces of undyed fabric measuring 10 cm by 4 cm and the three pieces are held together by stitching round the edges.

The composition of one of the colourless materials enclosing the specimen will be the same as the dyed sample and the other will be different (in our case it is wool).

The solution is made containing 4 g per litre of Syntapon LPG. The composite sample is then treated in a wash wheel (equivalent apparatus - scourotester) at 40°C for 30 minutes.

Atfer treatment the composite samples are rinsed in cold distilled water and then in cold running tap water. Then the pieces are opened out and dried in air at temperature of 55°C.

The changes in colour of the specimen is assessed with grey scale No 1 and the staining of the undyed materials with grey scale No 2.

The results are shown in the table bellow.

The degrees from 1 to 5 (in Table 25a and 25b) means:

1 : is for poor fastness

5 : is for excellent fastness

Table 25a

2			
PLE	The change in colour	Degree of	staining
SAMPLE	In colour	cotton	wool
a	3	3	4-5
b	3	3	4-5
C	4	3-4	4-5
d	4-5	4	5
е	2-3	2-3	4-5
f	2	2-3	4-5

Table 25b

PLE	The change in colour	Degree of	staining
SAM	In colour	cotton	wool
g	2	2-3	4-5
h	3-4	3-4	4-5
i	4	3-4	5
j	3	2-3	4-5

3.5.2. Test of fastness to perspiration according to ČSN 80 0165

The materials which are used : undryed cotton and wool clothes (standard type)

Instrument which is used: Perspirometer is used for test of fastness to perspiration. It gives a force equivalent to $4.5~\rm kg$. It has glass plated measuring $115~\rm mm \times 60~\rm mm \times 1.5~\rm mm$.

Two artificial perspiration solutions were made up as follows:

	SOLUT	TION
	(A)	(B)
L-Histidine mono-hydrochloride mono-hydrate	0.5 g	0.5 g
Sodium chloride	5 g	5 g
Disodium hydrogen orthophosphate	2.5 g	2.5 g
volume in distilled water [ml]	1000	1000
pH (adjusted with N/10 NaOH)	8	5.5

The material to be tested is placed between undyed pieces, one of which is of the same fibre and the other of an alternative composition. It is allowed to remain in the solution (A) for 30 minutes at room temperature, after which the liquid is poured off. The specimen is then placed between two glass plates pressed together with a force equivalent to 4-5 kg (10 lb) and allowed to stand in an oven 37° ± 2°C for 4 hours. After this the dyed sample and undyed cloths are separated and they are all dried at room temperature. And the procedure is repeated with solution (B). The changes in colour and staining are then assessed with grey scales for each test.

The results are shown in the tables bellow.

The degrees from 1 to 5 (in Table 26 and 27) means:

1 : is for poor fastness

5 : is for excellent fastness

Table 26

	SOLUTION (A)			SOLUTION (B)			
SAMPLE	Change degree of in staining		Change in	degree of staining			
SA	colour	cotton	wool	colour	cotton	wool	
o p q	5 5 5 5	3 4 4-5 3	5 5 5 5	5 5 5 5	3 3-4 4 3-4	5 5 5 5	

Table 27

	SOLUTION (A)				
SAMPLE	Change in	degree of staining			
SA	colour	cotton	wool		
k 1 m n	5 5 5 5	3-4 4 4 4-5	5 5 5 5		

See the total evaluation of fastness tests in chapter 4.3.

3.6. MIGRATION TEST

Skeins of white and coloured cotton of equal weight were treated under normal conditions in the so-called "blank bath" which contains all usual additions except for the dyestuff. The test was taken at 60°C and 80°C for 30 minutes. It is determined to what extent the dye is able to pass from the dyed onto the white fibre.

Its evaluation is given in chapter 4.4.

4.1. VISUAL EVALUATION

It was decided that to have a visual expression for the hue and saturation of the colour of the dyed samples, which are expressed by the figures 1 - 5. Figure 1 stands for the lowest degree and figure 5 for the highest degree of levelling and saturation.

The results are evaluted relatively by comparing the samples each other.

Discussion 1

Enfluence of electrolytes

When the liquor ratio of 1:50 was prepared, using NaCl (sodium chloride) as the electrolyte, a better hue was obtained than using the Na2SO4 (sodium sulphate) electrolyte.

Enfluence of dye auxiliaries

When 1.2 % and 5 % (according to the weight of the material to by dyed) of Slovasol 3520 was used, the colour of the resulting product was not uniform. Although a large amount of dye was taken from the dye liquor (heavy shade were apparent). (see the table 11, 12, 13, 14)

Due to the poor levelling effect produced by the Slovasol 3520, it was decided that other levelling agents should be tested to obtain the best possible results. Those to be tested were Slovasol O, Slovapon N and Syntegal V20.

A 1.2 % and 5 % of each agent (i.e. Slovasol 0, Slovapon N and Syntegal V20) was used. So observations could be made for determining of which agent to use.

Enfluence of levelling agents

For each levelling agent the 1.2 % gave the best results (the 5 % mixtures gave patchy results). The best evelling agents was the Syntegal V20 (i.e. it gave a good even and uniform colour distribution, but too small amount of dye was taken from the dye liquor (light shades were apparent). (see the tables 11, 12, 13, 14)

Then, it was decided that to use wetting agents Spolion 8 and Alfonal K.

Discussion 3

A 1.2 % and 5 % of each wetting agents (Spolion 8 and Alfonal K) was used in the dye liquor. (see the tables 11, 12, 13, 14). They were also used in combination with each levelling agents Slovasol 3520, Slovasol O, Slovapon N and Syntegal V20. The results are shown in the tables 15, 16, 17 and 18.

As can be seen from he tables, the best results came from the combination of 1.2 % Spolion 8 + 1.2 % Slovapon N.

For all the dyestuffs used the best combination was also the 1.2 % Spolion 8 with 1.2 % Slovapon N, followed by 1.2 % Spolion 8 and 1.2 % Slovasol O.

For better determination, it was decided that to have an instrumental match prediction.

4.2. INSTRUMENTAL MATCH PREDICATION

Some selected dyed samples, which are dyed by using different dye stuffs and dye auxiliaries, were tested by the Data-color (see chapter 3.1.).

Colour difference - DE, metric chroma difference - DC and hue difference - DH were measured.

The results are shown in the following table.

lighting = D65

[%] according to the weight of the material						
dye auxialiary	3% dyestuff	[%] of the auxiliaries	DC	DH	DE	
	Kh	1.2	0.1	0.3	0.7	
Slovasol O	Br1	1.2	0.0	0.1	0.2	
	Вр	1.2	0.2	0.2	0.7	
Elgara S., DC - vi	Kh	1.2	-0.1	0.3	0.4	
Slovapon N	Br1	1.2	-0.9		0.9	
	Вр	1.2	0.2	-0.2 -1.2	0.6	
combined dye auxiliaries	3% dyestuff	amount of each dyeauxiliaries in [%]	DC	DH	DE	
Slovasol O	Kh	1.2 and 1.2	-1.0	-0.5	2.3	
and Spolion 8	Br1	1.2 and 1.2	0.1	-0.2	0.7	
	Вр	1.2 and 1.2	-0.1	-0.2	0.5	
Slovapon N	Kh	1.2 and 1.2	0.9	-0.2	3,2	
and Spolion 8	Br1	1.2 and 1.2	-0.5	0.2	1.1	
	Вр	1.2 and 1.2	0.5	0.5	1.5	
Slovasol 3520 and Spolion 8	Br1	1.2 and 1.2	0.6	0.1	1.6	

EXPLANATORY: Kh - Saturn Khaki G

Br1 - Saturn Brown LT

Bp - Saturm pure Blue LB

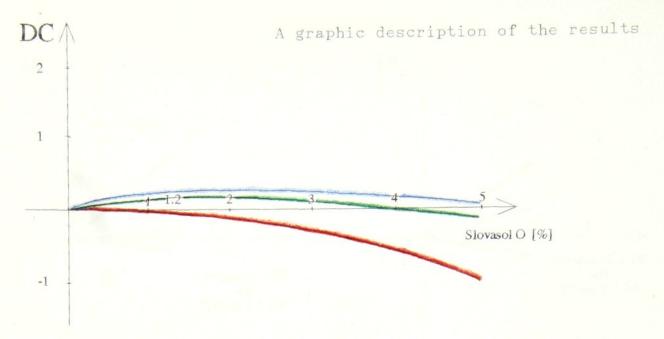


Figure 8 : DC - Metric chroma differences

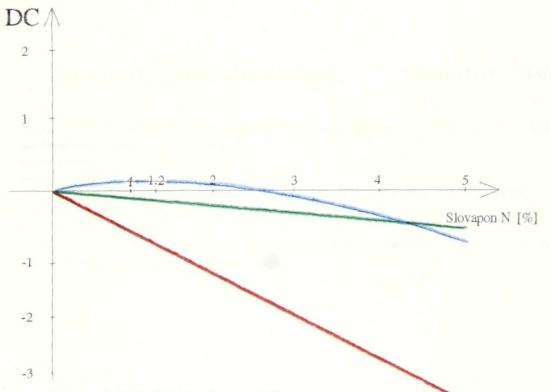


Figure 9: DC - Metric chroma differences

Dye stuffs which are used in both cases (Figure 8 and 9):

Saturn Khaki G

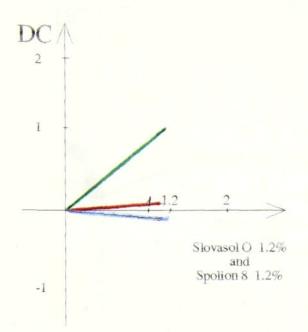
Saturn Brown LT

Saturn pure Blue LB

Levelling agents which are used: Slovasol O (Figure 8)

Slovapon N (Figure 9) and 5%

In both cases the samples were dyed by using 1.2% (according to the weight of the material to be dyed) of levelling agent. The standard was dyed by using no dye auxiliary.



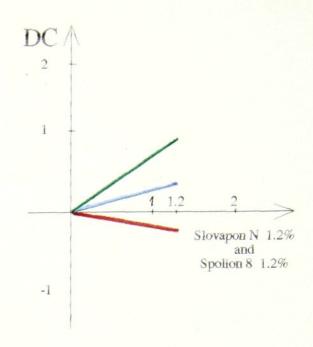


Figure 10 : DC - Metric chroma differences

Figure 11: DC - Metric chroma difference

Dye stuffs which are used in both cases (Figure 10 and 11):

Saturn Khaki G

Saturn Brown LT

Saturn pure Blue LB

And combinations of levelling agents SLOVASOL O with SPOLION 8 (Figure 10) and SLOVAPON N with SPOLION 8 (Figure 11) were used.

1.2% (according to the weight of the material to be dyed) of each dye auxiliary was used.

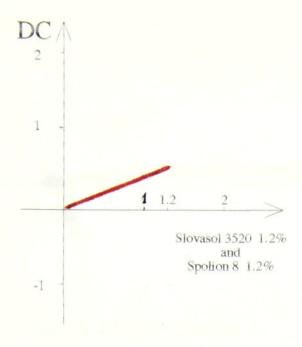


Figure 12: Metric chroma difference

Figure 12: Saturn Brown LT

The test was done on the samples, which are dyed with Saturn Brown LT by using levelling agent SLOVASOL 3520 with SPOLION 8. That was done to have same more information about SLOVASOL 3520, which had a poor levelling effect in previous procedure (by visual evaluation) (see discussion 1 on page 64).

1.2% (according to the weight of the material to be dyed) of each dye auxiliary was used. An objective predication was taken by Data-color and the result is shown on Figure 12.

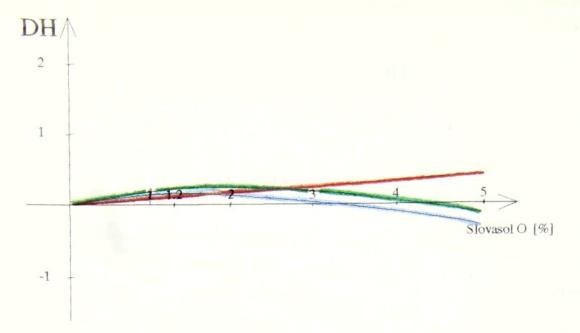


Figure 13: DH - Hue differences

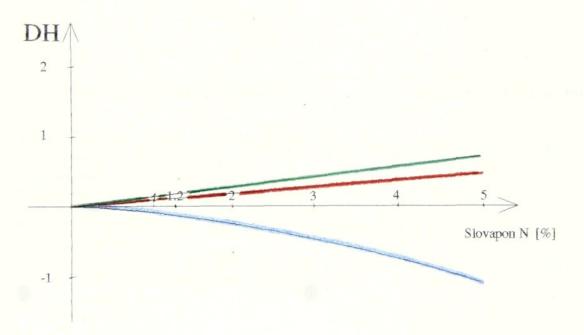


Figure 14: DH - Hue differences

Dye stuffs which are used in both cases (Figure 13 and 14):

Saturn Khaki G

Saturn Brown LT

Saturn pure Blue LB

Levelling agents which are used: Slovasol O (Figure 13)

Slovapon N (Figure 14) and 5%

In both cases the samples were dyed by using 1.2% V(according to the weight of the material to be dyed) of levelling agent. The standard was dyed by using no dye auxiliary.

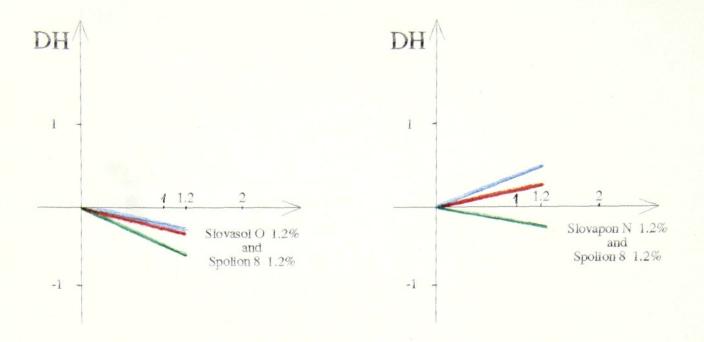


Figure 15: DH - Hue differences

Figure 16: DH - Hue differences

Dye stuffs which are used in both cases (Figure 15 and 16):

Saturn Khaki G

Saturn Brown LT

Saturn pure Blue LB

And combinations of levelling agents SLOVASOL O with SPOLION 8 (Figure 15) and SLOVAPON N with SPOLION 8 (Figure 16) were used.

1.2% (according to the weight of the material to be dyed) of each dye auxiliary was used.

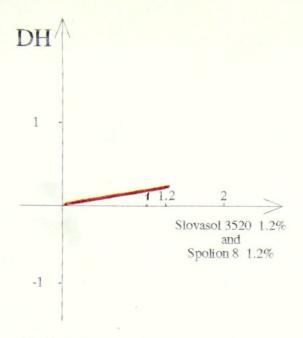


Figure 17: HD - Hue difference

Figure 17: ——— Saturn Brown LT

The samples were dyed with Saturn Brown LT by using levelling agent SLOVASOL 3520 with wetting agent SPOLION 8 1.2% (according to the weight of the material to be dyed) of each was used.

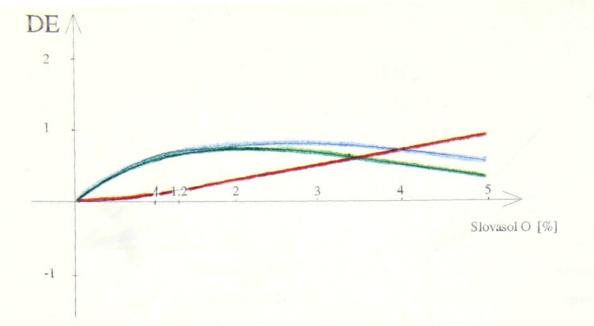


Figure 18: DE - Colour differences

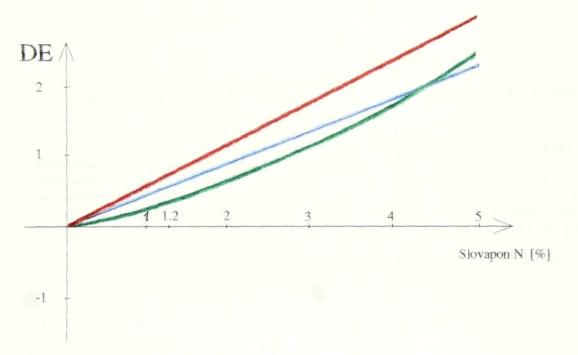
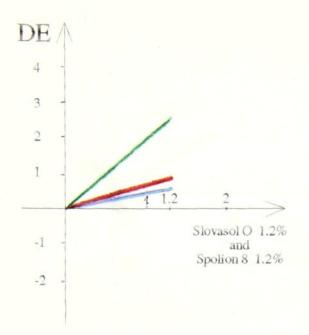


Figure 19: DE - Colour differences



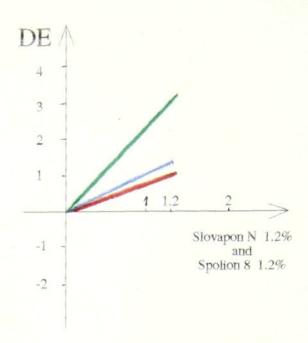


Figure 20 : DE - Colour differences

Figure 21: DE - Colour differences

Dye stuffs which are used in both cases (Figure 20 and 21):

Saturn Khaki G
Saturn Brown LT
Saturn pure Blue LB

And combinations of levelling agents SLOVASOL O with SPOLION 8 (Figure 20) and SLOVAPON N with SPOLION 8 (Figure 21) were used.

1.2% (according to the weight of the material to be dyed) of each dye auxiliary was used.

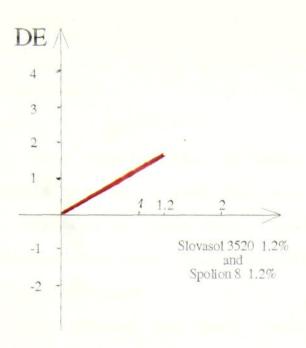


Figure 22: DE - Colour difference

Figure 22: Saturn Brown LT
The test was done on the samples, which are dyed with Saturn Brown LT by using levelling agent SLOVASOL 3520 with wetting agent SPOLION 8 1.2% (according to the weight of the material to be dyed) of each dye auxiliary was used. The result is shown on Figure 22.

Discussion 4

From the results in table 25a, we can see that the samples have get a good fastness to washing.

As can be seen from table 26 the best fastness to perispiration came from the test in alkaline solution than in acid solution.

To obtain the best result, it was decided that to repeat these tests on another samples.

Discussion 5

As it is shown in table 25b the best result (i.e. the best fastness to washing) is came from the sample i (the same as sample d in table 25a) which shows again the positive influence of the combination of 1.2 % Spolion 8 with 1.2 % Slovapon N.

But as we can see in table 26 and 27, there is a great difference in results of these two similar tests. It would be better if other samples were tested again to obtain the best possible results. But because of the shortage of the time that could not be done.

4.4. EVALUTION OF MIGRATION TEST

The two materials (dyed and undyed) were washed together at 60°C and 80°C. Washing liquor obtains all auxiliary chemicals but contained no dyestuff (blank bath).

The amount of dye which migrated to the undyed cloth, was observed.

The migration of the dye into the undyed material was greater at 60°C than at 80°c. At each temperature, there was no visible difference in the amuont of migration with different combinations of auxiliary chemicals.

This diploma work investigated the dyeing of cellulosic fibres at low temperatures and period in a dye bath.

Different types of levelling agents were used to give a better evenness at these low temperatures.

It is already known that good results can be obtained at a temperature of 100°C without levelling agents. To fulfil the aim of this diploma work, different types of levelling agents and their combinations are used and good results are obtained at temperature of 80°C. These good results are occured based on the following dyestuffs:

Saturn blue LBRR

Saturn violet L4B

Saturn khaki G

Saturn brown 2LG

and levelling agents :

Slovapon N in combination with Spolion 8

Slovasol O in combination with Spolion 8

In some exceptional cases, it is possible to get a good results even at 60°C, those are occured by the dyestuffs:

Saturn blue LBRR

Saturn brown 2LG

Saturn red LG

and levelling agents :

Slovapon N in combination with Spolion 8

Slovasol O in combination with Spolion 8

But the colour fastness is relatively poor and less amount of dye is taken from the dye liquor.

In general dyeing at low temperature may be taken, when there is an extraordinary energetic and other conditions (for example luck of steam), when it is necessary to take this operation with those negative results.

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