

1. Qualitative analysis of fibers in blended fabric

The analysis of fibers of blended fabrics is very often test in practice. The analysis is proceeding in two steps. The qualitative analysis is the first step. Second step is quantitative analysis, which is designed according with results of qualitative analysis. The mixture proportion of fibrous components in blend is analyzed in second step; the result can be e.g. "cotton/polyester 65/35".

1.1 Qualitative analysis of fibers

Experimental part

The aim: Determine the type of fibers in samples

Used chemicals:

Formic acid (HCOOH) conc. (dangerous caustic, cautious, be careful!)

Acetone

Used materials:

Samples of fibers (cotton, wool, polyester, polyamide, acetate and triacetate, viscose)

Two times fiber samples for qualitative analysis

Working procedure:

1. Prepare mount of fibers for the microscopy in samples 1 – 7. Estimate natural and synthetic fibers. Determine the type of natural fibers exactly.

The microscopy of fibers in lengthwise view:

ribbon = cotton (less clear in the case, when the cotton is mercerized)

lengthwise grooves = viscose or fibers with cross sections modification

scales = wool

without surface changes (smooth) = synthetic fibers (can contain small dots – it is matting agent in mass of fiber)

2. Dissolving test

Work on the following rules that you should work the most effectively to carry out of qualitative analysis. Testing is made only with minimal amount of fibers – e.g. unravel yarn with the length of 5 mm. Try the warp and weft separately. It can be made of different materials. During analysis you act according to following scheme:

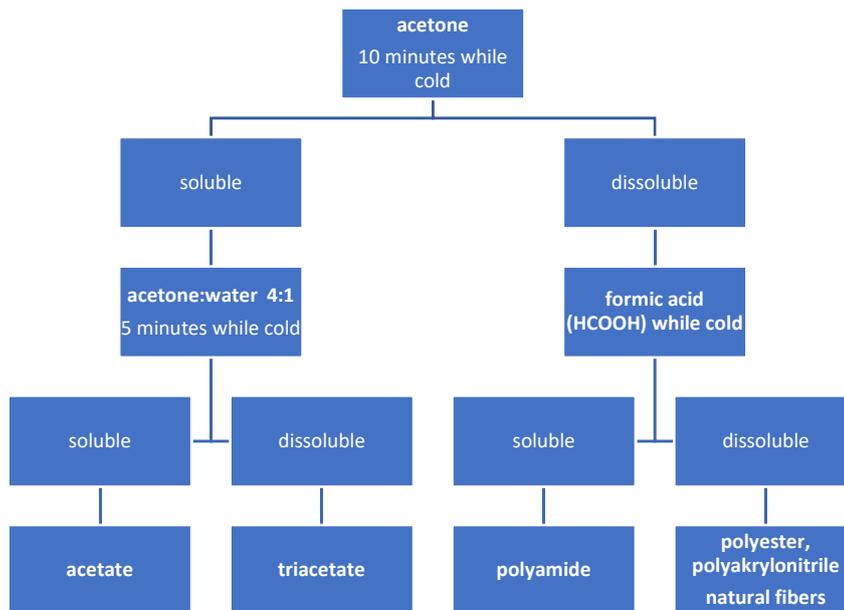


Figure 1. The scheme of qualitative analysis of fibers

Discuss your results with head instructor!

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1) Recommendation to drawing up a report from experiment

Make the report of used methods and your observation during experiment.

1.2 Quantitative analysis of fiber's blends

Analysis of fiber mixtures is very often a required test in practice. The analysis of the components must take place in two stages. The first is to perform a qualitative analysis and according to our components to choose a procedure for quantitative analysis. For time reasons, this exercise is based on the known composition of textiles.

Experimental part

Used chemicals:

Sulphuric acid (H_2SO_4) 75% (be carefull!)

Solution of ammonium (roztok 80 ml.l^{-1})

Material:

Sample of fabric polyester/cotton for quantitative analysis (1g)

standard ČSN EN ISO 1833

The aim: Determine the quantitative composition of the polyester/cotton fabric

Working procedure

1/ Quantitative analysis of polyester/cotton blend

Follow ČSN EN ISO 1833 (a similar procedure is also part of "Regulation (EU) No. 1007/2011 of the European Parliament and of the Council of 27 September 2011") for time reasons it is necessary to follow a simplified method: weigh the fabric after only a short drying (105 ° C, 15 min). Furthermore, assume that impurities and foreign matter are present in negligible amounts, so extraction with light petroleum can be omitted. Work with only one sample. These simplifications reduce the accuracy of the determination. The result of the analysis is to be considered as indicative only.

2/ Mathematical data processing

From the weighing results, determine the fiber contents in%, according to the description given in the general instructions in ČSN EN ISO 1833-1, both in the dried fabric and in the fabric with a standardized moisture content. For the calculation, use a moisture surcharge of 0.7% for polyester and 8.5% for cotton. ... Calculation from standard...

Procedure based on net dry weight

$$P = \frac{100m_1d}{m_0}$$

- P is the percentage of pure dry insoluble component
 m_0 is the dry mass of the test sample
 m_1 is the dry mass of the residue
 d correction factor for the change in mass of the insoluble component in the reagent ($d = 1$)

Procedure based on net dry weight with percentage moisture gain

$$P_M = \frac{100P(1 + 0,01a_2)}{P(1 + 0,01a_2) + (100 - P)(1 + 0,01a_1)}$$

- P_M is the percentage of pure insoluble component, including the percentage moisture surcharge
 P percentage of pure dry insoluble component
 a_1 moisture surcharge to the soluble component in percentage ($a_1 = 8.5 \%$)
 a_2 moisture surcharge to insoluble component in percentage ($a_2 = 0.7 \%$)

3/ Recommendations for elaboration of the protocol from the experiment

Describe in the protocol the procedure for the quantitative analysis of the polyester / cotton mixture. Present the calculation of the composition of the mixture and its result for the dry sample and for the sample with the moisture surcharge included.

2. Damage to wool and cotton fibers

Each type of fiber has a different chemical composition and therefore different chemical properties. From a practical point of view, there are significant differences in the resistance of the fibers to oxidative-reducing chemicals (possible bleaching problems) and in particular in the resistance to acids and alkalis (possible problems in dyeing, finishing and washing fabrics). Sensitivity to acids and alkalis differs markedly between cotton and wool fibers.

2.1 Evidence of wool fiber damage

Wool is sensitive to heat, alkalis and oxidation.

The structure of wool fibers is very complicated - the keratin macromolecules contained almost 20 different amino acids and the above-molecular structure of the fiber is also complex (epicuticula, cortex...). Some damage mechanisms only result in damage to selected parts of the fiber. As a result, a wide range of tests focusing on different fiber damage components and mechanisms must be used in the fiber damage analysis.

The most common principle used in chemical analysis of wool damage is the monitoring of its solubility in various solvents, which vary with the degree of damage. For example, caustic solubility is recommended for oxidative damage. Another method is the method of solubility in urea - pyrosulphite. It is important to note that the original raw wool should be available for safe conclusions of the dissolution methods.

Another approach to analysis is the reaction of metal ions with a damaged wave. An example is **the reaction with tin chloride (SnCl₂)**. This method is used to demonstrate the damage to wool fibers. The amino acid contained in keratin (cystine) is involved in the proof of damage to wool fibers. This amino acid contains a disulfide bridge. The action of the alkali disrupts the disulfide bridge and, following treatment in the tin chloride solution, the damage is become evident

by a brown to brown-black coloration of the coloration sulphide formed at the damaged sites.

Methylene blue dyeing is recommended for the proof of oxidative damage. Damaged sites become more dyed due to epicuticular damage and an increase in the number of available carboxyl groups in the wool.

2.2 Evidence of cotton fiber damage

Incorrect technological processes, unsuitable storage conditions or use of textile products can cause the damage, which is usually caused by damage to the fiber itself. The nature of the damage depends on both the factors of the damage of the product and the type of fibers and their properties. In practice, chemical damage of fibers is the most common.

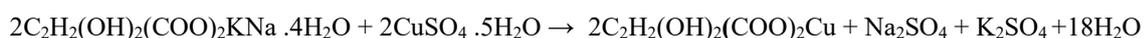
This part deals mainly with chemical tests. The damage-proving reactions serve to the identify the causes of the damage caused by improper handling of the textile material. This can be used for final and in-process control. In most cases, the reactions of the test material should be compared with the undamaged sample.

Cellulose fibers, both native, such as cotton, and chemical, such as viscose, are readily damaged by acids; they are further damaged by oxidizing agents and alkali solutions while boiling in the presence of atmospheric oxygen. All these factors cause the degradation of cellulose, which is characterized by shortening of macromolecular chains and also leads to a decrease in mechanical strength.

Acid damage

If cellulose fibers are damaged by acids, the reducing capacity of the cellulose increases as aldehyde groups —CHO are formed at the disrupted points of the chain, which can be used to prove cellulose damage. This mixture of differently degraded cellulose is called hydrocellulose. The degree of depolymerization is proportional to the intensity of the damage. The external feature is a decrease in strength to complete fiber breakdown. The fastest hydrolysis of cellulose is caused by inorganic acids (even considerably diluted).

The proof of reducing groups in hydrocellulose may be based on reaction with Fehling's reagent. The Fehling's reagent is an alkaline solution of a divalent copper salt which is maintained in solution by a complexing agent, in this case a Seignette salt, i.e. potassium sodium tartrate. It is prepared just before use by mixing Fehling I (60 g.l⁻¹ CuSO₄.5H₂O) and solution II (200 g.l⁻¹ sodium potassium tartrate + 100 g.l⁻¹ NaOH) solutions. in a 1: 1 ratio.



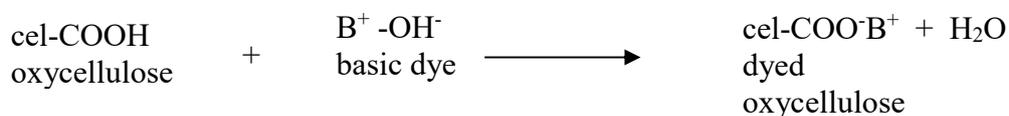
It is very sensitive agent to reducing effects. The reduction of the aldehyde (-CHO) group contained in the hydrocellulose by the Fehling reagent proceeds to form a carboxyl (-COOH) group in the cellulose and precipitation of cuprous oxide (Cu₂O) on the fabric. It shall be assessed visually.

Note: Alternatively, the test can also be evaluated quantitatively using the so-called copper number = the amount of copper that is reduced by 100 grams of absolutely dry material. In this case, the Cu₂O excreted sample is overlaid with a solution containing excess Fe³⁺ in a strongly acidic environment - under these conditions all Cu⁺ ions are oxidized to Cu²⁺ in cold conditions, thus reducing the equivalent amount of Fe²⁺ ions that can be titrated with KMnO₄ (Mn⁷⁺ is reduced by titration with ferrous ions to a colorless ion Mn²⁺).

Oxidative damage

If cellulosic fibers are damaged by oxidizing agents, aldehyde groups are formed initially at the ends of the individual cellulose chains, but they are subject to further oxidation and are converted to carboxyl groups —COOH. This mixture of oxidatively degraded and parent cellulose is called oxycellulose. Oxycellulose has the ability to bind well cationic or basic dyes, such as methylene blue.

The action of oxidizing agents on cellulose in an alkaline environment (by the action of airborne O₂ during alkaline cotton production, intensive hypochlorite bleaching, or staining of the material with a concentrated NaClO solution) produces acid-type oxycellulose containing carboxyl groups —COOH. These cause increased affinity of oxycellulose for cationic (basic) dyes:



Dyeing with methylene blue solution at elevated temperature, time 2 min, concentration 1 g.l^{-1} is recommended. Damaged areas become more stained.

Experimental part:

The aim 1: Damage of natural fibers by acids and alkalies

Chemicals:

Sodium carbonate (Na_2CO_3)

Sulphuric acid (H_2SO_4), solution 100 g.l^{-1}

Acetic acid (CH_3COOH), solution 20 g.l^{-1}

Tin(II) chloride (SnCl_2), solution 20 g.l^{-1}

Fehling agent I a II

Materials:

Cotton fabric (sample size approx. $5 \times 10 \text{ cm}$)

Wool fabric (sample size approx. $5 \times 10 \text{ cm}$)

Working procedure:

1) Exposure of fibers the action of acid and alkali

Take sample of cotton and wool fabric and unravel some threads from each side of sample. Then you have to measure the size of samples by the ruler precisely ($\pm 0.5 \text{ mm}$). Exposure samples the action of acid and alkali parallel.

A) Exposure the action of acid

Warm up 100 ml of sulphuric acid at conc. 100 g.l^{-1} at the beaker to approx. 80°C (beaker stands in boiling water on cooker). Put cotton and wool sample into the beaker for 10 minutes simultaneously. Then rinse samples in water and dry it at 105°C .

B) Exposure the action of alkali

Samples of cotton and wool fabric put into 100 ml solution of Na_2CO_3 20 g.l^{-1} at temperature of 80°C (beaker stands in water) for 10 minutes simultaneously. Then rinse sample in water and dry it at 105°C .

2) Determine the shrinking of sample

Measure the size of all samples of fabric after the action of acid/alkali. Quantify the size changes of textiles according this formula $\% \text{ of dimensional change} = 100 \times (\text{length of sample after treatment} - \text{length of sample before treatment}) / (\text{length of sample before treatment})$ in standard EN ISO 5077 ("Determination of dimensional change after washing and drying").

3) The proof of damage of wool by tin chloride

All three samples (undamaged wool and damaged wool from experiment 1A and 1B) dip suddenly into the beaker at 80°C for 10 minutes (beaker stands in boiling water). The volume of solution is 100 ml containing 2 g.l^{-1} acetic acid and 2 g.l^{-1} tin chloride. Then samples rinse in water intensively. Dry them in dryer at 105°C . Observe the color changes of samples after the testing.

4) The proof of damage of cotton by Fehling reagent

Prepare beaker with Fehling reagent by straining 30 ml of Fehling solution I and 30 ml of Fehling solution II. All three samples of cotton fabric (undamaged cotton, cotton from experiment 1A and 1B) dip into the beaker containing Fehling reagent I and II at 80°C for 10 minutes (beaker stands in boiling water). You can observe that damaged cotton fabric changes the color to rusty-brown. It is so-called hydrocellulose.

5) Recommendation to drawing up a report from experiment

Arrange samples of cotton fabric after the reaction with Fehling reagent and wool fabric after the reaction with tin chloride, including origin undamaged samples of woven fabrics in your report.

The aim 2: The proof of oxidative damage of natural fibers by methylene blue

Used chemicals:

Methylene blue 1 g.l⁻¹

Sodium hypochlorite (NaClO), solution, minimally 100 g.l⁻¹ active chlorine

Used materials:

Cotton fabric (size of sample approx. 5 x 10 cm)

Wool fabric (size of sample approx. 5 x 10 cm)

Working procedure:

1/ Damage of natural fibers by oxidative agent

Place samples of cotton and wool fabric on Petri dish. Spread small drop of sodium hypochlorite solution (undiluted) on cotton and wool fabric by glass rod or small pipette. Put Petri dishes into the dryer for 3 minutes at 105°C. Rinse samples in flowing water thoroughly.

2/ Dying test

Dye oxidative damaged samples of cotton fabric in 25 ml of cold water solution of methylene blue at conc. 1 g.l⁻¹ for 2 minutes. Place both sample into one bath. Then rinse samples in warm water as long as there is no dyestuff in rinsed water. It is possible to conclude to the level of chemical damage of sample according to the result saturation of coloration.

3/ Recommendation to drawing up a report from experiment

Arrange samples of cotton fabric and wool fabric after dyeing by methylene blue in your report.

3. Merceration and dyeing of cotton

3.1. Merceration and causticizing

Alkaline finish of cotton materials has great importance in the technology of cotton fabric processing and especially sewing threads, especially in view of the current high demands on the quality of the final products.

Mercerization is a finishing process in which cotton fabrics or yarns are treated with concentrated lye, i.e. sodium hydroxide NaOH at a concentration of about 25%, while stretching the material. The greatest shine can be achieved when the cotton material is stretched up to 5% above its original length. Tensioning of material may only be released after subsequent rinsing with water, when NaOH concentration falls below 10 %.

Short exposure times (approx. 1 minute) are sufficient for mercerization.

During the mercerization, the material is processed in cold caustic solutions, the optimal processing temperature being 15 ° C. For thicker fabrics, on the other hand, it is advisable to use higher temperatures (60 ° C), during which the mercerization takes place in the whole volume of the fabric faster than in cold conditions.

Mercerizing cotton fibers improves a number of their properties, namely:

gloss, tensile strength (up to 25 % - significant for cotton yarns), dyeability, cross-linking reactivity, hygroscopicity (up to 1.5 x) - common cotton contains 8 % water at 20 °C and 65 % relative humidity, mercerized around 12 %. Shrinkage (contraction) is reduced; Improves touch (soft and full) and dimensional stability. However, the abrasion stability is slightly worsened and the ductility is reduced.

These changes in properties are related to chemical transformation, transformations in supramolecular structure and shape changes of cotton fiber. These conversions are caused by the action of concentrated NaOH solutions on the cellulose fiber, which produces alkali cellulose and leads to strong swelling.

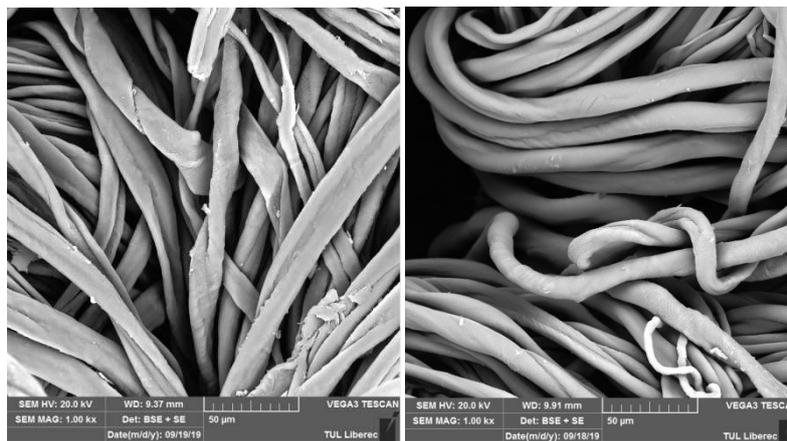


Figure: Effect of 25% NaOH treatment on cotton, original fibers on the left, stress-free alkali on the right (causticizing)

Mercerized cotton is essentially a hydrated cellulose having certain distinct properties. It is more hygroscopic and sensitive to elevated temperature. It has a higher content of amorphous content and an altered crystal lattice, which means an increase in the accessible inner surface of the fiber. The result is an increase in sorption capacity, i.e. an increase

in dye affinity. For the mercerization result, it is important that the infiltration of sodium hydroxide into the fiber is rapid and uniform. For better penetration of NaOH into the fiber, the mercerization baths contain an effective wetting agent with minimal foaming and excellent stability in alkalis.

Mercerization is always followed by efficient washing in warm water (60 ° C), neutralization by dilute acidification and washing again.

Leaching: For textiles that do not require increased gloss and strength, but only shrinkage and increased dye affinity, only leaching is performed. It is processing of cotton fabrics or knitted fabrics in cold sodium hydroxide, i.e. NaOH at a concentration of 20 to 25 %, without tension for about 1 minute. Under these conditions, swelling occurs with the precipitation of the fiber. The leaching is followed by washing, acid neutralization and re-washing as in mercerization. Leaching produces fabrics with high elasticity required for sportswear or crepe effect fabrics. It also serves to eliminate immature and dead cotton fibers, compensates for structural differences, reduces color streaking.

Experimental part

The aim: Observe the influence of causticizing on the dyeability of cotton

Used chemicals:

Acetic acid (CH₃COOH), 20g.l⁻¹

Sodium hydroxide (NaOH), 25%

Substantive dyestuff, e.g. Direct Blue L4G (C. I. Direct Blue 78), 10 g.l⁻¹

Sodium chloride (NaCl), 50 g.l⁻¹

Used materials:

Cotton fabric for soaking (size of sample approx. 5 x 5 cm)

Cotton fabric for fastness (size of sample approx. 8 x 8 cm)

Grey scale

ČSN EN ISO 105-X11 Test of color fastness – Ironing fastness

Working procedure:

1. Calculation of the amount of NaOH

Calculate the amount of solid sodium hydroxide NaOH for weighing to obtain 100 g of 25 % solution of NaOH. Consult the calculated result with head instructor.

2. Check up the concentration of solution of sodium hydroxide NaOH

You have to check up the concentration of sodium hydroxide by measuring of the density. Fill up the measuring cylinder (volume 10 ml) of tested solution of NaOH with volume 10 ml. Determine the weight of solution by using of the weight. At the end of this manual you can find the table with the concentration of NaOH in the solution.

3. Causticizing

Take Petri dish with diameter approx. 8 cm. Put on it sample of cotton fabric into 10 ml 25 % solution of NaOH for 1 minute. Take out the sample by tweezers and wash it at warm water

at 60 °C and make a neutralization with 50 ml of solution of acetic acid at conc. 20g/l. Then rinse sample in warm and cold water.

4. Dyeing test

Verify the increasing dyeability by dyeing test: soaking sample (wring thoroughly after last rinsing) and comparably size of sample un-causticized cotton fabric dye together for 2 minutes at 80°C (beaker stands in boiling water) in 100 ml dyeing bath containing:

2 g.l⁻¹ substantive dyestuff (e.g. Direct Blue L4G)

5 g.l⁻¹ sodium chloride NaCl

And rinse the sample after dyeing.

Notice the difference in dyeability. It can be observed immediately at the beginning of the dyeing process.

5. Testing of fastness (simplified „Ironing fastness“)

Put wet dyed textiles (size approx. 5x5cm) between two pieces of white cotton fabric (size approx. 8x8cm) and intensively iron by warm iron (or if it is possible to use the thermo press) at temperature of 110°C. Study the standard for ironing fastness. Evaluate results according to gray scale.

6. Recommendation to drawing up a report from experiment

Evaluate samples of soaked and un-causticize material and also both samples after dyeing and arrange them in your report.

Density g.cm ⁻³	Concentration NaOH %	Density g.cm ⁻³	Concentration NaOH %	Density g.cm ⁻³	Concentration NaOH %
1,000	0,159	1,180	16,44	1,360	33,06
1,005	0,602	1,185	16,89	1,365	33,54
1,010	1,04	1,190	17,34	1,370	34,03
1,015	1,49	1,195	17,80	1,375	34,52
1,020	1,94	1,200	18,25	1,380	35,01
1,025	2,39	1,205	18,71	1,385	35,50
1,030	2,84	1,210	19,16	1,390	36,00
1,035	3,29	1,215	19,62	1,395	36,49
1,040	3,74	1,220	20,07	1,400	36,99
1,045	4,20	1,225	20,53	1,405	37,49
1,050	4,65	1,230	20,98	1,410	37,90
1,055	5,11	1,235	21,44	1,415	38,49
1,060	5,56	1,240	21,90	1,420	38,99
1,065	6,02	1,245	22,36	1,425	39,49
1,070	6,47	1,250	22,82	1,430	40,00
1,075	6,93	1,255	23,27	1,435	40,51
1,080	7,38	1,260	23,73	1,440	41,03
1,085	7,83	1,265	24,19	1,445	41,55
1,090	8,28	1,270	24,64	1,450	42,07
1,095	8,74	1,275	25,10	1,455	42,59
1,100	9,19	1,280	25,56	1,460	43,12
1,105	9,64	1,285	26,02	1,465	43,64
1,110	10,10	1,290	26,48	1,470	44,17
1,115	10,55	1,295	26,94	1,475	44,69
1,120	11,01	1,300	27,41	1,480	45,22
1,125	11,46	1,305	27,87	1,485	45,75
1,130	11,92	1,310	28,33	1,490	46,27
1,135	12,37	1,315	28,80	1,495	46,80
1,140	12,83	1,320	29,26	1,500	47,33
1,145	13,28	1,325	29,73	1,505	47,85
1,150	13,73	1,330	30,20	1,510	48,38
1,155	14,18	1,335	30,67	1,515	48,90
1,160	14,64	1,340	31,14	1,520	49,44
1,165	15,09	1,345	31,62	1,525	49,97
1,170	15,54	1,350	32,10	1,530	50,50
1,175	15,99	1,355	32,58		

3.2. Dyes and dyeing of cotton

Dyes are divided into numerous groups, respectively. classes according to two fundamentally different approaches.

From the chemical point of view, i.e. according to the chemical structure of dyes (so-called chromophore is typical here), their chemical properties and method of application, we divide dyes into several chemical groups, some of which are: nitro-dyes and nitroso-dyes; azo dyes; anthraquinone dyes; vat dyes - indigoid and anthraquinoid; sulfur dyes; phthalocyanine dyes; thiazine dyes. For each group, synthesis or synthesis are common or very similar, and then their chemical resistance, e.g. all azo dyes are easily degradable by reduction. In addition to the shade, colorist, i.e. technological, application properties of the dyes - solubility, behavior in solutions and dyeing processes - are essential for dye users. Therefore, the classification into color classes according to the use of dyes is much more widely used in practice, which allows sufficient orientation in the application of dyes to textile or textile dyes or other materials. The common feature here is the nature of the bonding forces with the fibers, i.e. the same way of dyeing.

However, one colorist class, such as direct or acid dyes, cationic or vat dyes, may contain dyes from different chemical groups - only "linking groups" must be the same. A single chemical group of a dye can be present in multiple color classes, for example, azo dyes are among direct, reactive, acidic, metal complex, disperse and cationic dyes.

From a coloristic point of view, we therefore divide textile dyes into the following technological, ie, colorist classes: substantive or direct dyes; sulfur dyes; vat dyes; developed on the fiber; acid dyes; acidic metal complex dyes; reactive dyes; disperse dyes; cationic or basic dyes; pigments; (optically brightening agents); dyes with special purpose such as fat and wax soluble dyes, dyes for dyeing leather and fur, foodstuffs, metals, wood, etc.

3.2.1. Reactive dyes

Reactive dyes are an important group of dyes for dyeing cellulose fibers and, to a lesser extent, wool and polyamide fibers. They are anionic dyes, similar to acid or direct dyes. The reactive dyes contain one or two reactive groups or atoms in the molecule, which are capable of chemically reacting with the -OH groups of cellulose fibers or the -NH₂ groups of wool and polyamide fibers under appropriate conditions to form a strong covalent bond. Reactive dyes are characterized by high shade brilliance, ease of application and high wet fastnesses, which are due, among other things, to the covalent bonding of the reactive dye with cellulose, respectively. with keratin wool or polyamide.

The reactive dye has a complicated structure. In addition to the parts responsible for coloring (C), we can also find solubilizing groups - SO₃Na (S), which cause the colorant to dissolve in water. This colored part of the dye molecule is linked via a bridge (B) to a reactive system (R) (here based on 1,3,5-triazine), where we can find individual reactive groups or atoms (X) capable of chemically binding to cellulose.

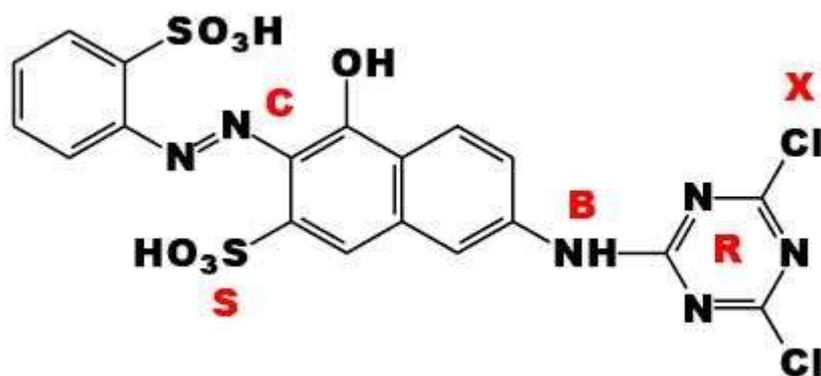


Figure: Labeling of basic reactive dye parts (https://www.researchgate.net/figure/Figure-3-Generic-structure-of-a-fiber-reactive-dye_fig1_324819332)

The mechanism of dyeing with reactive dyes is rather complicated. From a chemical point of view, it is a nucleophilic substitution. From a practical point of view, it is necessary to know that the reactive dye will only react with cellulose in an alkaline environment where the cellulose is present in the form of so-called alkalicellulose, in which some of the -OH groups are ionized (instead of the -OH groups are -O⁻). Alkalicellulose is a nucleophilic agent. Unfortunately, water molecules behave similarly in an alkaline environment (OH⁻ instead of H₂O), which also react with reactive dye molecules to form a dye hydrolyzate that is no longer capable of binding to cellulose and only stains cotton. For this reason, very intensive washing should be included after reactive dyeing. This is important so that the high color fastness is not only spoiled by the labile non-reactively adsorbed portion of the dye.

To minimize dye hydrolysis, alkaline dye solutions cannot be prepared beforehand and the textile material should be incorporated into the dye immediately after preparation. However, some of the reactive dye is hydrolyzed. Dyes with two reactive groups (bifunctional dyes) are used to suppress the effect of hydrolysis.

Experimental part

The aim: Dye cotton fabric by reactive dyestuff

Used chemicals:

Reactive dyestuff (e.g. Reactive red 198, attention: dyestuff can be toxic)

Sodium chloride (NaCl)

Sodium carbonate (Na₂CO₃)

Wetting agent (e.g. Spolion 8), 10 g.l⁻¹

Used materials:

Cotton fabric for dyeing (size of sample approx. 5 x 5 cm)

Cotton fabric for testing of fastness (size of sample approx. 8 x 8 cm)

Pattern book of reactive dyestuff (to browse through)

ČSN EN ISO 105-X11 Testing of color fastness - Ironing fastness

Gray scale

Working procedure:

1. Prepare dyeing bath for 1 gram sample of cotton fabric following content:

- 1 % reactive dyestuff (from weight of fabric), e.g. Reactive red 198
- 25 g.l⁻¹ sodium chloride NaCl (electrolyte)
- 20 g.l⁻¹ sodium carbonate Na₂CO₃ (alkali)
- 1 g.l⁻¹ wetting agent (e.g. Spolion 8)
- Liquor ratio 1:50

2. Dyeing of cotton fabric

Prepare the same volume of the bath without alkali parallelly! Add the alkali closely before dyeing of the cotton fabric. You will dye two samples of cotton at weight of fabric 1 g in the bath with 50 ml. One sample will be dyed in dyeing bath with alkali and the other without alkali. You will dye cotton samples at 60 °C for 20 minutes. After dyeing remove and rinse dyed cotton samples in cold and warm running water to remove unfixed dyestuff.

3. Scouring after dyeing

Make the soaping of dyed samples in the bath containing 2 g.l⁻¹ textile auxiliary agent (e.g. Syntapon ABA). The bath has to be boiled and sample will be scoured for 10 minutes. Then rinse samples in warm and cold warm water.

4. Fast test of fastness – Fastness in ironing

Place wet dyed cotton sample (size 5x5 cm) into pieces of white cotton fabric (size 8x8 cm) and iron (alternatively you could use the thermo-press). You will use the thermo-press and the temperature will be set to 150 °C for 15 seconds. Read and study the standard (ČSN EN ISO 105-X11) Textiles - Tests for color fastness - Part X11: Colour fastness to hot pressing. Evaluate degree of bleeding of dyestuff into dry accompanying cotton fabric according the gray scale.

5. Suggestion to drawing up the report

Attach dried colored cotton samples to your report and evaluate the influence of alkali to dyeability of cotton fabric. Demonstrate the result of fast test of fastness – Fastness in ironing.

3.2.2. Vat dyes

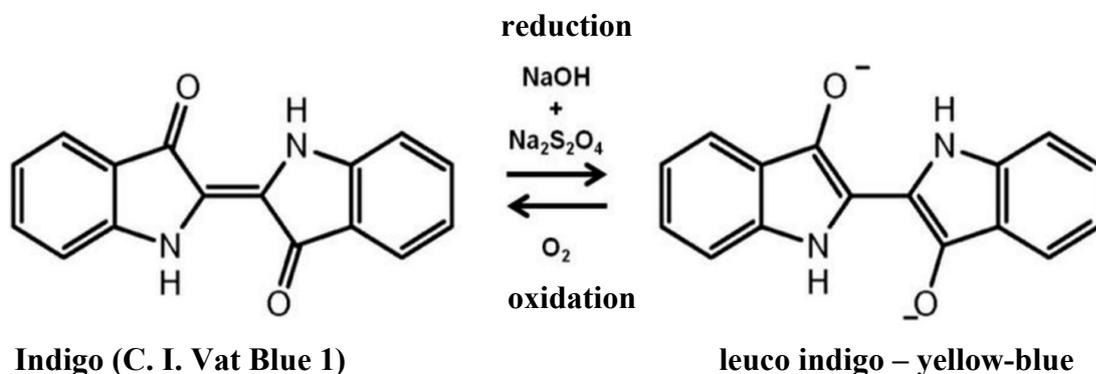
Vat dyes represent a shade-wide group of cellulose fiber dyes. They are characterized by high color fastnesses, both light fastnesses and so-called wet fastnesses, such as water fastnesses, wash fastnesses, etc. Furthermore, they are characterized by a high coloring ability and a wide variety of shades. It is suitable for demanding cotton assortments, which are repeatedly developed (bedding, tablecloths, towels, shirts) or must be maximally weather resistant (flags, beach assortment).

All vat dyes are practically insoluble in water and therefore, for dyeing, they must be converted by watering to a water-soluble cellulosic fiber affine form, the so-called leuco compound

of vat dye, which is, however, considerably different from the original dye. Sprinkling consists in the action of a reducing agent, almost always sodium dithionite $\text{Na}_2\text{S}_2\text{O}_4$, and an alkaline sodium hydroxide NaOH , thereby converting the leuco acid into a soluble sodium salt. The dye bath thus obtained is called **vat**. The leuco compound stretches substantively on the fiber - similarly as direct dyes.

After the "withdrawal" from the bath to the fiber, the leuco compound is converted by oxidation under a possible conditions with mild acidification back to vat dye insoluble in water. Oxidation of the leuco compound usually takes place already during washing with water by the action of air oxygen, it can be accelerated by the action of oxidizing agents. Surface-adhering dye is removed by additional soap. Due to the total water insolubility of this oxidized dye, which is finely dispersed in the pores of the fiber after dyeing and oxidation of the leuco acid, these dyes have high so-called wet stability even in hot baths.

Chemical reactions occur during the loosening process. First, the keto group $\text{C}=\text{O}$ is converted by reduction to the so-called enol group $\text{C}-\text{OH}$, and this is immediately converted to the $\text{C}-\text{ONa}$ group due to the NaOH present. The resulting leuco compound is converted to the original vat dye after oxidation staining. E.g. for indigo, the reactions are as follows:



Experimental part

The aim: The vat dyestuff for dyeing of cotton fabric

Used chemicals:

Vat dyestuff (C.I. Vat Yellow 2, dyestuff on the basis of anthraquinone, powder, **attention: dyestuff can be toxic**)

Sodium chloride NaCl (solid or solution 100 g.l^{-1})

Sodium carbonate Na_2CO_3 (solid or solution 50 g.l^{-1})

Sodium hydroxide NaOH 38 °Bé (32% !!!, **high concentration = dangerous**)

Sodium hyposulphite ($\text{Na}_2\text{S}_2\text{O}_4$)

Dispersing agent (e.g. Spolion 8, Synferol AH), 10 g.l^{-1}

Used materials:

Cotton fabric for dyeing (size approx. $5 \times 5 \text{ cm}$)

Cotton fabric for fast test of fastness (size approx. cca $8 \times 8 \text{ cm}$)

Pattern book of vat dyestuffs (find the color of leuco-compound)

ČSN EN ISO 105-X11 Dye fastness – Fastness in wet ironing

Working procedure:

1. Prepare dyeing bath for vat dyestuff according the recipe:

0,2 g dyestuff (stir it with dispersing agent in the beaker)
2 ml solution of dispersing and wetting agent 10 g.l⁻¹ (e.g. Spolion 8)

Pour:

30 ml warm water temperature at 60 °C

Add:

0,8 ml sodium hydroxide NaOH 38 °Bé
0,4 g sodium hyposulphite Na₂S₂O₄ (**do not forget to stir it!**)

Give the beaker into the water and let it at the temperature of 60 °C for 10 minutes. Find the color of leuco-compound of used dyestuff in the pattern card.

2. Dyeing of cotton sample

Put the cotton sample into the dyeing bath at 50°C for 15÷ 20 minutes. Then remove the sample and rinse it in the running water. This step supports the oxidation process of dyestuff.

3. Finish treatment of cotton sample

Put dyed cotton fabric into beaker with the solution of acetic acid at conc. 2 g.l⁻¹. It removes the rest of alkali from the sample. Launder sample in the soaping bath at the boiling for 1 minute. The soaping bath contains 1 g.l⁻¹ of wetting agent (Syntapon ABA). At the end rinse the sample in the water a dry it on the air or in the laboratory drier.

4. Fastness in wet ironing - the same process at previous.

You will try fastness in wet ironing of your vat dyed cotton fabric. The process will be the same as in previous case.

6. Suggestion to drawing up the report

Attach dried colored cotton samples to your report and evaluate vat dyed cotton fabric. Demonstrate the result of fast test of fastness – Fastness in ironing.

4. Synthetic fibers

Heat setting of polyamide fibers

The crystallinity of polymers refers on a group of usable and manufacturing properties of synthetic fibers. The main role has the content of crystallize regions of polymer in the fibers. The amorphous regions of polymer are not accessible to dyestuffs and other chemicals. So-called heat setting can influence the crystallinity of polymer. Fiber is exposed to high temperature (from 140 up to 200 °C) for short time (approx. 30 seconds). It causes the re-crystallization of polymers at the same time. A lot of tests can prove heat setting of fibers e.g. iodine sorption value. Iodine can easily penetrate into fibers at ambient temperature from aqueous solution into amorphous regions of fibers. The content of amorphous proportion of synthetic fibers is decreased due to thermal fixation. Iodine sorption value is easy way to evaluate the level of the fixation. There is possible to make quantitative determination of the level of heat setting.

Experimental part

The aim: Make a fixation of polyamide fabric and observe the changes of its properties

Used chemicals:

Disperse dyestuff for dyeing of polyamide (e.g. C. I. Disperse blue 56) or acid dyestuff for polyamide (e.g. C. I. Acid Red 118)

Used materials:

Un-fixed polyamide woven fabric for “testing of dimensional change” (sample size approx. 20 x 3 cm)

Un-fixed polyamide woven fabric for “testing of dyeing” (sample size approx. 5 x 5 cm)

Working procedure:

Izotonic fixation of polyamide fabric

You will take one sample for “testing of shrinking” and one sample for “testing of dyeing”. You can make cut-in of both samples for better identification (approx. 10 mm).

You will expose both samples of polyamide fabric to effect of high temperature in free state in the drier. You will use temperature at 190 °C for 5 minutes.

Treatment of samples after testing of dimensional change in washing and drying

You will measure the length of polyamide fabric for “test of shrinking” after fixation. Also, you will measure the length of polyamide fabric for “test of shrinking” without fixation. To prevent the split of fabric, you have to unweave the fabric. Do not cut project ends of threads from the fabric.

Determination of dimensional change in washing and drying

You will put both samples of polyamide fabric for “test of dimensional change in washing and drying” to boiled water for 5 minutes. Then dry them in dryer and measure the length of each of them. Quantify the relative dimensional change of sample in washing and drying in boiled water in % of size change= $100 \times \frac{\text{the length of sample after fixation} - \text{the length of sample before fixation}}{\text{the length of sample before fixation}}$.

Examine the standard ČSN EN ISO 5077 “Textiles - Determination of dimensional change in washing and drying”.

Dyeing of polyamide fabric in dyeing bath

You will put samples of polyamide fabric “for dyeing” (original and after heat setting) in one dye bath for 10 minutes simultaneously. The temperature is about 80 ° C (beaker standing in boiling water). Prepare a dye bath by mixing 0.10 g of disperse dye with 100 ml of water, or 0.10 g of acid dye and 0.10 g of $(\text{NH}_4)_2\text{SO}_4$ and 100 ml of water. You will dry samples at 105 ° C. You will observe the color shade of the samples.

Suggestion to drawing up the report

You will present the value of dimensional change in washing and drying of your samples at heat setting in your report. Discuss whether heat setting has improved the resistance of polyamide fabric against to level of dimensional changes in washing and drying. Adjust dyed samples in your report. Discuss the effect of heat setting on dyeability of polyamide fabric.

Proof of depolymerisation of polyamide fibers by dyeing test

Synthetic fibers exhibit relatively high chemical resistance. Damage to the synthetic fibers is most pronounced in the reduction of the polymerization degree, which leads to a decrease in the strength of the fibers.

Polyamide is dyed with almost all dye groups. Acid and disperse dyes have practical importance for dyeing of polyamide. You will use these dyestuffs for this test. Polyamide depolymerization shortens the polymer chain and increases the concentration of terminal amino groups.

Acid dyes bind to $-\text{NH}_2$ (or $-\text{NH}_3^+$) groups, the existence of which can only be expected to exist at the 1 end of the polyamide molecule. The maximum amount of dye in the polyamide therefore depends on the length of the macromolecules. Polyamide damage by acids is hydrolytic - it breaks down peptide bonds, thus shortening macromolecules and thus increasing the number of chain ends = binding points for the dye. In order to achieve real results, it is necessary to stain in **a neutral environment at about pH = 7 (without acidification of the bath)**.

Consequence: Acid-damaged areas of polyamide with acid dyes dye more intensely. In acidic dyes with more sulfo groups, the difference between damaged and undamaged sites is most pronounced.

Disperse dyes behave quite differently: hydrolytic damage to the polyamide is virtually not reflected in this dye class. This is due to the method of binding the dye molecules to the fiber (non-ionic interactions).

Acid and dispersion dyes are simultaneously applied in the actual tests used, while the damaged areas are more contrasted - not only a change in color saturation but also a significant shade shift.

Experimental part

The aim: Simulate the acid damage of polyamide fabric and demonstrate changes of dyeability properties caused by polymer hydrolysis

Used chemicals:

C.I. Acid Red 27 (acid dyestuff)

C. I. Disperse blue 56 (disperse dyestuff for dyeing of polyamide)

10 % H₂SO₄

Used materials:

Heat-set polyamide fabric (size of approx. 6 x 6 cm)

Working procedure:

1. Simulate the acid damage of polyamide fabric

Drip a drop of 10 % sulphuric acid (H₂SO₄). Let it wet to „natural“ forces. The area of damaged polyamide will be much more regular in this case. Put the sample into the laboratory drier at 105°C for 3 minutes.

The word "dry" is enclosed in quotation marks, because at 100 °C the water is only distilled from the solution and the acid concentration is increased. Interestingly, the azeotrope contains 98.7% H₂SO₄ and 1.3% H₂O and boils at about 338 °C. Under the above-mentioned "drying" conditions, it is expected to heat the H₂SO₄ solution to about 90 °C and increase the H₂SO₄ concentration to about 70%.

The "correctly" damaged polyamide is slightly spatially deformed in the dripping region - due to the contraction caused by the disruption of the supramolecular structure of the polyamide. Slightly visible edges of the stain are not a defect. If the polyamide is absent (dissolved) or there are significant structural changes in the fabric, you have damaged the polyamide too much - relax the conditions! (Note: evidence of damage on non-existent polyamide is not relevant).

Damaged sample of polyamide should be washed with water (no matter what temperature) and neutralized with the solution of soda (Na₂CO₃) with a concentration of about 10 g.l⁻¹. It is also advisable to rinse the sample with cold water again.

2. Dyeing tests

A) Viktoriarubin O (C.I. Acid Red 27, acid dyestuff with three sulpho groups)

Approx. 0.2 g.l⁻¹; 25 ml, 95°C, approx. 2 min, stir it

B) C. I. Disperse blue 56 (or other disperse dyestuff for polyamide fabric with contrasting shade)

Approx. 0.2 g.l⁻¹; 25 ml, 95°C, approx. 2 min, stir it

C) pour A) +B) together 25 ml, (0.2 g.l⁻¹ Viktoriarubin O and 0.2 g.l⁻¹ C. I. Disperse blue 56)

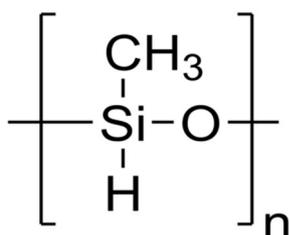
5. Wetting and surfactants

Modification of wetting properties of fibers

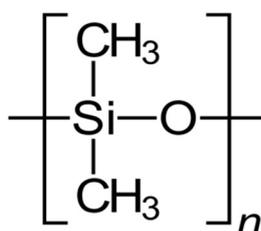
The behavior of textiles towards water is one of the key properties of textiles that affect not only the processability of textiles but especially their utility value. For fabrics in direct contact with the human skin of the application, it is advisable to use hydrophilic (wetable) fabrics that can drain and absorb large amounts of sweat. It is advisable to use hydrophobic (non-wetable) textiles for rain protection. The natural behavior of the fibers is modified for specific applications using final finishes.

Hydrophobic treatment

The wettability of the fabric is suppressed by the hydrophobic finish. Water repellency is provided to treated fabric. For garment purposes, it is used the finish keeping of air permeability of the fabric. The following chemicals are the most commonly used for these treatments: paraffin emulsions with aluminum or zirconium salts, higher fatty acid derivatives, silicones and perfluoroalkanes. Hydrophobic agents based on polysiloxanes (silicones such as dimethylpolysiloxane or hydrogenmethylpolysiloxane) are the most widely used because they give high-quality water-repellent effect. Treated fabrics have a soft and smooth feel and at the same time improve the creasing. Their disadvantage is the relatively high cost and sensitivity to the pretreatment of materials.



hydrogenmethylpolysiloxan



dimethylpolysiloxan

The evaluation of the hydrophobic finish is carried out according to a number of different procedures, such as the so-called Spray test or orientation drop test.

The drop test is the only indicative testing and serves to quickly assess the hydrophobicity / hydrophilicity of the fabric.

Hydrophilic treatment

A wide range of organic and inorganic substances with high polarity are used as hydrophilic finishes. Some of these finishes are durable and resist to washing cycles. Chemical surface treatment is an interesting solution for the hydrophilization of textiles by means of plasma. Similarly, alkaline hydrolysis of the polyester, in which sodium hydroxide (or a similar alkali) is applied, results in surface etching of the fiber associated with an increase in the number of hydrophilic end groups (-COOH, -OH) in the fiber's surface. This finish is specific to polyester and it is permanent finish.

Experimental part

The aim: Quantify the change of wettability of polyester fabric by drop test

Used chemicals:

Hydrophobizing agent based on silicones (possibly based on perflouralkanes)

NaOH (solution 50 g.l⁻¹, Caution! It is a highly corrosive / hazardous chemical. In case of contact with skin or eye, rinse with plenty of water.)

Used materials:

Polyester fabric (size of samples approx. 6 x 6 cm)

pH paper

Working procedure:

1. Hydrophobization of polyester fabric

Immerse the sample of polyester fabric in the hydrophobic solution. When using LUCOSIOL E35, use this preparation at a concentration of 1 g.l⁻¹, which can be prepared, for example, by mixing 0.10 g of LUCOSIOL E35 in 100 ml of water. Immerse the polyester fabric sample in this solution and remove the sample using a rubber roller, for example. Dry the sample at 105 ° C and then fix it at 150 ° C for 5 minutes.

If another hydrophobizing agent is available, refer to the supplier's instructions. For example, for Baygard EFN (flourocarbon) use a concentration of 20 g.l⁻¹, dry and fix the sample at 150 ° C for 5 minutes.

2. Hydrophilization of polyester fabric

Immerse the sample of polyester fabric in a 50 g.l⁻¹ NaOH solution. Immerse the polyester fabric sample in this solution and remove the sample using a rubber roller, for example. Dry the sample at 105 ° C and then expose it to high temperature (for 5 minutes at 150 ° C) to degrade the fiber's surface by alkaline hydrolysis. Wash the sample thoroughly under running water. Make sure that the alkali is removed with a pH paper.

3. Drop test

The core of the test is to apply a small drop of water (25 micro-liters) to the surface of the test fabric using a micropipette. The evaluation is performed by measuring the time required to inject the drop into the fabric. If the drop does not absorb into the fabric within 10 minutes, stop the experiment. Use the drop test to compare the wettability of the original polyester fabric, the hydrophobic fabric and the hydrophilic fabric.

4. Suggestion to drawing up the report

Present the absorption time of water drop in the report. You can take pictures of each water drops and present them in your report.

Determination of surfactant ionicity

Surfactants are substances whose common characteristic is surface activity. Therefore they are also called surfactants (SAA – surface active agent).

Surfactants reduce the surface tension of liquids and facilitate surface wetting. These specific properties of surfactants are determined by the chemical and physical structure of the molecules having the hydrophilic and hydrophobic moieties. The hydrophobic, i.e. the non-polar part, forms a usually long carbon chain. The hydrophilic, i.e. polar part, forms an ionic group, most commonly -COO^- , -SO_3^- , -OSO_3^- or a non-ionic group, eg $\text{-(CH}_2\text{CH}_2\text{O)}_n$ - n-oxyethylene (polyglycol ether) group.

The surfactant molecules diffuse to the surface of the water, adsorb on the phase boundary and form a monomolecular oriented layer of the surface film.

Depending on the ionic character, the surfactants are divided into ionic and nonionic. Ionicity is distinguished by the electrical charge that remains on the organic portion of the surfactant's molecule after it dissociates in water.

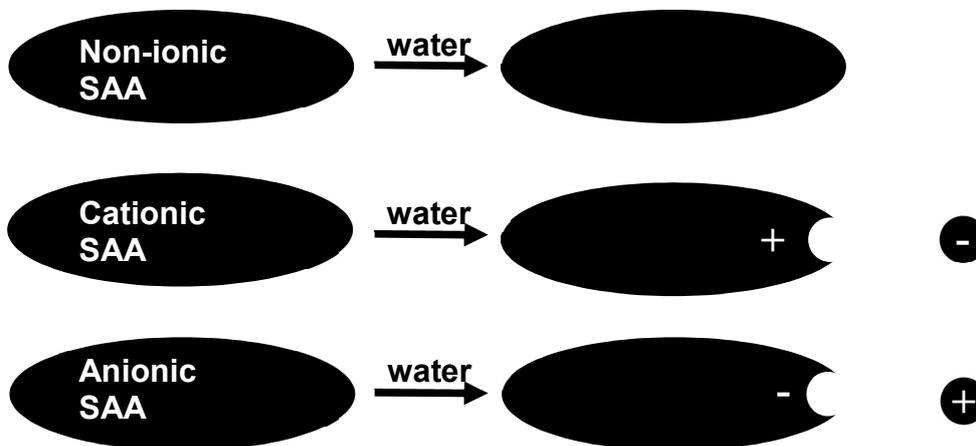


Figure: Schematic behaviour of surfactants of different ionicity in water

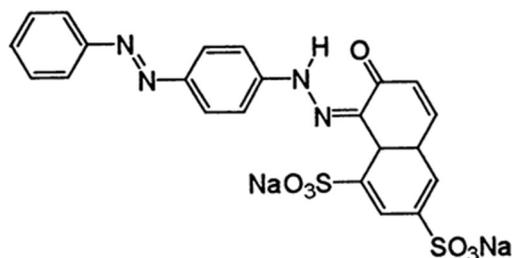
Ionicity is very important in terms of chemical combinability because anionic and cationic chemicals collide/inactivate each other. This is important in the finishing liquor and it is also crucial in the washing process. During the washing, the fibers are charged negatively, and therefore cationic surfactants which precipitate on the fibers cannot be used. This is used in fabric softeners in turn.

The qualitative method of determining ionicity consists in the precipitation of aqueous SAA solutions by substances of opposite ionicity. 1 % aqueous solutions of the following dyes have proven to be ideal as precipitants:

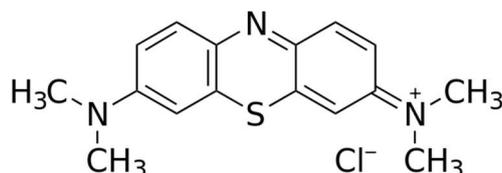
C.I. Acid Red 73 as an anionic agent to detect cationic SAA;

Methylenblau 2B (C.I. Basic Blue 9) as a cationic agent to detect anionic SAA

C.I. Acid Red 73



Methylenblau 2B (C.I. Basic Blue 9)



The aim: determine the ionicity of the 3 presented surfactants (surface-active agents)

Used chemicals:

1 % solution of C.I. Acid Red 73

1 % solution of C.I. Basic Blue 9

1 % solution of unknown surfactant (first sample)

1 % solution of unknown surfactant (second sample)

1 % solution of unknown surfactant (third sample)

Working procedure:

The determination of the ionic character of an unknown surfactant. Use two testing tubes and pour 5 ml of the prepared 1% solution of unknown surfactant into each of them. Pour a maximum of 5 ml of 1% solution of C.I. Acid Red 73 into one, and a maximum of 5 ml of a 1% solution of C.I. Basic Blue 9 into the other. Add both solutions of the above eyes to the tubes first dropwise, then in small portions, observe the formation of a precipitate.

To better evaluation the precipitate in dark solutions, pour the contents of the tube into a Petri dish.

If a precipitate is formed in a test tube with C.I. Acid Red 73, the test substance is cationic; if it is formed in a test tube with C.I. Basic Blue 9, the substance is anionic. If no precipitate forms in either tube, the substance is nonionic.

In some cases, clearer results can be obtained when using more concentrated PAL solutions, e.g. 10%.