Lahden ammattikorkeakoulu
Lahti University of Applied Sciences

Coating of textile structures with ultra fine cellulose powder

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PENNANEN, HELMI:

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ABSTRACT

The aim of this thesis was to research how coating with ultra fine cellulose powder will influence the textile to be coated and how it affects the penetration of dyestuff. Acid and cationic dyes were used in this project to research how they behave in dying cotton and which one of them is more suitable for this particular use.

Another objective was to study the need of binder for a functional coating paste and how binder influences the penetration of dyestuff. The aim was to find an ideal balance between the substances needed in a fuctional coating paste containing cellulose powder.

In addition to researching the behaviour of different dyestuffs and coating with ultra fine cellulose powder, some of the samples were washed in different phases of the coating process. This was done to examine the effect of washing on the final color fastness and color strength. The information of the washing experiments was also used when comparing the acidic and cationic dyestuffs.

The practical work of the thesis was executed in the laboratory of Polytechnic University of Catalonia, in Terrassa, Spain. This project is part of a bigger study concerning research work concentrating on ultra fine cellulose powder in Polytechnic University of Catalonia. The work was supervised by Professor Josep Canal.

Key words: nanocellulose, textile coating, textile dyeing, ultra fine cellulose powder

Lahden ammattikorkeakoulu
Tekstiili- ja vaatetustekniikka

PENNANEN, HELMI:

Tekstiilimateriaalien pinnoittaminen nanoselluloosajauheella

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TIIVISTELMÄ

Opinnäytetyössä tutkittiin tekstiilien pinnoittamista selluloosasta valmistetulla jauheella. Tutkimuksen tavoite oli selvittää, kuinka nanoselluloosalla pinnoittaminen vaikuttaa pinnoitettavaan tekstiilimateriaaliin. Tutkimuksen kohteena oli myös selluloosalla pinnoitetun tekstiilin värjääminen sekä happo- että emäspohjaisella väriaineella ja selvittää, kumpi näistä väriaineista sopii paremmin kyseiseen käyttötarkoitukseen.

Opinnäytetyössä pyrittiin myös löytämään ideaali koostumus selluloosaa sisältävään pinnoitusmassaan. Projektin aikana tutkittiin toimivaan pinnoitusmassaan tarvittavien aineiden suhteita ja niiden vaikutusta lopulliseen pinnoitustulokseen.

Tekstiilien pinnoituksen ja värjäyksen lisäksi tekstiilimateriaalille tehtiin pesututkimuksia tavoitteena selvittää, miten tekstiilin pesu eri vaiheissa valmistusprosessia vaikuttaa väriaineiden lopulliseen kiinnittymiseen sekä värin vahvuuteen. Pesututkimuksia käytettiin myös vertailukohtina eri väriaineiden toimivuuden määrittelyssä.

Tutkimuksellinen osuus suoritettiin Katalonian teknillisessä yliopistossa, Terrassassa, Espanjassa, osana suurempaa tutkimuskokonaisuutta keskittyen tekstiilien nanoselluloosalla pinnoittamiseen. Tutkimustyön ohjaajana toimi Professori Josep Canal.

Avainsanat: tekstiilien pinnoitus, nanoselluloosa, tekstiilien värjäys

CONTENTS

1 INTRODUCTION

This project deals with ultra fine cellulose powder (UFC) produced by a company J. Rettenmaier & Söhne. Two coating pastes were made containing UFC cellulose powder, thickener Lutexal GP ECO, Helizarin binder TOW and crosslinker Luprintol CF. To determine the required amount and behavior of UFC powder and binder, the two pastes had different amounts of these substances. The main difference between the pastes is the amount of binder.

To evaluate the behavior of the coatings during wash, some of the samples were washed before dyeing. Samples were also dyed with two different dyestuffs. The dyestuffs used were acid dye, Azul Bemacid, and cationic dye, Verde Malaquita. Washing was also executed for some of the samples after dyeing to see the behavior of the dyestuffs. The color strength was analyzed from the goodface and the backface of the fabric using colorimetry. All researching has been performed on cotton fabric.

2 MATERIALS AND METHODS

2.1 Coating procedure

Coating is a procedure used to do a finishing to textile materials. It adds value to the fabric and enables the fabric to adopt functional properties that are not characteristic for the material. By using the right mixtures of chemical products and the right application system, a fabric can be made waterproof, UV resistant, stain-proof, fireproof, etc.

The coating process consists of applying a polymeric layer directly to one or both surfaces of the fabric. Then the coated fabric is dryed and polymerized. In order to achieve optimum results for the finished fabrics in terms of both aesthetic appearance and technical quality, the layer of the chemical product must be applied as evenly as possible both in regard to its quantity and spreading throughout the entire width of the fabric to be treated.

There are several processes for the application of coating to the textile material depending upon the requirement of the end product. Some of these processes are described below.

2.1.1 Direct coating

One of the simplest coating methods is direct coating. In direct coating the fabric has to be extremely even so the fabric is stretched flat. The application of coating paste is performed by knife or stationary doctor blade. The knife scrapes the fibers forward while moving over the fabric and adding the polymer resin smoothly over the surface. For this type of coating method to succeed best, the weave structure should be quite tight and the fabric capable of being held taught. It is used for example for car seat fabrics, inflatable materials and waterproof fabrics. (Gross Martin 2010, 532-553.)

2.1.2 Transfer coating

The basic principle of transfer coating is to spread the polymer layer or layers on to release paper or separator to form a film and then transfer this film to the fabric.

The main uses of this type of coating technique are polyurethane fabrics in the waterproof protective clothing and for example artificial leather. Other usages for transfer coated polyurethane include upholstery, luggage, footwear, gloves and waterproof mattress covers. (Fern, Alam, Touaiti & Toivakka 2012, 181-187.)

2.1.3 Screen printing

This is the coating technique used in this project. Instead of ink, coating paste is applied through a mesh frame.

In screen printing a mesh is used to transfer the ink to the surface. Some of the areas of the mesh frame are made inpeneratable to the ink. A blade or squeegee is moved across the frame to fill the penetrable mesh areas with ink. The reverse stroke causes the mesh frame to touch the fabric and it causes the ink to wet the substrate. (Print wiki 2017)

Screen printing can also be performed using stencil method of print making in which a design is imposed on a screen of polyester or other fine mesh, with blank areas coated with an impermeable substance. Ink is forced into the mesh openings by a fill blade or squeegee, and by wetting the substrate, transferred onto the printing surface during the squeegee stroke. As the screen rebounds away from the substrate, the ink remains on the substrate. It is also known as silk-screen, screen, serigraphy, and serigraph printing. One color is printed at a time, so several screens can be used to produce a multicoloured image or design. (Sefar Inc. 1999.)

2.2 Nanocellulose materials

Nanocellulose offers possibilities for the future by being a completely natural and environmental friendly resource with many favourable features such as renewability, high surface area, low density and biodegradability (Figure 1). Nanocellulose has been used in many different purpouses, for example to increace wet and dry strength in paper and packaging, to add strength and durability to vicose and other yarns and to make see-through packaging for food industry. (Brodin, Gregersen & Syverud 2014, 156-166; Iwamoto, Isogai & Iwata 2011, 831-836; Hitoshi and Akira 2007, 334-335)

Nanocellulose materials have a natural tendency to form a film. Films made of nanocellulose are strong, thin and smooth. These are great features for a textile coating material. It might be possible to add textile's wearing resistance by coating it with nanocellulose. (Tammelin & Vartiainen 2014, chapter 13.)

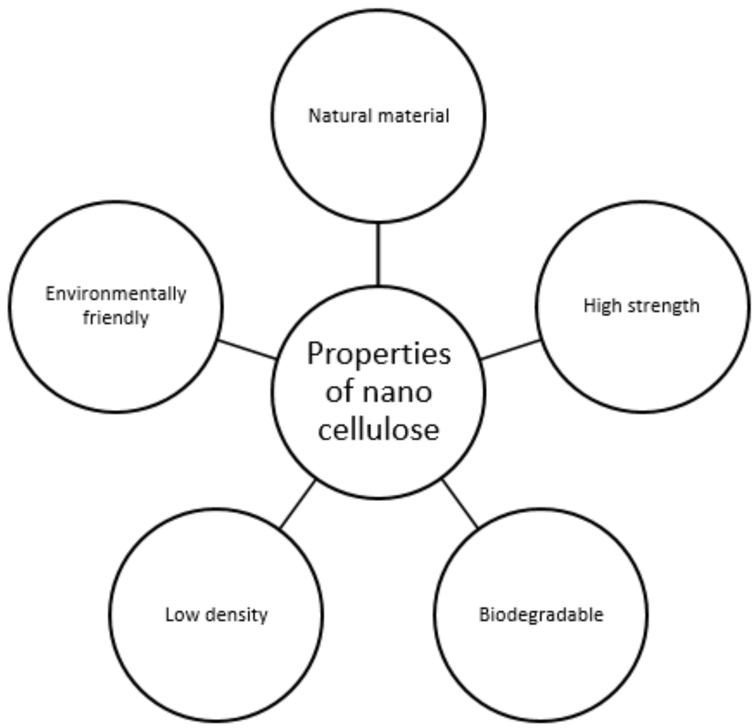

Figure 1. Properties of nano cellulose

2.3 UFC cellulose powder

ARBOCEL UFC is today the finest grade of cellulose available in the industrial scale worldwide. It is made of 100% cellulose coming from wood, which means it is a neutral product coming from natural and renewable resources. UFC stands for Ultra Fine Cellulose and is a product of the company J. Rettenmaier & Söhne.

Physical data of Arbocel UFC:

- Cellulose, no chemistry
- Ultra fine powder
- Average particle d50 from 6 to 12 µm
- Insoluble in water
- High water binding capacity
- Renewable resources

Adding ARBOCEL UFC 100 to the coating material can result in uniform and flexible coatings and good adhesion to the substrate, which results in no air bubbles or cracks. The haptic of UFC coating is also less sticky. Higher addition rates of hydrophilic particles increase the waterproofing effect. Particles of UFC are clearly visible in SEM microscopy (Figure 2). (J. Rettenmaier & Söhne Gmbh + Co. KG (JRS) 2017)

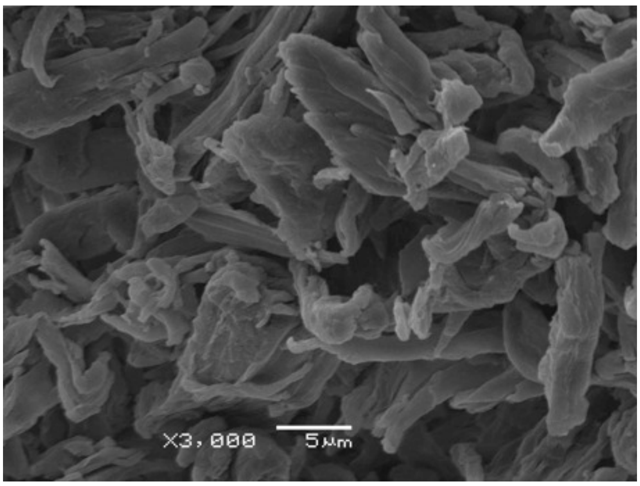

Figure 2. UFC particles in SEM microscopy

Advantages of Ultra Fine Cellulose include:

- Reduction of polymer binder: less penetration means less overdosing of binder, and thus UFC helps to reduce the main cost driver
- Higher smoothness and better printability: fine UFC particles level out any unevenness in the substrate
- Improved abrasion resistance: due to the reinforcing property of UFC abrasion resistance is improved
- Matting agent at higher dosage (5-10 wt.%): high matting efficiency without negative influence on viscosity.

2.3.1 UFC in textile coating

UFC 100 gives excellent water retention levels at higher temperature and under pressure pulses. Apart from the natural water retention of UFC, the water insoluble UFC particles form a barrier layer, which enables quick immobilization of coating substrate interface. This means reduced penetration of the aqueous dispersion of poymer binders. Above this barrier layer, coating remains flowable and easily evens out the surface.

In conclusion UFC powder enables quick immobilization of coating color and this improves the coverage and smoothness of the substrate.

2.4 Thickener, Lutexal GP ECO

The thickener used in the project was a synthetic thickener micro granulates, no powdery form. (Agro Biobase 2017)

Advantages of Lutexal GP ECO are:

- Storage stability
- Excellent profiling and equalization
- Very good cut overlays
- High stability to electrolytes
- Low doses of employment (cca 10 g/kg)

- Extremely low VOC values
- Less pollution of wastewater
- It does not produce smoke in thermal processes
- Lower consumption of paste (up to 25%)

2.5 Binder, Helizarin TOW

Helizarin Binder TOW is an acrylic binder for pigment printing. It is thermally crosslinkable and allows the production of prints with a pleasingly soft appearance and very good general use fastness properties. It has very little effect on the viscosity of synthetic thickenings. It is recommended for use with Lutexal. (Agro Biobase 2017)

2.6 Crosslinker, Luprintol CF

Luprintol CF is a crosslinker-based auxiliary combination for pigment printing. Mixture is based on emulsifier and resin. It is a yellow liquid with a characteristic odor. (Chemitex 2017)

Luprintol CF has the following technical advantages:

- Emulsifier with fixative and dispersant
- Suitable for automatic metering
- Greatly simplifies the recipe
- Often makes it possible to reduce the amount of synthetic thickener used
- The print pastes have very good running properties
- The standard of fastness is considerably increased (especially on synthetics, regenerated cellulose and their blends with cotton)
- The prints are sharply defined and the color yield is often significantly increased
- Contains no silicone

2.7 Dyes

Two different dyestuffs were used in this project. Different dyestuffs were used to see the differences in the behavior of UFC and to determine which dye is best for dyeing UFC.

2.7.1 Verde Malaquite, 110%

Verde Malaquita is a cationic dye. Cationic dyes are a class of synthetic dyes, which act as bases and when made soluble in water, they form a colored cationic salt, which can react with the anionic sites on the surface of the substrate. The cationic dyes produce bright shades with high tinctorial values, on textile materials. The chemical structure of Verde Malaquita is seen in the following Figure 3:

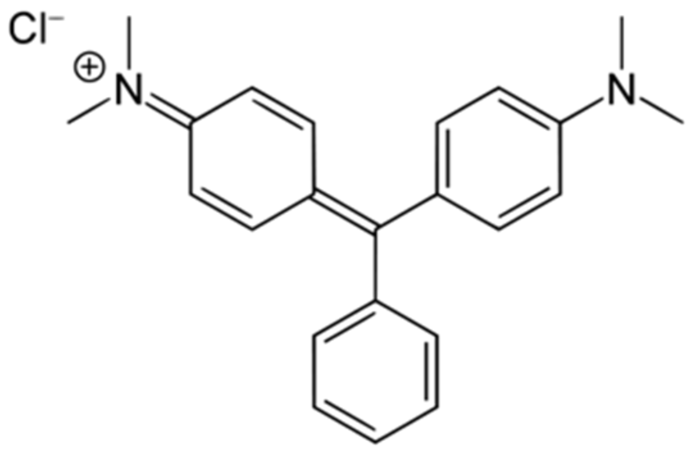

Figure 3. Chemical structure of Verda Malaquita

Cationic dyes are powerful coloring agents. They are applied to wool, silk and acrylic fibers. Usually acetic acid is added to the dye bath to help the take-up of the dye onto the fiber. Cationic dyes are also used in the coloration of paper.

2.7.2 Azul Bemacid 5R

Azul Bemacid 5R is an acid dye. An acid dye is a dye which is a salt of a sulfuric, carboxylic or phenolic organic acid. The chemical structure of acid blue is shown in Figure 4. The salts are often sodium or ammonium salts. Acid dyes are typically soluble in water and possess affinity for amphoteric fibers while lacking direct dyes' affinity for cellulose fibers. When dyeing, ionic bonding with fiber cationic sites accounts for fixation of colored anions in the dyed material. Acids are added to dyeing baths to increase the number of protonated amino-groups in fibers. Some acid dyes are used as food colorants. (PubChem, National Center for Biotechnology Information 2017)

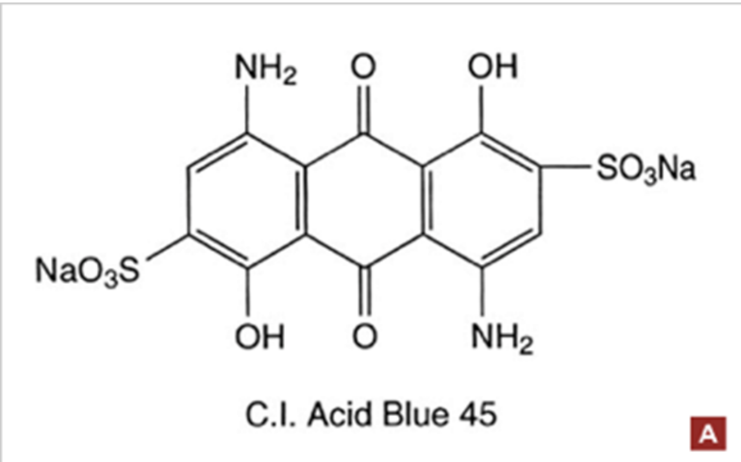

Figure 4. Chemical structure of Acid Blue

In the laboratory, home, or art studio, the acid used in the dye-bath is often vinegar (acetic acid) or citric acid. The uptake rate of the dye is controlled with the use of sodium chloride. In textiles, acid dyes are effective on protein fibers, i.e. animal hair fibers like wool, alpaca and mohair. They are also effective on silk. They are effective in dyeing the synthetic fiber nylon, but of minimum interest in dyeing any other synthetic fibers. (World Dye Variety 2017)

3 EXPERIMENTAL PART

The goal of experimental part was to test the amount of UFC needed in a good coating paste and the behavior of the coating during washing and dyeing. Two pastes were made and samples were coated, washed and dyed. Afterwards the colors of the samples were analyzed using colorimetry. The experiments were all performed in laboratory conditions and distilled water was used in all parts of the experiments.

3.1 Schematic presentation of the experimental plan

From the following Figure 5 can be seen the the whole experimental process.

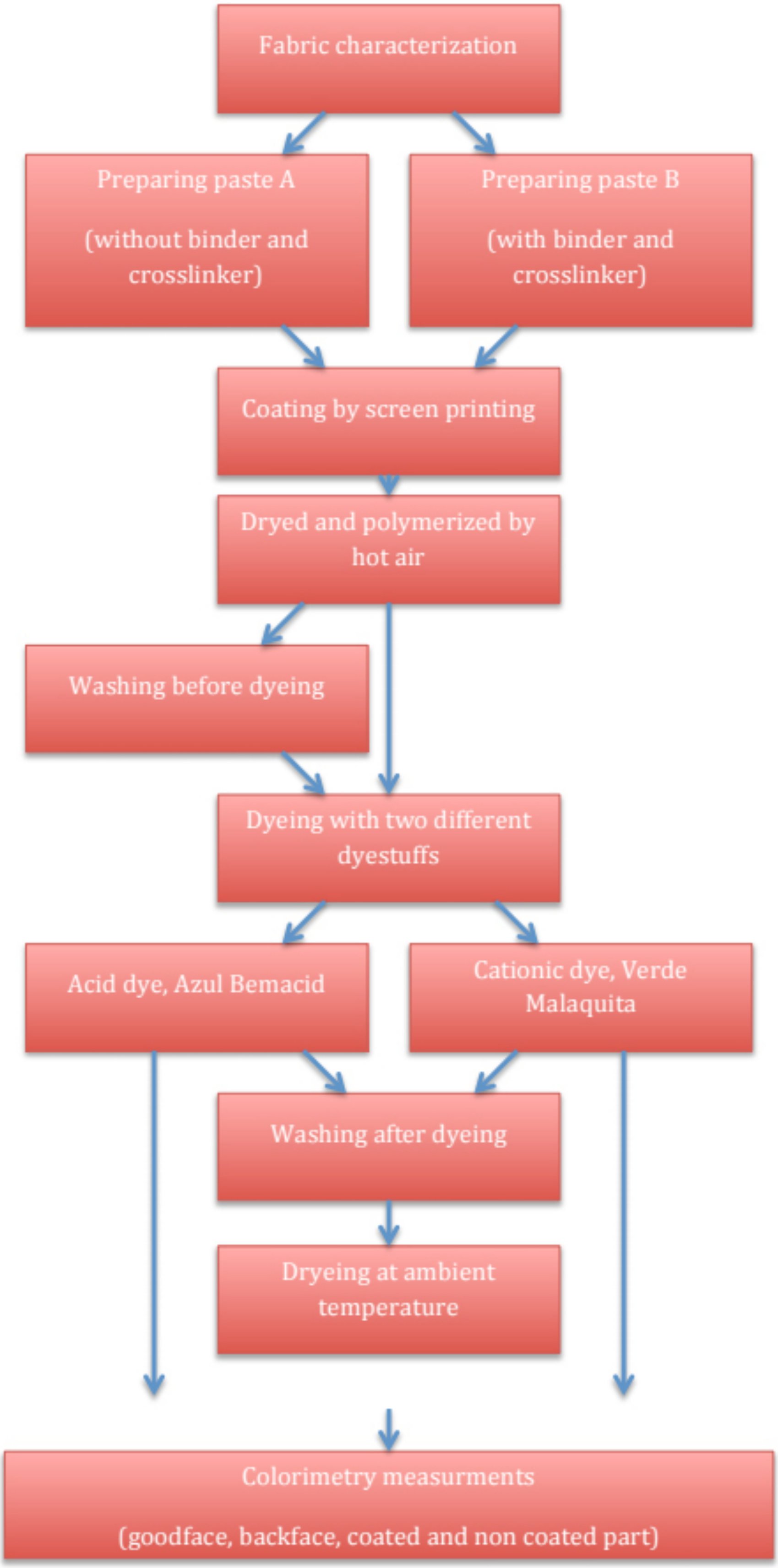

Figure 5. Experimental plan

3.2 Codification of samples

All experiments were performed on cotton fabric. In total, 16 samples were made, eight samples for each paste. Every sample was the same size 10cm x 10cm.

The samples were marked as:

(M) = Verde Malaquita, (B) = Azul Bemacid

Table 1. Presentation of all the samples

Sample	**Treatments made**
1.1	Coated, dried, polymerized, dyed (M)
1.2	Coated, dried, polymerized, washed, dyed (M)
1.3	Coated, dried, polymerized, washed, dyed (M), washed
1.4	Coated, dried, polymerized
1.5	Coated, dried, polymerized, washed
1.6	Coated, dried, polymerized, dyed (B)
1.7	Coated, dried, polymerized, washed, dyed (B)
1.8	Coated, dried, polymerized, washed, dyed (B), washed
2.1	Coated, dried, polymerized, dyed (B)
2.2	Coated, dried, polymerized, washed, dyed (B)
2.3	Coated, dried, polymerized, washed, dyed (B), washed
2.4	Coated, dried, polymerized
2.5	Coated, dried, polymerized, washed

Table 1 shows the number of each sample and what treatments were made to it. In the table, (M) means dyed using colorant Verde Malaquita and (B) means dyed using Azul Bemacid. From the chart can be seen the colors and the numbers of the samples for each paste.

3.3 Coating pastes

Fabrics were coated with two different pastes. One containing only UFC powder and thickener. The other, in addition to the above, contained Helizarin TOW binder and Luprintol CF crosslinker. The machine used in coating was Ahiba Polymat. After coating, fabrics were dried in 110ºC for 3 minutes and polymerized in 150ºC for 4 minutes. For drying and polymerization, Werner Mathis AG machines were used.

3.3.1 Paste A; containing UFC and thickener

The paste was made using 16% UFC powder liquid, which has been mixed before. All substances were mixed carefully using a Heidolph stirrer with 3000u/min force. The paste was used right after mixing it. Table 2 shows the constitution of the paste for 1g of UFC/1000g coating paste.

Table 2. Constitution of paste A

Substance	**G/1000g**
Water	888.3
UFC	110
Lutexal GP ECO, thickener	1.7

3.3.2 Paste B; containing UFC, thickener, binder and crosslinker

Beforehand mixed 16% UFC liquid was also used in this paste. The paste was mixed using the Heidolph stirrer with 3000u/min power and used soon after mixing it.

Table 3. Constitution of paste B

Substance	**G/1000g**
Water	729
UFC	90
Lutexal GP ECO, thickener	5
Helizarin TOW, binder	160
Luprintol CF, crosslinker	16

The constitution of the paste for 1g of UFC/1000g coating paste is shown in Table 3. Cosslinker and binder were prepared the day before but there were changes in the structure after the night and the mix of binder and crosslinker was unusable (Figure 6). The fluid form of the binder and crosslinker had changed overnight to an uneven mass. Therefore, crosslinker and binder should be mixed only when preparing the paste.

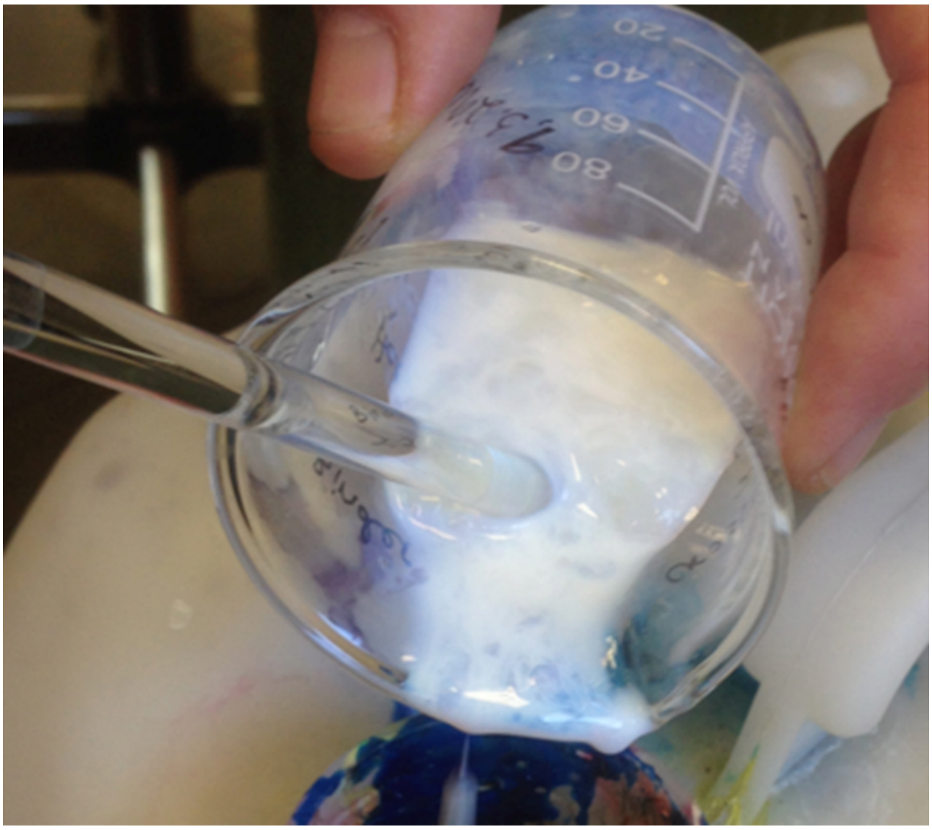

Figure 6. Crosslinker and binder after one day

3.4 Coating procedure

Coating was done using Johannes Zimmer (Klagenfurt-Austria, Mini MDF/734). The machine worked with magnetic stick, which was controlled by hand. The coating technique used is called screen printing. The coating frame was not long enough for the whole fabric so the coating was done in two parts.

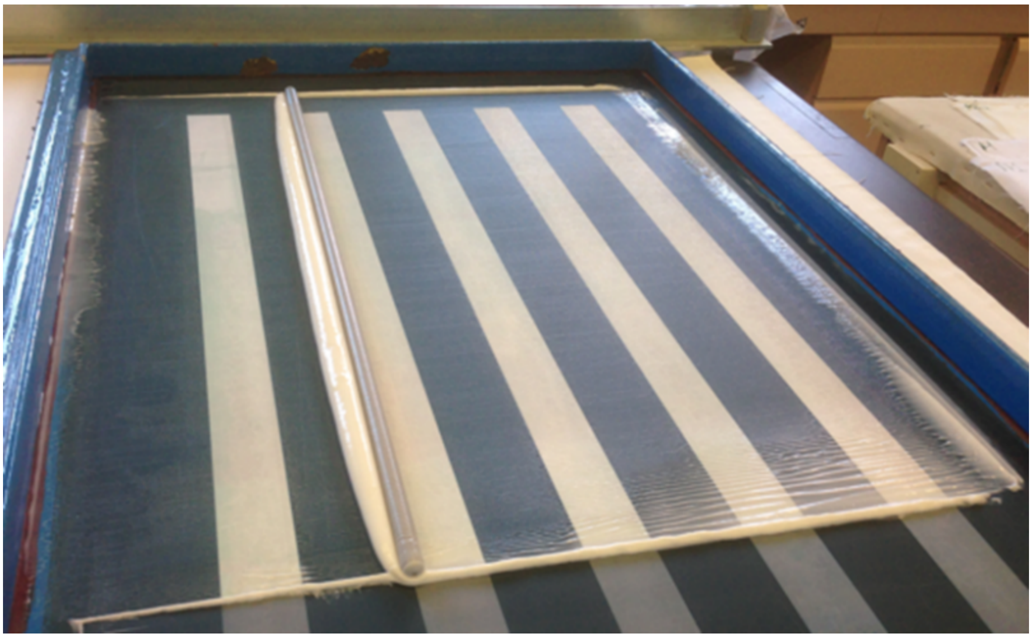

Figure 7. Magnetic stick applying the paste

The magnetic stick applied the coating paste (Figure 7). First the paste was added to the left length of the frame. The applying with magnetic stick started from the left end of the frame and went back and forth applying the paste to the whole length of the stick. It is important that there is the same amount of paste at the whole length of the stick so the final coating will be evenly thick. Slightly too much paste was applied to both ends of the stick (Figure 7). The applying process should also flow through without stopping in the middle of the frame. After the applying is ready, the frame can be removed carefully and used in other parts of the fabric.

3.5 Drying and polymerization

Samples were dried and polymerizated using Werner Mathis AG. Samples were dried in 110°C for 3 minutes and polymerizated in 150°C for 4 minutes.

3.6 Washing before dyeing

Part of the samples were washed before dyeing to see if it has an effect on the final color of the sample. Samples were washed using Ahiba Polymat (Figure 8). Time for the washing procedure was 30 minutes and temperature 60ºC.

Figure 8. Ahiba polymat

Table 4. Constitution of washing liquid

R/b	1g fabric/50ml washing liquid
Washing liquid	ECE detergent 5g/l

The washing liquid consisted of water and ECE reference detergent (Figure 9) with relation of 5 g/l. The relation of fabric and washing liquid used in washing was 1g fabric per 50ml of washing liquid (Table 4).

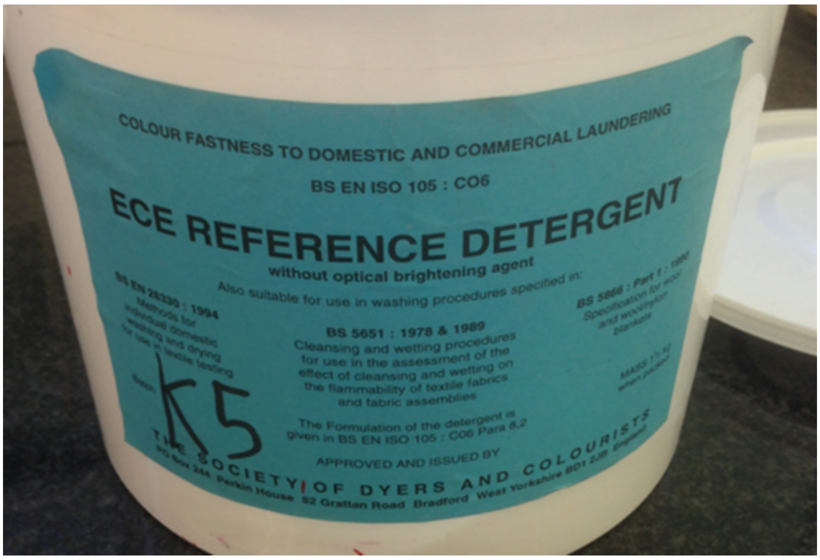

Figure 9. Substance used in the washing liquid

3.7 Dyeing

Two different dyes were used in dyeing, Azul Bemacid 5R and Verde Malaquita 110%. Samples were dyed in 80ºC for 60 minutes. The liquid used in dyeing had to have pH 4.5-5 so a mixture was made consisting of water, acid acetic 60% and sodium acetate. Final pH of the liquid was 4.93. The relation used in dyestuff was 1/1000.

Table 5. Constitution of dyeing liquid

Substance	**Amount per liter**
Acid acetic 60%	0.8g/l
Sodium acetate	1.2g/l
Final pH	**4.93**

The constitution of dyeing liquid for one liter is shown in Table 5. For the actual dye bath 0.5g/100g of dyestuff and 1/30 of pH 4.93 were mixed.

Figure 10. Lines from the grid can be seen in the sample

The first samples dyed were dried naturally on a grid but the grid affected the sample and some of the color was attached to the grid. The lines of the grid can be seen in the sample after drying (Figure 10).

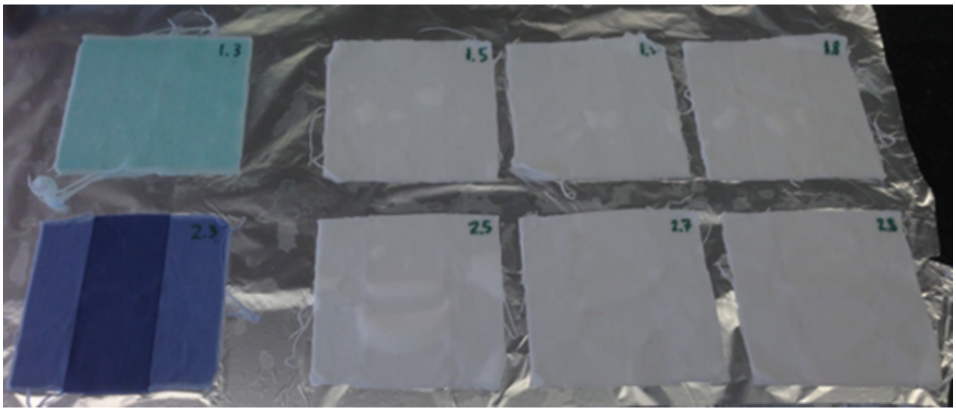

Figure 11. Other samples were dried on a tinfoil

To minimize the influence of the drying platform, tinfoil was used in the next drying (Figure 11).

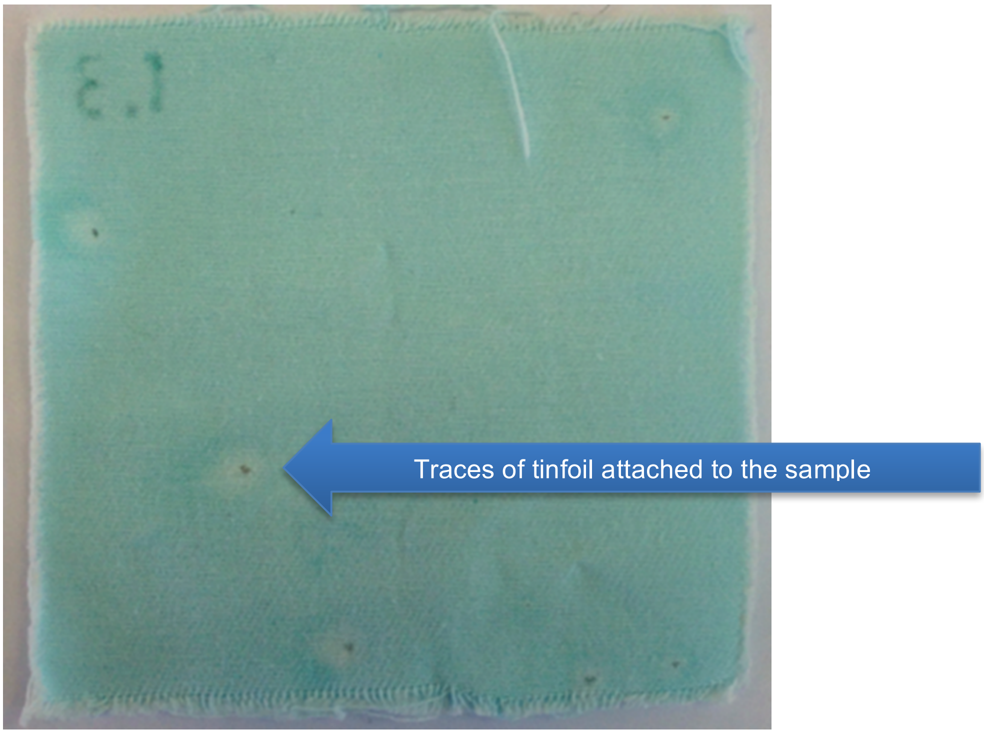

Figure 12. Backside of a sample influenced by tinfoil

After all tinfoil also affected one of the samples. Small parts of tinfoil (Figure 12) managed to stick to the sample and the sample ended up losing color in these parts of the sample. For future experiments different drying platform should be used.

3.8 Washing after dyeing

Samples washed after dyeing were washed three times to see how much they lose color and how the coating is affected by multiple washes. Ahiba Polymat was also used for this washing. Time for the washing procedure was 30 minutes and temperature 60ºC.

Table 6. Washing procedure after dyeing

	Temperature °C	**Time min.**	**Liquid**	**Other**
1. Wash	60	30	5g/l ECE	-
2. Wash	60	30	water	10 metal balls
3. Wash	60	30	water	10 metal balls

Table 6 shows the washing procedure done to the samples after dyeing. The first wash was done with the same procedure as in the washing executed before dyeing. The second and third wash were done with the same time and temperature but only water was used as washing liquid and 10 metal balls were added to make the wash more mechanical.

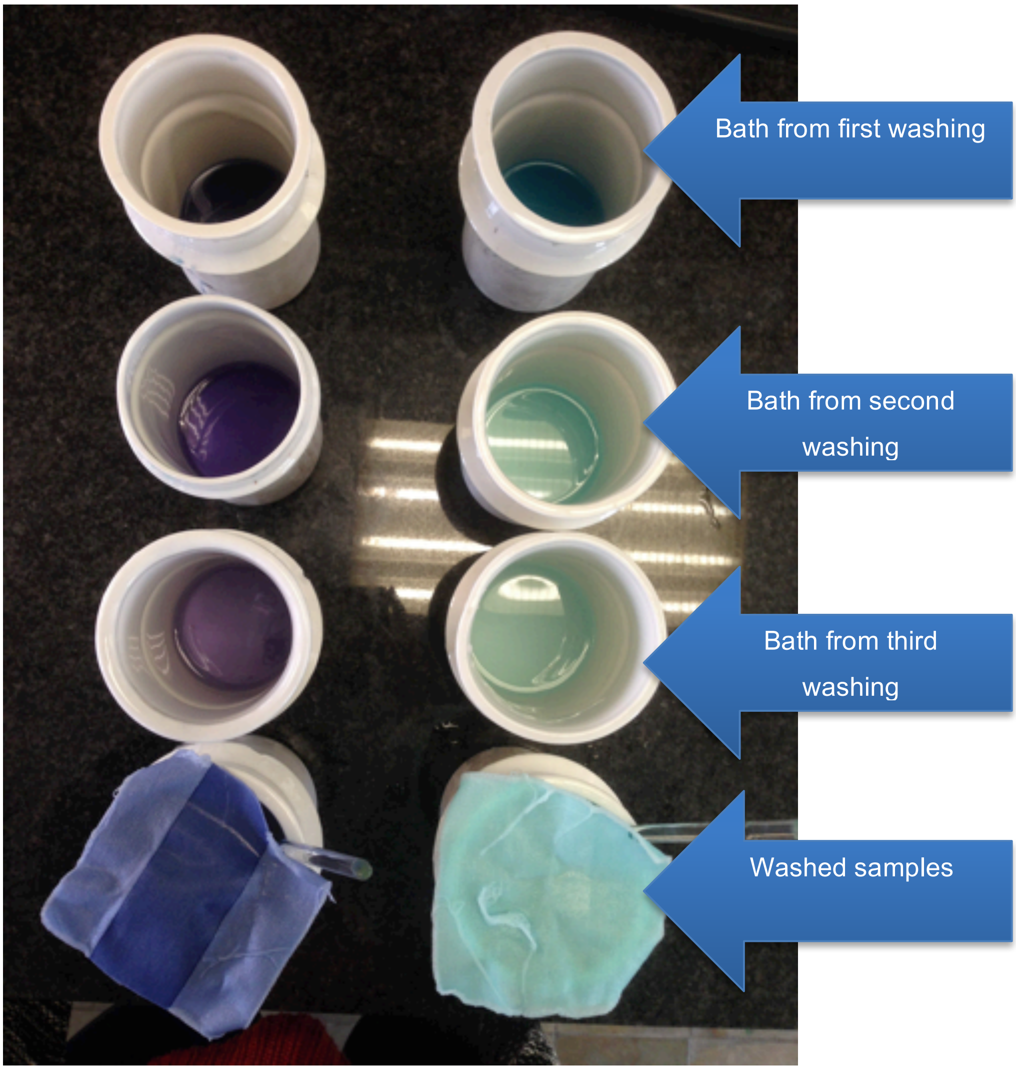

Figure 13. Washing baths from three washes for sample 1.3 and 2.3

Figure 13 shows the washing baths of the three separate washes. The top baths are from the first wash, the second baths from the top from the second wash and the third baths from the third wash and at the bottom are the samples after three washes. The decrease of the dyestuff left in the washing baths can be seen in the picture. Notable is the high amount of dyestuff left in the washing baths. Also sample 1.3 dyed with Verde Malaquita and coated with paste A lost almost all the coating during the washes.

4 RESULTS OF THE EXPERIMENTS

From the two coating pastes the one containing binder conducted well and the coating was smooth, solid and received great amount of dyestuff. The other paste not containing binder was not able to bind color the same way and the coating did not attach to the textile and therefore the coating was broken and UFC was lost.

4.1 Colorimetry

Colors of the samples were measured using colorimetry. Colorimetry calculated the spectral values of the samples and whiteness degrees of not dyed samples. Colors were measured from four different surfaces: from coated and non-coated surface of the goodside and the backside. The following picture will represent the measuring points at the sample (Figure 14). The picture is from the sample's cross-section. The points are marked in the picture the following way:

A= Goodface, non-coated

B= Goodface, coated

C= Backface, non-coated

D= Backface, coated

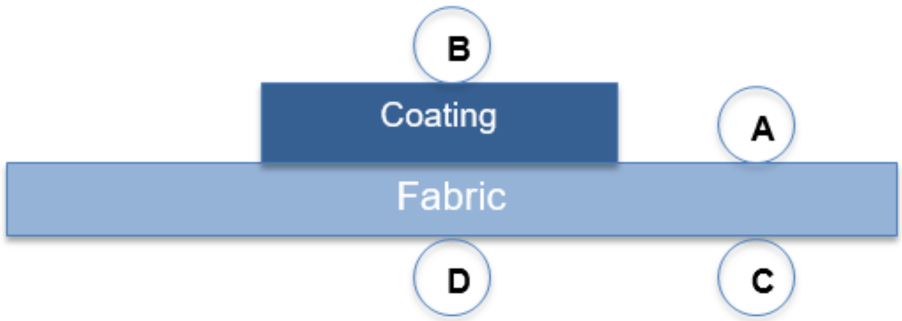

Figure 14. Cross-section of a sample, dots descriping the points of measurements using colorimetry

4.1.1 Whiteness degree

One of the purposes of colorimetry is to see differences in whiteness and color.

Table 7. Whiteness degree of coating

Sample	Whiteness degree
Sample 1.4 (Paste A, no binder, non-washed)	87.1
Sample 1.5 (Paste A, no binder, washed)	86.7
Sample 2.4 (Paste B, with binder, non-washed)	81.0
Sample 2.5 (Paste B, with binder, washed)	82.9

As seen in Table 7, coating done with paste A not containing binder has a higher whiteness degree than paste B containing binder. Judging by the result it can be stated that adding binder and crosslinker to the paste decreases the whiteness of the coating. This may be due to the fact that the surface of the coating is different and affects the colorimetry. Binder and crosslinker may also affect the color of the coating because they are not white or clear substances.

Washing had a different effect on the whiteness degrees. In the case of paste A, the whiteness degree decreased during wash and in paste B it increased. These results may be due to the fact that samples coated with paste A lost a lot of coating during wash so the whiteness degree decreased. Also samples with paste B might have lost some of the binder and crosslinker during the wash, which would increase the whiteness.

4.1.2 K/S values

All the samples were analyzed using colorimetry. Colors were analyzed from goodside, backside and coated and non-coated parts of the samples. The K/S value means the color strength of the sample and it is important to calculate it from different parts of the sample to see the differences between coated and non-coated parts. By calculating the K/S values it is also possible to reliably compare different samples. The K/S values were calculated with the following Formula 1:

$$(\mathrm{K/S}) = \frac{(1-0.01\,R)^2}{2(0.01R)}$$

Formula 1.

In the formula, R is the reflectance, K is the absorbance and S is the scattering. R is always the lowest value of the spectral values which were received from the colorimetry. The following tables illustrate the K/S values for some of the samples. The highest K/S value of all the samples is highlighted and the highest value for every sample is underlined. The K/S values ranged from 0.286 to 21.307.

K/S values pointed out a clear difference between the coating pastes. Samples coated with paste B containing binder had the highest values always at the coated part of the goodface but the opposite effect is seen at the other samples coated with paste A not containing binder. The lowest K/S value in these samples was at the coated part of the goodface. This verifies the fact that the coating A without binder was not able to receive dyestuff the same way as other parts of the sample. The difference between the goodface and the backface in non-coated areas was not remarkable at any of the samples.

4.1.2.1 K/S tables

The following tables show the calculated and corrected K/S values for each sample from four different surfaces of the sample.

Table 8. K/S values for sample 1.1 Paste A not containing binder

Sample	**R values**	**Corrected K/S values**
1.1 goodface, non-coated, dyed with Malaquita	$(\%\mathrm{R})_{dyed}$=0.204 $(\%\mathrm{R})_{white}$=0.759	$(\mathrm{K/S}\ )_{600}^{corr}$=1.518
1.1 goodface, coated, dyed with Malaquita	$(\%\mathrm{R})_{dyed}$=0.310 $(\%\mathrm{R})_{white}$=0.820	$(\mathrm{K/S}\ )_{600}^{corr}$=0.747
1.1 backface, non-coated, dyed with Malaquita	$(\%\mathrm{R})_{dyed}$=0.195 $(\%\mathrm{R})_{white}$=0.757	$(\mathrm{K/S}\ {}_{600}^{corr}$=1.626
1.1 backface, coated, dyed with Malaquita	$(\%\mathrm{R})_{dyed}$=183 $(\%\mathrm{R})_{white}$=0.807	$(\mathrm{K/S}\ )_{600}^{corr}$=1.801

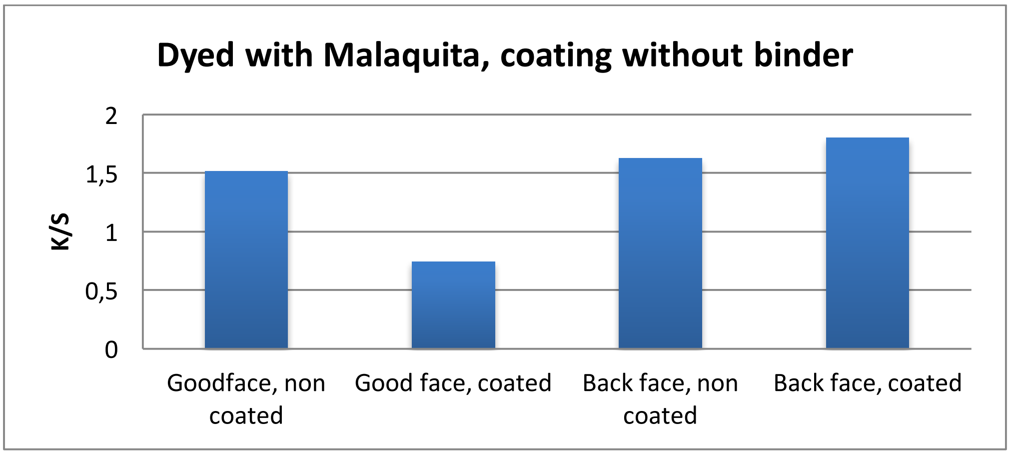

Figure 15. K/S values of sample 1.1

Sample 1.1, the K/S values of which are shown in Table 8 and Figure 15, was coated with paste A, and the only treatment was dyeing with Verde Malaquita. The highest K/S value is found from the backface's non-coated area and the lowest value was in the coated area of the backface.

Table 9. K/S Values of sample 2.6, Paste B containing binder

Sample	**R values**	**Corrected K/S values**
2.6 goodface, non-coated, dyed with Malaquita	$(\%R)_{dyed}$=0.094 $(\%R)_{white}$=0.751	$(K/S)_{600}^{corr}$=4.302
2.6 goodface, coated, dyed with Malaquita	$(\%R)_{dyed}$=0.025 $(\%R)_{white}$=0.797	$(K/S)_{600}^{corr}$=19.394
2.6 backface, non-coated, dyed with Malaquita	$(\%R)_{dyed}$=0.092 $(\%R)_{white}$=0.751	$(K/S)_{600}^{corr}$=4.422
2.6 backface, coated, dyed with Malaquita	$(\%R)_{dyed}$=0.061 $(\%R)_{white}$=0.780	$(K/S)_{600}^{corr}$=7.237

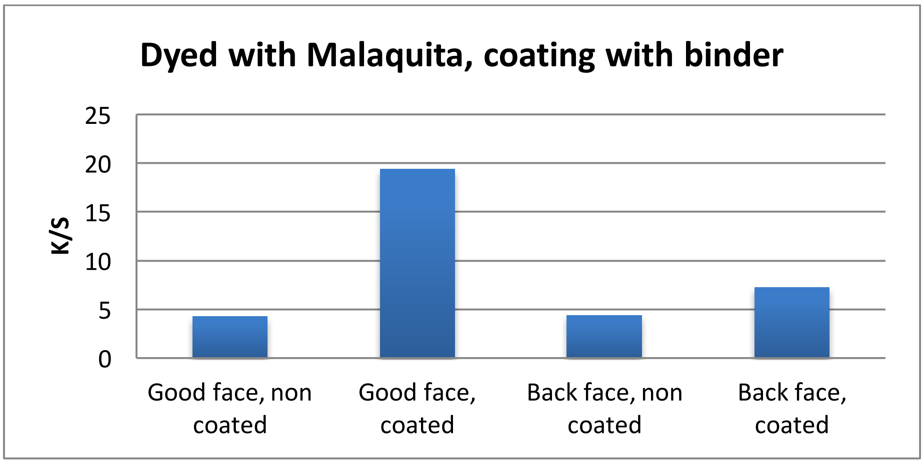

Figure 16. K/S Values for sample 2.6

Sample 2.6, the K/S values of which are shown in Table 9 and Figure 16, was coated with paste B, and the only treatment was dyeing with Verde Malaquita. The highest K/S value is found from the goodface's coated area and the lowest value was in the non-coated area of the goodface.

Table 10. K/S values of sample 1.2, Paste A not containing binder

Sample	**R values**	**Corrected K/S values**
1.2 goodface, non-coated, dyed with Malaquita	$(\%R)_{dyed}$=0.132 $(\%R)_{white}$=0.759	$(K/S)_{600}^{corr}$=2.820
1.2 goodface, coated, dyed with Malaquita	$(\%R)_{dyed}$=0.216 $(\%R)_{white}$=0.817	$(K/S)_{600}^{corr}$=1.399
1.2 backface, non-coated, dyed with Malaquita	$(\%R)_{dyed}$=0.131 $(\%R)_{white}$=0.757	$(K/S)_{600}^{corr}$=2.858
1.2 backface, coated, dyed with Malaquita	$(\%R)_{dyed}$=0.187 $(\%R)_{white}$=0.814	$(K/S)_{600}^{corr}$=1.740

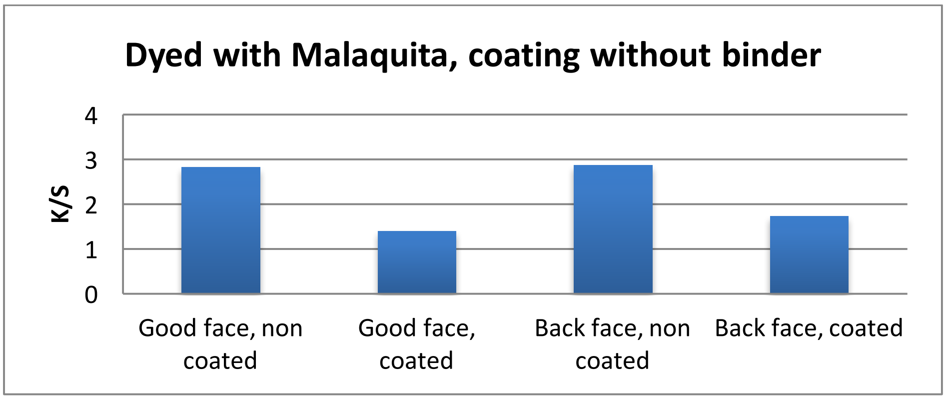

Figure 17. K/S values for sample 1.2

Sample 1.2, the K/S values of which are shown in Table 10 and Figure 17, was coated with paste A, and washed before dyeing and dyed with Verde Malaquita. The highest K/S value is found from the backface's coated area and the lowest value was in the coated area of the goodface.

Table 11. K/S Values of sample 2.7, Paste B containing binder

Sample	**R values**	**Corrected K/S values**
2.7 goodface, non-coated, dyed with Malaquita	$(\%R)_{dyed}$=0.079 $(\%R)_{white}$=0.754	$(K/S)_{600}^{corr}$=5.297
2.7 goodface, coated, dyed with Malaquita	$(\%R)_{dyed}$=0.022 $(\%R)_{white}$=0.797	$(K/S)_{600}^{corr}$=21.307
2.7 backface, non-coated, dyed with Malaquita	$(\%R)_{dyed}$=0.082 $(\%R)_{white}$=0.751	$(K/S)_{600}^{corr}$=5.097
2.7 backface, coated, dyed with Malaquita	$(\%R)_{dyed}$=0.051 $(\%R)_{white}$=0.783	$(K/S)_{600}^{corr}$=8.761

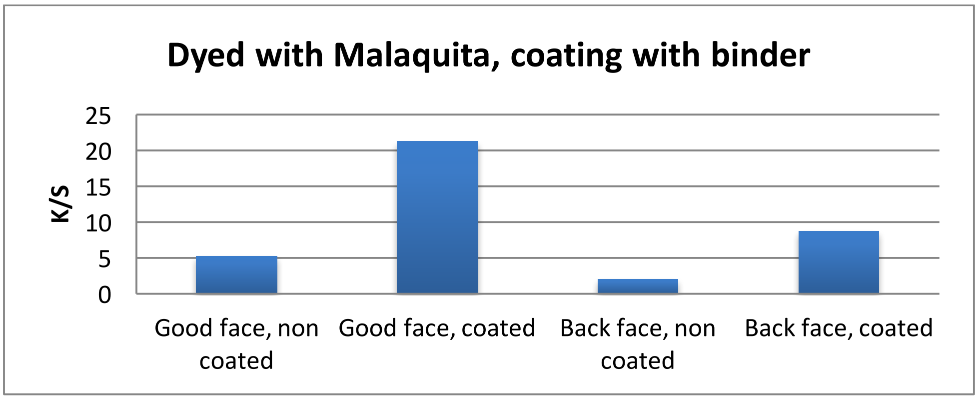

Figure 18. K/S values for sample 2.7

Sample 2.7, the K/S values of which are shown in Table 11 and Figure 18, was coated with paste A, and washed before dyeing and dyed with Verde Malaquita. The highest K/S value is found from the goodface's coated area and the lowest value was in the non-coated area of the backface.

Table 12. K/S values for sample 1.3, Paste A not containing binder

Sample	**R values**	**Corrected K/S values**
1.3 goodface, non-coated, dyed with Malaquita	$(\%R)_{dyed}$=0.442 $(\%R)_{white}$=0.761	$(K/S)_{600}^{corr}$=0.315
1.3 goodface, coated, dyed with Malaquita	$(\%R)_{dyed}$=0.466 $(\%R)_{white}$=0.820	$(K/S)_{600}^{corr}$=0.286
1.3 backface, non-coated, dyed with Malaquita	$(\%R)_{dyed}$=0.424 $(\%R)_{white}$=0.760	$(K/S)_{600}^{corr}$=0.353
1.3 backface, coated, dyed with Malaquita	$(\%R)_{dyed}$=0.427 $(\%R)_{white}$=0.810	$(K/S)_{600}^{corr}$=0.361

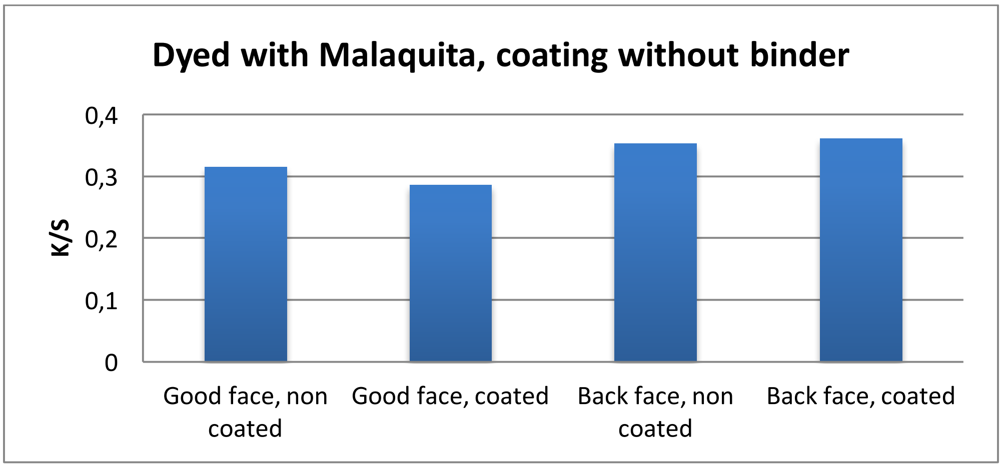

Figure 19. K/S values of sample 1.3

Sample 1.3, the K/S values of which are shown in Table 12 and in the Figure 19, was coated with paste A, washed before and after dyeing and dyed with Verde Malaquita. The highest K/S value is found from the backside's coated area and lowest value is in the coated area on the goodside. The low value of the coated area is explained by the fact the coating without binder did not receive much dyestuff and after three washes the sample appears almost white.

Table 13. K/S values for sample 2.8, Paste B containing binder

Sample	R values	Corrected K/S values
2.8 goodface, non-coated, dyed with Malaquita	$(\%R)_{dyed}$=0.298 $(\%R)_{white}$=0.751	$(K/S)_{600}^{corr}$=0.783
2.8 goodface, coated, dyed with Malaquita	$(\%R)_{dyed}$=0.030 $(\%R)_{white}$=0.793	$(K/S)_{600}^{corr}$=<u>15.436</u>
2.8 backface, non-coated, dyed with Malaquita	$(\%R)_{dyed}$=0.284 $(\%R)_{white}$=0.751	$(K/S)_{600}^{corr}$=0.864
2.8 backface, coated, dyed with Malaquita	$(\%R)_{dyed}$=0.110 $(\%R)_{white}$=0.780	$(K/S)_{600}^{corr}$=3.578

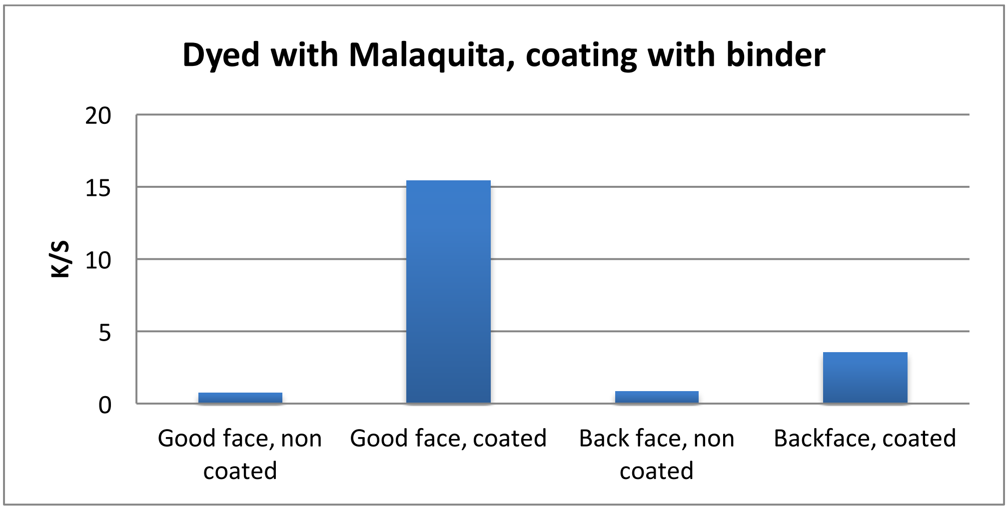

Figure 20. K/S values of sample 2.8

Sample 2.8, the K/S values of which are shown in Table 13, was coated with paste B, washed before and after dyeing and dyed with Verde

Malaquita. The highest K/S value is found from the goodface's coated area, which is also the highest value of all the samples and the lowest value was in the non-coated area of the goodface. This sample was washed three times after dyeing and from Figure 20 it can be seen how the color has been lost from the non-coated areas but in the coated areas the color strength is still high.

Table 14. K/S values for sample 1.6, Paste A not containing binder

Sample	**R values**	**Corrected K/S values**
1.6 goodface, non-coated, dyed with Bemacid	$(\%R)_{dyed}$=0.164 $(\%R)_{white}$=0.765	$(K/S)_{600}^{corr}$=2.095
1.6 goodface, coated, dyed with Bemacid	$(\%R)_{dyed}$=0,343 $(\%R)_{white}$=0.824	$(K/S)_{600}^{corr}$=0.610
1.6 backface, non-coated, dyed with Bemacid	$(\%R)_{dyed}$=0.166 $(\%R)_{white}$=0.763	$(K/S)_{600}^{corr}$=2.056
1.6 backface, coated, dyed with Bemacid	$(\%R)_{dyed}$=0.174 $(\%R)_{white}$=0.814	$(K/S)_{600}^{corr}$=1.939

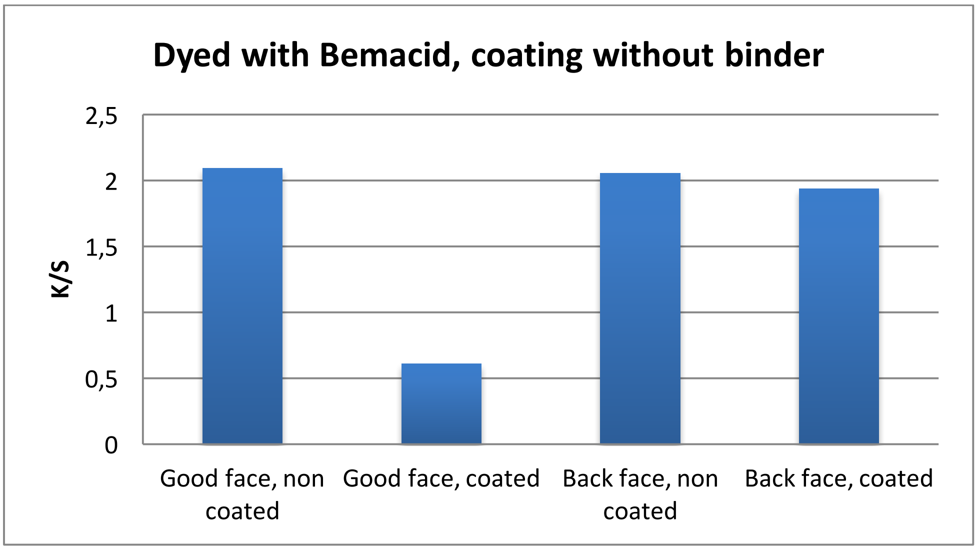

Figure 21. K/S values of sample 1.6

Sample 1.6, the K/S values of which are shown in Table 14, was coated with paste A, and the only treatment was dyeing with Azul Bemacid. The highest K/S value is found from the goodface’s non-coated area and the lowest value was in the coated area of the goodface. Also in this case, the coated part has the lowest value because the coating without binder was not able to receive much dyestuff as can be seen in Figure 21.

Table 15. K/S values for sample 2.1, Paste B containing binder

Sample	**R values**	**Corrected K/S values**
2.1 goodface, non-coated, dyed with Bemacid	$(\%R)_{dyed}$=0.187 $(\%R)_{white}$=0.757	$(K/S)_{600}^{corr}$=1.723
2.1 goodface, coated, dyed with Bemacid	$(\%R)_{dyed}$=0.080 $(\%R)_{white}$=0.800	$(K/S)_{600}^{corr}$=<u>5.249</u>
2.1 backface, non-coated, dyed with Bemacid	$(\%R)_{dyed}$=0.181 $(\%R)_{white}$=0.758	$(K/S)_{600}^{corr}$=1.808
2.1 backface, coated, dyed with Bemacid	$(\%R)_{dyed}$=0.133 $(\%R)_{white}$=0.787	$(K/S)_{600}^{corr}$=2.808

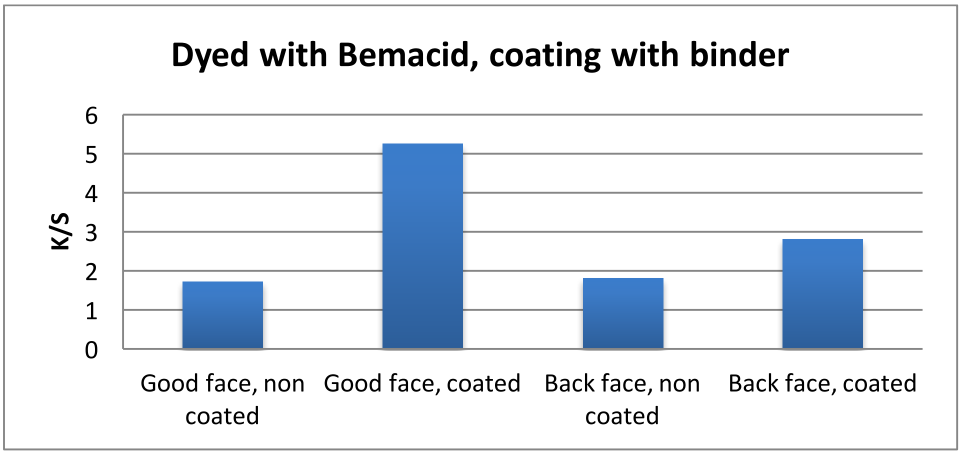

Figure 22. K/S values of sample 2.1

Sample 2.1, the K/S values of which are shown in Table 15, was coated with paste B, and the only treatment was dyeing with Azul Bemacid. The highest K/S value is found from the goodface's coated area and the lowest value was in the non-coated area of the goodface. In Figure 22 it can be seen that the coating including binder can receive more dye than the non-coated fabric.

Table 16. K/S values for sample 1.7, Paste A not containing binder

Sample	**R values**	**Corrected K/S values**
1.7 goodface, non-coated, dyed with Bemacid	$(\%R)_{dyed}$=0.179 $(\%R)_{white}$=0.765	$(K/S)_{600}^{corr}$=1.851
1.7 goodface, coated, dyed with Bemacid	$(\%R)_{dyed}$=0.369 $(\%R)_{white}$=0.824	$(K/S)_{600}^{corr}$=0.521
1.7 backface, non-coated, dyed with Bemacid	$(\%R)_{dyed}$=0.178 $(\%R)_{white}$=0.763	$(K/S)_{600}^{corr}$=1.858
1.7 backface, coated, dyed with Bemacid	$(\%R)_{dyed}$=0.185 $(\%R)_{white}$=0.814	$(K/S)_{600}^{corr}$=1.772

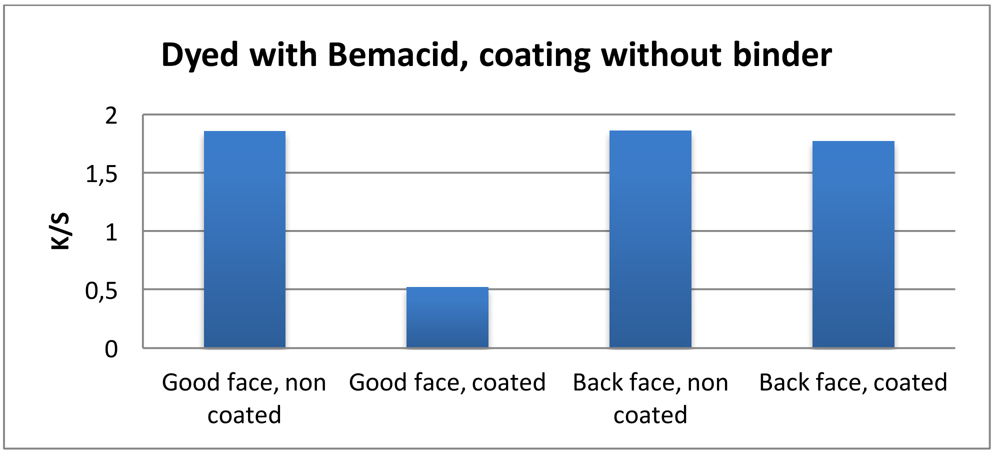

Figure 23. K/S values of sample 1.7

Sample 1.7, the K/S values of which are shown in Table 16, was coated with paste A, and was washed before dyeing and dyed with Azul Bemacid. The highest K/S value is found from the backface's coated area and the lowest value was in the coated area of the goodface. From Figure 23 it can be seen that the coated part was not able receive much dye.

Table 17. K/S values for sample 2.2, Paste B containing binder

Sample	**R values**	**Corrected K/S values**
2.2 goodface, non-coated, dyed with Bemacid	$(\%R)_{dyed}$=0.174 $(\%R)_{white}$=0.759	$(K/S)_{600}^{corr}$=1.921
2.2 goodface, coated, dyed with Bemacid	$(\%R)_{dyed}$=0.076 $(\%R)_{white}$=0.800	$(K/S)_{600}^{corr}$=5.575
2.2 backface, non-coated, dyed with Bemacid	$(\%R)_{dyed}$=0.172 $(\%R)_{white}$=0.758	$(K/S)_{600}^{corr}$=1.948
2.2 backface, coated, dyed with Bemacid	$(\%R)_{dyed}$=0.119 $(\%R)_{white}$=0.787	$(K/S)_{600}^{corr}$=3.229

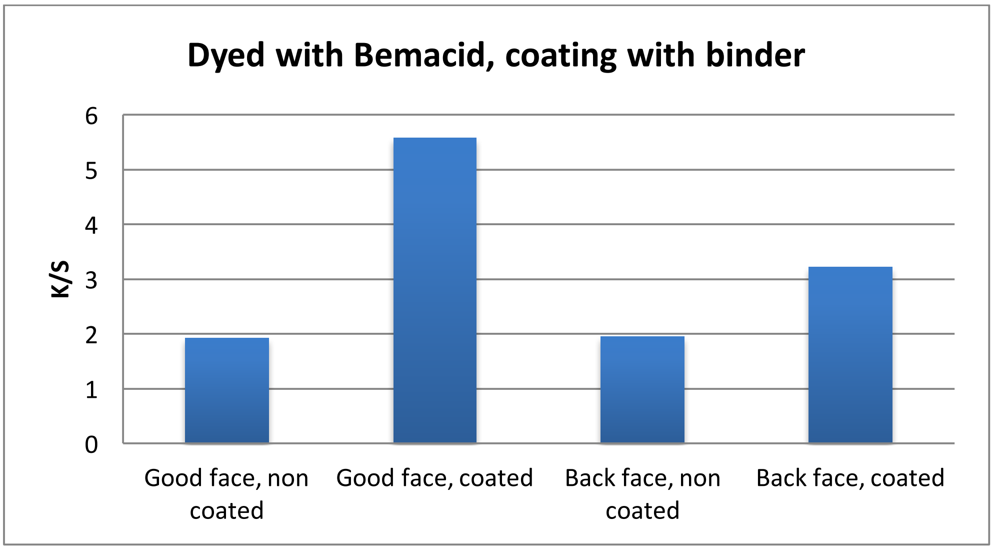

Figure 24. K/S values of sample 2.2

Sample 2.2, the K/S values of which are shown in Table 17, was coated with paste B, and washed before dyeing and dyed with Azul Bemacid. The highest K/S value is found from the goodface's coated area and the lowest value was in the non-coated area of the goodface. Figure 24 shows that the coated part received more than double amount of dyestuff compared to the non-coated part of the fabric.

4.2 Differences in washing

Some of the samples were washed before dyeing to see the behavioral differences of the two coatings during wash, and to see if it affects the penetration of dyestuff.

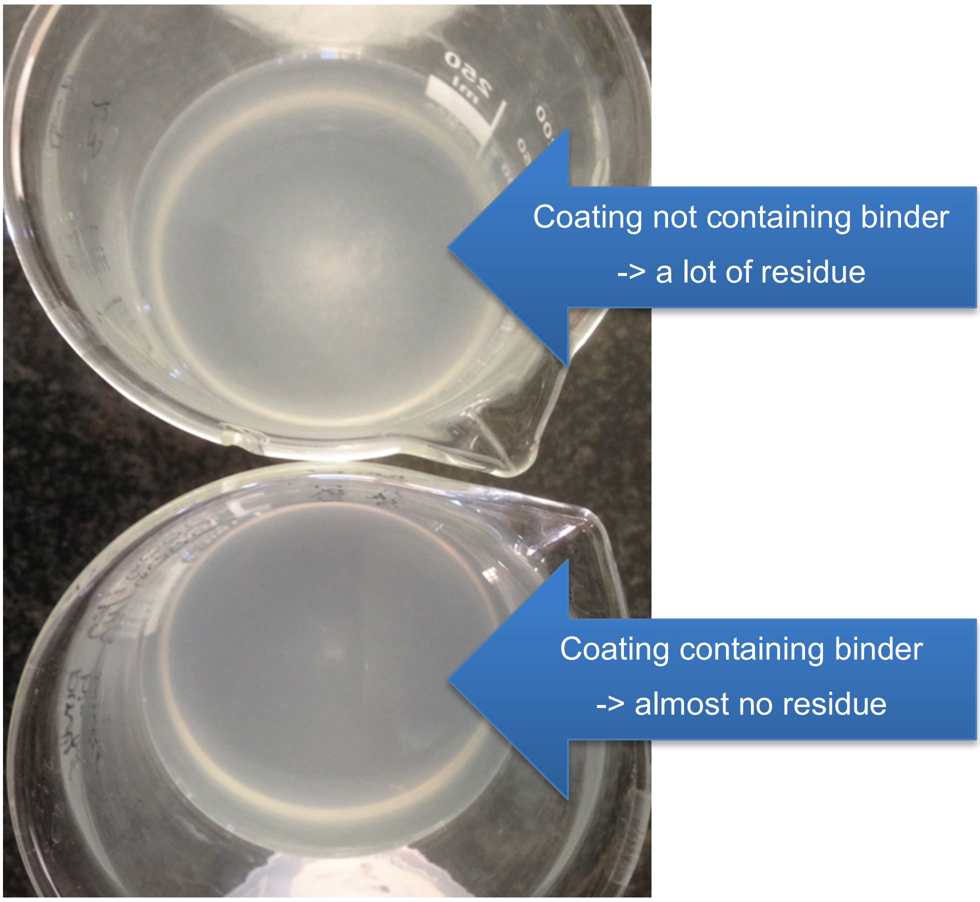

Figure 25. Two washing baths, upper one from a sample without binder and lower with binder

There were clear differences between the washing baths with and without binder as can be seen from Figure 25. The samples with no binder lost a great amount of UFC during the wash before dyeing. The loss of UFC can be seen in the upper washing bath of the picture since there is a lot of visible UFC at the bottom of the washing bath. The coating including binder and crosslinker did not lose UFC equally and the difference can be seen at the lower washing bath in the picture.

The difference could also be seen from the samples. The coating without binder was no longer solid and there were visible cracks on the coating. The coating with binder was still solid and there was not any visible change. As a conclusion can be said that a coating without binder and crosslinker does not maintain its structure in washing and therefore is not a functional formula for coating. However coating including binder, crosslinker and UFC powder performed well in washing and is can be used in fuctional coating paste.

4.3 Color difference in samples washed and not washed before dyeing

Some of the samples were washed before dyeing to see if the washing before dyeing affects the penetration of dyestuff. The K/S values showed different results. In three cases of four the colour strength increased in the samples washed before dyeing compared to samples not washed before. Only in one case the color strength was higher in the sample not washed before dyeing (in samples 1.6 and 1.7).

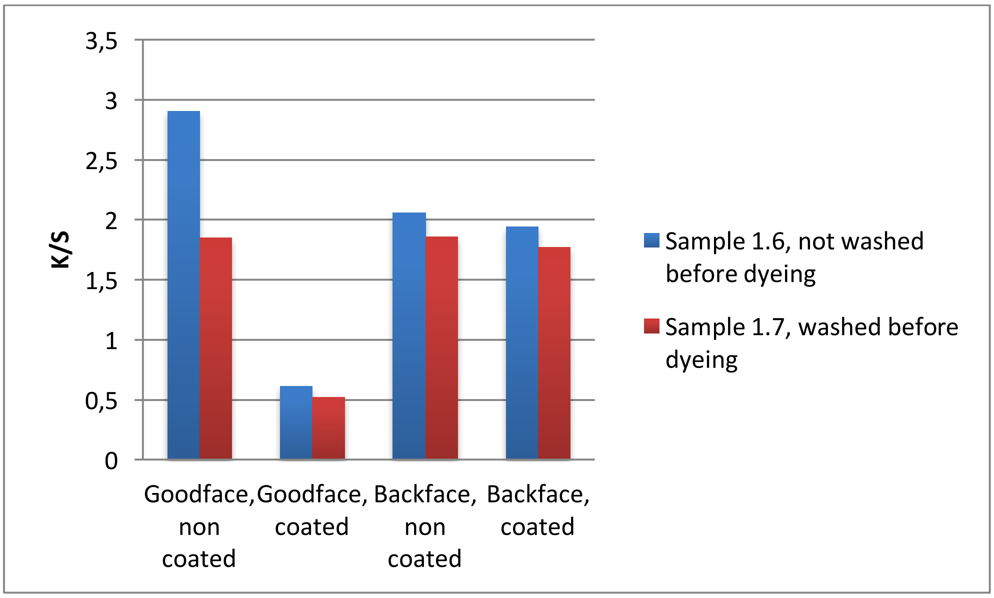

Figure 26. K/S values for samples 1.6 and 1.7, dyed with bemacid, coating without binder

In this case the samples that were not washed before dyeing had stronger color strength, as seen in Figure 26. This was the only case where color strength values did not increase in the sample washed before dyeing.

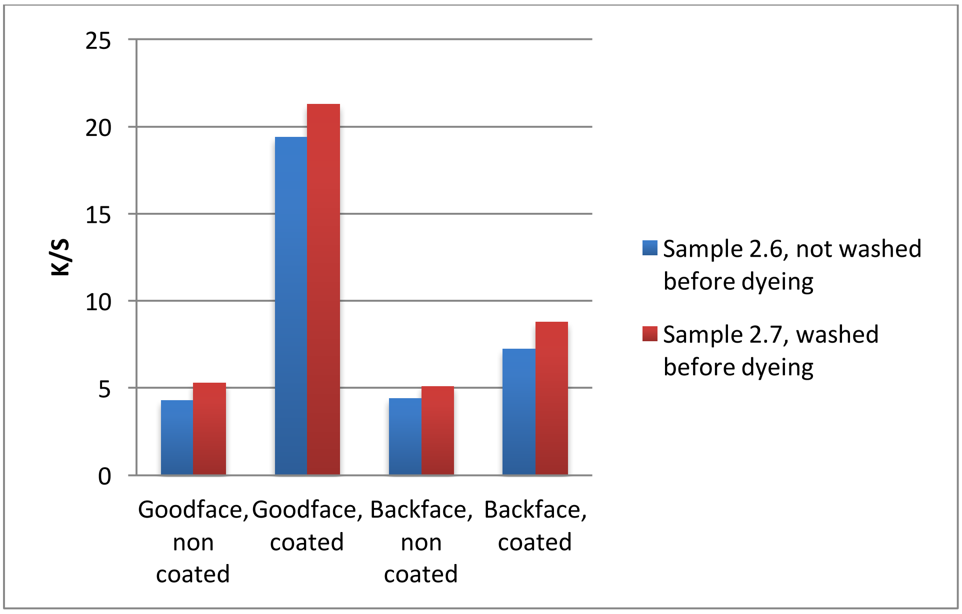

Figure 27. K/S values of samples 2.6 and 2.7, dyed with Malaquita, coating with binder

The color difference between the samples washed and not washed before dyeing was not major as can be seen from Figure 27 but was still continuous in every face of the sample. In all the faces of the samples the color strength increased in the samples washed before dyeing.

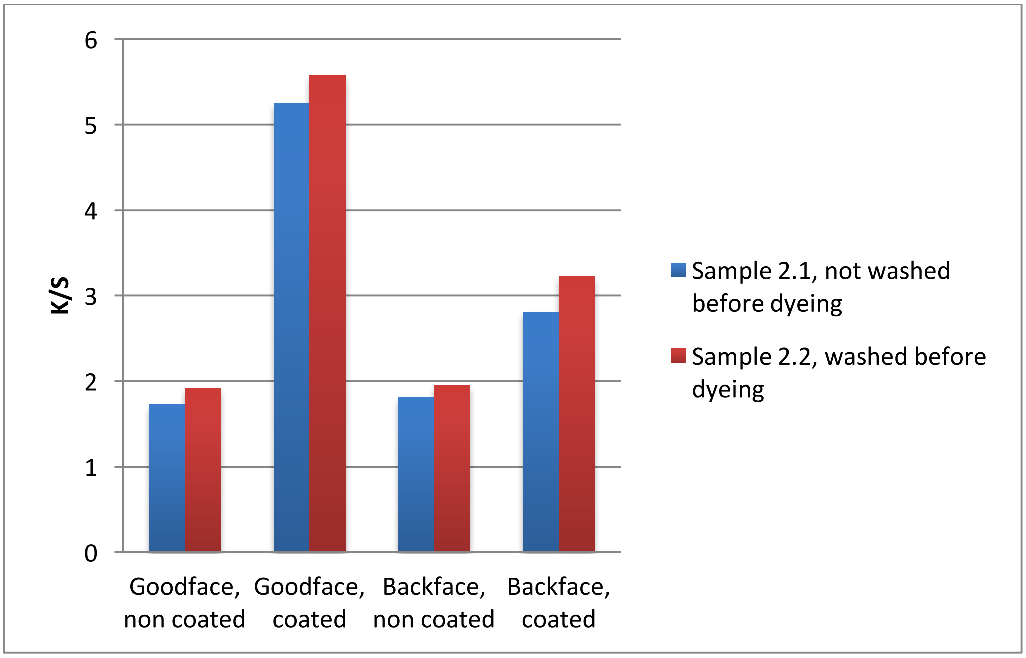

Figure 28. K/S values for samples 2.1 and 2.2, dyed with Bemacid, coating with binder

The color difference between the samples washed and not washed before dyeing was not major as can be seen from Figure 28 but was still continuous in every face of the sample. In all the faces of the samples the color strength increased in the samples washed before dyeing.

Considering these results, it can be said that in most cases washing before dying increases the color strength of the dyed textile. The difference is not remarkable and it is not seen from the textiles without calculations. The increasing color strength might be due to the fact that the washing removes residues of other substances left on the textile from previos treatments and cleans the possible stains.

4.4 Differences in dyeing

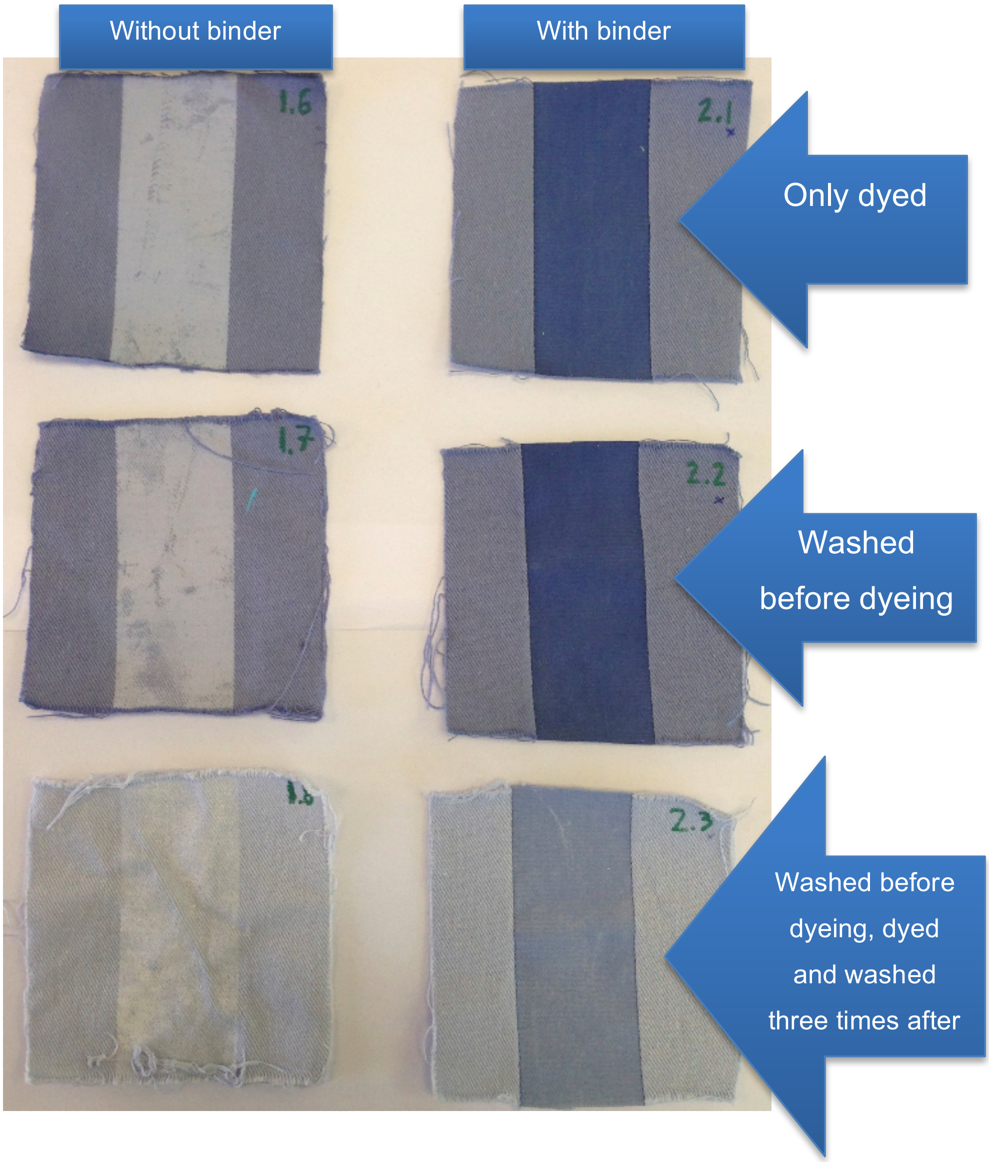

Figure 29. Samples dyed with same dyestuff, different treatments and two different coating pastes

There were clear differences in the samples during dyeing, which can be seen in Figure 29. Samples 1.6-1.8 were coated with paste not including binder and samples 2.1-2.3 were coated with paste including binder and crosslinker. The dyestuff used in these samples was Azul Bemacid. In samples 1.6-1.8 the dyestuff has not penetrated the coating, leaving it almost white and only the fabric is dyed properly. The effect is opposite in the samples coated with paste including binder. The coated part has a bright color and it has received most of the dyestuff.

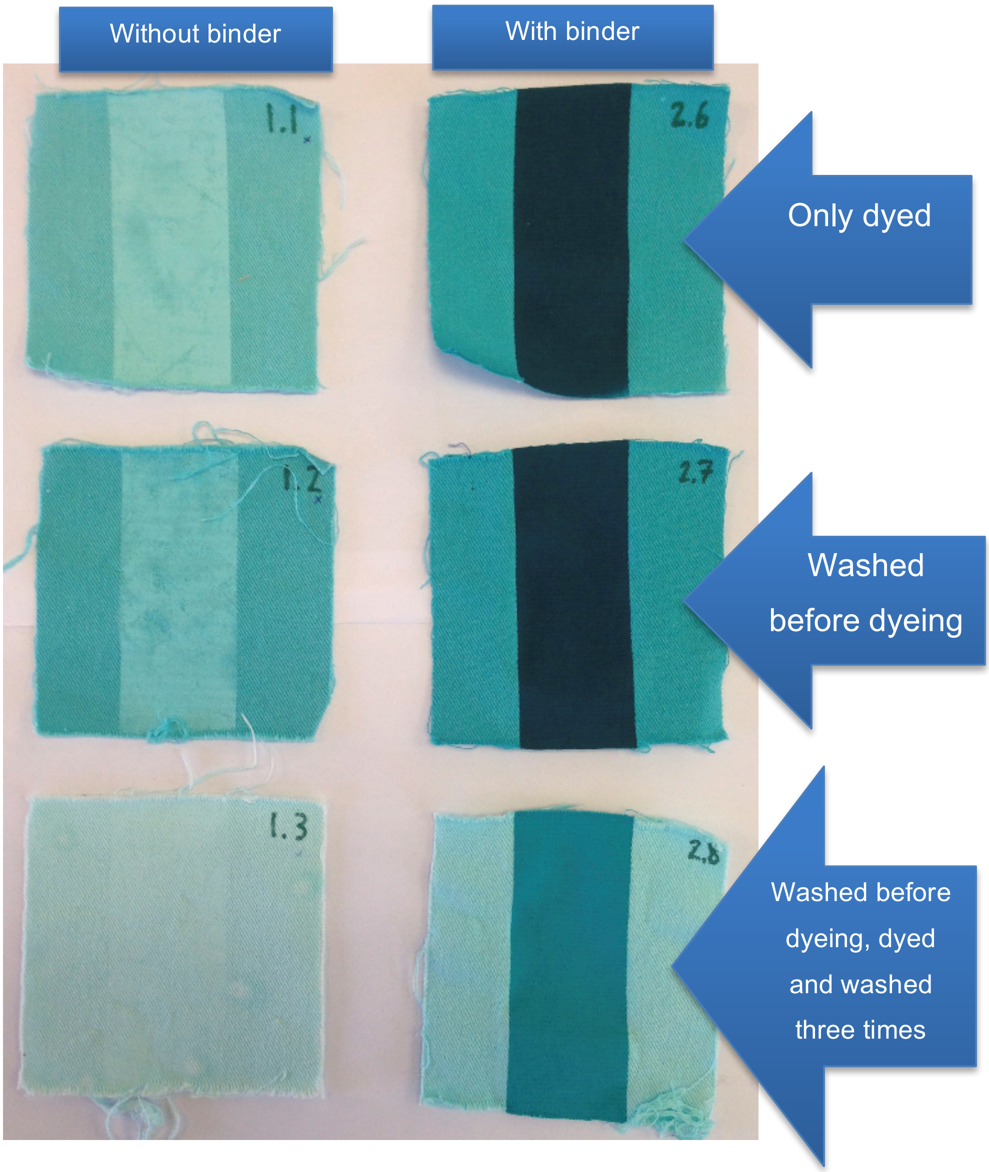

Figure 30. Samples dyed with same dyestuff, different treatment and two different coating pastes

The same behavior continued with different dyestuff, Verde Malaquita, as seen in Figure 30. Samples 1.1-1.3 were coated with paste A not including binder and samples 2.6-2.8 with paste B including binder. Coating without binder did not receive any dyestuff and coatings with binder were the opposite.

The penetration of dyestuff in coating including UFC and binder was notable; the color in these coatings was strong and bright while the coatings without binder remained almost white. Hereby it can be pointed out that binder has more influence on the degree of the penetration of the dyestuff than UFC.

The figures also show the big difference between the middle samples and the lowest samples. The lowest samples were washed three times after dyeing and the middle ones only before dyeing. A lot of dyestuff was left in the washing baths after dyeing and samples lost a lot of color insensitivity. There was also some UFC left in the dye baths of the samples coated using paste without binder.

Considering these differences it can be concluded that a functional coating paste needs binder for attaching the coating to the fabric and to enable successful penetration of dyestuff.

4.5 Comparison of K/S values

Samples 1.2 and 2.7 were dyed with Verde Malaquita and washed before dyeing. The only difference between the two samples is different coating paste. Sample 1.2 was coated with paste without binder and sample 2.7 with coating paste including binder. In sample 1.2 without binder the highest K/S values were found from the non-coated areas of the sample but in sample 2.7 the highest values were at the coated parts. Comparison of the K/S values for these samples can be seen in Figure 31.

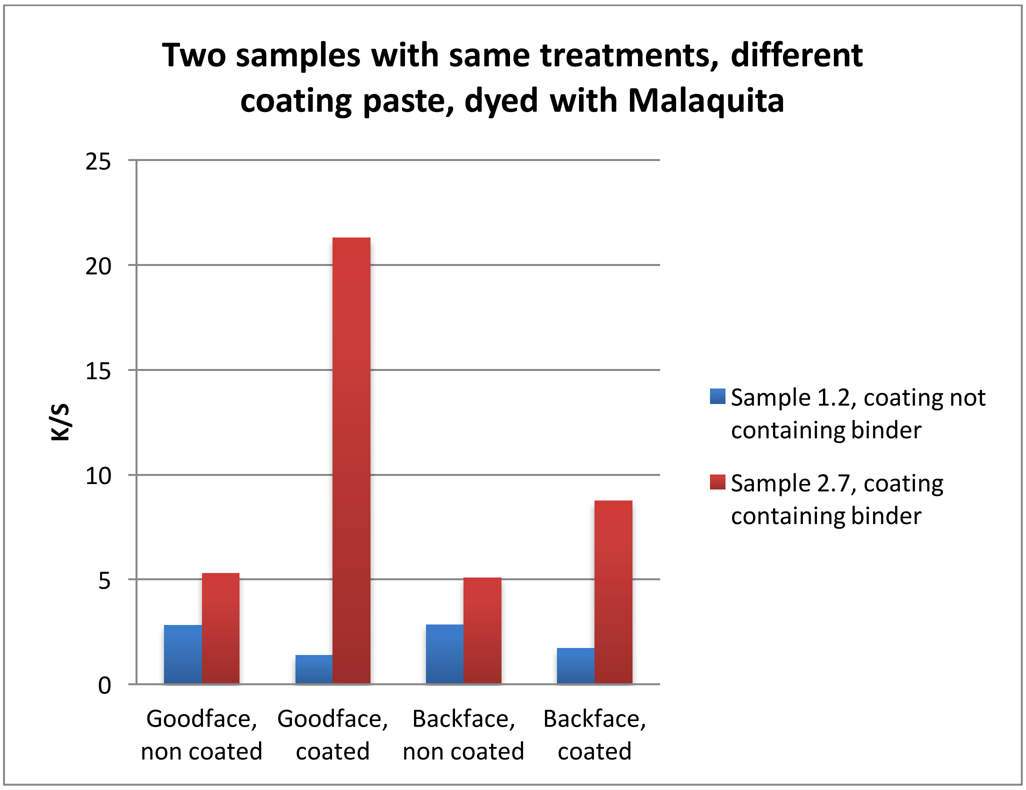

Figure 31. K/S values of samples 1.2 and 2.7, same treatments only difference coating paste

Samples 1.6 and 2.1 were dyed with Verde Malaquita. The only difference between the two samples is different coating paste. Sample 1.6 was coated with paste without binder and sample 2.1 with coating paste including binder. In sample 1.6 without binder the highest K/S values were found from the non-coated areas of the sample but in sample 2.1 the highest values were at the coated parts. Comparison of the K/S values for these samples can be seen in Figure 32.

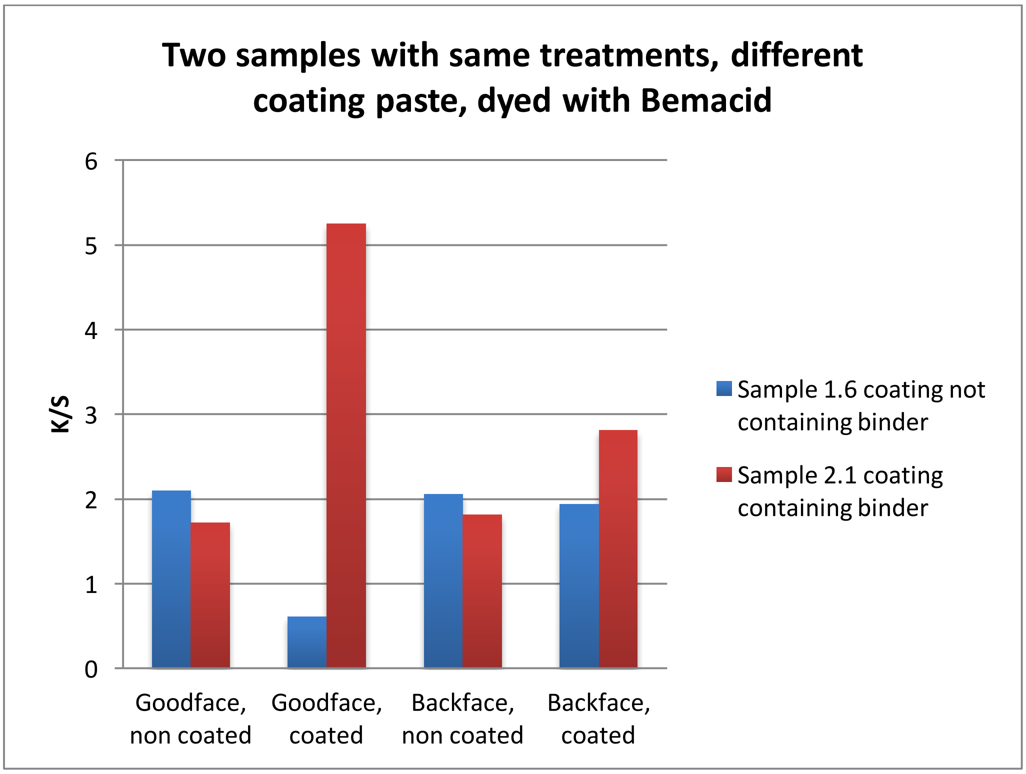

Figure 32. K/S values of samples 1.6 and 2.1, same treatments only deifference coating paste

These results can be explained by the fact that the coating paste without binder was not able to bind to the fabric and UFC polymer by itself is not able to receive much dyestuff. This results as low K/S values at the coated areas, but the samples also have different K/S values at the non-coated parts which should not be possible because they were dyed with the same procedure. The dyeing of the samples was executed in two parts so that might explain the changes.

From Figures 31 and 32 it can also be seen how much effect the coating paste with binder also has on the coated part of the backside. In samples coated with paste A without binder there were not big differences between the coated and non-coated part of the backface but the color difference at the backside was clear on the samples coated with paste B containing binder.

This can be explained by the lack of binder at paste A and therefore the coating paste not attaching to the fabric and penetrating it. In the samples coated with paste including binder the penetration of the coating was significant as can be seen from Figure 33. Depending on the use of the coated fabric it can be a positive or a negative factor but should, however, be considered.

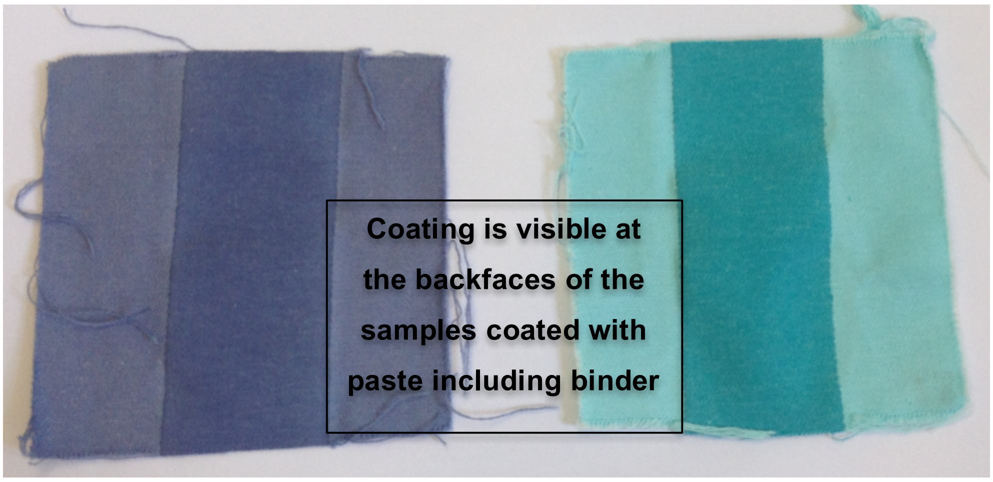

Figure 33. Backfaces of samples 2.2 and 2.8, both coated with paste including binder

4.6 Differences between acid dyestuff and cationic dyestuff

The samples were dyed with acid dye and cationic dye. Experiments showed some differences between the behaviors of the dyestuffs.

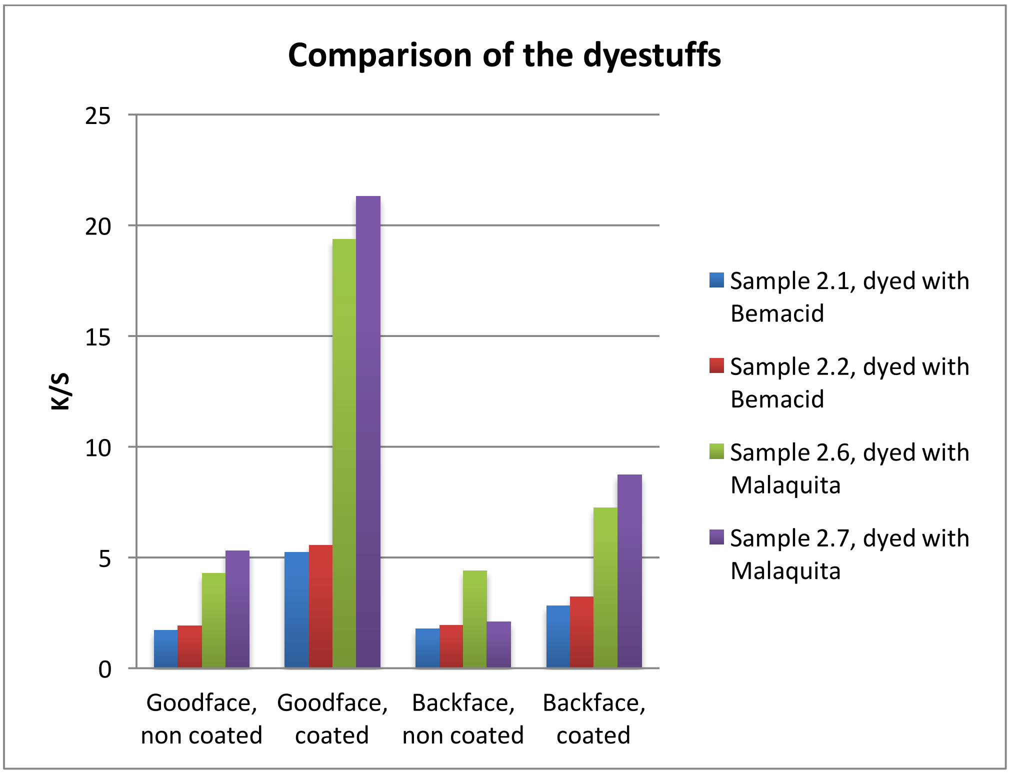

Figure 34. K/S Values for samples 2.1, 2.2, 2.6 and 2.7

Samples dyed with Verde Malaquita had always higher color srtenght values than samples dyed with Azul Bemacid. Figure 34 shows the K/S values for some samples. Samples 2.6 and 2.7 are dyed with Verde Malaquita and samples 2.1 and 2.2 with Azul Bemacid. Samples 2.1 and 2.6 were treated the same way, as well as samples 2.2 and 2.7. The only difference between the samples is the dyestuff.

Hereby the samples can be compared and it can be concluded that Verde Malaquita resulted in higher color strength values and therefore is more suitable for this use. This proves the fact that cationic dyes are more effective for dyeing cotton. Acid dye is more effective on protein fibers.

In this project the dyebath used was acidic to make the dyestuffs able to bind but usually the dyebath used for cotton is alkaline. All samples washed after dyeing also lost a lot of dyestuff during the wash. Acidic dyebath might have reduced the dyestuff’s ability to attach to the textile and reduce the color fastness.

5 CONCLUSIONS

The results of the experiments conducted in this project show that Ultra Fine Cellulose polymer in a coating paste by itself is not able to bind to the textile surface. UFC coating without binder does not remain solid during washing and dyeing procedures and a great amount of UFC is lost to the washing and dyeing baths. For a functional coating paste containing UFC, binder is always needed to bind the coating to the fabric.

Binder is also the key factor to a successful penetration of dyestuff. UFC coating paste without binder is incapable of receiving dyestuff. Samples coated with paste including binder had the highest K/S values in the coated parts of the sample with both acid and cationdic dyestuff. The K/S values ranged from 5.2 to 21.3. The samples without binder had the lowest K/S values in the coated parts of the sample with both dyestuffs. The K/S values in the samples without binder ranged from 0.3 to 1.4.

Washing before dyeing increases the color strength of the textile in most cases because the K/S values are higher in the samples washed before dyeing. Washing removes residues of substances left on the textile in previous treatments and possible stains are also removed during the washing procedure. The anionic washing detergent used in the washing bath can also affect the sample and improve the penetration of the cationic and acid dyestuffs.

Cationic dye is better for dyeing cotton than anionic dyestuff. Acidic dyebath used to dye cotton reduces the penetration of the dyestuff in both coated and non-coated parts of the textile. This can be seen as a great amount of dyestuff left in the dyeing baths and weak color fastness in washing. Cationic dyestuff resulted in higher K/S values (ranged from 4.4 to 21.3) than acid dye (ranged from 1.7 to 5.6).

Future experiments could concentrate on experimenting more with cationic dyestuff and specify the needed amount of binder for functional nanocellulose coating paste. A lot of research has been done about nanocellulose materials for other purposes but there are still a lot of undiscovered possibilities for cellulose in the textile industry. An environmental friendly and natural choice for raw material is needed in the future of textile structures.

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