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Treatment of Kynol fiber materials – Part 1: dyeing processes

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ABSTRACT

*Kynol fibers are excellent flame-retardant materials used for manifold applications. These fibers exhibit originally an orange coloration. Up to now, dyeing processes for Kynol fibers are less reported, and the functionalization of these fibers by finishing processes is less investigated. With this background, the main aim of this paper is the evaluation of different dyeing processes for Kynol fiber materials – a woven fabric and a non-woven material. The following types of dyestuff are evaluated – direct dyes, vat dyes, disperse dyes and color pigments. The reached color quality is determined by CIE L*a*b* indices. Dry and wet rubbing fastness is measured. Finally, best results are gained by application of disperse dyes. The gained results could be used in future also to develop other functional treatments of Kynol fibers in different fields for, e.g., light protection, UV-stability, water repellency or antistatic properties.*

Keywords
Kynol fiber,
novoloid fiber,
High performance fibers,
dyeing,
disperse dye,
color pigment

1 Introduction

Fibers based on novoloid resins are offered under the brand name Kynol fibers [1]. Kynol fibers are used, e.g., for preparation of sealing materials and in heat shielding applications [1-3]. Kynol fibers contain originally a strong orange coloration, which is related to a low reflection of UV light and blue light [4]. This orange coloration is a property of the Kynol fibers by themselves and caused by the aromatic chemical structure of the fibers. The orange coloration cannot be removed by bleaching processes without drastically damaging the fiber. Kynol fibers belong to the group of high performance fibers [5]. High performance fibers are fibers containing at least one outstanding property, which conventional fibers do not have. These outstanding properties can be e.g. high strength, high chemical stability, high thermal stability or flame-retardant properties [6]. Kynol fibers are flame-retardant fibers.

The chemical structure of this fiber contains 76 wt.% carbon (C), 18 wt.% oxygen (O), and 6 wt.% hydrogen (H) [7], compare also the chemical structure depicted in Figure 1. Due to the high carbon content, this fiber can be used as precursor in production processes for carbon fibers. Such carbon fibers are especially used as activated materials dedicated for the absorption of chemicals [8, 9]. Kynol fiber comprises a lot of bulk aromatic phenolic groups. For this, the thermal conductivity of this fiber is low. Further, this fiber has high LOI-values (Limiting Oxygen Index) of 30–34% and a maximum temperature of usage (T_{max}) of 150 °C [5]. In other references also a maximum continuous operating temperature of 200 °C is mentioned [10]. Due to the 3-dimensional cross-linked chemical structure, Kynol fibers do not melt. The smoke generated from this fiber comprises water vapor, carbon dioxide and other carbon-based components. However, due to the absence of nitrogen, sulphur or halogens in the chemical structure, the burning gases from Kynol fibers do not contain toxic components such as NO_x, SO_2, HCN, HCl or HF [11, 12].

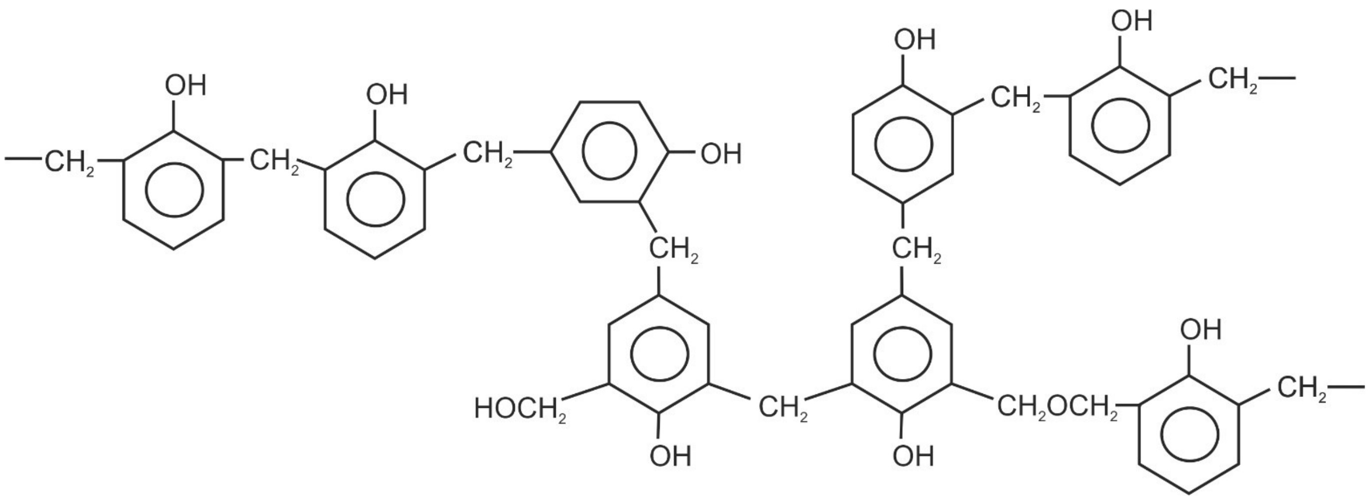

Fig. 1 Chemical structure of novoloid resin building up the Kynol fibers.

Similar to other high-performance fibers, Kynol fibers are difficult to dye with conventional dyeing processes [13-16]. The objective of the actual investigation is to develop the dyeing and finishing procedures for coloration with good fastness properties to Kynol fiber materials. Up to now dyeing processes for Kynol fibers are less reported. Nevertheless, there are several interesting patents from the year 1974 reporting on the dyeing of Kynol fibers with disperse dyes, cationic dyes and anionic dyes [17-20]. The main inventor in that times was James Economy [17-19]. For Kynol fiber materials, its orange coloration has to be considered in the formulation of dyes to achieve a desired shade. Beside the achievement of new coloration, a second aim for dyeing of Kynol fibers is the possibility to gain a better sunlight and UV light stability after dyeing [21-23].

By view on the chemical structure of Kynol fibers (Figure 1), it is clear that different types of dyeing strategies could be explored for Kynol fibers. The aim of dyeing processes is a high up-take of dye by the fiber resulting in a strong coloration. Further, the fixation of the dye on the fiber aims at leading to a strong rubbing and washing fastness. Dye up-take and fixation are reached if there is a strong fiber-dye-interaction [24, 25]. The chemical structure of Kynol fibers exhibits many hydroxy groups (-OH) which usually lead to a certain affinity to direct dyes or vat dyes. Both these types of dyes are used in conventional dyeing process for cotton materials. Also, Kynol fibers exhibit a huge number of aromatic units, which are related to hydrophobic properties. For this, the dyeing with hydrophobic disperse dyes could as well be possible. With this background, in the current study direct dyes, vat dyes and disperse dyes are applied and evaluated for dyeing of Kynol fibers. Further, color pigments are applied, which are fixed on the fiber by using a binder.

Dyeing trials are first performed to establish the shades that could be expected with individual dyes. The wet and dry rubbing fastness of dyed Kynol fiber materials are measured afterwards for promising samples. The color properties of the dyed Kynol woven and non-woven fabric samples are documented by using CIE L*a*b* indices.

2 Experimental Section

2.1 Textile Materials

For dyeing experiments, two different types of Kynol materials are used – a non-woven fiber felt and a woven fabric. These materials are supported by the company Kynol Europa GmbH (Hamburg, Germany). Both materials contain a significant gold or orange coloration. The weight per area is 209 g/m^2 for the non-woven material and 297 g/m^2 for the woven fabric. Microscopic images of used Kynol fiber materials are presented in Figure 2. Also, the typical orange coloration of those materials is shown here.

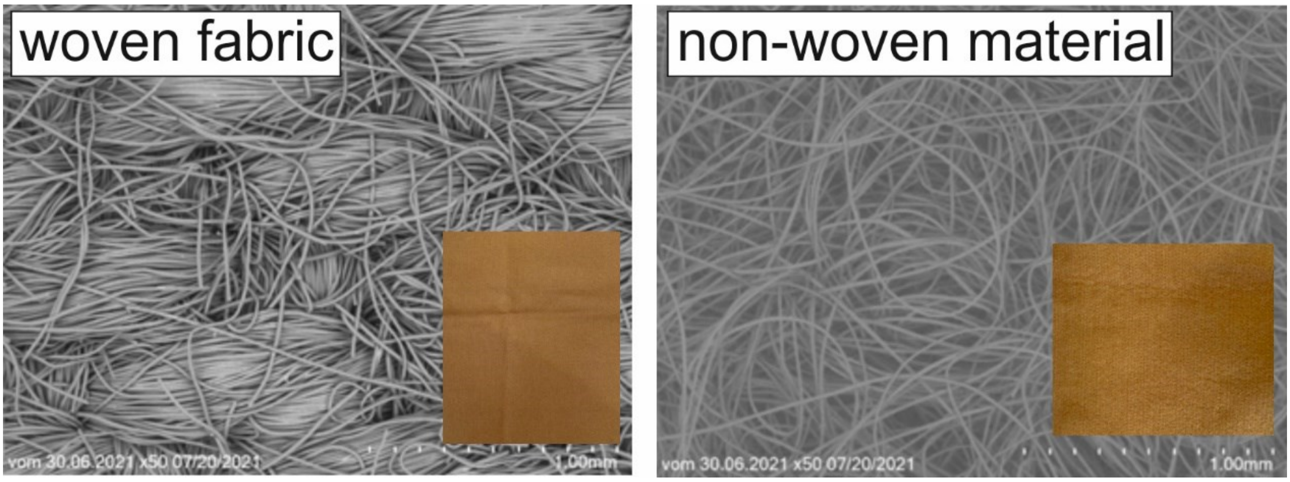

Fig. 2 Microscopic images of investigated woven fabric and non-woven materials. Images taken by scanning electron microscope (SEM). The inlays show photographs of these materials.

2.2 Dyeing procedures

Four different types of dyestuffs are evaluated for dyeing Kynol fiber materials – vat dyes, direct dyes, disperse dyes and color pigments. The vat dyes used are TECOTHREN DUNKELBLAU DB U.D. and TECOTHREN BRILL. GREEN FFB U.D. from Textilcolor GmbH (Pfullingen, Germany). The colors are dark blue and green. The direct dyes used are SIRIUS® Light Red F4BL and SIRIUS® Royal Blue S from DyStar. The colors are light red and blue. The disperse dyes used are Dianix Red AC-E and Dianix Blue AC-E supplied by DyStar. The colors are red and blue. The color pigments used are COLORMATCH 400 GREEN and COLORMATCH 360 DENIM from CHT Switzerland AG. The colors are green and denim. As dyeing machine for application of the vat dyes, direct dyes and disperse dyes, a Polycolor supplied by Zeltex AG is used. The liquor ratio for dyeing in this machine is set to 1:10.

The recipes used for application of the vat dyes are shown in Table 1 and Table 2. Beside the vat dyes, the following additives are used: Meropan DPE as dispersing agent (from CHT Germany GmbH), Kollasol CDS as surfactant (from CHT Germany GmbH) and Sarabid VAT as equalizing agent (from CHT Germany GmbH). The compositions of these recipes are developed with the help of the software WellVat supported by CHT. After keeping the Kynol materials for 25 minutes at 25 °C in these vat dye recipes, the temperature is increased to 60 °C. The heating rate is 3 K/min. The process duration at 60 °C is 45 minutes, before a cold reduction is performed with 2 ml/l caustic soda and 2 g/l sodium hydrogensulphite. The rinsed fabrics are then passed through oxidation with Sera Con M-LU (supplied by DyStar). At last, the samples are cleaned in a Mathis SOAPY by soaping with Beixon AB (dispersing agent supplied by CHT Germany GmbH). Finally drying at room temperature is performed.

Table 1. Vat dye recipe of TECOTHREN DUNKELBLAU – given are the concentrations of the different components and the amounts of components put together for recipe realization.

component	Concentration	Amount
MEROPAN DPE	2.00 g/l (1:10)	4.0 g
KOLLASOL CDS	0.5 g/l (1:50)	5.0 g
TECOTHREN DUNKELBLUE	3% (1:10)	3 g
Caustic soda	28.4 ml/l	5.68 g
SARABID VAT	0.5% (1:20)	2 g
Sodium hydrogensulphite	4.44 g/l	0.888 g
Soft water	----	177.32 g

Table 2. Vat dye recipe of TECOTHREN GREEN FFB U.D. – given are the concentrations of the different components and the amounts of components put together for recipe realization.

component	Concentration	Amount
MEROPAN DPE	2.00 g/l (1:10)	4.0 g
KOLLASOL CDS	0.5 g/l (1:50)	5.0 g
TECOTHREN BRILL GREEN	3% (1:10)	3 g
Caustic soda	21.6 ml/l	4.32 g
SARABID VAT	0.5% (1:20)	2 g
Sodium hydrogensulphite	4.31 g/l	0.888 g
Soft water	----	178.68 g

The recipes containing direct dyes for treatment of 20 g woven Kynol fabric contain the following components – 12 g direct dye, 25 g sodium sulphate and 163 g soft water. For treatment of 10 g non-woven Kynol material, a recipe is used containing 6 g direct dye, 25 g sodium sulphate and 169 g soft water. The used recipes are developed following the recommendations of the supplier of the direct dyes. For application of these recipes, a dyeing temperature of 95 °C with a process duration of 35 minutes is used. The heating rate is 3 K/min. After the drying process, the fabrics are rinsed with cold water and then dried at room temperature.

The recipes used for application of the disperse dyes are shown in Table 3 and Table 4. Beside the disperse dyes, the dispersing agent Sera gal PLP (DyStar) is used. The used recipes are developed following the recommendations of the supplier of the disperse dyes. For application of the recipes with the disperse dyes, a dyeing temperature of 135 °C is used with a process duration of 60 minutes. For this, the temperature is quickly raised in the Polycolor device to 85 °C, following the heating rate is set to 1.5 K/min. After dyeing process, the bath is cooled down to 60 °C for 10 minutes. Then a reductive cleaning is done with a reductive bath containing 1 g/l Sera gal PLP, 5 ml/l (1.36 g/ml) NaOH 33%, 4 g/l sodium hydrogensulphite and 1 liter of soft water. This reductive bath is applied at 85 °C for 15 minutes. Finally, rinsing with water and drying at room temperature is performed.

Table 3. Disperse dye recipe of Dianix Red AC-E – given are the concentrations of the different components and the amounts of components put together for recipe realization.

Product name	**Concentration**	**Amount**
Dianix Red AC-E	3 % (1:10)	3 g
Sera gal PLP	1 % (1:20)	2 g
Acetic Acid	0.5 g/l (1:10)	0.5 g
Soft water	----	94.5 g

Table 4. Disperse dye recipe of Dianix Blue AC-E – given are the concentrations of the different components and the amounts of components put together for recipe realization.

Product name	Concentration	Amount
Dianix Blue AC-E	3 % (1:10)	6 g
Sera gal PLP	1 % (1:20)	2 g
Acetic Acid	0.5 g/l (1:10)	0.5 g
Soft water	----	91.5 g

The application of color pigments on Kynol materials is done by a padding process. The recipes used for application of color pigments are shown in Table 5. The components Sera Fix C-PD (acrylate binder) and Sera Pad M-PD (antimigration agent) from DyStar are added as textile auxiliaries. After stirring the recipe together, the pH of this solution is set to 6 by adding acetic acid. The used recipes are developed following the recommendations of the suppliers of color pigments and textile auxiliaries. The treatment by padding is done twice for each fabric. After padding, drying is performed at 120 °C for two minutes. Finally, thermofixation is done for 45 seconds at 190 °C.

Table 5. Color pigment recipe for treatment of Kynol fiber materials in a padding process – color pigments: COLORMATCH 400 GREEN or COLORMATCH 360 DENIM – given is the concentration of the different components and the amounts of components put together for recipe realization.

Product name	Concentration	Amount
Soft water	----	100 ml
Sera Fix C-PD	35.0 g/l	7g
Sodium sulfate	7.5 g/l	1.5 g
Sera Pad M-PD	15.0 g/l	3 g
Color pigment	10 g/l	2g

2.3 Analytical methods

For determination of dyeing results of the prepared samples, the CIE L*a*b* indices are measured [26]. These measurements are done with a device DATA Color 400 (Rotkreuz, Switzerland). The wet and dry rubbing fastness is measured using a crockmeter according to ISO 105-X12. For this rubbing test, specimens of the textile are rubbed with a dry rubbing cloth and with a wet rubbing cloth. The change of coloration is determined using a Gray Scale evaluating the staining of the rubbing cloth. The microscopic images shown in Figure 2 are recorded with a scanning electron microscope (SEM) Tabletop TM4000 from Hitachi (Japan).

3 Results and Discussion

3.1 Color Properties

After the dyeing process, the coloration results are evaluated by color measurements determining the CIE L*a*b* indices. However, these measurements are not done for the samples treated with the vat dyes, because of the strong irregularities in color up-take (please compare Figure 3). Due to this irregular up-take of the vat dyes, vat dyes are further no longer considered and probably not useful for dyeing of Kynol fibers.

For treatment with the other dye and color pigment recipes, the CIE L*a*b* indices are determined and compared to the indices of the untreated Kynol materials. Figure 4 shows the results for the woven Kynol fabrics, while Figure 5 presents the results for the non-woven Kynol materials. To support a complete view on the realized coloration in the following Figures 6 to 9 also the photographs of the dyed Kynol fiber materials are presented. The index L* represents the brightness of the fiber material. The decrease of index L* after dyeing is related to a darker coloration. The index a* represents a color shift from red to green coloration. A decrease of index a* is related to a decrease of red coloration of Kynol fibers.

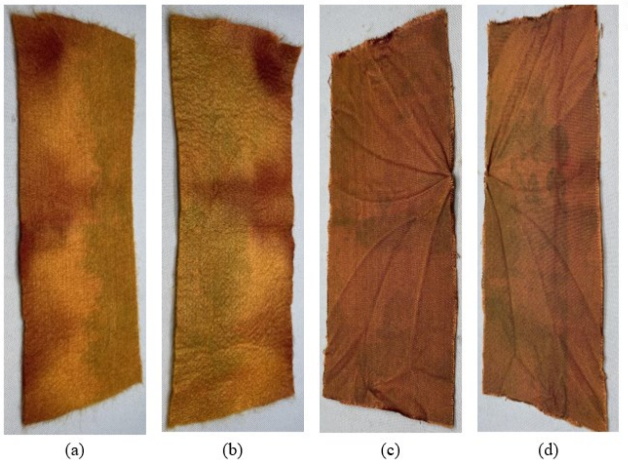

Fig. 3 Vat dyeing results (TECOTHREN BRILL. GREEN FFB U.D.), (a) face side of Kynol non-woven fabric, (b) reverse side of Kynol non-woven fabric, (c) face side of Kynol woven fabric, (d) reverse side of Kynol woven fabric.

The index b* describe the color shift from yellow to blue, with positive values for yellow and negative values for blue coloration [15].The typical orange coloration of untreated Kynol fiber material is documented by L* = 48, a* = 22 and b* = 48 for woven fabrics (Figure 4). The non-woven material exhibits nearly the similar indices (Figure 5). By application of both direct dyes, the indices L* and b* are only slightly changed, while the index a* is not changed (compare also photographs in Figure 6). The coloration gained from applications with direct dyes is slight, probable because of low dye up-take by the fiber materials. For this, direct dyes are probably not useful for application on Kynol fibers. In contrast, after application of the disperse dyes a clear color change is obvious, which is also determined by significant changes of CIE L*a*b* indices. The indices L* and a* are decreased, especially after use of the blue disperse dye. These results are related to a darker and greener coloration (compare also photographs in Figure 7). The dye up-take is significant. The main difference between the red and blue disperse dye is shown with the index a*. After application of disperse red, index a* is even slightly increased. Here, the original orange coloration of Kynol fibers is intensified to red by the up-take of the red dye. However, because of the similarity in coloration of naturally orange fibers and the red disperse dye, the realized change in coloration is not that strong. In comparison, by application of disperse blue the change in coloration is more significant, with a decrease of index a* down to a value of 9 for the non-woven material. Here, a clear shift from the originally orange coloration to a darker blue/brown coloration can be stated.

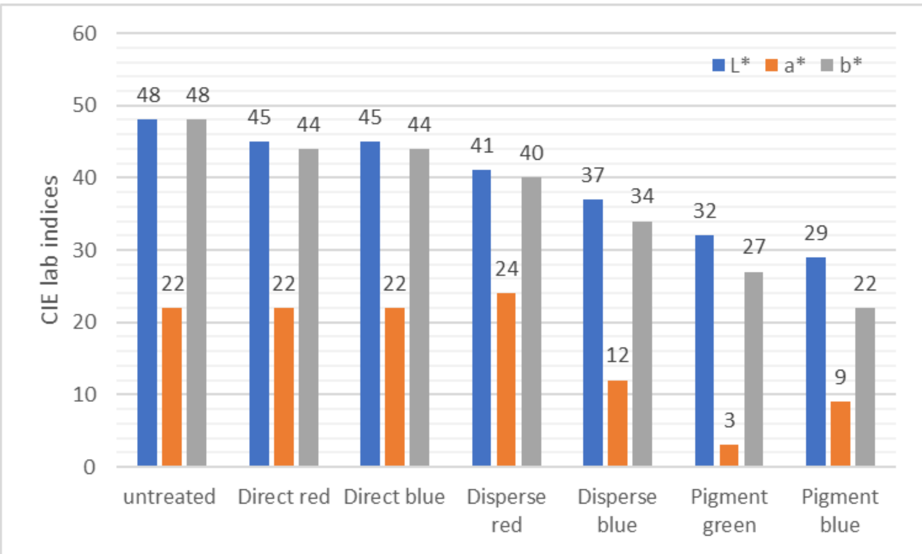

Fig. 4 CIE lab indices of woven Kynol fabrics after treatment with different dyestuffs.

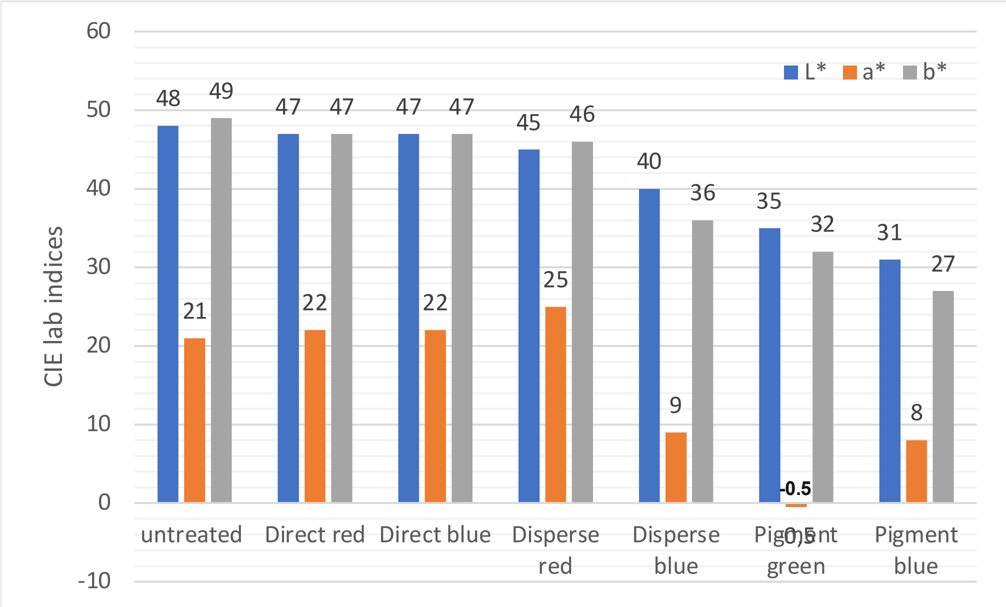

Fig. 5 CIE lab indices of non-woven Kynol material after treatment with different dyestuffs.

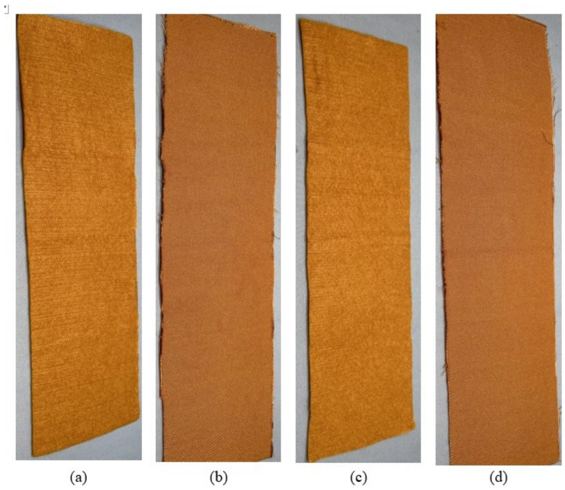

Fig. 6 Direct dyeing results, (a) SIRIUS® Light Red F4BL 154 % dyed Kynol non-woven fabric, (b) SIRIUS® Light Red F4BL 154 % dyed Kynol woven fabric, (c) SIRIUS® Royal Blue S dyed Kynol non-woven fabric, (d) SIRIUS® Royal Blue S dyed Kynol woven fabric.

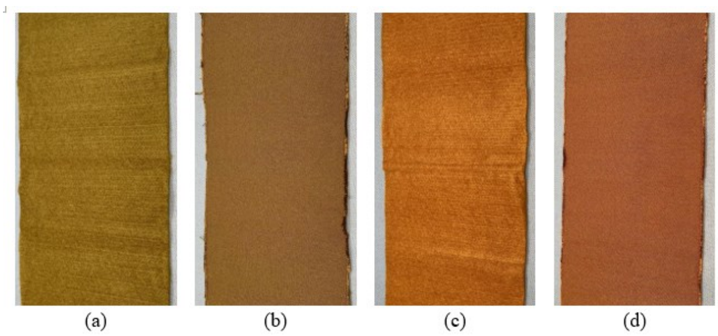

Fig. 7 Disperse dyeing results, (a) Dianix Blue AC-E dyed Kynol non-woven fabric, (b) Dianix Blue AC-E dyed Kynol woven fabric, (c) Dianix Red AC-E dyed Kynol non-woven fabric, (d) Dianix Red AC-E dyed Kynol woven fabric.

An even stronger coloration effect is reached by the application of both color pigments (Figures 4 and 5), compare also photographs in Figures 8 and 9. This strong change is probable also related to coverage of the Kynol fibers by the color pigments. Here, the orange fiber coloration is covered behind the applied pigments.

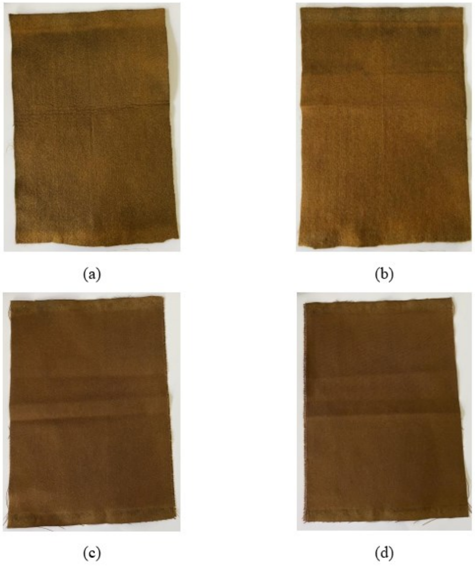

Fig. 8 Blue pigment application results (COLORMATCH 360 DENIM), (a) face side of Kynol non-woven fabric, (b) reverse side of Kynol non-woven fabric, (c) face side of Kynol woven fabric, (d) reverse side of Kynol woven fabric.

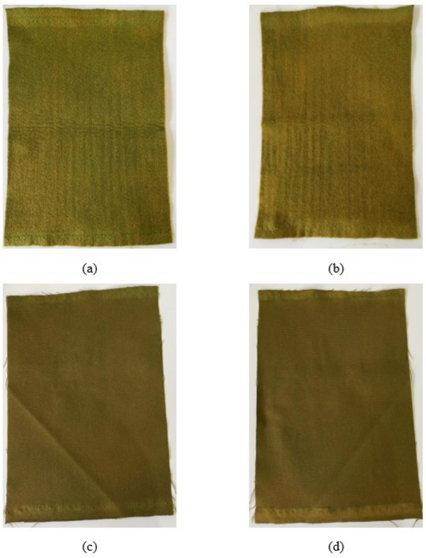

Fig. 9 Green pigment application results (COLORMATCH 400 GREEN), (a) face side of Kynol non-woven fabric, (b) reverse side of Kynol non-woven fabric, (c) face side of Kynol woven fabric, (d) reverse side of Kynol woven fabric.

3.2 Rubbing fastness

The fixation of the dyes and color pigments on the fiber materials is determined by rubbing under dry or wet conditions. The rubbing fastness is only evaluated for samples treated with disperse dyes or color pigments (Figures 10 and 11). Only for those recipes a regular and significant change in coloration is received. The grade of rubbing fastness is determined in the gray scale in the range of 1 to 5, with grade 5 as best value. This grading system is related to a color transfer from the dyeing Kynol material to a white cotton fabric sample, which is rubbed on the Kynol fabric. In case of grade 5, there is no color transfer, while a sample with grade 1 shows a strong color transfer to the cotton sample. Usually the wet rubbing fastness, determined by rubbing with a wet cotton fabric, leads to lower grade in rubbing fastness.

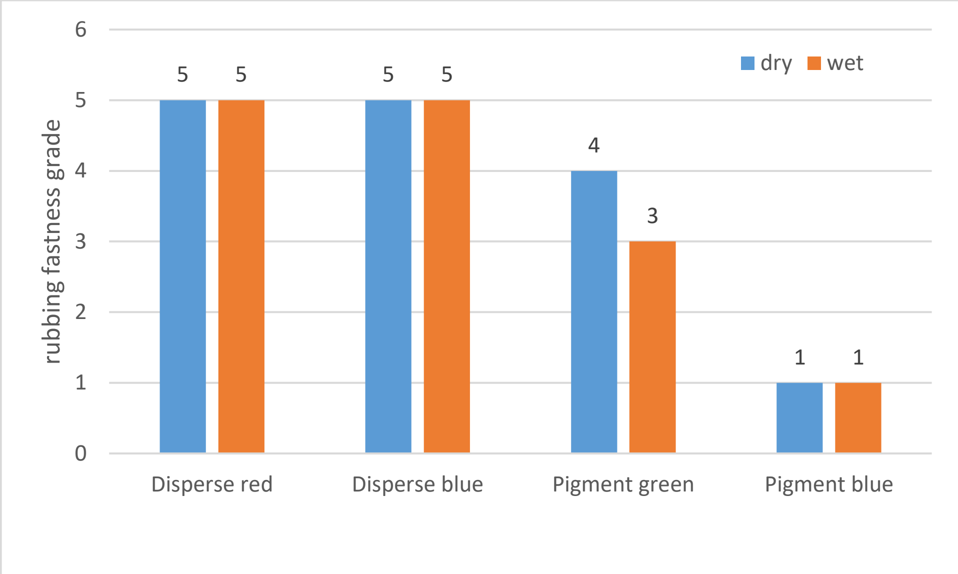

Fig. 10 Rubbing fastness of woven Kynol fabrics after treatment with different disperse dyes and color pigments. Determined is the color transfer to a white cotton fabric.

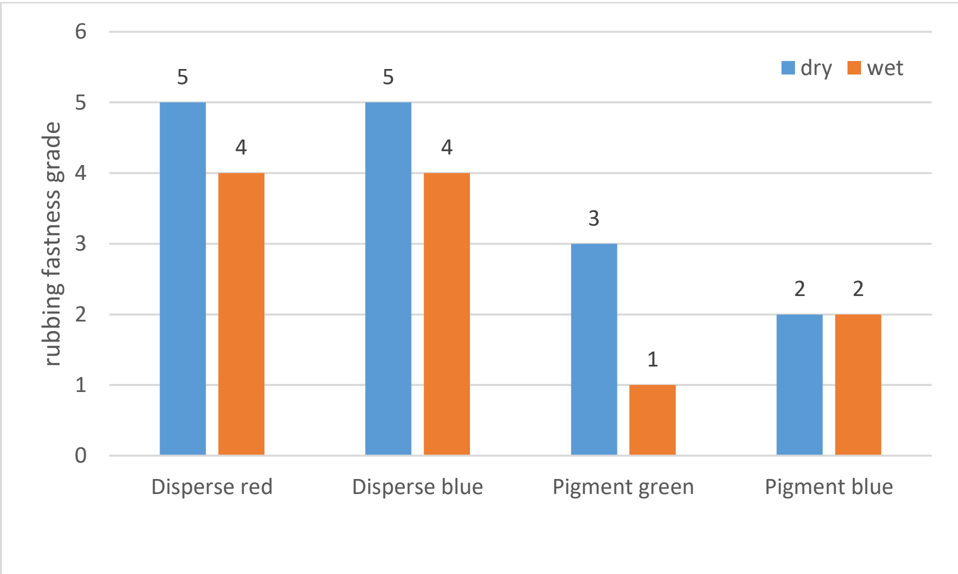

Fig. 11 Rubbing fastness of non-woven Kynol material after treatment with different disperse dyes and color pigments. Determined is the color transfer to a white cotton fabric.

By view on the rubbing results, it is clear that samples dyed with the disperse dyes exhibit excellent rubbing fastness, while for the pigment containing samples the grades are weaker (Figures 10 and 11). Also, the wet rubbing fastness is weaker compared to the dry rubbing fastness. The different results for disperse dyes and color pigments could be explained by the hydrophobic disperse dyes being probably able to penetrate into the hydrophobic fiber structure under the chosen process temperature for dyeing. At room temperature the dye molecules are physically embedded into the polymer structure of the resin fiber and cannot be removed by simple rubbing. In contrast, the color pigments are placed on the fiber surface and fixed by a binder on the fiber. On the fiber surface, the applied pigments are strongly exposed to the mechanical influence of rubbing. For samples with pigment application, only the one with the green color pigment under dry rubbing test exhibits a sufficient fastness grade. These different grades of rubbing fastness of both pigment applications could be explained by different interaction between the used binder and the different used color pigments. Nevertheless, it has to be stated that only with the disperse dye application a significant coloration depth and fastness properties can be reached on Kynol fibers.

4 Conclusions

The dyeing properties of Kynol fibers are evaluated with three different types of dyestuffs – vat dyes, direct dyes and disperse dyes. Also, the application of color pigments is evaluated. Only by application of disperse dyes a significant color change and a rubbing fastness are reached. The application of a red disperse dye intensifies the originally orange coloration of Kynol fibers towards a red coloration. By applying a blue disperse dye, also dark coloration can be reached for Kynol fibers. The mechanical properties (as textile strength or modulus) are not investigated after the dyeing process. A detailed investigation of the mechanical properties goes beyond the scope of this paper. Nevertheless, it can be stated that the dyeing processes mainly do not influence the hand feeling of the Kynol fiber materials.

The gained results can be useful in future not only for dyeing purposes. By this, also the stability of Kynol fibers against UV-light could be improved, due to UV absorption properties of dyestuffs. The dyeing processes can be also taken as a proof-of-concept for the application of conventional hydrophobic UV-absorbers which could be applied in similar processes as described for the disperse dye application.

Author Contributions

The presented results are based on the master thesis of Juan Wang performed at the Hochschule Niederrhein in the year 2021. Boris Mahltig acted as supervisor of this master thesis and wrote the current article based on the results of the master thesis. Both authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest

The authors declare no conflict of interest.

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