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Treatment of Kynol fiber materials – Part 2: antistatic and water-repellent functionalization

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ABSTRACT

Kynol fibers are flame retardant high performance fibers. Without further treatment they do not exhibit significant antistatic or water-repellent properties. With this background, several conventional antistatic agents and one hydrophobic finishing agent are evaluated to introduce these properties to Kynol fiber materials. Also, the combination of both properties is investigated to realize bifunctional fiber materials. By application of antistatic finishing agents, the electric surface resistance of Kynol fiber materials can be decreased to values smaller 10^8 Ω, which is a good value related to antistatic properties. By application of the hydrophobic agent, water repellent properties can be introduced to Kynol fiber materials. However, simultaneously the antistatic properties are decreased. The combination of both types of agents in a kind of bifunctional finishing can lead to intermediate antistatic effects combined with an intermediate water repellency. Nevertheless, in bifunctional application no single excellent property is gained. The flame retardant properties are tested on selected samples after the finishing processes. No change in flame retardant properties is determined, if the finishing agents are applied. For this, the presented results can be the starting point for development of functionalized flame retardant Kynol fiber products.

Keywords
Kynol fiber,
Novoloid fiber,
high performance fibers,
antistatic,
water-repellent,
hydrophobic,
double functional finishing
bifunctional fiber material

1 Introduction

Kynol fibers are flame resistant high-performance fibers based on novoloid resins which are prepared by condensation reaction of formaldehyde with phenol [1-6]. These phenol resins are also named as phenoplastics. A schematic polymer structure of such a phenol-formaldehyde resin is presented in

Figure 1. This Figure 1 gives a simple description of the main repeating unit of the resin without showing the polymer end-groups or the also possible three-dimensional network formation [7]. The chemical composition of Kynol fibers is determined by the ratio of the contained chemical elements carbon and oxygen. Compared to the oxygen content, the content of carbon is four times larger. This is a high value especially in comparison to polyester fibers and related to the aromatic structure of Kynol fibers [8]. Applications for Kynol fibers are developed in the area of fire-resistant clothes and heat insulation materials with self-extinguishing properties [9-12]. The flame resistant properties are determined with a Limiting Oxygen Index (LOI) value in the range of 30-34 according to supplier information [13]. Additional to this high LOI value, Kynol fiber materials contain further several advantages for flame resistant applications, such as a non-melting behavior, minimal evolution of smoke, nearly no shrinkage and no development of halogen containing gases in the smoke [14]. Also, no HCN or NO_x are developed in the flame gas of Kynol fibers [14]. Kynol fibers exhibit originally a typical orange coloration and a specific density of 1.27 g/cm^3 [15]. The coloration of Kynol fibers can be modified by different dyeing processes as presented in several patent and in an earlier comparative investigation [16-20]. For dyeing of Kynol fibers, especially the application of disperse dyes is advantageous [20]. At standard conditions of 20 °C and 65% relative humidity, these fibers exhibits a moisture regain of 6% [13,15]. This moisture regain is quite high compared to other synthetic fibers, especially polyester based fibers as PET or PLA. This high moisture regain of Kynol fibers is probably caused by the hydroxy functional groups of the novoloid structure. Conventional polyester fiber materials from PET exhibit in comparison only a moisture regain smaller 0.5% [15, 21]. PET fibers and Kynol fibers exhibit both aromatic elements in their chemical structure, but only Kynol fibers contain also significant amounts of hydrophilic hydroxy groups.

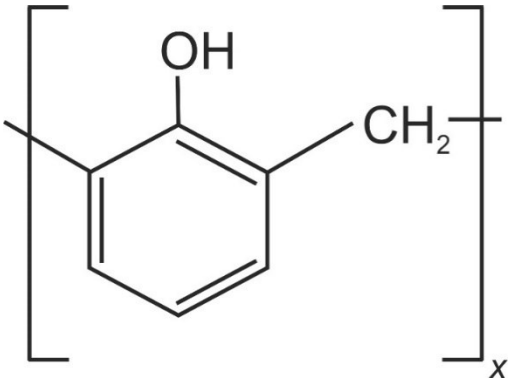

Fig. 1 Schematic polymer structure of phenol-formaldehyde resin. Simple description without showing the also possible three-dimensional network formation.

The antistatic property of a fiber material describes its ability to avoid the building up of static electric charges, caused e.g. by rubbing on another material [22,23]. Such static charging should be avoided for different reasons [24,25]. Especially after spontaneous discharging and spark formation, damages of electronic devices can happen, or the ignition of explosive gases is possible [24,25]. Also, increased staining of textile materials is possible, caused by electrostatic attraction of dust and dirt particles [25]. This increased staining is especially discussed for filter materials and carpets [26]. For textile materials, there are different methods to realize antistatic properties. One often used method is the treatment with hydrophilic finishing agents. Once applied, these hydrophilic agents increase the humidity up-take of synthetic fibers [22]. The up-taken humidity decreases the electric surface resistance of the fiber material due to the conductivity of up-taken water [25]. In contrast to the hydrophilic agents, hydrophobic agents are applied to realize water-repellent and soil-repellent textiles [27].

Kynol fiber materials exhibit originally only slight antistatic or water-repellent properties. For this, the aim of this study is to evaluate several conventional finishing agents for the realization of both properties on Kynol fibers. Also, experiments are performed with the aim of combining both antistatic and hydrophobic properties together on Kynol fibers.

2 Experimental Section

2.1 Textile Materials

For finishing experiments, two different types of Kynol fiber materials are used – a non-woven fiber felt and a woven fabric. Both materials are supported by the company Kynol Europa GmbH (Hamburg, Germany). The weight per area is 209 g/m^2 for the non-woven material and 297 g/m^2 for the woven fabric. The surface electric resistance of the Kynol non-woven fabric is 3.2×10^{11} Ω, that of Kynol woven fabric is 3.5×10^{13} Ω. Both materials do not exhibit antistatic properties or significant water-repellent properties.

2.2 Finishing processes

Finishing processes are performed with different finishing agents to realize antistatic properties or water-repellent properties. Also, a combination of both types of finishing agents is evaluated to realize both functional properties together. For padding application, a laboratory padding equipment is used (CH-8155 Niderhasli/Zürich Laboratory padding Equipment, Werner Mathis AG, Switzerland). For sample drying a device Labor-Universal-Dämpfer Typ DHe (Werner Mathis AG, Switzerland) is used.

2.2.1 Antistatic finishing

The antistatic agents used in this experiment are ZEROSTAT FC NEW from Huntsman Textile Effects (Germany) GmbH, AVISTAT G 100 and AVISTAT AZ NEW from CHT Switzerland AG. These three different antistatic finishing agents are selected for evaluation of anionic, cationic and non-ionic compounds on the Kynol fiber materials. ZEROSTAT FC NEW is an anionic phosphorus organic compound. It is a clear and colorless liquid which can be applied by padding method. According to supplier information, it is compatible with oil- and water-repellent finishes. AVISTAT G 100 is a non-ionic agent based on fatty acid and polyglycol esters. It is a clear liquid with brownish-yellow coloration. It can be applied by padding method. AVISTAT AZ NEW is a cationic agent based on a fatty acid condensation product. According to supplier information, this antistatic agent can also lead to softening properties. The actual antistatic finishing applications are performed with three different concentrations for each of the three antistatic agents. These concentrations are chosen according to the supplier recommendation given in the product data sheet and listed here in Table 1. These concentrations are chosen to support a comparison of these finishing agents under the aspect of industrial application. For padding application, the supplied finishing agents are diluted with soft water to realize the aimed concentration. The pressure at padding machine application is set to 2 bar. The wet pick up ratio of antistatic finished Kynol woven fabrics is around 60 wt.% and for the non-woven materials the wet pick up is around 100 wt.%. After the padding application, the samples are dried at 150 °C for 2 minutes.

Table 1. Concentration of antistatic agents used in the padding application. The used concentrations are chosen according to the recommendation of the supplier of these finishing agents.

Finishing agent / Concentration	ZEROSTAT FC NEW	AVISTAT G 100	AVISTAT AZ NEW
Low	5 g/l	3 g/l	5 g/l
Medium	7.5 g/l	4 g/l	7.5 g/l
High	10 g/l	5 g/l	10 g/l

2.2.2 Water repellent finishing

The hydrophobic finishing agent RUCO-DRY DHY is used. This agent is supplied by Rudolf GmbH (Geretsried, Germany). The agent RUCO-DRY DHY is based on hyperbranched polymers in a hydrocarbon matrix and it is cationic. It can be also described as dendrimer containing finishing agent [28,29]. It is a white emulsion containing a pH in the range of 3 to 6. The agent RUCO-DRY DHY is free

of organic halogenated compounds including fluorinated carbon compounds. For current investigation RUCO-DRY DHY is applied in three different concentrations 100 g/l, 125 g/l or 150 g/l according to the recommendation of the supplier given in the product data sheet. For padding application, the supplied finishing agents are diluted with soft water to realize the aimed concentration. After the padding application, the samples are dried at 150 °C for 2 minutes.

2.2.3 Bifunctional finishing

For combination of antistatic and water-repellent properties on Kynol fiber materials, antistatic and hydrophobic finishing agents are applied both. For this, at first the antistatic agents are applied by a padding process as described above. Following the hydrophobic finishing agent RUCO-DRY DHY is applied in a concentration of 150 g/l by padding. This concentration of 150 g/L is the highest earlier investigated concentration for the application of the hydrophobic agent. This high concentration is chosen, because after application of lower concentration of hydrophobic agents, the gained water repellency is less effective. After the padding application, the samples are dried at 150 °C for 2 minutes.

2.3 Analytical methods

Antistatic properties are determined as electrical surface resistance according to the standard ISO EN 1149-1. For these measurements, a Tera-Ohmmeter TO-3 is used. Before measurement of antistatic properties, the samples are stored at 65% humidity at 20 °C. The water repellent properties are tested by using a spray test according to the AATCC Test Method 22-2017 Water Repellency: Spray Test. The water/alcohol repellency of the fabrics is tested by using a drop test according to the AATCC Test Method 193-2017 Aqueous Liquid Repellency: Water/Alcohol Solution Resistance test. The flame retardant properties of Kynol fiber samples are evaluated by using a bottom-edge ignition test according to ISO 15025:2016. For this, a Flammability Tester FlexiBurn is used. The sample size is 20 cm × 8 cm. The gas type for combustion is propane. Flame height is about 40 mm and the burning duration is set to 10 seconds.

3 Results and Discussion

3.1 Antistatic functionalization

The surface electric resistance of the investigated untreated Kynol non-woven fabric is 3.2×10^{11} Ω and that of the untreated Kynol woven fabric is 3.5×10^{13} Ω, respectively. Due to these high values, the untreated Kynol fiber materials do not exhibit antistatic properties. The three different antistatic agents are applied with increasing concentration on the two Kynol fiber materials. To determine the reached antistatic properties, the electric surface resistance is measured and depicted as function of the concentration of applied finishing agents (Figures 2 and 3).

The decrease of surface resistance is clearly correlated to the increasing amount of applied antistatic finishing agents. With the highest applied concentration for all three types of agents a resistance smaller than 10^9 Ω is reached. This value of 10^9 Ω is often set to describe good antistatic properties [30]. However, other sources stated even sufficient antistatic properties for fiber materials if the resistance is smaller 10^{11} Ω [31]. For this, with all three finishing agents antistatic Kynol fiber materials can be realized. Best results are gained for application of the agent Zerostat FC New, which leads on the non-woven material even with medium applied concentration to a resistance value less than 10^8 Ω.

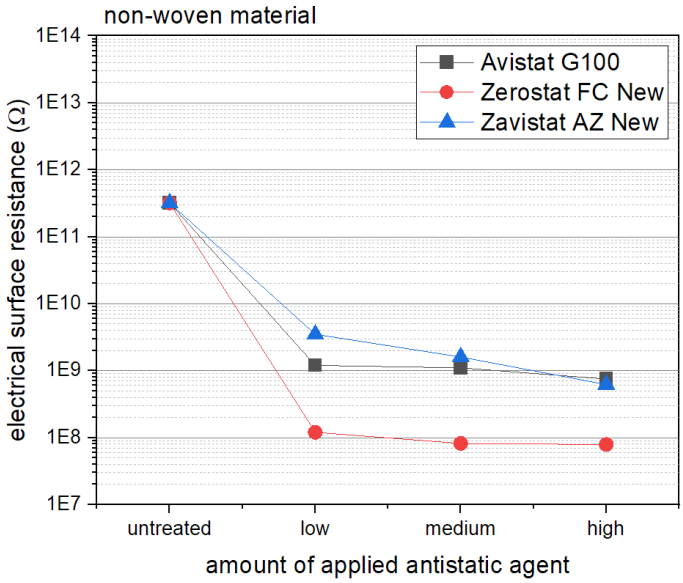

Fig. 2 Antistatic performance of different antistatic finishing agents on non-woven Kynol fiber material. The lines shown are a guide for the eyes.

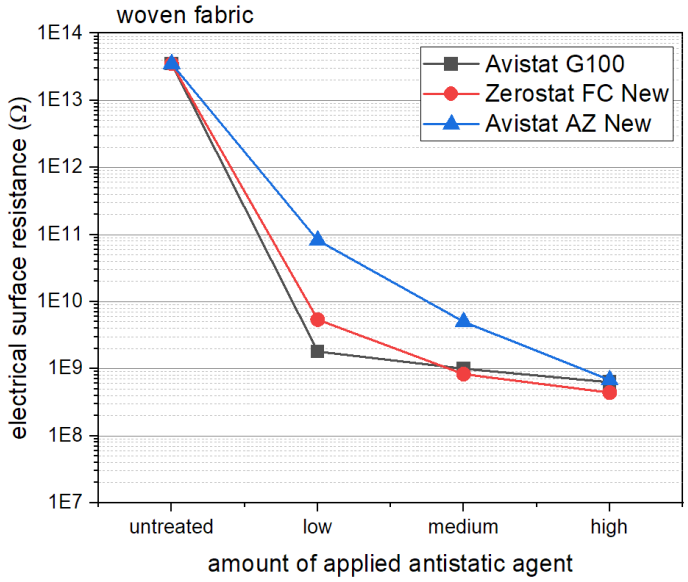

Fig. 3 Antistatic performance of different antistatic finishing agents on the woven Kynol fabric. The lines shown are a guide for the eyes.

3.2 Water repellent functionalization

Kynol fibers are synthetic fibers with certain hydrophobic properties due to their chemical structure containing huge amounts of aromatic structural elements [32]. However, for investigated untreated Kynol non-woven material no water-repellent property can be determined by testing with the water spray test – spray test result ISO 0. Also, with the water/alcohol drop test, grade 0 is determined. For untreated Kynol woven fabric slight water repellency can be determined with the spray test result of ISO 1 and the drop test result of grade 2.

The water repellency – determined by water spray test – is depicted as a function of increasing amount of applied hydrophobic agent (Figure 4). A clear effect increasing with the applied amount of agent is determined. In general, on the woven fabric stronger water repellency is reached, probably because of the denser fabric structure leading to better repellency against sprayed water drops. Similarly, the water/alcohol repellency can be introduced by the application of the hydrophobic agent (Figure 4). For this, water repellency and also a certain soil repellency can be introduced to the Kynol fiber materials. The test results

for water/alcohol repellency done by the drop tests can be correlated to repellency for water and alcohol based soils, as e.g. coffee, tea or red wine [33].

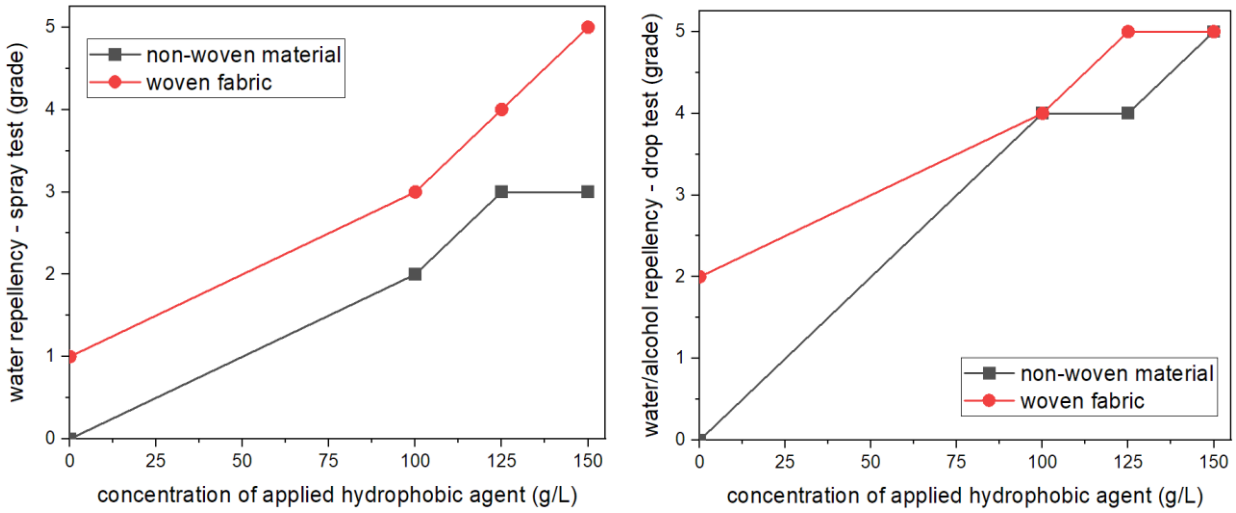

Fig. 4 Water repellency (left) and water/alcohol repellency (right) of Kynol fiber materials after application of hydrophobic agent RUCO-Dry DHY. The lines shown are a guide for the eyes.

The effect of the hydrophobic agent on the electrical surface resistance is presented in Figure 5. After the application, the resistance of the non-woven material is drastically increased to values higher 10^{13} Ω. For the woven fabric, also values higher than 10^{13} Ω are determined. With view on these numbers, it is absolutely clear that this hydrophobic agent does not support any antistatic property. To realize both hydrophobic and antistatic properties, a combination of two different types of finishing agents has to be performed to reach bifunctional properties. This issue is discussed in the following section 3.3.

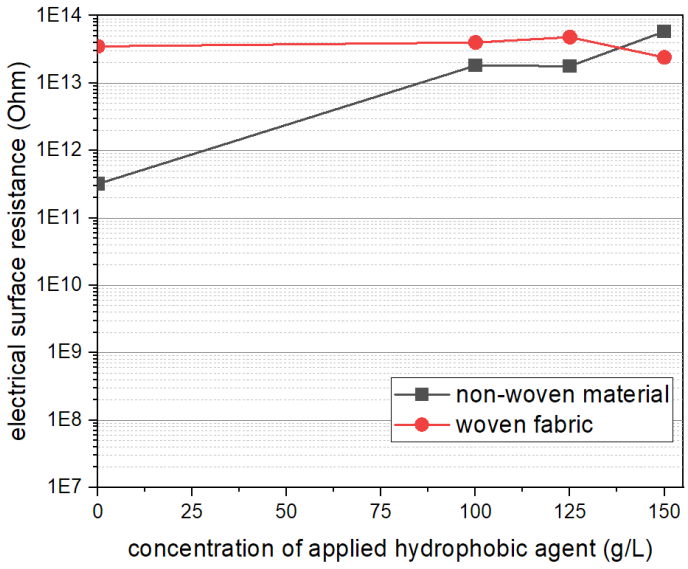

Fig. 5 Electrical surface resistance of Kynol fiber materials after application of hydrophobic agent RUCO-Dry DHY. The lines shown are a guide for the eyes.

3.3 Bifunctional treatment

To realize antistatic and water repellent properties together, at first the Kynol materials are treated with the different antistatic agents and three different concentrations are evaluated. Afterwards the hydrophobic agent is applied in the highest concentration (150 g/L), because this led previously to the strongest water repellency. Also, for this combined application a certain decrease in electric resistance as a function of applied concentration of finishing agent is determined (Figures 6 and 7). However, no

combination is observed which leads finally to a resistance smaller than 10^9 Ω. For this, a certain antistatic effect can be realized, but the reached values are not excellent.

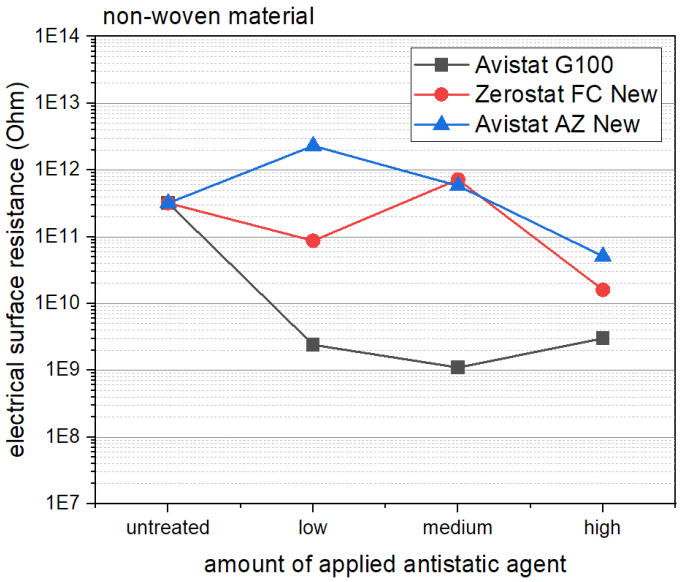

Fig. 6 Antistatic performance of different antistatic finishing agents on non-woven Kynol fiber material previously treated with the hydrophobic agent RUCO-Dry DHY. The lines shown are a guide for the eyes.

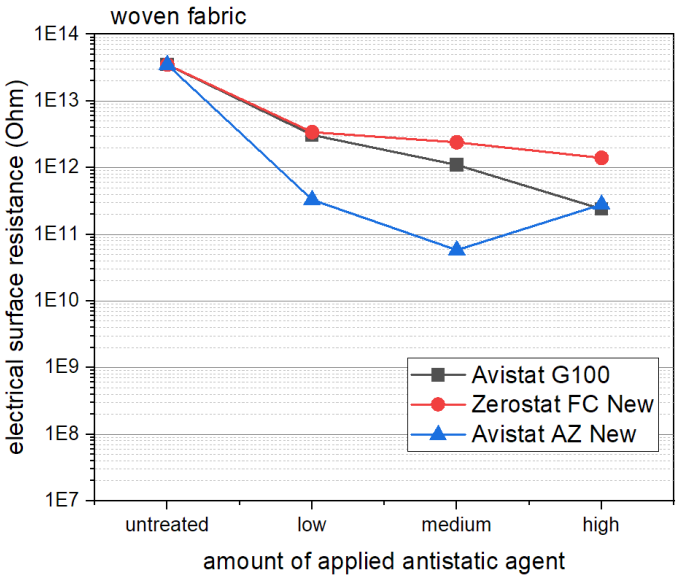

Fig. 7 Antistatic performance of different antistatic finishing agents on the woven Kynol fabric previously treated with the hydrophobic agent RUCO-Dry DHY. The lines shown are a guide for the eyes.

By view on the water and water/alcohol repellency, a decrease in properties with increasing amount of applied antistatic agent can be stated (Figure 8). Only for the non-woven Kynol material a high repellency with grade 4 is reached for the combination with lowest concentration of the antistatic agent Avistat G 100. In this combination the repellency grade is 4 and by this significant, while the resistance value is quite low with $2x10^9$ Ω. In this combination, there is probably a good compromise between the reached antistatic effect and the reached water repellency. Unfortunately, this recipe is only working on the investigated non-woven material and not with the woven fabric. A possible explanation for these different results for both fiber materials could be found in the different surface structure of woven and non-woven materials, e.g. a different surface roughness may lead to different results in water-repellency [29,34].

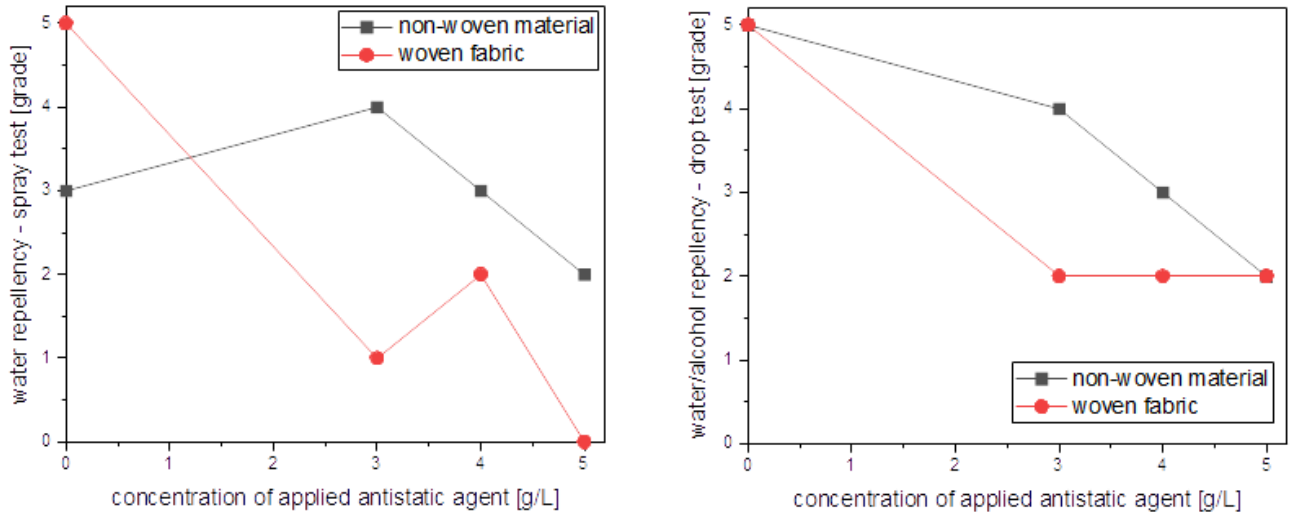

Fig. 8 Water repellency (left) and water/alcohol repellency (right) of Kynol fiber materials after application of hydrophobic agent RUCO-Dry DHY in a concentration of 150 g/L. The Kynol fiber materials are pretreated with the antistatic agent Avistat G 100 before application of the hydrophobic agent. The lines shown are a guide for the eyes.

3.4 Fire retardant properties

The flame retardant property is the key-functional property of Kynol fibers as high-performance fiber. For this reason, it has to be determined if the applied antistatic and hydrophobic finishing agents have a negative influence on the flame retardant property of Kynol fibers. For this, the fire retardant properties are determined by a bottom-edge ignition test. As reference, this ignition test is applied on untreated Kynol fiber materials as received by the supplier. As representative for functionalized Kynol fiber materials, bifunctionalized fiber materials treated with 10 g/L Zerostat FC New and 125 g/L Ruco-DRY DHY are investigated with the ignition test. The materials after the ignition tests are presented in Figure 9. After removing the ignition source from both untreated Kynol materials, the flame immediately extinguishes. There is also no after-glowing. The obtained char length is only around 3 to 5 cm. The flame retardant properties do not change by application of the mentioned finishing agents. A second ignition test is performed on a disperse dyed Kynol fiber material with even higher amount of applied finishing agent (10 g/L Zerostat FC New and 150 g/L Ruco-DRY DHY) (Figure 10). Even for this Kynol material with higher applied amount of finishing agent and the applied dye, similar flame retardant properties are determined. It can be concluded that the important flame retardant properties of Kynol fibers are not diminished by the currently applied finishing agents.

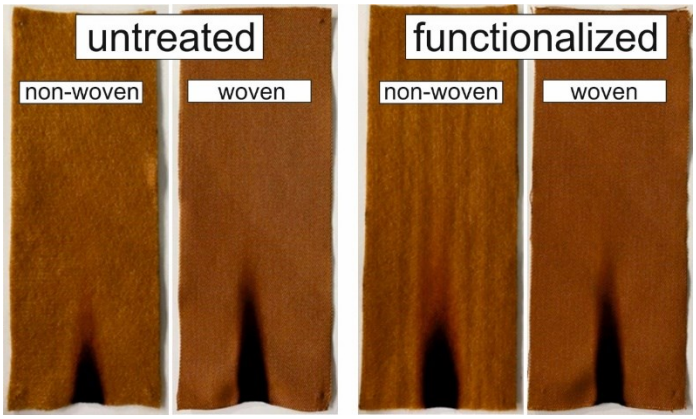

Fig. 9 Bottom-edge ignition test results. Shown are results from untreated Kynol fiber materials (left) and functionalized Kynol fiber materials (right). The functionalization is performed by a combined application of the hydrophobic agent RUCO Dry DHY (125 g/L) and the antistatic agent Zerostat FC NEW (10 g/L).

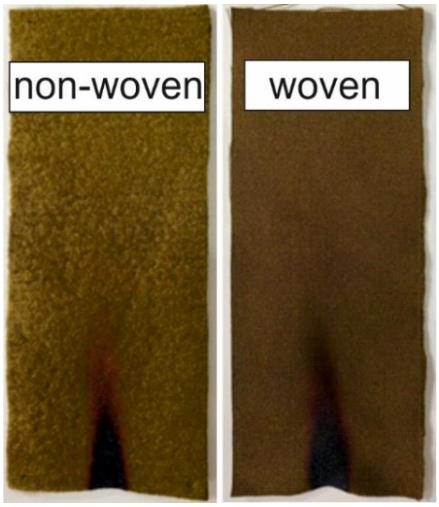

Fig. 10 Bottom-edge ignition test results. Shown are results from dyed Kynol fiber materials (disperse dye) after functionalization. The functionalization is performed by a combined application of the hydrophobic agent RUCO Dry DHY (150 g/L) and the antistatic agent Zerostat FC NEW (10 g/L).

4 Conclusions

It is possible to modify Kynol fiber materials to reach excellent antistatic or water-repellent functional properties. The gained effects are correlated to the applied amount of functional finishing agent. A combination of both antistatic and water-repellent properties is only possible if a compromise in the final performance can be accepted. However, no compromise has to be done due to the key-property of Kynol fibers. Their excellent flame retardant properties are not decreased by application of the used functional finishing agents. The shown results are a proof-of-concept for the functional treatment of Kynol fiber materials and could be a first step for a future practical application. However, for practical application also investigations on the durability of the functional finishing have to be performed.

Author Contributions

The presented results are based on the master thesis of Juan Wang performed at Niederrhein University of Applied Sciences 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 and no funding for the presented research.

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