Scientific paper

A Natural Based Method for Hydrophobic Treatment of Natural Fiber Material

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Received: 30-01-2017

Abstract

A treatment for hydrophobic functionalization of natural fiber materials is developed. This hydrophobic treatment is based mainly on natural products. As hydrophobic component the natural Tung Oil is used, which is originally a compound used for wood conservation purposes. The application on textile is done in a padding process under presence of an oxidative agent. For the current investigations a fiber felt from linen was used. The hydrophobic effect is determined by the concentration of Tung Oil and the duration of a thermal drying process. The hydrophobic effect is investigated by capillary rise tests and contact angle measurements. Scanning electron microscopy SEM is used to investigate the surface topography of the fiber material and the deposited hydrophobic material. Altogether, an interesting and promising method for hydrophobisation of natural fibers is developed, which could especially be used as part of a production process of a fiber reinforced composite material, mainly based on natural products.

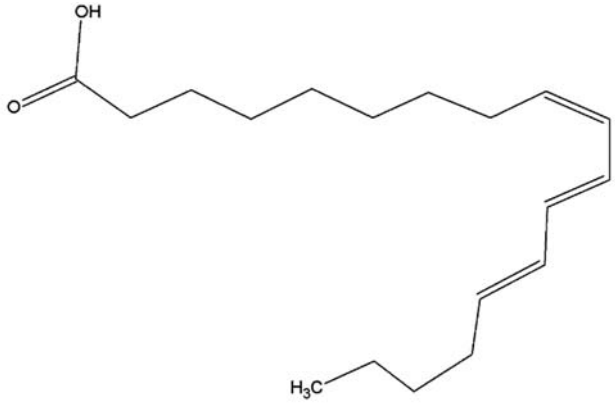
Keywords: Tung Oil, contact angle measurements, natural fiber, fiber felt, non-woven

1. Introduction

Natural fiber materials are based on fibers from plants or animals.[1,2] They can be used as traditional textiles for clothes but also as non-woven materials for technical purposes. Natural fibers are also used as fiber component in fiber reinforced composite materials used for example in the automotive sector.[3,4] For composite applications natural fibers have to compete with composites containing glass or carbon fibers. The mechanical properties of these competing composite materials are excellent. However also natural fibers exhibit important advantages, if they are used in composites. These advantages are related to material properties, economic reasons and ecologic concerns. From the material point of view, natural fibers are of lower density compared to glass fibers, so composite materials of lower weight can be produced.[4] The use of low weight composite materials in automotive sector will reduce for the final product – the car – the fuel consumption.[5] From the economic point of view, natural fibers are low cost materials, if their price is compared to glass and carbon fibers.[6,7] From the ecologic point of view, the recycling of materials containing natural fibers is simple compared to the recycling of composite materials with glass or carbon fibers.[4,8] One significant property of natural fibers is their hydrophilicity. Natural fibers are able to take up significant amounts of water.[9–11] For technical applications, this hydrophilicity is often disadvantageous, due to several reasons. The presence of water on a natural fiber can support the growth of fungi and bacteria, which can be the starting point of a bio corrosion. Also the up-take of water can lead to change in fiber volume, so crack formations in a composite material are promoted. Actually there are many excellent chemicals on the market, which are especially developed for a hydrophobic treatment of textile materials.[12–14] The main aim of those treatments is the realization of water- and soil-repellent textiles, usable as rain clothes or as home textiles.[14,15] The most effective chemicals in that field are based on fluorine-carbon compounds. Fluorine-carbon compounds are from the technical point of view excellent materials but in the last years significant concerns arise, due to potential environmental and health risks.[16] Other hydrophobic chemicals used in the textile field are polysiloxanes, which are also under discussion.[17,18]

With this background, there is a certain demand for a natural based hydrophobic agent usable for the hydrophobic treatment of natural fiber materials. This statement

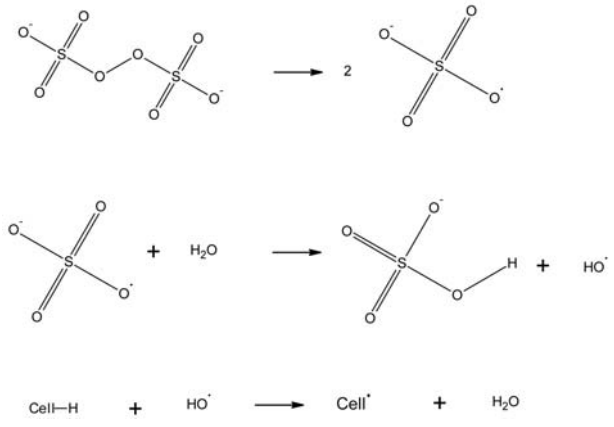
is especially valid, if a fully "bio-based" product is wished. This product should be built up from natural fibers and a treatment which is as well gained from natural products. For this, in the current work a hydrophobic treatment for natural fibers based on a natural product is developed. As natural product the natural oil – Tung Oil – is used. Tung Oil is a natural product, sometimes also named as China wood oil or China nut oil. It is produced from seeds of the Tung tree. This Tung tree is originated from China, there it is known and cultivated since centuries. Today Tung Oil is used for the surface treatment of wood.[19] The Tung Oil is used for wood conservation and protection in Europe in the 1920th.[20,21] Tung Oil-treatment on wood is used for protection against wood-decay fungi and to decrease water uptake of wood.[22] This decreased water uptake is reported for laboratory but also for long term field tests. The Tung Oil treatment is as well mentioned as an effective mean to replace traditional biocidal treatment for wood, which are under consideration, because of possible hazardous potential.[22] The use of Tung Oil for textile treatment is mentioned in some older patent references.[23,24] However, in those references the main focus for the Tung Oil is not to realize water-repellent textiles. The Tung Oil is reported for the improvement of crease and shrink resistance of textiles.[23] Also mentioned is the realization of high flex abrasion resistance of cotton textiles by Tung Oil.[24] Tung Oil is from chemical point of view a triglyceridic ester composed mainly from alpha-elaeostearic acid (*cis*-9, *trans*-11, *trans*-13-octadecatrienoic acid) (structure see Scheme 1).



Scheme 1: Chemical structure of alpha-elaeostearic acid.

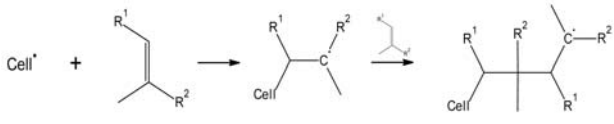
The content of this unsaturated organic acid is around 84% in the Tung Oil.[25–27] However other references reports lower content of only 64% alpha-elaeostearic acid in the Tung Oil.[28] It has to be kept in mind, Tung Oil is a natural product, and its composition can be influenced by the climate or other local conditions surrounding the originating plant.

The appearance of multiple conjugated carbon/carbon double bonds makes the Tung Oil a monomer thermally polymerizable at higher temperatures.[25,29] Such a thermal polymerization after application onto a fiber material can be part of a hydrophobic treatment process. However, due to the thermal sensitivity of natural fibers, the use of a temperature driven polymerization process could lead to partly fiber decomposition. Alternatively to the thermal polymerization, also oxidative processes are suitable for crosslinking of the unsaturated carbon-carbon double bonds.[30] By such a process also a crosslinking to the surface of a cellulosic based fiber is possible.[31,32] This connection to the fiber surface is especially necessary, if a long-term stability of the hydrophobic treatment onto the fiber material is wished. Persulfates are reported to initiate radical grafting reactions onto cotton fibers in aqueous media, however the formation of carbon or oxygen centered secondary radicals on the cellulose remains unclear.[33] However, using persulfate most authors imply a localization of the intermediate radical on the cellulosic oxygen (Scheme 2).[31,34,35]

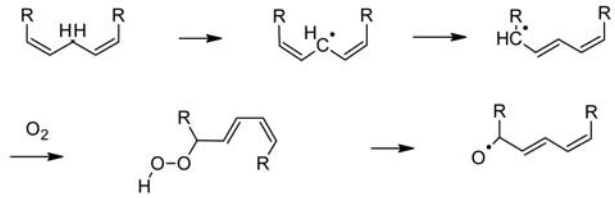


Scheme 2: Schematic drawing of persulfate reaction to form intermediate radicals on the cellulose.

Thakur et al. mention both options, but give no further evidence on the mechanism favoring one or the other.[36] The cellulosic radical can then undergo a grafting reaction with an unsaturated hydrocarbon, as described in Scheme 3. Natural oils like linseed oil or Tung Oil undergo a natural polymerization by atmospheric oxygen. The mechanism is proposed by Mallegol et al. and is summarized in Scheme 4.[30] The polymerization of these oils can consequently be enhanced by introducing an oxidizing agent. By using sodium persulfate the crosslinking can be accelerated combined with the option of a radical grafting reaction onto the cellulosic fiber.



Scheme 3: Drawing of cellulosic radical reacting with unsaturated hydrocarbon.



Scheme 4: Polymerisation step for unsatured natural oil.

Based on this background, in the actual investigation a hydrophobic treatment based on Tung Oil and an oxidative agent is developed for application onto natural fibers. This demonstration is done on linen fiber felt. It is shown in a first approach that by this method natural oils can be used for hydrophobic modification of natural fiber materials.

2. Experimental Section

2. 1. Materials

As textile material a nonwoven felt from linen fibers is chosen. This is a natural material and has to be cleaned before a further wet chemical treatment is performed. This cleaning is done with an enzymatic pretreatment, which is able to remove unwished substances and dirt from the fiber surface. This pretreatment is done in a dyeing machine (Then from Fong Europe GmbH, Schwäbisch Hall) usable for laboratory scale. For the pretreatment a water based recipe with following components is used – 30 liter water, 390 ml PERIZYM DBS from the company Dr. Petry GmbH, 60 ml PERIPLEX AHL from the company Dr. Petry GmbH and 60 ml KOLLASOL OCE from CHT.R. BEITLICH GmbH (Tübingen, Germany). PERIZYM DBS is a ready-made aqueous solution with enzymatic components of pectinase and amylase. PERIPLEX AHL is an aqueous yellow and transparent liquid. It contains complexing agents for heavy metal ions and ions from calcium and magnesium. KOLLASOL OCE is a clear transparent liquid containing wetting agents. It is used to improve the wetting of the aqueous recipe on the fiber material and to suppress the formation of foam during the application. The pH-value of the complete recipe is 7.2. With this recipe an amount of 569 g linen fibers is treated for 2 hours at 60 °C in the above mentioned dyeing machine. Afterwards the linen fibers are treated with 30 liter of water at 95 °C for 20 minutes, following by a washing step at 70 °C for 10 minutes. In the end the fiber felt is dried at room temperature. After the complete process a weight loss of 7% is determined for the fiber felt, probably related to the removed components from the fiber surface. For hydrophobic treatment of the linen fiber felt recipes containing following components are used. Tung Oil which is a yellow and viscose substance gained from Sigma-Aldrich, sodiumperoxodisulfate from VWR and sodiumoleate form Sigma-Aldrich.

2. 2. Preparation

For hydrophobic functionalization, the linen fiber felt is treated with a recipe containing 45 mL water, 5 g sodiumperoxodisulfate, 0.5 g sodiumoleate and Tung Oil in an amount of 5 g, 10 g or 20 g. Before application on the fiber material all components are stirred together for a duration of 15 minutes. The application onto the fiber felt is done in a horizontal padder – 2-Walzen-Labor-Foulard supplied by the company Wichelhaus GmbH (Germany). During application the roller pressure is set to 0.3 MPa and the speed is set to 18 m/min. Each sample is treated twice. The wet pick-up of the fiber samples treated this way is 93 wt-%. Afterwards the samples are dried in an oven (from Memmert) at a temperature of 90 °C. The duration for this drying process is set in a range from 5 to 60 minutes.

2. 3. Analytics

The hydrophobic properties of the prepared samples are determined by two methods, the capillary rise test and contact angle measurements. The capillary rise test is used to determine the capability of a textile sample to soak up water.[37] The actual measurements are performed according to DIN 53924. For this, the fiber felt samples are cut in strips of 30mm width and one end of the strip is placed vertically in contact with a testing liquid. This testing liquid is an aqueous solution of 0.5% of the blue dye C.I. Direct Blue 086. After the contact with the testing liquid, the textile sample soaks up the liquid and after 30 minutes the distance is measured as capillary rise. Each sample is tested twice and the average value is reported. Contact angle measurements are performed with a device Drop Shape Analyzer DSA25 supplied by KRÜSS GmbH (Hamburg, Germany). For measurement, a 30 µl drop of water is placed on the sample and after 10 seconds the contact angle is recorded. For each sample this measurement is repeated 5 times and the average value is reported. For testing the stability against rinsing, the textile samples are stirred into 250 mL water containing 1 mL of the surfactant Triton X. This procedure is done for 10 seconds at room temperature. To evaluate the mechanical properties of prepared textile samples, the elongation at break and the breaking strength are determined according to DIN EN 29 073. The measurements are performed with a device ZmartPro supplied by the company Zwick/Roell. The surface topography of the fiber materials is investigated by scanning electron microscopy, SEM. For this microscopic measurements a microscope Tabletop TM3000 supplied by Hitachi is used.

3. Results and Discussion

3. 1. Material Properties

The morphology of the linen fiber felt is at first investigated by SEM (Figure 1). This morphology is recorded

before and afterwards of the enzymatic treatment for cleaning the linen felt. These measurements show that the enzymatic cleaning does not effected the arrangement and diameters of the linen fibers. However, by these SEM-investigations a more detailed statement concerning the structure and the surface topography of the fibers cannot be done. The hydrophobic treatments containing the Tung Oil are all applied onto the linen fiber felts after the enzymatic treatment is done.

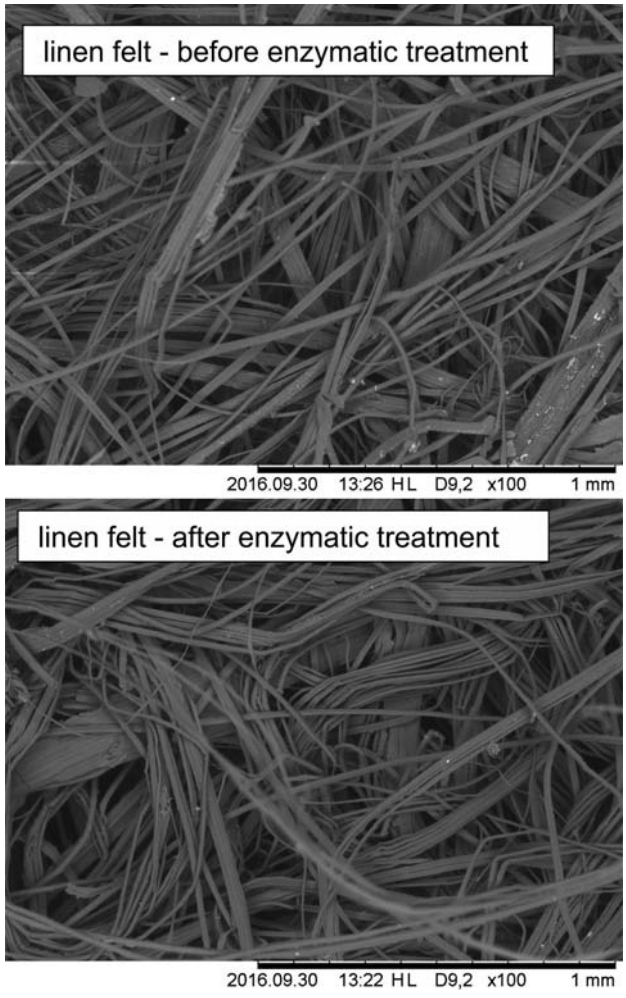


Figure 1: SEM-images of linen fiber felt before application of the hydrophobic recipe.

The mechanical properties of fiber samples after application of the hydrophobic recipes are investigated as function of the duration of thermal treatment after application of a hydrophobic recipe (Figure 2). This recipe contains with 20 g the highest amount of used Tung Oil. The thermal treatment is performed at 90 °C with a maximum duration of 60 minutes.

The determined elongation at break is drastically decreased after 30 minutes of thermal treatment. This result can be explained by the cross-linking reaction of the Tung Oil by an oxidative process. The progress of the crosslinking reaction is indicated by the change in elongation at break as function of duration of thermal treatment. After 30 minutes the crosslinking is progressed in a way that the elongation of the treated linen fiber felt is drastically reduced. Similar to this, the crosslinking leads to a promotion of the hydrophobic effect of the Tung Oil application, as it is presented in the following sub-section.

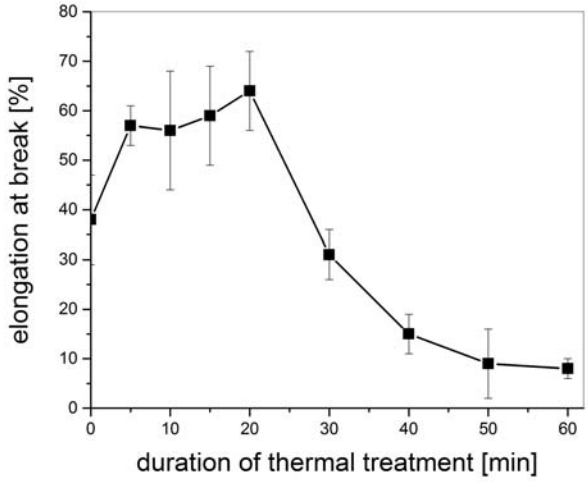


Figure 2: Mechanical properties as function of the duration of thermal treatment after application of a hydrophobic recipe containing 20 g Tung Oil. Shown is the elongation at break.

The breaking strength of fiber samples without the Tung Oil treatment is determined to be 60 N and can be significantly decreased by the Tung Oil treatment to values in the range of 20 N to 50 N (Figure 3). However compared to the determined elongation at break (Figure

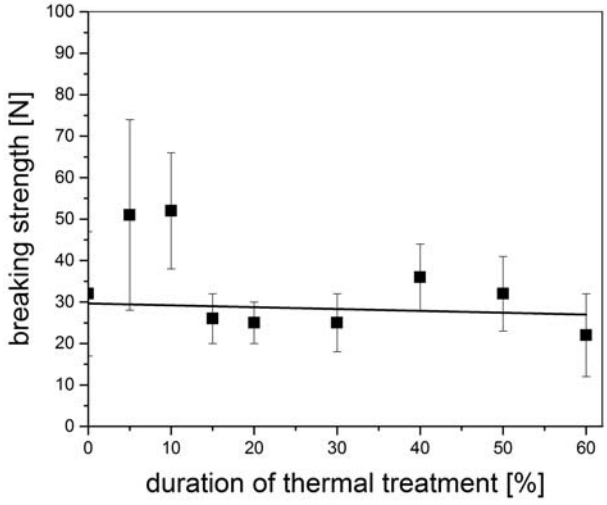


Figure 3: Mechanical properties as function of the duration of thermal treatment after application of a hydrophobic recipe containing 20 g Tung Oil. Shown is the breaking strength. The solid line is a guide for the eye based on a linear fit of measurement points.

2), it has to be remarked, that the duration of thermal treatment after the Tung Oil application has a less significant influence on the breaking strength (Figure 3). Evaluating the measurement data of breaking strength as function of duration of thermal treatment by using a linear fit, only a small decrease in the breaking strength can be estimated, if the duration of thermal treatment is expanded to 60 minutes. The oxidative crosslinking of the Tung Oil obviously glue the fibers together and decrease for this the elongation at break. However, the strength of this oil impregnation is less influenced by the progress of crosslinking driven by the thermal treatment.

The morphology of linen fiber felt after application of the Tung Oil recipe is presented in figure 4. By this SEM-images it can be clearly identified that the Tung Oil is especially up-taken by the interspace between the linen fibers. The change in the mechanical properties of the fiber samples could be also explained by a gluing together of the single linen fibers by the crosslinked Tung Oil.

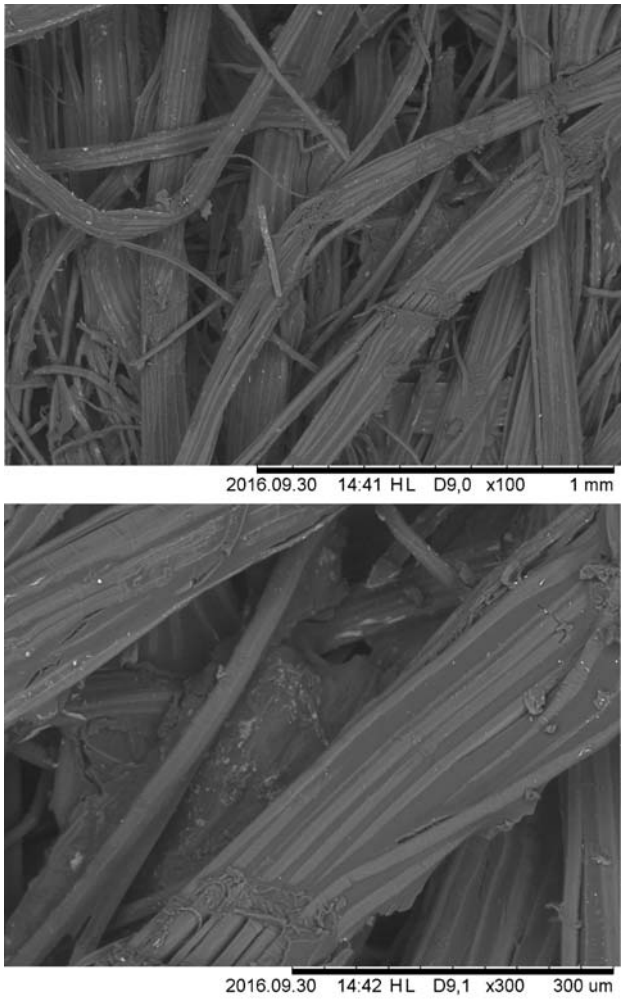


Figure 4: SEM-images of linen fiber after application of the hydrophobic recipe containing 20 g Tung Oil and dried for 30 minutes at 90 °C.

3. 2. Hydrophobic Properties

To optimize the hydrophobic treatment at first an investigation is performed as function of the duration of thermal treatment after application of the Tung Oil recipe, analogously to the investigation of mechanical properties presented in Figure 2.

This recipe contains with 20 g the highest amount of used Tung Oil. The thermal treatment is performed at 90 °C with a maximum duration of 60 minutes. The hydrophobic properties of the prepared linen samples is determined as capillary rise (Figure 5). The capillary rise of the linen fabric without the Tung Oil application is around 85 mm after 30 minutes measurement time. After Tung Oil application without further thermal treatment a capillary rise of around 10 mm is reached after 30 minutes, so a clear hydrophobic effect is gained even without a further drying procedure. This capillary rise is decreased, if the linen samples with the Tung Oil are thermal treated and this effect is stronger as function of the duration of thermal treatment. After 30 minutes of thermal treatment the capillary rise is nearly zero, so here strong hydrophobic properties are realized.

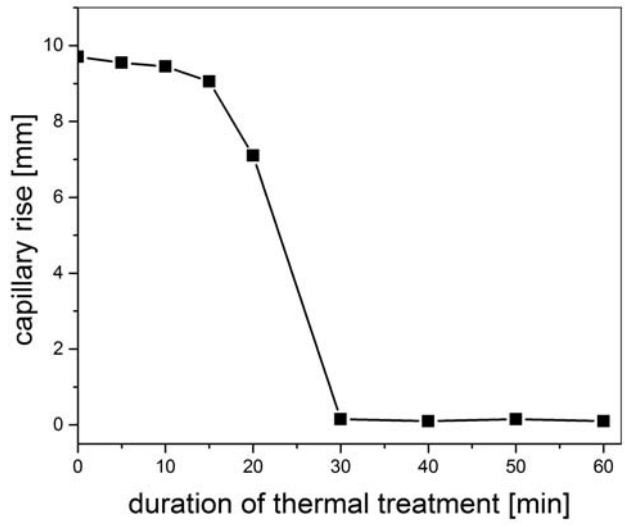


Figure 5: Hydrophobic properties as function of the duration of thermal treatment after application of a hydrophobic recipe containing 20 g Tung Oil. Shown is the capillary rise after 30 minutes testing time.

These results of determined capillary rise are in certain agreement with the determined mechanical properties of the investigated samples shown in Figure 2.

Additionally the reached hydrophobic effect is investigated as function of Tung Oil concentration in the applied recipe (Figures 6 and 7). Here it is of special interest, if also lower Tung Oil concentrations could lead to significant hydrophobic properties. The hydrophobic properties are determined by two methods, the capillary rise (Figure 6) and the contact angle measurement with water (Figure 7).

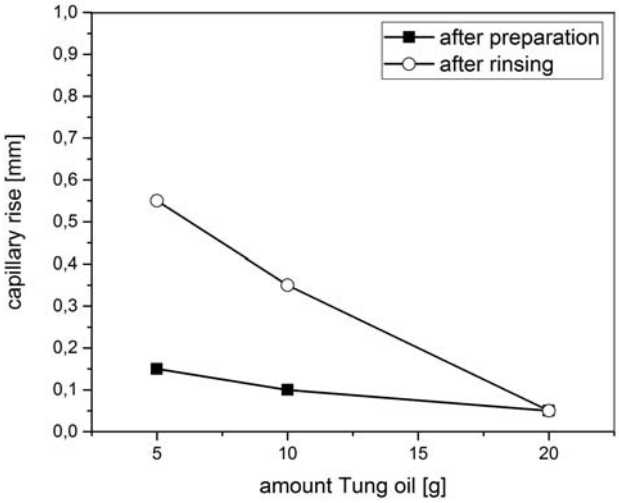


Figure 6: Hydrophobic properties as function of the content of Tung Oil in the hydrophobic recipe. After application of the hydrophobic recipe, the samples are dried for 30 minutes. Shown is the capillary rise after 30 minutes testing time.

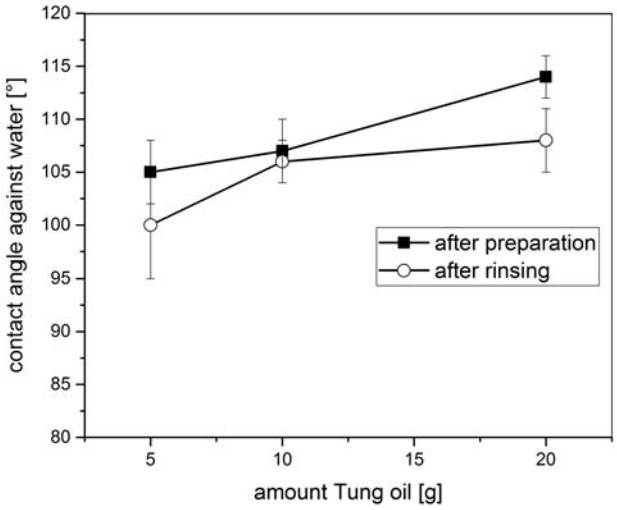


Figure 7: Hydrophobic properties as function of the content of Tung Oil in the hydrophobic recipe. After application of the hydrophobic recipe, the samples are dried for 30 minutes. Shown is the contact angle of water.

Even with the lowest applied amount of Tung Oil with 5 g in the recipe a very low capillary rise can be reached, so significant hydrophobic effect is realized even if smaller amounts of Tung Oil are applied. However it can be also stated that this hydrophobic effect gained with smaller amounts of Tung Oil is less stable against a rinsing process. Probably, only small amounts of Tung Oil are needed to modify the surface of linen fibers with hydrophobic properties but larger amounts of this oil are necessary to support suitable crosslinking which is probably a necessary requirement for a certain rinsing stability. Similar results are gained with the contact angle measurements (Figure 7). The contact angle against water of the treated linen fiber samples increases as function of the amount of Tung Oil applied (Figure 7). All treated samples exhibit contact angles of more than 100° and are therefore clearly hydrophobic. Highest values are gained in the current investigation with highest amount – 20g recipe – of applied Tung Oil. However, also with the application of lower Tung Oil amounts of 5g hydrophobic fiber samples are realized. Determined contact angles are around 115°, which is similar to hydrophobic sol-gel agents supporting an intermediate hydrophobic effect on textiles.[38,39] However, with advanced hydrophobic sol-gel systems, containing long-chained alkylsilanes or even fluorinated additives, contact angles around 140° are reached.[38]

By wet chemical application on textile substrates with different perfluorinated recipes in different approaches even contact angles of water in the range of 150° to 160° are reported.[40–42] These applications are based on perfluorinated polysiloxanes, fluorinated acrylic lattices and perfluoroalkylacrylate compounds combined with silica nanoparticles.[40-42]

With different plasma deposition techniques also contact angles of water in the range of 150° to 170° are reached on textile substrates.[43,44] In this technique the type of monomer used for plasma deposition is significant for the gained hydrophobic effect. Suitable monomers are here hexamethyldisiloxane HMDSO and tetrafluoromethane CF_4.[43,44]

Such high contact angle values are often summarized under the terms superhydrophobic properties or superhydrophobic textiles.[45–47] Therefore, in relation to other hydrophobic recipes for textile treatment the actual developed Tung Oil recipe can be ranked in the range of moderate hydrophobic agents but not used for realization of superhydrophobic textiles. However, it should kept in mind that for the application in fiber reinforced composites a moderate hydrophobic fiber functionalization could be advantageous, because it decreases the water up-take of the fiber material but contains still enough hydrophilic groups on the fiber surface necessary for the interaction with the polymer resin forming the matrix of a fiber-reinforced composite.

Altogether it was shown in a first proof of concept, that the natural Tung Oil can be successfully used as hydrophobic agent for natural fibers. This natural based method could be promising for future developments of fully bio based materials as for example fiber reinforced materials – so called bio composites.

4. Conclusions

A natural based method for hydrophobic treatment of natural fiber materials is realized by recipes of natural oil. As natural oil the Tung Oil originally used for protec-

tion of wood is used. By this natural recipe moderate hydrophobic properties can be introduced on natural fibers as linen. Suitable recipes contain beside the Tung Oil also sodiumperoxodisulftate to promote the crosslinking reaction of the Tung Oil and sodiumoleate to stabilize the recipe in a water based application recipe. The Tung Oil concentration is varied in a broad range and even with lower concentration a suitable hydrophobic effect is reached. However, it has to be remarked that even with the highest amounts of Tung Oil applied no superhydrophobic properties are reached for the treated fiber materials. Nevertheless, the realized materials are promising for future developments of fully bio based fiber and composite materials.

5. Acknowledgements

For funding of the electronmicroscopic equipment the authors acknowledge very gratefully the program FH-Basis of the German federal country North-Rhine-Westphalia NRW. All product and company names mentioned in this chapter may be trademarks of their respective owners, also without labeling. For many helpful discussions and technical help in the lab many thanks have to be acknowledged to Thomas Heistermann from Niederrhein University of Applied Sciences.

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Povzetek

Razvili smo postopek za hidrofobno funkcionalizacijo vlaken naravnih materialov, ki temelji na uporabi naravnih produktov. Za hidrofobno komponento smo uporabili tungovo olje, ki se sicer uporablja za zaščito lesa. Tungovo olje smo na tekstil nanašali v prisotnosti oksidanta. V raziskavi smo uporabili lanena vlakna. Hidrofobni učinek določa koncentracija tungovega olja in čas procesa tremičnega sušenja. Hidrofobnost smo preučevali s testom kapilarnega dviga in meritvami stičnega kota. Za preučevanje topografije površine vlaken in nanešenega hidrofobnega materiala smo uporabili vrstično elektronsko mikroskopijo (SEM). Razvili smo zanimivo in obetajočo metodo za hidrofobno funkcionalizacijo naravnih vlaken, ki jo lahko uporabimo že v procesu priprave vlaken in temelji na uporabi naravnih produktov.