Obtaining, Microstructure and Morphology of Nonwoven Fabric Based on Camel Wool

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Abstract

A three-layer non-woven composite material for the lining of insulated shoes has been obtained by an adhesive bonding method. The thickest middle layer of the material consists of evenly laid coarse camel fibers, the top and bottom layers consist of cotton jersey, polymer glue is located between the layers. The layers are bonded by thermal duplication at a temperature of 150 ± 5 ° C for 2.0 ± 0.2 minutes. The microstructure and morphology of camel hair, polymer glue and composite were studied by FT-IR spectroscopy, SEM and X-ray structural analysis. The chemical interaction between wool fibers and polymer glue, the geometric dimensions and shape of the fibers, the cross-section of the layers of the composite material, and the change in the degree of crystallinity of the material have been established. The diameter and internal structure of the investigated camel wool fibers differ significantly from cellulose and silk fibers. The average diameter of full, not hollow wool fibers is about three times that of cotton and four times that of silk. Camel wool is more suitable for the production of non-woven textile material. In the process of thermal exposure, the molten polymer diffuses into the structure of both wool and knitted fabric, which leads to a decrease in density and an increase in the thickness of the adhesive layer. Diffusion and excellent adhesion of the molten polymer to the fibers ensures the strength of the composite.

Key words: camel wool, nonwoven fabric, microstructure, diffusion, adhesion.

Highlights:

- obtained a three-layer material based on camel wool with cotton jersey;
- under thermal action chemical bonding of acrylic polymer with wool fibers occurs;
- diffusion of polymer melt into the structure of wool and cellulose was established;
- adhesive interaction of the components ensures the strength of the composition.

1.Introduction

In the modern textile and light industry, non-woven and multilayer textile materials are http://annalsofrscb.ro 3748

certainly in demand and have their rightful place and significance. In recent years, nonwovens have become an extremely important segment of the textile industry. Technical developments in the fields of polymers, nonwovens processing and fabric finishing have led to significant improvements in the physical and mechanical properties of materials [1]. Nonwovens are a part of technical textiles used to make apparel and footwear, mainly for functional purposes, where aesthetic properties are of little importance. They are mainly used for keeping warm, supporting and reinforcing shoes [2].

Nonwovens are made using various bonding technologies, such as needle-punched bonding [3], chemical bonding [4], thermal bonding [5], thermochemical hybrid bonding [6, 7]. Nonwoven fabrics based on chicken feathers with the addition of wool [8], a nonwoven composite with a sandwich structure, which consists of an upper and a lower layer, were obtained by needle-punched method. The layers are made of nylon fibers, recycled Kevlar® fibers and low melting polyester fibers [9, 10].

Nonwoven materials are used to reinforce polymer composites [11, 12, 13], for electromagnetic shielding in electrical installations [14, 15], in membrane materials for clothing and footwear [16]. Material based on silk fibroin and silk fibers can be used as a biodegradable scaffold for tissue restoration and regeneration [17]. Bio-based materials such as calcium alginate [18] and collagen [19, 20] have excellent fire resistance.

A large group is represented by insulated nonwoven materials with heat-retaining and temperature-regulating properties. To improve the heat content of fabrics, it is proposed to alloy the nonwoven material with microcapsules [21]. Reversible thermoregulated nonwoven fabric for footwear is based on the use of particles containing phase change materials that can absorb or generate heat depending on environmental conditions [22]. Thermal comfort and thermal regulation of special clothing is achieved using modern electronic heating devices in traditional textile material. The use of heat-retaining, electrically conductive materials with a phase change, membranes increases thermal comfort and the efficiency of thermoregulation [23, 24, 25, 26]. Composites with isotropic fibrous elements have high physical and mechanical properties, in contrast to anisotropic and brittle layered composites [27]. The biopolymer is considered a promising encapsulating agent for textile materials due to its biocompatibility, lack of toxicity, antibacterial activity, high availability, and low cost [28]. A new multilayer low-temperature protective composite based on microcapsules with a phase transition [29] from polyamide 6 [30] has been prepared. The thermal and mechanical properties of all microcapsule composites are related to the reinforcement morphology, fiber volume fraction, and layer orientation.

Fibrous raw materials are the basic component for nonwovens, which determines the main properties of the final products, not only in terms of chemical properties, but also in terms of physical or other functional properties. In technical nonwovens, natural, chemical and modified fiber raw materials are used for use in various fields [31]. Nonwovens used for footwear must have both high sorption and permeability [32]. Bioactive composites [33] and natural fibrous materials meet these requirements to the greatest extent. Waste wool fibers are a potential source of thermal and acoustic applications due to their natural properties [34, 35].

Among natural fibers, sheep wool is one of the oldest textile fibers used by mankind. Woolen fibers with improved functional properties and technical characteristics using finishing technologies can be used in various technical fabrics [36]. Camel wool has been widely used since the 70s and 80s of the last century for the manufacture of rough carpets, bags for transportation, etc., as well as for yarn mixed with other natural and artificial fibers [37].

Camel wool production includes collection, sorting, refining, spinning, weaving or knitting and dyeing. Camel hair is mainly divided into three main classes: 1st level includes short staple fibers with a soft and silky texture close to the skin, 2nd level includes short coarse outer fibers from 1.5 to 5 inches, and 3rd the level includes coarser outer fibers up to 15 inches. The fibers are characterized by strength, gloss and smoothness, excellent insulation, warmth and round or oval cross-section. They are used to make garments, including sweaters, coats, as well

as industrial belts, awnings, fabrics, and blankets [38].

The influence of the structure of camel wool on its properties, including length, strength, fineness, crimp, felting, moisture absorption, etc., is analyzed. [39]. To improve the hand-spinning process, fineness and strength of the yarn, camel wool was blended with merino wool in three different proportions. Abrasion resistance and hand-knitting performance depend on the ratio of the different wool fibers [40]. Camel wool and some other animal fibers are a classic two-sided structure. Down and guard hair fibers have different temperatures and enthalpies of denaturation, which probably explains the large difference in mechanical characteristics between actually two types of fibers [41]. The researchers examined the qualitative characteristics of camel hair, collected from the shoulders, middle side and humps, in both calves and adults.It is noticed that the fibers of the middle part have the best textile properties, making them ideal for clothing such as sweaters, coats and shawls, as well as various other household items [42].

The camel is an integral part of animal husbandry in countries with hot and dry climates. Livestock farms for camel breeding, camel milk and wool production appeared in the Republic of Uzbekistan. At the same time, enterprises specialized in the processing of camel wool appeared. Camel wool is much coarser than other fibrous materials; factories accumulate a sufficient amount of coarse wool fibers that are not spun and knitted. On the other hand, camel wool, being a heat insulator, perfectly retains warmth. Meanwhile, there is a shortage of warm, comfortable, lightweight, soft lining materials for shoes based on natural raw materials. Camel wool fully meets these requirements. The main purpose of this study is to assess the potential use of coarse camel wool as a non-woven thermal insulation layer in shoe lining. In such materials, the issues of structure and ensuring the strength of the bonding of the components are of particular importance. To clarify this, we investigated the obtaining, microstructure and morphology of the insulated composite material based on camel hair.

2. Experimental

2.1.Materials

Washed, free from impurities, ready for use, but not suitable for spinning, camel wool fibers were kindly provided by the private enterprise "M. Sayfullaeva" in Tashkent. Cotton knitted fabrics of two types, the characteristics of which are presented in the table, were provided by the enterprise JV LLC "UZTEX" in Chirchik, Tashkent region. Dry film acrylic glue was purchased in the market of the city of Tashkent. Table

Properties	Knitted fabrics	
Thickness, mm	0.33	0.45
Weight, g / m^2	160.3	157.5
Breaking strength, N		
by length	269	401
in width	203	296
Elongation at break,%		
by length	39	18
in width	71	68
Air conductivity, $dm^3 / m^2 \cdot s$	160.3	157.5
Abrasion resistance, cycles	9500	14500

Characteristics of knitted fabrics.

2.2. Obtaining a multilayer material

A film of dry glue is laid on cotton jersey, and on it is a uniform layer of camel wool, which makes up the bulk and bulk of the composition (Fig. 1). Another layer of glue and cotton jersey is placed on the surface of the woolen layer.



Fig. 1. Construction of multilayer insulated nonwoven fabric: 1 - knitted fabric, 2 - glue layer, 3 - camel wool, 4 - knitted fabric.

The composite material is formed by simultaneous duplication of all layers under pressure and with heating. For this, the complete material is passed through heated pressure rolls. The shaft temperature is 150 ± 5 °C, the contact time of the material with the shaft is 2.0 ± 0.2 min. The layers are attached to each other under the pressure of the heated shafts of the installation. In fig. 2 shows the front and back sides of the composite material.





2.3. Characterizations

The structure of the samples was determined using infrared spectroscopy with Fourier transform (FT-IR) Nicolet IN10 Company Thermo Fisher Scientific (USA) in the scanning range of 500-4000 cm⁻¹. Samples were prepared using KBr tablets; sample identification was performed using OMNIC Spectra software and from literature.

The surface morphology and cross-section of the material and fibers were examined using a scanning electron microscope SEM-EVO MA 10 (Carl Zeiss, Germany). To carry out the process of preparing the samples, samples of fibers or material were fixed on the stage of the microscope. The sample surface was not sprayed with conductive carbon or other metal. Next, the stage was installed in the working chamber of the microscope, and air was evacuated from the chamber. For the measurement, an accelerating voltage of up to 10 kV was applied in the SI (secondary electrons detector) detection mode. Theworkingdistancewas 8.5 mm.

X-ray phase analysis of the initial substances and the products of their interaction was carried out on a diffractometer of the EMPYREIN system by the X-ray powder method in the range of $^{\circ}2\theta$ angles from 5 to 90 with a step of 0.0130 in the scanning mode. Scan time approximately 40min, scan type - continuous. The sample length is 10 mm, the anode material is copper, the research temperature is 25 ° C. The system automatically registers the Bragg angles

of radiation $^{\circ}2\theta$ during diffraction by a given radiation, the values of the interplanar distances (d_{hkl}) and the corresponding intensities of the line (I) of the X-ray spectrum.

Physical and mechanical properties of materials were tested at the certification center "CENTEXUZ" TITLI, room temperature $20 \pm 3 \circ C$, relative air humidity $65 \pm 5\%$. The breaking load of materials was determined on a dynamometer "AO-1" (tensile machine), the maximum breaking force of the device is 1000 N. The air conductivity of materials was determined using an instrument for determining air conductivity "AP-36OSM", the sample size is 160x160 mm. The abrasion resistance of the materials was determined on a device for determining the strength of fabrics to abrasion M 235/3, the rotation speed of the abrasives was 47.5 \pm 2.5 rpm, the sample size was a circle with a diameter of 50 mm.

3. Resultsanddiscussion

3.1. FT-IR

The FT-IR spectrum of camel hair contains absorption bands related to the bonds of the protein macromolecule (Fig. 3). The broad absorption band at 3278 cm-1 refers to stretching vibrations of N - H and O - H bonds of associated peptide and amino groups, hydroxyl groups. There is an absorption band of stretching vibrations $v_{C=0}$ at 1633 cm⁻¹ (band amide I), bending vibrations δ_{NH} at 1515 cm⁻¹ (band amide II) and at 1393 cm⁻¹ (band amide III). The absorption band at 1235-1202 cm⁻¹ refers to $\delta_{NH} + v_{CN}$. The absorption band at 1041 cm⁻¹ refers to vibrations of C - O bonds. Stretching vibrations of C - H bonds show an absorption band at 2924 cm⁻¹, deformation vibrations of these bonds - at 1447 cm⁻¹.



Fig. 3. FT-IR spectrum of camel wool fibers.

The FT-IR spectrum of the polymer glue contains absorption bands of asymmetric (2915 cm⁻¹) and symmetric (2849 cm⁻¹) stretching vibrations, asymmetric (1468 cm⁻¹) and pendulum (717 cm⁻¹) deformation vibrations of CH_2 groups (Fig. 4). The spectrum of the glue lacks absorption bands in the region of 3500-3000 cm⁻¹, therefore, there are no hydroxyl, carboxyl and amino groups in its composition. The absorption band at 1697 cm⁻¹, apparently, refers to $v_{C=0}$, the absorption band at 1256 and 937 cm⁻¹ refers to the stretching vibrations of C - O bonds.



Fig. 4. FT-IR spectrum polymer glue.

Now let us consider the FT-IR spectrum of the product of interaction between camel hair and polymer glue after thermal exposure (Fig. 5). The spectrum of the product shows changes in comparison with the spectra of the starting substances: disappear two absorption bands of the glue at 1256 and 937 cm⁻¹ and the absorption band of wool at 1447 cm⁻¹. A shift of the absorption band $v_{C=0}$ and deformation vibrations of the wool peptide group is observed. At that time, the absorption bands attributed to the stretching vibrations of the O - H andN - H bonds of wool, CH_2 groups of the glue remain unchanged. The observed changes indicate that a chemical interaction occurs between the glue polymer and the camel wool macromolecule. It is possible that the reaction involves the carbonyl groups of the polymer and the hydrogen atom at the tertiary carbon of the protein molecule. Chemical interaction provides strong bonding of the components of the composite material.



Fig. 5. FT-IR spectrum of the product of interaction of camel wool fibers with polymer glue.

3.2. SEM

Very interesting results were revealed from the analysis of SEM images. Wool contains fibers of varying thickness and length (Fig. 6).



Fig. 6. SEM images of camel wool fibers.

The average diameter of wool fibers is about 50 μ m (Fig. 7), this is about three times the diameter of cotton fibers and about four times the diameter of silk fibers. As is known, wool fibers with a diameter of 14-19 microns are recognized as the most suitable for spinning [43].Many natural fibers have a hollow space (lumen) resulting in low densities, and have nodes at irregular distances that divide the fibers into individual cells. The surface of natural fibers is rough and uneven and provides good adhesion to the matrix in a composite structure [44, 45]. In contrast to cotton and silk fibers, the investigated woolen fibers are not in the form of a tube, but in the form of a dense bulk fiber (Fig. 8). Coarse, dense and irregular in diameter camel wool fibers are difficult to spin. Therefore, it will be expedient to form a non-woven layer of insulated composite material from camel wool fibers.



Fig. 7. Average diameter of camel wool fibers.



Fig. 8 Cross-section of camel wool fiber.

The polymer adhesive has a uniform, smooth structure with a thickness of about 98 μm (Fig. 9).



Fig. 9. Cross-section of polymer adhesive.

In fig. 10 shows the morphology of a cross-section of a multilayer composite material.



Fig. 10. Cross-sectionofamultilayercompositematerial.

As can be seen from Fig. 10, the main thickness of the material is occupied by a woolen layer, in which the fibers are located, to a greater extent, longitudinally. Cotton knitted fabrics are located on both sides of the fabric. There is a thin adhesive layer between the wool and knitted layers. Here you can see that the diameter of the camel wool fibers is much larger than the cotton fibers in knitwear. The thickness of the adhesive layer is greater than the thickness of the polymer adhesive film itself (Fig. 11).

As can be seen from Fig. 11, the thickness of the knitted fabric layer is about 500 μ m, which is slightly larger than the thickness of the original knitted fabric. The glue melted when heated diffuses into both the woolen and the knitted layer, so its area of distribution is wider than the thickness of the original film. When you zoom in on the image, you can see a more accurate value for the thickness of the adhesive layer (Fig. 12).



Fig. 11. The thickness of the knitted and adhesive layers of the composite material.



Fig. 12. Thickness of the adhesive layer of the composite material.

Polymer glue combines very well with both wool and cotton fibers. The adhesive bond firmly binds the main wool layer with the fibers of the surface knitwear (Fig. 13). The adhesive strength of the layered nonwoven material is provided by the adhesive bonding of the layers [46]. Cohesion within the fabric is the result of greater entanglement and friction between the fibers and bundles that make up the nonwoven fabric [47].



Fig. 13. Adhesive bond of woolen fibers of non-woven layer and cotton fibers of knitted layer.

The laminated material provides high adhesion strength of the fiber-adhesive bond. The polymer adhesive lacks polar functional groups such as hydroxyl, carboxyl or amino groups. Despite this, the adhesive film is well wetted with water (Fig. 14).



Fig. 14. Angle of wetting of the surface of the adhesive film with a drop of water.

As can be seen from Fig. 14, the angle of wetting the surface of the adhesive film with water is in the range of $30-50^{\circ}$. Such an indicator of the contact angle refers to the surfaces wetted by this liquid. This is probably why the hot melt glue wets the camel wool fibers well (Fig. 15).

Fig. 15 shows that some wool fibers "sink" in the melted glue. You will notice good adhesion of the adhesive to the fiber surface. The fact that in some places of contact there is no interface, once again proves the formation of a chemical bond between the polymer of the glue and the protein molecule of camel hair. Diffusion of melted glue into the fibers of wool and knitwear, adhesion of the adhesive layer on the surface of the fibers, and finally, the chemical interaction of the polymer of the glue and wool are factors of solidity and high strength of a multilayer insulated composite material based on camel wool.



Fig. 15. Interaction of camel wool fibers with melted glue.

3.3. X-ray phase analysis

In order to reveal the influence of the interaction of the polymer with wool, X-ray phase analyses of polymer glue, wool fibers and the product of their interaction were carried out (Fig. 16).



Fig. 16. Radiographs of camel hair (a), polymer glue (b) and the product of their interaction (c).

As can be seen from Fig. 16 all three substances are amorphous substances with a certain region of crystallinity. The crystalline phase of wool exhibits an intense reflex at an angle of diffraction ($^{\circ}2\theta$) of 26.5, for polymer glue at an angle of 21, and the product of their interaction at an angle of 21.5. In the product of the interaction of the components, the crystalline region of the wool disappears; it is possible that the protein molecule is incorporated into the structure of the polymer. The intensity of the reflection of the crystalline phase of the interaction product decreases in comparison with the intensity of the crystalline phase of the polymer. This may be due to an increase in the porosity and volume of the polymer after thermal interaction with wool. The result to some extent explains the increase in the width of the polymer film in the composite material found in the SEM studies.

4. Conclusions

Coarse, non-spinning camel wool fibers as well as wool waste are suitable fiber components for insulated shoe liners. A nonwoven composite material was obtained, the outer layers of which are made of cotton knitted, and the inner main layer is made of camel wool. The thickness of the obtained material is 2.4 ± 0.1 mm, about 75% of which is a non-woven woolen layer. The layers are held together by means of polyacrylic glue. In the process of thermal exposure at a temperature of 150 ± 5 ° C for 2.0 ± 0.2 minutes, a chemical interaction occurs between the molten polymer and protein macromolecules of wool.

The average diameter of non-uniform in size, dense, bulky wool fibers is about 50 microns, so they are difficult to spin. In the process of forming the composition, the melted gluepolymer diffuses into the woolen and knitted layers; the melt is well combined with woolen and cotton fibers. Judging by the intensities of the X-ray spectra signal, the crystalline phase of the interaction product decreases in comparison with the initial components, this leads to an increase in the porosity and volume of the composite. Diffusion of melted glue into wool and knitwear fibers, adhesion of the polymer layer on the fiber surface, and finally, the chemical interaction of polymer and wool macromolecules are factors of solidity and high strength of a multilayer insulated composite material based on camel wool.

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