Investigation on the thermal degradation, moisture absorption characteristics and antibacterial behavior of natural insulation materials

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Abstract

The demand for natural insulation materials is increasing with special attention to the use of such materials for exploiting renewable energy. Natural insulation materials tremendously influence the sustainability development and energy efficiency enhancement in the buildings. Natural fibers from animal's origin absorb great amount of moisture on exposed to the environment which significantly affects the performance and thermal insulation properties. The thermal degradation of such material strongly influences the accidental burning characteristics, an important selection criteria for building materials. In the present study, three different kind of natural insulation materials namely sheep wool, goat wool and horse mane have been characterized in terms of moisture absorption, thermal degradation and morphology using thermogravimetric analysis (TGA), differential scanning calorimetry techniques, and scanning electron microscopy, respectively. In addition, antibacterial behavioral study has been also carried out for untreated raw wool and treated wool (copper nitrate). These properties are vital for a holistic evaluation of the insulation material. Moisture absorption results indicate that the sheep wool and goat wool absorb less moisture content as compared to horse mane. Unlike this horse mane shows great stability than goat wool and sheep wool in the temperature range not exceeding 470 °C. TGA data indicate 50% mass loss ($T_{50\%}$) at 306 °C, 322 °C and 318 °C for sheep wool, goat wool and horse mane, respectively. In addition the tests show that the content of fire retardant elements like nitrogen and sulphur is more in horse mane as compared to sheep wool and goat wool. The treated wool samples showed excellent antibacterial properties as compared to untreated wool samples.

Keywords Natural insulation materials · Thermal degradation · Moisture absorption · Thermogravimetric analysis (TGA) · Antibacterial properties

Abbreviations

CHNS	Carbon, hydrogen, nitrogen and sulphur
DSC	Differential scanning calorimetry
DTA	Differential thermal analysis
DTGA	Derivative thermogravimetric analysis
Gw	Goat wool
Hm	Horse mane
SW	Sheep wool
TGA	Thermogravimetric analysis
TIS	Thai Industrial Standard

w_d	Dry weight
w_w	Wet weight

Introduction

Insulation materials are the key element for energy saving in the building sector. For thermal and sound absorption applications, the synthetic materials such as stone wool, glass wool, basalt fiber etc. are widely used. The glass wool based insulation is widely used but it is very health hazardous as it contains silica and other synthetic fiber based on petroleum products. Glass wool insulation material is known to have carcinogenic effect [1] and is difficult to recycle. Thus the demand for natural insulation (e.g. flax, hemp, wood wool, sheep wool, goat wool etc.) is growing.

Natural wool based insulation materials (e.g. sheep wool, goat wool and horse mane) [2] have the affect of influencing

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the energy efficiency in the building sector on account of their porous structure and cheap cost. Wool has high hygroscopic, low thermal conductivity and good acoustic properties. On account of high degree of water absorption, wool fibers produce a difficult microclimate around for the growth of microorganism. The thermal insulation properties are rarely affected to 20% by the moisture content. Absorption of large amount of moisture considerably affects the properties of insulation material, particularly the thermal properties. As such it is important to evaluate moisture absorption characteristics of the insulation material before its possible use as a building insulation material.

Many authors have studied the moisture absorption characteristics of natural fibers. Korjenic et al. [3] studied the moisture absorption performance of renewable insulation materials such as jute, flax and hemp fiber and compared the results with the conventional insulation material. They found that the hemp flux and jute fiber absorb less moisture content as compared to wool, wood etc. Volf et al. [4] investigated sorption properties of treated and untreated sheep wool, wood fiber and hemp fiber and compared the result with synthetic insulation material (mineral wool). They observed that the natural fiber absorb more moisture humidity than mineral wool due to its complex organic structure. Luamkanchanaphan et al. [5] investigated the moisture absorption properties of narrow-leaved cattail fiber based insulation material. They found that the moisture content of the insulation board is between 11 and 13% for the density of board 200–400 kg/m³.

Thermal degradation of natural insulation materials must be studied before their use in the building sector. In addition, in the building sector use of new materials with good thermal and acoustic performances is being investigated. The thermal analysis [TGA, differential scanning calorimetry (DSC)] are used to investigate the thermal stability of the materials [6, 7]. In the past decade, thermogravimetric analysis (TGA) has been widely used in cases involving insulation materials exhibiting change in mass as the function of increasing temperature. TGA and DSC hyphenated technique is an efficient method for studying thermal decomposition of insulation materials.

Many authors have investigated the thermal degradation of insulation materials in different ambient conditions. Forouharshad et al. [7] investigated the TGA behavior of raw and treated wool. Their results indicate the first endothermic process occurs from 30 to 160 °C involving 7% of mass loss. The second stage of endothermic occurs from 190 to 350 °C involving 38% of mass loss. The third stage of exothermic process takes place at 350 °C. Their results further indicate that the treated sample show high decomposition temperature with greater mass loss than the raw wool. Pakkaner et al. [8] carried TGA and DTGA studies of wool, oxidized wool and keratose wool samples. They found three major stage of

mass loss for all the samples and found 2% of residue at the end of 1000 °C. The first endothermic process took place at about 75 °C in which about there was 7% of mass loss. The second and major endothermic process was observed between 200 and 400 °C involving 36% of mass loss for defatted wool, 37% for oxidized wool and 45% for keratose. Jiao and Sun [9] studied the thermal degradation behavior of extruded polystyrene (XPS) using TDA-DSC technique. Their result shows that the XPS follows two stages of mass loss of the samples under nitrogen and three stage of mass loss under air. Thermal degradation of XPS material is fast in ambient air as compared to nitrogen. Chetehouna et al. [10] investigated the thermal degradation of insulation material e.g. wheat straw and barley straw and two binders from the temperature of 50–100 °C at a heating rate of 20°/min. Their TGA results indicate that the thermal degradation of composite samples show 3–4 stages of degradation as compared to 1–2 step for the basic materials. In addition, it is mainly affected by the binder nature of the materials. Sisti et al. [11] carried TGA, of retted and unretted hemp fiber between 50 and 800 °C at a heating rate of 10°/min under nitrogen atmosphere. Their results indicate that both the samples loss their mass to about 3% in the temperature range of 50–160 °C. The second stage of degradation begins at 200 °C for unretted and retted hemp fiber. The degradation temperature seems to be quite higher with 44% of mass loss for the unretted hemp fiber and 68% for the retted fiber.

Natural wool belongs to a group of proteins known as keratins which act as nutrients and energy sources for microorganisms and bacteria under certain conditions. Soil, dust, sweat and scraps resulting during the finishing of fibers can also be nutrient sources [12]. Wool fiber produces a large number of bacteria and molds during the course of shearing, spinning, transportation, and storage, which severely damage the quality of wool fiber, resulting in failure for textile processing and great economic loss [13]. The growing interests for the personal health, hygiene and economic loss have necessitated the trend for improving the antibacterial properties of wool fabrics. Several different types of antimicrobial agents are in vogue in the textile industry for enhancing antimicrobial properties of wool fabrics. Some commonly used agents for arresting the bacterial and microbial activities in the fibers are chitosan, dyes, triclosan, metals and metal compounds, poly(hexamethylene biguanide), quaternary ammonium salts, regenerable *N*-halamine compounds and peroxyacids [14]. It is pertinent to mention that copper ions, either alone or in copper complexes, have been used as a biocide for centuries [15].

The objective of the present study is the investigation of the moisture absorption properties, thermal degradation and morphology using TGA, DSC, chemical analysis (CHNS) and scanning electron microscope (SEM) of the three different types of natural insulation material namely sheep wool,

goat wool and horse mane. In addition a comprehensive antibacterial behavioral investigation of the untreated (raw) and treated has been carried. The selection of materials in the current study is based on the fact that very few studies are available in the literature for these materials.

Materials and methods

Material selection

Three different natural raw materials namely sheep wool, goat wool and horse mane (horse's neck hair) have been selected for the investigation. Sheep and goat wool natural raw material are obtained from the sheep and goat, respectively. Mane is obtained from the upper part of the horse's neck. The application of goat and horse mane is very less as compared to their availability which results in its wastage. There are lots of grease and scraps in the raw wool, and the fibers are interlocked with each other. So it is very essential to remove all the scraps from the fleece and unlock the wool. Initially, wool is unlocked manually followed by the carding machine. This ensures all the impurities like dry plants are removed and fibrous raw wool is obtained.

Preparation of wool samples for antibacterial study

For carrying the antibacterial study procedure adopted by Heliopoulos et al. [16] has been used in the present study. Aqueous solutions of 2500 mg/500 ml was prepared by dissolving copper nitrate $\text{Cu}(\text{NO}_3)_2 \cdot 5\text{H}_2\text{O}$ powder in distilled water. The washed wool samples were cut into pieces and immersed into separate flasks of aqueous solutions, each containing 1: 30 ratio (liquor-to-fiber sample) and subjected to agitation in an orbital shaker at 200 rpm at 27 °C for 24 h. After the stirring, the wool samples were rinsed with distilled water and dried at room temperature. Finally the wool samples are ready for the antibacterial study.

Preparation of bacterial suspension

Agar well-diffusion method has been adopted for determining the antibacterial activities in the present study. The Nutrient Broth (NB) agar was prepared as the ratio of 8 g: 1 L and Muller Hinton Agar (MHA) as 38 g: 1 L. In the preparation, 8 g of nutrient broth powder is added to 1 L of distilled water taken in a beaker. The suspension is boiled to dissolve all the medium completely. The dissolved medium is then autoclaved at 121 °C for 15 min. Once the nutrient agar has been autoclaved, it is allowed to cool. The media is then poured into sterile petri plates under sterile conditions and incubated overnight at 37 °C. Ten streaks of the diluted inoculum are made over a standard petri dish with nutrient

agar (NA), without refilling the loop. The wool samples were placed over the streaks, ensuring intimate contact with the agar surface. The petri dishes were incubated for 48 h at 37 ± 2 °C. The level of antibacterial activity is assessed by the examination of the extent of bacterial growth in the contact zone between the agar and the test specimen.

Moisture absorption properties

Organic insulation material plays a vital role in the moisture absorption characteristic affecting the thermal performance of the material. Wool has to ability to absorb huge amount of water as compared to other fibers without affecting the thermal properties on account of its complex organic structure. In the present study the moisture absorption properties of the three insulation wool samples namely sheep wool, goat wool and horse mane have been evaluated. The method of moisture absorption has been carried out according to the Thai Industrial Standard TIS 876–2547 in view of the approach used by several researchers [5, 17]. All the samples were dried in sunlight for one day followed by drying in an oven at 110 °C for 1 h. The samples were weighed with a weighing balance and dry weight was obtained. The samples were conditioned at room temperature at a relative humidity of $75 \pm 5\%$ for 72 h. The samples were weighed again and wet weight was obtained. The procedure was repeated thrice. The moisture absorption of the samples is determined by the following equation:

$$\frac{w_w - w_d}{w_d} \times 100, \quad (1)$$

where w_w is the wet weight of the sample and w_d is the dry weight of the sample.

Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC)

TGA is a technique that measures the change in weight of a sample as it is heated, cooled or held at constant temperature. Simultaneously DSC detects thermal events such as melting and crystallization in addition to providing accurate and precise transition temperatures. TGA and DSC were performed with Mettler Toledo ultra-micro balance (TGA/DSC 3 + STAR^c System) as shown in Fig. 1 to study the thermal behavior and stability of the three different samples of wool. Wool samples were heated from room temperature to about 500 °C at the heating rate of 10 °C/min in ambient air atmosphere. The resulting thermogram plots indicate percentage residual weight against temperature. The derivatives of TGA are obtained directly from the microprocessor data analysis system coupled to TGA data at different decomposition temperature. The decomposition temperature $T_{50\%}$ is used as the

Fig. 1 Photograph of the Mettler Toledo TGA/DSC

temperature at which the material has lost 50% of its original weight. A differential thermogravimetric analysis (DTGA) profile is generated from the TGA analysis data.

Elemental analysis

Elemental analysis of all the wool samples has been performed using CHNS organic elemental analyzer Euro Vector Instruments & Software (Euro EA 3000 Elemental Analyser) shown in Fig. 2. Elemental analysis is used to investigate the elemental percentage of carbon, hydrogen, nitrogen and sulphur in the wool samples. Elemental analysis can be qualitative (determining what elements are present) and it can be quantitative (determining how much of each are present).

Fig. 2 Photograph of the CHNS Euro EA elemental analyzer

Scanning electron microscope (SEM)

The surface morphology of dried wool fiber samples has been studied with Zeiss EVO 50 operated at a typical acceleration voltage of 20 kV with a working distance of 9–11 mm. The samples were sputter-coated with a thin layer of gold before carrying SEM observations.

Antibacterial study of the wool fiber

The antibacterial properties of untreated (raw wool) and treated with copper nitrate $\text{Cu}(\text{NO}_3)_2 \cdot 5\text{H}_2\text{O}$ were studied. The antibacterial properties of fabrics were evaluated according to the ATCC standards. ATCC test method is a qualitative method for determining antibacterial activity of treated materials against both Gram-positive and Gram-negative bacteria. Treated material is placed in nutrient agar which is streaked with test bacteria. Bacterial growth is determined visually after incubation. In the present study, the antibacterial properties were determined against *Staphylococcus aureus* ATCC 25923, a Gram positive bacterium and *Aeromonas hydrophila* ATCC 7966A, a Gram negative bacterium. Agar was used as the culture medium for the two bacteria types.

Results and discussion

Moisture absorption characteristics

The Table 1 shows dry weight, wet weight and moisture absorption percentage of each category of wool. The average value of moisture content must be in the range of 4–13% according to the TIS 876-2547 standard. From the Table 1 it is observed that the moisture content of sheep wool is 10.12%, goat wool is 10.85% and horse mane is 11.53%, which agrees well with TIS 876-2547 standard. It is observed that horse mane absorbs more moisture than that of goat and sheep wool indicating that the horse mane is coarser and thicker. Sheep and goat wool absorb less moisture content due to thinner and fine fibers of wool.

Table 1 Moisture absorption properties of various insulation samples

Sample	Dry weight w_d (g)	Wet weight w_w (g)	Moisture absorption $\frac{w_w - w_d}{w_d} \times 100\%$
Sheep wool	42.55	46.86	10.12
Goat wool	45.59	50.54	10.85
Horse mane	52.44	58.49	11.53

Thermogravimetric analysis (TGA) and derivative thermogravimetric analysis (DTGA)

Figure 3 represents the TGA and DTGA of sheep wool, goat wool and horse mane. As seen from the Fig. 3 the characteristics feature of thermograms are similar in the three samples of wool used. During the heating process the thermal degradation in air atmosphere takes place in three stages for all the three insulation samples. The initial stage of degradation starting from 30 to 135 °C for sheep wool involving mass loss of 14.2%. For goat wool the first stage of degradation occurs between 31 and 140 °C with the mass loss up to 14.8%. Similarly for horse mane first stage of degradation occurs between 31 and 133 °C with the mass loss up to 13.8% as shown in the Table 2 and Fig. 3. The first stage of degradation can be attributed to the loss of water from the wool samples as reported by other authors [18, 19]. There are three categories of water present in the wool fiber namely free water, chemically bonded water and loosely bonded water all are lost in the first stage of degradation [20].

The second most important stage of degradation appears between 218 and 390 °C for sheep wool with a mass loss of 51.1%, for the goat wool a mass loss of 46.6% in the temperature range of 220–383 °C and for horse mane 41.2% mass loss in the temperature range of 224–370 °C. In this stage the mass loss is caused by the breakdown of microfibril-matrix structure and disulphide bonds of wool. In the third stage of degradation remaining mass loss takes place. In this stage the degradation occurs in the temperature range of 390–500 °C for sheep wool, 383–500 °C for goat wool and 370–500 °C for horse mane with the mass loss of about 23.5%, for sheep wool, 16.3% for goat wool and 21.9% for horse mane. This may be attributed to loss of various side chains on account of severe breakage of the peptide of the wool fiber [21]. It has been observed from the Table 2 and Fig. 3 that the sheep wool loses higher mass in the second and third stages of degradation while as horse mane loses less mass in the first and second stages of degradation. The major DTGA peaks reach the temperature of 60 °C and 280 °C for sheep wool, 62 °C and 275 °C for goat wool and 50 °C and 271 °C for horse mane. Similar results of TGA curve has been observed by Pakkaner et al. [8] for the different wool samples.

Table 3 presents the 50% ($T_{50\%}$) of mass loss and mass residue at 500 °C for the three wool samples. It is found that the 50% of mass loss occurs at the temperature of 306 °C for sheep wool, 322 °C for goat wool and 318 °C for horse mane. The mass residues at the heating temperature of 500 °C are 5.3%, 21% and 18% for sheep wool, goat wool and horse mane respectively.

Figure 4 clearly demonstrates the degradation comparison of the three wool samples. It can be seen from the figure that the goat wool shows higher mass loss than the sheep wool

Fig. 3 TGA-DTGA curves for three different insulation wool samples **a** sheep wool **b** goat wool **c** horse mane

and horse mane in the first stage of decomposition but at the end of the first stage, sheep wool sample loss the mass very rapidly than other two types of wool samples. From Fig. 4 it is found that the horse mane has higher thermal stability upto a temperature of 460 °C. At temperature above 460 °C

Table 2 Mass loss (%) at different stages of degradation under air atmosphere

Sample	First stage of degradation temperature (°C)	Mass loss (%)	Second stage of degradation temperature (°C)	Mass loss (%)	Third stage of degradation temperature (°C)	Mass loss (%)
Sheep wool	30–135	14.2	218–390	51.1	390–500	23.5
Goat wool	31–140	14.8	220–383	46.6	383–500	16.3
Horse mane	31–133	13.8	224–370	41.2	370–500	21.9

Table 3 Decomposition temperature (°C) at 50% of mass loss (%) and mass residue

Sample	Decomposition temperature (°C) at ($T_{50\%}$)	Mass residue (%) at 500 °C
Sheep wool	306	5.3
Goat wool	322	21
Horse mane	318	18

Fig. 4 Thermogravimetric analysis (TGA) curves for three different insulation wool samples

the horse mane lose slightly more mass as compared to goat wool.

Differential scanning calorimetry (DSC)

Figure 5 shows DSC curve for the three different insulation samples and Table 4 reports the corresponding endothermic peak of the samples. The endothermic reaction occurs with the release of sulphur compounds due to the breaking of cystine disulphide bonds and simultaneous release of hydrogen sulphide. The first endothermic peak for sheep wool is seen at 57 °C, for goat wool at 53 °C and for the horse mane at 60 °C. The second endothermic peak appears at 232 °C for sheep wool, 227 °C for goat wool and 228 °C for horse mane. The third endothermic

Fig. 5 Differential scanning calorimetry (DSC) curves for three different insulation wool samples**Table 4** The endothermic peaks of three different insulation materials

Sample	Peak	T_{max} (from DSC curve) (°C)
Sheep wool	First peak	57
	Second peak	232
	Third peak	380
Goat wool	First peak	53
	Second peak	227
	Third peak	375
Horse mane	First peak	60
	Second peak	228
	Third peak	400

peak occurs at 380 °C for sheep wool, 375 °C for goat wool and 400 °C for horse mane. The temperature ranges and trends of TGA, DTGA and DSC curve are very well matched with other researchers [19, 22–24] for the insulation materials.

Elemental analysis

Elemental analysis of all the wool samples has been performed using CHNS organic elemental analyzer and the results are displayed in Table 5. It can be seen that all wool

Table 5 Elemental analysis of three different wool samples

Sample	Carbon %	Hydrogen %	Nitrogen %	Sulphur %
Sheep wool	44.807	7.647	14.268	2.860
Goat wool	45.594	7.040	15.533	3.463
Horse mane	45.477	7.189	15.939	3.755

samples contain carbon, nitrogen, hydrogen and sulphur. From the Table 5, it is clear that sheep wool has less carbon, nitrogen and sulphur content as compared to the of goat wool and horse mane. It is well known fact that nitrogen and sulphur content in wool fibers are essential for the fire retardant performance of the wool fibers. This is on account of the fact that the existence of nitrogen requires large quantities of oxygen from the environment for combustion.

Morphological examination

Wool fibers contain two types of cells: cuticle cells (the outer layer of the fibre) and cortex (the inner layer of the fiber) as reported by [25]. Coarse wool fibers (diameters > 35 mm) contain a third type of cell called medulla. Cuticle cells overlap both along and around the circumference of each fiber like tiles on a roof, and can be seen clearly in the SEM (see Fig. 6). This structure makes wool unique in the class of the textile fibers. It is because of the wettability and tactile

behavior of cuticle cells the wool can be converted into felts on exposure to moist conditions [26]. The cuticle cells provide a tough exterior, and thus protect the fiber from damage. The cuticle and cortex differ in configurations in their polymer chains as well as amino acid compositions. The cortex is the most important component of wool covered by the cuticle. A large volume of air traps between medullar cells which improve the thermal insulation properties. Figure 6 shows the morphology of the wool samples. Sheep wool and goat wool is smooth, exhibiting sharp scales edges and more relaxed, intact and clear edges. Unlike this the horse mane sample shows coarse wool fiber with the presence of very tiny scales. The scales act as barriers for water, dye and finishing agents applied on wool fibers and adversely affects the sorption behavior. Some impurities are also seen on the surface of wool fiber, but no surface damages are observed.

Antibacterial properties

Antibacterial behavior of untreated (raw wool samples) and treated samples of three different kinds of wool are shown in Fig. 7a–d. *S. aureus* (Gram-positive) of untreated wool is seen in Fig. 7a, *A. hydrophila* (Gram-negative) of untreated wool in Fig. 7(b), *S. aureus* (Gram-positive) treated with $\text{Cu}(\text{NO}_3)_2 \cdot 5\text{H}_2\text{O}$ in Fig. 7(c) and *A. hydrophila* (Gram-negative) treated with $\text{Cu}(\text{NO}_3)_2 \cdot 5\text{H}_2\text{O}$ in Fig. 7(d). Visible bacteria are clearly seen in the Fig. 7a, b in all the raw

Fig. 6 SEM images of the three wool samples **a** sheep wool **b** goat wool **c** horse mane

Fig. 7 Antibacterial behavior of untreated and treated samples of sheep wool, goat wool and horse mane **a** untreated wool against *Staphylococcus aureus* **b** untreated wool against *Aeromonas hydrophila* **c** treated wool against *Staphylococcus aureus* **d** treated wool against *Aeromonas hydrophila*

samples of untreated wool (sheep wool, goat wool and horse mane) and as such the samples are ineffective against both *S. aureus* and *A. hydrophila*. Unlike this, Fig. 7c, d of treated wool samples show excellent antibacterial properties. From the Fig. 7c, d it is seen that treated wool samples exhibit antibacterial activity against *S. aureus* and *A. hydrophila*, with activity against *A. hydrophila* being more prominent. Sheep wool and goat wool exhibited large inhibition zone than horse mane against *A. hydrophila* while against the *S. aureus* all the samples show large inhibition zone. This type of antibacterial activities is in line with the literature [16, 27, 28] and it becomes evident that bacterial growth seems unrestricted in the untreated raw wool samples while as the treated wool possess efficient antibacterial effects.

Conclusions

Three natural insulation wool materials (sheep wool, goat wool and horse mane) have been selected for the investigation of thermal degradation, moisture absorption and elemental analysis. The main conclusions drawn from the study are:

1. Horse mane shows higher moisture content (11.53%) than sheep wool (10.12%) and goat wool (10.85%) due to high coarseness and thickness.
2. TGA curve indicates more stability for horse mane up to a temperature of 460 °C as compared to sheep wool and goat wool and above 460 °C temperature horse mane loses more mass followed by goat wool.
3. It has been observed that the 50% ($T_{50\%}$) of the mass loss occurs at 306 °C for sheep, 322 °C for goat wool and 318 °C for horse mane. The mass residue at 500 °C of

sheep wool is 5.3%, goat wool is 21% and horse mane is 18%.

4. DSC curve indicates that the three endothermic peaks observed for sheep wool are at 57 °C, 232 °C and 380 °C, for goat wool at 53 °C, 227 °C and 375 °C and for horse mane 60 °C, 228 °C and 400 °C.
 5. It is found that all the wool samples contain carbon, hydrogen, nitrogen and sulphur. It is also observed that the sheep wool has less carbon, nitrogen and sulphur content than the goat wool and horse mane.
 6. Horse mane has higher nitrogen and sulphur content as compared to goat wool and sheep wool.
 7. The morphology of the wool samples shows that the sheep wool and goat wool fibers are smoother and more relaxed while the horse mane samples show coarse and rough surface.
 8. Untreated wool samples are ineffective against *Staphylococcus* and *A. hydrophila* while as treated wool shows efficient antibacterial effects.
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