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Tannery Shavings and Mineral Additives as a Basis of New Composite Materials

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Abstract

This paper analyses the possibility of using tannery shavings as a basis for new composite materials with specified properties. The new material was made by combining tannery shavings with an adhesive medium. Mineral additives, such as dolomite, kaolin and bentonite were used in an amount of 5% and 10% of the total mass of shavings as a filler. In order to point out the influence of mineral additives on the final composite structure, some physico-mechanical properties, such as tensile strength and elasticity were examined with the use of static tensile tests. The second part of the investigation involved the analysis of wetteability by immersion, because this property depends on several aspects, such as surface roughness and the material structure. Thus, changes in wetteability values can provide information about the impact of mineral additives on the material properties of composites. With regard to physico-mechanical and sorption properties obtained on the basis of experimental results, it was possible to define areas of possible applications of these materials in comparison with current methods.

Key words: *composites, tannery shavings, mineral fillers.*

Introduction

Tannery wastes are actually a major environmental problem [1, 2]. In 2016 the production of leather in Poland generated about 46 thousand tonnes (excluding municipal waste), of which about 2% was recycled and 4% neutralised (in the manufacturer's own way). At present, it is estimated that the total amount of waste generated by the leather industry which is deposited at landfills controlled by the industry stands at around 28.4 thousand tonnes. It is projected that in the next few years, the total amount of waste will grow. This growth is a consequence of the increase in leather production from 15 thousand tonnes in 2010, through 17.2 in 2015, to 21.1 tonnes in 2016 [3]. European tanneries produce about 2.14 kg of waste per each square metre of leather.

According to the list of wastes drawn up by the European Commission (Commission Decision 2000/532/EC), tannery wastes can be classified as follows:

- Fleshings and lime split waste,
- Liming waste,
- Degreasing waste containing organic solvents without a liquid phase,
- Tanning liquour containing chromium,
- Tanning liquour free of chromium,
- Sludge containing chromium,
- Sludge free from chromium,
- Waste tanned leather (sheetings, shavings, cuttings, dusts),
- Waste from dressing and finishing processes,
- Not otherwise specified.

The type of waste and its amount vary depending on the processes used for leather production and methods of the waste water treatment used in the tannery.

Tanning processes are very important in the leather manufacturing cycle, because they protect raw leather materials from harmful environmental effects (such as heat, sweat or moisture). About 80-90% of world tanneries use salts of chromium III in these processes [4, 5], which is nontoxic in a trivalent state, but toxic in a hexavalent state [6]. The literature shows different methods for safe and environmentally sound disposal of chromium–containing solid waste [7] due to the "green route" [8] and "wealth from waste" [9] ideas. For example, Erdem in paper [10] investigated the process of chromium recovery from chrome shaving wastes (solid waste containing chromium) by oxidation with air, oxygen and Na_2O_2. Chromate ions were reduced with the use of Na_2SO_3, which is one of the ways to avoid the formation of chromium (VI). Pillai and Archana [11] describe an effective method of utilisation of tannery shavings by using chromium resistant bacterium – Baccilus subtilis.

Aftab et al. [12] demonstrated the enzymatic hydrolysis of chrome shavings in order to obtain amino acids, polypeptide products and other [products], which may be chemically treated or recycled. This is important from the recovery point of view because leather wastes are rich in protein and collagen fibres. For example, protein products can be used in the pro-

Table 1. Particle size distribution of shavings.

Fraction, mm	Mass fraction, %
0-1.0	34.0
1.0-2.5	25.5
2.5-8.0	22.0
8.0-15.0	18.5

duction of cosmetics, adhesives, printing, photography [13], microencapsulation or films.

The other way is to use them as an additive in finishing products utilised in the leather industry [14]. ŁUKASIEWICZ Research Network the Institute of Leather Industry is researching new seed coating technology based on collagen hydrolysates derived from tannery wastes. The coating process is conducted in order to improve drought and pest resistance especially during the germination period [15-17].

In this paper the authors focused on the possibility of utilising tannery shavings because they constitute about 25% of the total mass of raw leather. These are small particles of irregular shape, usually consisting of collagen fibres with chromium III complexes [7, 14, 18]. The literature shows that the organic matter content in the dry weight of tannery shavings is between 85 and 90%. The average moisture content is around 50-60%. Moreover, chrome shavings have a significant amount of proteins (78.6-75.2%) and chromium oxide (4.4-4.3%) [19] and are used in different branches. For example. Przepiórkowska and co-authors used recovery collagen from chrome-tanned wastes as fillers for isoprene rubber mixes [20]. As an effect of this procedure, some mechanical properties such as the tensile strength and elongation at break were improved [21]. Nowadays biocomposites are gaining increasing environmental importance [22]. Using materials based on renewable sources will make it possible to solve a lot of ecological issues [23].

Experimental results obtained in this paper are aimed at proposing a new way of utilising tannery shavings by creating new composite materials. In order to find possibilities of application, innovative materials with mineral additives as fillers were examined in the field of physico-mechanical properties, such as strength and wetteability. This paper is a continuation of the authors' previous research [24, 25], with the difference that the adhesive medium, i.e., the water dispersion of polyvinyl acetate, was replaced by naturally occurring glue.

Materials and method

Tannery shavings from the conventional chrome – tanning process (intermediate material, wet-blue from a Polish tannery) were used in order to form new composite materials. Particle sizing distribution was conducted by sieve analysis [26-34] (***Table 1***). Shavings of dimensions smaller than 8 mm (constituting less than 80% of all fractions) were blended with collagen-based adhesive. The ratio between the mass of shavings to that of the adhesive medium was equal to 60:40.

At the end of the mixture preparation process, mineral additives were added in a proportion of 5% and 10% in relation to the shavings mass. The following materials were used as mineral fillers: dolomite (with a fineness below 0.045 mm) and silicates: kaolin (with a kaolinite content equal to 81% and particle size between 0.2 and 0.002 mm) and bentonite (with an 81% montmorillonite content and particle size less than 0.056 mm). The mixture prepared was pressed with a hydraulic press at a constant pressure of 20 MPa, and composite materials were formed as squares with dimensions 280 x 280 mm. The materials prepared were dried for over 24 hours in a laboratory drier at 25 °C. The materials were conditioned for 72 hours in a well-ventilated room at ambient temperature prior to testing and their physico-mechanical parameters were obtained with the use of a Zwick/Roell Z010 material testing system by means of uniaxial static strength tests with a constant crosshead speed of 50 mm/min. These tests were done in order to indicate how the material reacts to forces being applied in tension. The applied load and elongation were measured. Results of these mechanical tests were used to determine the modulus of elasticity, elongation under load and the tensile strength.

In order to determine the sorption properties of these materials, moisture absorption was measured with the use of smaller samples (3 x 3 cm). These samples were placed in glass beakers with distilled water. The water column over the samples was set at 30 mm. The soaking time was equal to 15, 30, 60 and 150 min. After this time, samples were removed from the beakers, drained between two filters for one minute and weighted.

Water absorption X (%) for the times measured was calculated using the following ***Equation (1)***:

$$X = \frac{m_1 - m}{m} \cdot 100\,\% \qquad (1)$$

where:
m_1 – mass of moistened sample, g,
m – mass of sample before immersion, g.

The final result was an arithmetic mean of two parallel samples.

Results and discussion

Mechanical properties

In order to determine the material characteristics, Young's modulus was calculated with the use of the tensile strength and elongation values. Values of Young's modulus were used in order to classify the materials in a group of materials with similar properties, because it is a measure which describes the elongation or compression of a material in an area when the stress load is less than the yield strength of the material. Aggregated results describing mechanical properties of the composites formed were put together in ***Table 2***.

According to the aggregate data from ***Table 2*** it can be observed that the highest values of tensile strength are achieved by samples with 5% bentonite addition (5.7 MPa) and with 10% dolomite (3.7 MPa). On the other hand,

Table 2. Physico-mechanical properties of composites with mineral additives (5% and 10% of dolomite, kaolin and bentonite).

Mineral filler	Mineral content, %	Tensile strength, MPa	Maximum load F_{max}, N	Elongation for maximum load F_{max}, mm	Young's modulus, GPa
Dolomite	5	3.7	756.0	5.2	0.0297
	10	5.0	904.3	4.3	0.0464
Kaolin	5	2.7	692.0	5.3	0.0261
	10	4.5	1008.7	4.3	0.0428
Bentonite	5	5.7	1025.0	6.1	0.0375
	10	4.2	884.0	4.4	0.0389

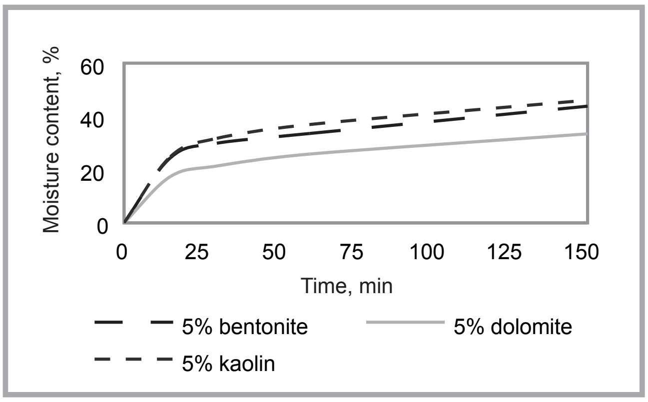

Figure 1. Moisture absorption for tannery shavings based composites with 5% mineral filler.

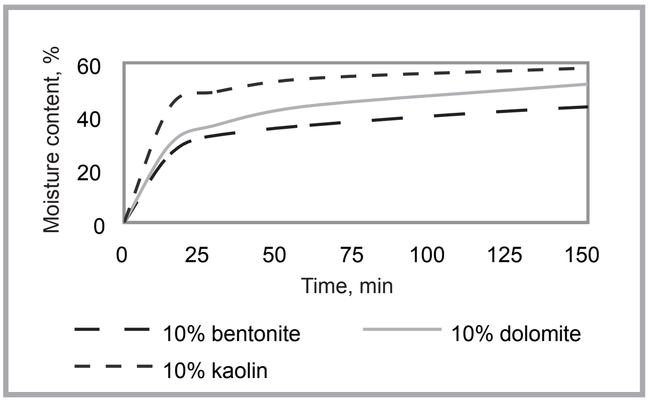

Figure 2. Moisture absorption for tannery shavings based composites with 10% mineral filler.

a reduction in tensile values to 2.7 MPa was observed for 5% kaolin addition. The analysis demonstrated that the type and proportion of mineral filler is related to the mechanical properties of the composites (such as tensile strength, elongation and maximum load). This method was confirmed by statistical analysis of differences between results with the use of the t-Test with a confidence interval of $\alpha = 0.05$. For example, when the composite with a 5% bentonite content was compared with 5% dolomite, the differences were statistically significant, with a p-value equal to $0.00145 < \alpha$. On the other hand, materials with a 5% mineral filler addition reached higher values of elongation than composites with a 10% filler content for all kinds of fillers. Maximum values of elongation were noted for the bentonite composite. The impact of the type of mineral filler on elongation values was confirmed by statistically significant differences for composites with 5% bentonite and kaolin (with a p-value equal to $0.0318 < \alpha$). Statistically significant differences were also observed for another parameter examined, F_{max}. They appeared for materials with 5% and 10% kaolin when the p-value reached 0.0243, which was less than the confidence level α.

Moreover, maximum values of F_{max} were achieved for materials with 5% bentonite (1025 N) and 10% kaolin (1008.7 N), in contrast to 5% kaolin filler, when the maximum load was the smallest from the set of data (692 N). As one can find in the literature, it is possible to change mechanical characteristics, such as hardness and abrasion resistance with the use of silica, dolomite or aluminium oxide as fillers [35, 36]. The authors of paper [37] modified footwear leather materials by the addition of zeolite and montmorillonite in order to improve some physico-mechanical (i.e., tensile strength) and hygienic parameters (i.e., water vapour permeability and water vapour permeability coefficient).

The values of such parameters as tensile strength and elongation at break made it possible to calculate Young's modulus for each material, which was very important in order to classify these materials in groups with similar properties. The values of Young's modulus lie between 0.0261 GPa (for a 5% kaolin filler content) and 0.0464 GPa (for a 10% dolomite filler content). Therefore, the classification of these materials places them in the group of polymers (EVA) and certain materials from the group of elastomers, for example, rigid polymer foam [38]. The properties of these materials provide information about the possibility of their application. For example, rigid polymer foams are used in thermal insulation, such as the insulation of buildings. Polyurethane foams with a high closed-cell structure are moisture resistant and can be used for buoyancy in boats.

Moisture absorption

The results of moisture absorption during water exposure are shown in ***Figures 1*** and ***2***. Moreover, the moisture absorption rate is an important characteristic for a qualitative assessment of the final product. Water absorption rates are shown in ***Table 3***. They can be interpreted by the tangent of the angle of inclination of the line passing through the measuring points for neighbouring time intervals, i.e.: 0-15 min, 15-30 min, 30-60 min and 60-150 min.

As a result of analysis of moisture content for each material, it can be observed that the composites with kaolin are the best absorbers, where the moisture content reached the value of 46% for a 5% mineral content and 58% for a 10% mineral content over the 0-150 min time interval. In contrast, the weakest absorption property was noted for the composite with a 5% dolomite filler (33%). The amount and rate of water absorption depend on the structure of materials. These properties can provide qualitative information about storage and drying processes and other characteristics such as pore size distribution. In the case of the mineral additives used in this experiment, it is possible to find a source of differences between materials based on the type of mineral filler. For example, for dolomite, the presence of such oxides as MgO or CaO results in this mineral having an anhydrous property. Thus, the

Table 3. Moisture absorption rate of materials with mineral fillers for neighbouring periods (0-15 min, 15-30 min, 30-60 min and 60-150 min).

Mineral filler	Time intervals, min			
	0-15	15-30	30-60	60-150
5% bentonite	1.2576	0.3173	0.1491	0.1067
5% dolomite	1.6293	0.3592	0.1218	0.1152
5% kaolin	1.1565	0.2672	0.1485	0.0848
10% bentonite	1.6597	0.4565	0.1744	0.1007
10% dolomite	1.7169	0.4174	0.1524	0.0759
10% kaolin	1.965	0.4822	0.2373	0.0903

composite material with a dolomite filler is characterised by smaller moisture absorption [39, 40]. On the other hand, the ability to swell of kaolin and bentonite [41-43] correlates with higher absorption ability. The literature provides many examples of improved sorption properties of materials. For example, in paper [44] the water sorption of polypropylene – zeolite composites was studied as a function of the zeolite content. Huuhilo et al. [45] studied the impact of mineral fillers such as calcium carbonate, talc and wollastonite on the moisture resistance of wood-plastic composites.

One of the potential applications of the composite materials examined may involve the production of insoles, which must be characterised by an intensive sorption property, because it determines the fast discharge of sweat from the foot surface [46, 47]. Analysis of moisture absorption as a ratio between the mass of the sample after and before immersion to that before immersion shows that all composite materials with mineral fillers have better properties than the minimum acceptable values for footwear insoles (i.e., 10% after 30 min immersion and 20% after a two hour immersion). On the other hand, it can be pointed out that values of moisture absorption obtained for cellulose leather such as Uniflex, Texon or paperboard with different thicknesses [46] are very similar to those obtained in this research. Moreover, materials with a 10% kaolin addition have higher absorptivity than cellulose leathers. Therefore, it can be concluded that the materials examined, after certain treatment, can be used for the production of insoles.

Conclusions

Experimental results described in this paper make it possible to predict that the new composite materials investigated in this paper could provide a new way to utilise and reuse tannery shavings in a "close loop". Changing adhesive media or the total quantity of mineral additions can influence the physico-mechanical properties of these materials. Another area where optimisation actions could provide different solutions is the forming process. It is possible to change such control parameters as the temperature of pressing, pressure or time of pressing. Also sorption – drying processes can be carried out at different depths of the water column or at different temperatures and relative humidity. Thus, the range of possible applications is wide and depends on individual requirements. Young's modulus values classify these materials in the group of polymers (EVA) and certain materials from the group of elastomers, for example rigid polymer foam. Hence, thanks to the varied properties, they can be used in several applications, such as insoles or building materials.

The investigation in this paper constitutes a multi-stage cycle of research in the field of recycling and reusing wastes generated by tanneries. Their principal objective is to create a new systematic approach in order to form new materials with properties in dependence on the expectations of industry segments. They fit into the current trend connected with eco-friendly technologies.

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