Comparison of Granulation Methods for Tannery Shavings

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Abstract
The work presents a comparison of methods of granulating waste – tannery shavings – from the leather industry. This waste is often used for the production of leather composites, but it creates a number of problems in transport and storage. It seems that granulation of this waste together with mineral additives needed in their further processing is a solution. However, the traditional method of granulation involving the gradual moistening of a loose deposit on a moving surface of the granulator does not give the desired results for the granulation of material with such unusual physical properties. Therefore, new methods have been proposed to eliminate the disadvantages and limitations of traditional granulation. The methods proposed differ in the way the binder liquid and mineral material are applied. They consist in soaking the shavings with binding liquid beforehand, removing excess liquid, and then granulating the wet pulp with the addition of selected fine-grained mineral material. Analysis of the results was based on a comparison of the granulometric compositions and compressive strength of the agglomerates obtained.

Key words: granulation, tannery shavings, mineral fillers.

Introduction

The management of tanning shavings as a waste product of leather expedition technology is an important issue from the environmental point of view, including the circular economy, because they can constitute up to 20% of the total mass of tanned leather. The known directions of processing, utilisation and recycling of tanning waste are as follows: production of secondary leather, composites based on collagen fibres [1-3], other fibrous materials, protein hydrolysates [4-6], composting, agriculture (fertilizers), thermal treatment [7], and storage.

Tanning shavings are waste which is resistant to biological degradation. They are characterised by many unfavourable physical properties which make their storage, transport, pouring out of containers, dosing and loading difficult i.e. low bulk density, irregular shapes, and different fractions of grains. These properties also cause difficulties in the implementation of other unit processes, including comminution, milling [8-10] and sieving [11-20] (blocking the sieve holes).

Giving the shavings a regular, spherical shape and thus creating a loose deposit could be a solution to these problems. This effect can be obtained by non-pressure disk [21, 22], continuous [23] or drum [24-27] agglomeration processes.

The aim of granulation is to minimise environmental burdens associated with the storage and transport of tanning shavings. These agglomerates in sizes of 1-14 mm can be used instead of the previously used loose, moist and dusty shavings, e.g. in processes of producing composite materials based on collagen fibres.

Traditional granulation methods involve adding raw material, e.g. shavings and mineral material (additive) to a granulator plate, their initial mixing during the rotational movement of the plate, and then moistening in the form of drops or spurts [28] using nozzles with a solution of binding liquid. Sometimes during such granulation there is the unfavourable phenomenon of the segregation of fine-grained shavings and mineral material during the rotational movement of the plate. This segregation results from the different humidity and bulk density of both components of the mixture and may result in their separate granulation after moistening. A non-uniform granulate is obtained in which individual grains differ greatly in size, density and composition (content of shavings and mineral additive). The aim of the research was to develop a method of agglomerate production from waste tanning shavings without the disadvantages of the technology described above.

Test methods and apparatus

The first stage of the study was to screen the starting material (raw shavings) in a 2.5 mm sieve to screen the largest fraction, consisting of the longest fibres, with the most irregular shapes. These fibres cannot be treated by the process of plate granulation. Sieving time in a laboratory vibrating shaker was 20 min. As a result, two grain fractions of shavings were obtained:

- the lower fraction (grains smaller than 2.5 mm), which constituted approx. 60% by mass, which was sent directly to granulation,
- the upper fraction (grains over 2.5 mm in size), constituting 40% by mass, which was stored for later refining and re-screening.

Tests were carried out in a disc granulator [29, 30]. Three basic elements can be distinguished in the design of the granulator used for the granulation tests: a granulator plate, an electric motor with rotation speed control, and a wetting system for the granulated deposit.

The moisturising liquid consisted of water glass, i.e. an aqueous solution of sodium and potassium silicates (so-called mixed water glass). Water glass is a viscous liquid whose viscosity increases rapidly as the concentration of silicates increases. A 50% aqueous solution of water glass was used in the tests, as it was found to have optimal viscosity for the granulation of waste shavings (viscosity 0.004 Pa·s).
Figure 1. Diagram of test stand: 1 – electric motor, 2 – disk, 3 – the drive system 4 – tank with liquid, 5 – spray nozzles, 6 – rotameter, 7 – device frame, 8 – drive shaft, 9 – inverter, 10 – driver.

The 3 kW electric motor (1) is connected to the drive shaft (8) via a V-belt. At the end of the shaft a disk (diameter 0.5 m, height of the rim 0.1 m) (2) is mounted, inside which the granulated material is placed. The rotation speed (15 rpm) is controlled by means of the inverter (9) by setting the desired value on the driver (10). The binding liquid is fed directly to the deposit of granulated material through the spraying nozzles (5). The liquid flows by gravity from the liquid container (4), located 2 meters above the spraying nozzles. The liquid flow rate is controlled by means of a float flow meter – rotameter (6). The design of the device frame (7) allows to adjust the angle of the disc (45°). A diagram of the entire installation is shown in Figure 1.

Granulation with moistening of the granulated deposit

Initially, dry waste tanning shavings from chromium leather tanning processes were introduced into the granulator disc in such an amount that the ratio of their volume to the disk volume was 0.15-0.3, which ensures optimal conditions for the process. Then, a mineral additive was poured in, and the two dry ingredients were mixed for 3 minutes. The deposit was subsequently sprayed with humidifier nozzles using a 50% solution of water glass at 20 °C. The nozzle droplet size was in the range of 0.01-0.5 mm, and the wetting time was 5 minutes. After moisturing, further granulation was carried out with the disk rotation unchanged until granules were formed. The agglomerate was subjected to drying at 50 °C for 24 hours. Granulation was carried out by two methods with deposit spraying, using two mineral additives with a grain size less than 0.05 mm: gypsum and dolomite.

Method 1. Dry shavings in the amount of 200 g and 600 g of gypsum with 17% humidity, coming from the flue-gas desulphurisation process (Belchatów Power Plant), were poured into the granulator. After mixing the ingredients, they were moisturised with a 50% water glass solution. Moisturising was completed after 1300 g of solution was added, and granulation continued without moisturising. Since no granulate was observed, 800 g of gypsum was added. The total granulation time was 20 minutes.

Method 2. Dry shavings in the amount of 150 g and 600 g of dry dolomite were poured into the granulator. After mixing the ingredients, they were moisturised with a 50% water glass solution. After adding 500 g of solution, granulation was continued without moisturising. An additional 780 g of dolomite was added during granulation. The granulation time was 15 minutes.

Granulation of wet pulp

Dry shavings in the amount of 150-200 g, were mixed with 50% water glass until they were completely moisturised. A cylindrical mixer with a low-speed anchor stirrer was used, designed for mixing structures of high viscosity and density. Then, the wet mixture was transferred to a drain sieve and excess binding liquid drained by gravity until so-called static humidity was reached (this is the maximum humidity that granular material may have).

The granular deposit at rest can be characterised by the WS static moisture ratio, which is the amount of moisture (water) that a granular deposit (granular layer) is able to contain during rest. However, when this deposit is found on, for example, a vibrating (moving) sieve, a certain amount of water is quickly separated from this deposit, obtaining the so-called WD dynamic moisture ratio, which is always smaller than static one. Thus, the WD dynamic moisture ratio of the granular deposit will represent the moisture content remaining in the material despite the inertial forces (driving forces of the process) on the material. Above the W_D - W_S border area, determined by dynamic and static humidity coefficients, begins the drainage and rinsing area of the granular materials (Figure 2).

Obtaining material with the highest humidity is crucial in the further stage of the granulation process, because no binding liquid is added during the formation of granules (deposit) on the rotating surface of the disc.

The wet pulp was placed on a disc and granulation carried out with the addition...
of dry, powdered mineral. After completing the dosing of the mineral additive, further granulation was carried out with unchanged disc rotation for at least 5 minutes. The resulting agglomerate was dried at 50 °C for 24 hours.

The wet pulp was granulated on a rotating disc by four different methods, each time with the addition of completely dry or wet (17%) gypsum ( grain size less than 0.05 mm) derived from the flue-gas desulphurisation process.

Method 1. Pulp from shavings in the amount of 150 g and 50% water glass in the amount of 450 g were prepared in the mixer. After transferring the pulp to a drainage sieve, no leachate was found, which means less humidity than the static moisture of the deposit. After placing the pulp in the granulator, granulation was carried out by adding dry gypsum gradually in the amount of 500 g (granulation time – 6 min).

Method 2. A mixture of shavings in the amount of 150 g and 1000 g of 50% water glass were prepared in a mixer. After transferring the pulp to the drainage sieve, a slight leachate was found, which meant that the moisture content of the deposit was equal to the maximum static humidity. After placing the pulp in the granulator, granulation was carried out by adding wet gypsum (17%) gradually in the amount of 500 g (granulation time – 10 min).

Method 3. A mixture of shavings in the amount of 150 g, wet (17%) gypsum in the amount of 500 g, and 50% water glass (1000 g) were prepared in the mixer. No leachate was found after transferring the pulp to the drainage screen. After placing the pulp in the granulator, granulation was carried out until granules formed, and then another 280 g of dry gypsum was added to cover the surface of the granules with mineral material (granulation time – 10 min).

Method 4. Using a mixer, pulp was prepared from 150 g of dry shavings and 800 g of 70% water glass solution. No leachate was found after transferring the pulp to the drainage screen. Then the pulp was placed in the granulator, and after 2 minutes from the start of granulation, 500 g of wet gypsum (17% humidity) was added. Small amounts (100g) of water glass solution were added during granulation (granulation time – 10 minutes).

![Figure 3. Granulate grain-size (Method 2 with deposit wetting).](image)

### Research results

#### Granulation results with moistening of granulated deposit

The agglomeration process resulted in a granulated deposit, which was then subjected to sieve analysis. Agglomerates were obtained with a granulometric composition in the range of 1-12.5 mm, being non-dusty and non-caking and having a compressive strength exceeding 20 N (using an Intron testing machine).

In Method 1 of granulating shavings and gypsum, the mineral additive with a moisture content of 17% granulated too quickly, without connecting dry shavings. Adding a second portion of gypsum partially solved this problem, because the shavings and the entire deposit were already moisturised. As a result, however, uneven granules with different solid components in individual grains were obtained. However, it should be noted that the total amount of gypsum (additive) was 1400 g per 200 g of shavings, and only such a high content of the mineral allowed the granulation of tanning shavings to be carried out.

Similar observations were noted in the granulation method 2: the mineral additive (dolomite) initially agglomerated separately, and only the addition of its second portion made it possible to obtain the granulate. The total amount of mineral additive was 1380 g per 150 g of shavings. Figure 3 presents the results of granulometric analysis of the agglomerate obtained by the method described. The large amount of fine granules and lack of larger agglomerates are noteworthy. Figure 4 shows the deposit on the disc after granulation – a small amount of ungranulated material is visible, and the smallest grains, which are the most numerous, have an irregular shape.

In the granulation tests conducted by the traditional method, noteworthy is a large amount of the mineral additives: gypsum and dolomite, which should be considered the biggest disadvantage of this solution. Moreover, the granulometric composition was irregular and contained a disproportionately large amount of fine grains (Figure 3).

#### Pulp granulation results

As a result of the agglomeration of tanning shavings, granulate was obtained, which was then subjected to sieve analysis. A deposit was obtained with a granulometric composition in the range of 1-25 mm, being non-dusty and non-caking, and characterised by high powder flow and a compressive strength exceeding 20 N (except for Method 1).

The initial moisturising of tanning shavings in the mixer turned out to be a key parameter for conducting granulation. This was confirmed by granulation...
are subjected to granulation. The above-mentioned problems do not occur if pre-moisturised shredded shavings in the form of a pulp clump into large agglomerates which do not properly absorb the binding liquid. The total addition of the mineral component (gypsum) in this method was 500 g per 150 g of shavings.

Method 3 was characterised by the production of a complete pulp (with the addition of gypsum) already at the mixing stage in the mixer. However, the addition of dry gypsum during granulation alone also proved necessary to cover the surfaces of the forming agglomerates in order to prevent them from sticking together.

Method 4 is analogous to method 2 in the pulp preparation technique, but in this case a smaller amount of binding liquid was added at the pulp production stage, hence the pulp’s too low moisture content.

In Methods 2, 3 and 4, stable and non-sticky granules with a high grain content, above 10 mm, were obtained. A comparison of the results of sieve analysis of agglomerates from these three tests is presented in Figures 5.

In Method 2 of pulp granulation, it was ensured that the maximum static moisture ratio of the shavings was achieved before granulation, which gave much better results in the form of a stable agglomerate, which also retained its properties after drying. In this case the content of grains larger than 14 mm reached above 20%, and the non-granulated material (grains below 1 mm) did not exceed 2% by mass content. Figure 6 shows a deposit after granulation obtained by Method 2.

The above-mentioned problems do not occur if pre-moisturised shredded shavings in the form of a pulp are subjected to granulation.

Based on the tests, it can be concluded that the best quality granules are obtained from shavings previously mixed with a water glass solution. Too dry shavings do not granulate well. It is, therefore, recommended to first mix them with a water glass solution and then conduct granulation without squeezing excess solution, along with using gravitational drainage only.

The fact that some of the mineral additives (e.g. gypsum) used in the granulation methods proposed are, similar to shavings, waste products from industry is an additional advantage. The agglomerate formed in disc granulation is durable, mechanically stable, as well as easy to transport and store.

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References


