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# ANALYSIS OF THE PRODUCIBILITY OF MINERAL FERTILIZERS FROM **INDUSTRIAL WASTE**

Summary

Most industrial waste in the form of sludge materials or dusts characterised by a high content of potassium, phosphorus, calcium or sulphur can, upon proper pretreatment, become complete fertilizers for agriculture, vegetable farming or forestry. The Department of Manufacturing Systems of AGH carried out research tests on the dry granulation of selected waste. The first group of materials included post-filtration sludge materials from the chemical industry which contained significant amounts of magnesium and potassium compounds and did not contain harmful substances and heavy metals. The other waste was the FGD gypsum which is calcium sulphate dihydrate. The third material which underwent initial granulation tests consisted of ash which was a biomass combustion side product containing mainly calcium oxide and silica. The article shows test research results which prove that is possible to produce proper granules from selected waste.

**Key words:** industrial waste, dry granulation, mineral fertilizers

# ANALIZA MOŻLIWOŚCI WYTWARZANIA NAWOZÓW MINERALNYCH Z ODPADÓW PRZEMYSŁOWYCH

Streszczenie

Część odpadów przemysłowych w postaci szlamów lub pyłów, charakteryzujących się dużą zawartością potasu, fosforu, wapnia lub siarki, po odpowiednim przygotowaniu może stanowić pełnowartościowe nawozy dla rolnictwa, ogrodnictwa lub leśnictwa. W Katedrze Systemów Wytwarzania AGH przeprowadzono badania dwustopniowej granulacji wybranych odpadów. Pierwszą badaną grupą materiałów były szlamy pofiltracyjne z przemysłu chemicznego charakteryzujące się tym, że posiadają znaczne ilości związków magnezu oraz potasu i nie zawierają szkodliwych substancji i metali ciężkich. Drugą grupą odpadów był gips IOS stanowiący dwuwodny siarczan wapnia. Trzecim materiałem, który poddano wstępnym próbom granulowania był uboczny produkt spalania biomasy, jakim jest popiół zawierający przede wszystkim tlenek wapnia i krzemionkę. W artykule przedstawiono wyniki badań, które dowodzą możliwości wytwarzania odpowiedniego granulatu z wytypowanych odpadów.

Słowa kluczowe: odpady przemysłowe, granulacja dwustopniowa, nawozy mineralne

### 1. Introduction

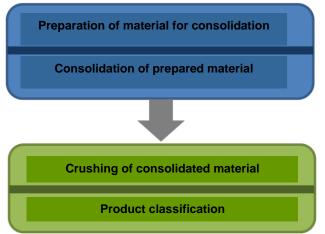
Various industrial processes produce waste in the form of sludge materials or dusts. These include, among others, post-filtration sludge materials, the FGD gypsum from fluegas desulphurisation systems, dusts from gas dedusting systems, ashes from the combustion of solid fuels, including biomass etc. Some of them are successfully reused after being properly pretreated. In Poland, there are possibilities of using them to a wider extent. This is exemplified by a group of waste materials which can become mineral fertilisers. This, however, requires preparing them properly by giving them a solid forms [6]. In this case, it is preferable to use the dry granulation method [3, 11]. From the point of view of the usability in agriculture, vegetable farming or forestry, the waste containing large quantities of magnesium, potassium, phosphorus, calcium as well as sulphur are particularly valuable. The article focuses on three kinds of waste materials, the composition of which shows that they might be used as mineral fertilisers.

The first group of materials includes post-filtration sludge materials from the chemical industry which contain significant amounts of magnesium and potassium compounds and do not contain harmful substances and heavy metals. The other waste was the FGD gypsum which is calcium sulphate dehydrate, whose purity often exceeds 95%. It has already been used in the American agricultural sector. Initially natural gypsum was used but its production and transport costs made the profitability of this undertaking questionable. As a result of the development of the flue desulphurisation method and its common use in various industry branches, cheap synthetic waste gypsum has become easily available. Its properties are often better from the point of view of the reasonability of using it in agriculture as compared to the natural material [12]. Therefore scientists carrying out research in the area of plant cultivation and farmers again focused their interest on the gypsum [10, 13]. It is one of the few raw materials which demonstrate positive effects in all three areas of the plant cultivation process: improvement, fertilisation and nutrition [9]. In Poland there is a shortage of knowledge about the usability of the FGD gypsum in agriculture and the benefits which result from it. Its advantages show that it might contribute to the improvement of the quality of soils in Poland. It requires carrying out specialised research tests domestically to prove this concept true. This is related to the need to properly prepare a specific quantity of the FGD gypsum. The third material which underwent initial granulation tests consisted of ash which was a biomass combustion side product. Due to the high content of calcium oxide up to approx. 35 %, it can be successfully used for soil deacidificaThis article presents the results of the granulation capability tests for 3 selected waste materials.

### 2. Description of Dry Granulation

The dry granulation is a process in which a loose material assumes the form of granules of a specific grain size [1, 2, 4, 5, 7, 11]. It is carried out in two operations consisting in:

- properly preparing a dusty material and then consolidating it under pressure,
- crushing the product of consolidation and classifying it.
  Fig. 1 presents a flowchart of a dry granulation of fine-grained material.



Source: own work / Źródło: opracowanie własne

Fig. 1. Diagram illustrating the dry granulation of loose material

Rys. 1. Schemat obrazujący przebieg dwustopniowej granulacji materiału sypkiego

The advantage of the dry granulation as compared to the conventional granulation consists in the possibility of using that method to consolidate not only dusts but also powders and fine-grained materials as well as their blends [1, 5, 7]. Furthermore, it is not necessary to use a significant amount of a moisturising and binding agent. Cases of making granules by dry method without using it are also known. The product of the dry granulation shows smaller moisture content and higher mechanical strength as compared to the conventional granules. It is a product of a wasteless process since the grains of a size different than required are reconsolidated. In Poland, the dry granulation method for the production of compound fertilizers is used [3, 4]. Its advantages were an inspiration to start the research on the usability of the dry granulation to process selected production waste materials which come from domestic plants and are suitable for use as mineral fertilizers.

## 3. Test Methods and Results

Selected waste materials were consolidated using the experimental system for the briquetting process tests, the diagram of which is presented on Fig. 2. It consists of, among others, a mixer with a heated jacket, a roll press with rolls with a diameter of 450 mm and stations for determining the quality of briquettes. The following were selected for the tests: post-filtration sludge from chemical plants, the FGD gypsum from a power plant and ashes from the com-

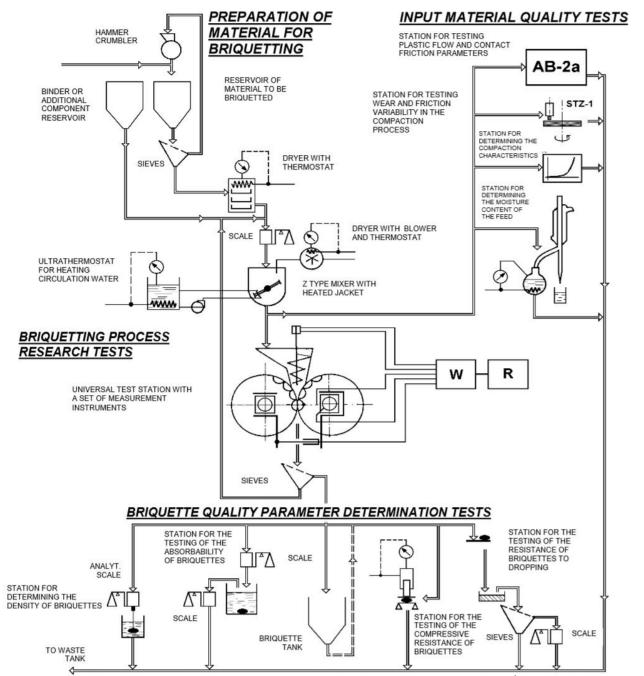
bustion of biomass for energy production purposes. There were 2 kinds of post-filtration sludge selected and marked with the symbols "PS1" and "PS2".

The sludge materials were prepared for briquetting by being dried to a specific moisture content and accurately averaged. To do that, a Z type mixer with a heated jacket was used. It was also used to change the moisture content of the FGD gypsum in order to determine the range of its favourable values. It was also used to prepare the ash. The preparation consisted in adding a specified amount of water to it and spreading it carefully. After being properly pretreated, the materials were fed into the compaction zone of a laboratory roll press which was equipped with a gravity feeder. The post-filtration sludge materials and the FGD gypsum were briquetted with the use of moulding elements used to make briquettes in the shape of a saddle with a volume of approx. 6.5 cm<sup>3</sup> [2]. Their model is presented on Fig. 3b. In case of ashes, a different set of working elements was used. They make it possible to obtain briquettes of a similar shape but with a smaller volume reaching 4 cm<sup>3</sup> (Fig. 3a). The tests were carried out with a value of the circumferential speed of rolls of 0.15 m·s<sup>-1</sup> while maintaining the width of the gap between them of a  $\approx 1.5$  mm.

It was decided that the measure of quality of the briquettes would be their resistance to dropping and their compressive strength. The resistance of the briquettes to dropping was tested by simultaneously dropping 9 briquettes from a height of 2 m onto a 120 mm thick steel plate. The test was repeated three times and then the briquettes were sieved through a 18x18 mm sieve. The sieve hole size was selected so as to ensure that was about 2/3 of the average calculated based on the two largest briquette sizes. The resistance of the briquettes to dropping is represented by the share of the sifting in the weight of 9 briquettes expressed as a percentage. The average drop resistance of briquettes of 3 tests has been determined to be representative. On the other hand, the compressive strength is actually the force destroying a briquette and it is determined in course of a monoaxial compression test performed between parallel flat surfaces. The average of 9 tests has been determined to be a representative force destroying a briquette.

The consolidated materials were crushed in a hammer crusher with the use of a clearance grate 5 mm. The type of the crusher and the value of the perimeter speed of hammers  $v_k=7\ m\cdot s^{\text{-}1}$  was established experimentally. The crushed briquettes were sieved to select 2.0-5.0 mm class grain which was recognised to be the final product.

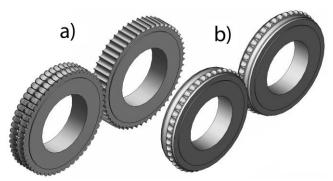
The post-filtration sludge dry granulation research tests were started from determining the limit moisture content of the material identified with the symbol "PS1". Based on the initial test results, it was determined that it should not exceed 16%. Otherwise, the material adheres to the working surface of the moulding elements of the press. This makes it difficult for the briquettes to fall out of the cavities. While continuing the tests, it was found that the material dried up to the moisture content level within the range of 13.7-15.9% can be processed in a roll press to produce briquettes of high mechanical resistance. Reducing the "PS1" sludge moisture within the given range causes a significant increase of the compressive strength of the briquettes which is illustrated on Fig. 4. However, their resistance to dropping continues to be about 100 % which means that it is maximum for the adopted criterion.



Source: own work / Źródło: opracowanie własne

Fig. 2. Diagram of the experimental system for fine-grained material briquetting process tests

Rys. 2. Schemat obrazujący przebieg dwustopniowej granulacji materiału sypkiego



Source: own work / Źródło: opracowanie własne

Fig. 3. Models of the LPW 450 press forming elements for making briquettes in a form of a saddle with a volume of: a) 4 cm<sup>3</sup>, b) 6.5 cm<sup>3</sup>

Rys. 3. Modele elementów formujących prasy LPW 450 do wytwarzania brykietów w kształcie siodła o objętości: a) 4 cm³, b) 6,5 cm³

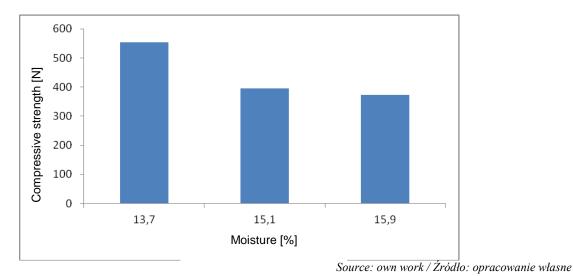


Fig. 4. Impact of the material moisture on the compressive strength of the briquettes made of post-filtration sludge "SP1" Rys. 4. Wpływ wilgotności materiału na wytrzymalość na ściskanie brykietów ze szlamu pofiltracyjnego "SP1"

The appropriate mechanical resistance of the "PS1" sludge briquettes makes them suitable for crushing immediately after production. The biggest yield of the demanded 2.0 – 5.0 mm grain size, reaching 70%, was obtained as a result of crushing briquettes made of the "PS1" sludge with a moisture of 13,7%. The phenomenon of spontaneous granulating of post-filtration sludge in the drying process is worth noting. The performed tests show that the material so consolidated is also crushed in a hammer crusher and the yield of the desired grain class reaches 46%. In both cases, the sub-grain formed in the drying process was successfully returned to the post-filtration sludge while it was being prepared for briquetting.

Positive results were also obtained for the "PS2" sludge. Based on the analysis of the research results, the required relative moisture of this materials was determined to range from 19 to 27%. When the moisture content was lower, the briquettes easily crushed and changed into rubble. On the other hand, excessive moisture made the briquettes remain in the moulding cavities which had been observed earlier during the "PS1" sludge briquetting process test runs. Considering the elastic nature of the newly formed briquettes, they should be crushed after at least 24 hours following their production. Adding the return materials in the amount of 20% to the moist sludge has a positive effect on the mechanical resistance of the briquettes. These were the briquettes which, while being crushed, provided the highest yield of the demanded 2.0-5.0 mm grain size which reached 73%. In both analysed cases, granules were obtained granules dissolved in water.

The FGD gypsum briquetting tests were started from consolidating it in such a form in which it had been supplied. The average relative moisture of this waste material was approx. 6%. The briquettes showed insufficient resistance to dropping immediately after being made. As time went by, it significantly improved. After 48 hours of being seasoned in a roofed room, the resistance of the briquettes to dropping exceeded 90 %. The density of the briquettes was 1.96 g·cm³ which, in turn, ensured their low absorbability which did not exceed 4.1%. While continuing the tests, the FGD gypsum of a lower and a higher moisture content was briquetted. In both cases, the quality of the briquettes deteriorated which contributed to the formation of

rumble. The preferable range of the waste gypsum moisture content which guaranteed a sufficient resistance of briquettes after being seasoned was estimated to be 5-7%. The briquettes were successfully crushed to produce granules.

The ash used for tests and produced as a result of biomass combustion had a density of 2,3 g·cm<sup>3</sup> and featured approx. 0.3% moisture and a grain size below 256 µm [8]. Distilled water was used as a binder. After properly mixing the dusts and water, they were briquetted. Also, their consolidation was tested twice to render the operational conditions of a roll press equipped with a screw feeder. In both cases, positive results were obtained. After 24 hours of being seasoned, the briquettes underwent a crushing test in a hammer crusher. The granules obtained in this way show high mechanical strength and resistance to dissolution in water. This is caused by the formation of strong pozzolanic bonds resulting from the presence of calcium oxide and silica in the ash. Research is currently carried out to find a way to weaken these bonds and improve the solubility of the granules in water. Their results show that this problem can be solved.

#### 4. Summary

The article focused on the laboratory research on the possibility to produce 2-5 mm grain size granules from waste materials containing chemical compounds which can be the source of nutritional components for plants. The dry granulation of properly prepared post-filtration sludge from chemical plants, the FGD gypsum from power plants and ashes from the combustion of biomass for energy purposes gave positive results. In practice, their use must be preceded by specialised tests performed by duly authorised units. To apply for a permit to place a new fertilizer on the market, it is required to present:

- results of tests and opinions about the fulfilment of quality and contamination related requirements,
- report on agricultural research tests and the opinion on suitability.

Based on one's own experiences, it is possible to make assumptions and produce test batches of fertilizers necessary to carry out relevant tests.

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