Research on the properties and assessing vulnerabilities of CaF_2 to briquetting in a roll press

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Introduction

Providing a consolidated form for powdery and fine-grained materials in many cases enables their further utilization in various processes – also those which are realized in their original technologies. However, compacting and then compressing such materials, carried out intentionally with the use of suitable processes and equipment is not always possible. An important, and in many cases even decisive role in the assessment of the possibility of providing a consolidated form to fine-grained materials and of applying (or not) a selected agglomeration technology is played by the properties of these materials. They may be generally characterized using two notions: compressibility and compatibility [1÷5]. The first is explained as the ability of a grain bed to decrease its volume as a result or pressure caused by e.g. deadweight or external forces. The second notion should be understood as the ability of the grain centre to create, on its own or under the influence of external factors, a durable consolidated structure with a required shape and size and with desired quality indicators determined by e.g. its mechanical strength. Taking into account a specific process and device, one may e.g. mention the susceptibility of the grain material to the briquetting process (briquettibility) in a roll, ring or stamp press, etc. An integral part of this notion includes also obtaining the required agglomerate quality [4] which is specified by conducting e.g. a compressive strength test, drop strength test, abrasive strength test, etc.

Four basic factors are taken into account when assessing the material's compressibility and compatibility: material properties, the manner of its preparing for agglomeration, the adopted compaction method and the type of equipment in which this process will be realized [4].

Among the physical values which characterize the material and which have a direct impact on its behaviour during compression and compaction, the external and internal friction factors are taken into account as well as the radial pressure factor, grain composition, size, shape and grain hardness [4,5]. When assessing the susceptibility of fine-grained materials to consolidation, compaction characteristics are also taken into account [2,4,6]. It is assumed that it takes into account, to a suitable degree, all the above-mentioned material properties (also the unknown ones) which decide about the material's capability to create a consolidated structure. Its nature, the maximum obtained material compaction degree, as well as so-called specific work (surface area under the compaction curve) decide about the material's susceptibility to the agglomeration process.

In the case of many fine-grained materials (e.g. powdery and fine-grained industrial waste), often the necessity occurs to improve their qualities (susceptibility to compaction and consolidation) in order to be able to use a suitable agglomeration technology. In order to obtain e.g. the required material flow in dosing equipment, to obtain high process output or a specified agglomerate shape and size, measures are undertaken to change material properties by e.g. changing its grain composition, moisture, applying a binder or changing the process temperature [4].

Aim and scope of work

This article presents selected results of testing properties of calcium fluoride in order to determine its susceptibility to compaction and consolidation in a roll press.

Tests were carried out in which the following was determined:

- Qualitative and quantitative nature of changes of static and kinetic external friction factors in the compaction process
- Compaction characteristics of CaF_2
- Plastic flow parameters (cohesion, kinetic and effective internal fraction angle, resistance to uniaxial compression, flow function) for five levels of consolidating tension.

Experimental verification of the susceptibility of CaF_2 to briquetting was carried out in a laboratory roll press and some of its results were also presented and discussed in this article.

Methodology of tests

An assessment of susceptibility to consolidation in a roll press was performed on a discarded fine-grained calcium fluoride which may constitute a substitute for fluorite natural for metallurgy. At the stage of initial tests, a modification of its properties was performed when necessary. In order to improve susceptibility to briquetting in a roll press, favourable moisture of feed and grain size composition were sought as well as suitable components and/or binder were added. Next, mixing was performed in order to unify the composition and properties. Burnt lime, constituting a discard from dust collectors, was also used as an additional component.

Compaction characteristics were determined on a testing machine with 0 – 100 kN pressure. The rate of stamp movement during tests equalled 5 mm/min. A closed die with a 30-mm diameter was used for tests. The amount of sample material was selected in such a way so as to obtain briquette (in the form of a roller), whose height will constitute approximately 2/3 of the die's diameter. The volume of such briquette from the closed die corresponds in such a case to the volume of briquettes with a separation surface obtained in an LPW-450 laboratory roll press using a classic shape of forming grooves [4, 7].

Plastic flow parameters were set experimentally according to a modified procedure and in a Jenike shear apparatus. These parameters, among other things, impact the free flow of the feed in dosing and transportation equipment [9] and, as a consequence, on the even filling of the roll press's forming rings and the proper course of the agglomeration process. Direct shearing of samples was performed employing multiple shearing [8] which uses all the advantages of the Jenike procedure and, at the same time, considerably increases the speed of the process for determining the sought parameters. In the multiple shearing method, the initial plasticization curve $YL = YL(\rho_i)$ (yield locus) is determined by performing shearing only on one sample while the Jenike method requires an amount of samples corresponding to the number of measurement points which determine the YL curve (approx. 4 ÷ 5 samples). The multiple shearing method does not allow for (by stopping the shifting of the ring) the creation of a deformation of the tested sample and prevents the decrease of its strength; thus, subsequent measurements may be performed for lower normal pressures (Fig. 1). In the case of the traditional Jenike method, the process of sample shearing, with a set normal pressure, is conducted until its complete destruction (shearing). Measurements at lower normal pressures require further material samples and, most importantly, achieving in each case the same output condition of the sample after consolidation as in the first sample. The shearing force value in the function of ring shifting of the Jenike apparatus was registered on an XY/t recorder.

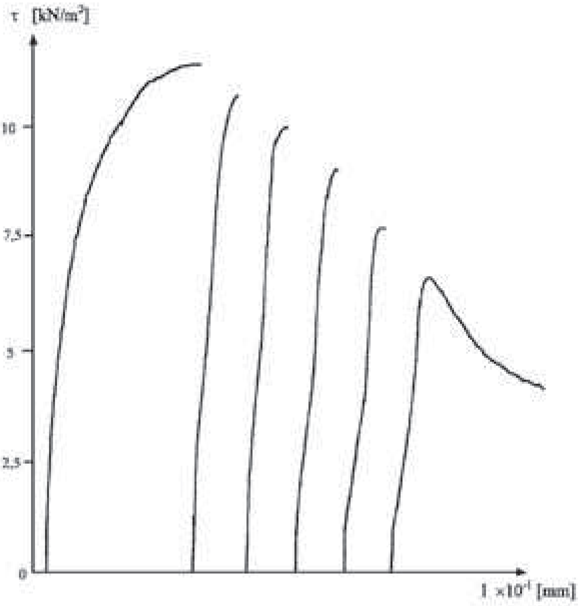

Fig. 1. Graphs of shear CaF_2 by using the modified procedure in the Jenike shear apparatus

For the purpose of assessing the free gravitational discharge ability of CaF_2 and, in consequence, the even filling of grooves of the press's forming elements, the flow index ff_c proposed by Jenike was used. This index expresses the dependence of the material's limiting strength (resistance to uniaxial compression) f_c on the value of the largest consolidating tension σ_1. Based on the values of this index it is possible to classify fine-grained materials in terms of their flow capability in the following way:

	ff_c	<2	– strongly cohesive, not flowing,
2≤	ff_c	<4	– cohesive, flowing difficult,
4≤	ff_c	<10	– weakly cohesive, flowing,
10≤	ff_c		– incohesive, freely flowing

Determining the qualitative and quantitative nature of changes of the static and kinetic external friction factor of the steel – consolidated material frictional pair in the compaction process was carried out on a specially designed and constructed workstation at the AGH laboratory. It enables a relatively precise recreation of surface phenomena which occur during briquetting in a roll press at the point of contact between the powdery centre and the forming tool [4]. A sample of the consolidated material was placed in a closed die with a 30-mm diameter and its amount was selected experimentally according to the rule presented above. A pressure of 0 – 100 kN was applied to the stamp, which allowed for conducting the test with unit pressures from the scope of 0 – 140 MPa. The relative speed value was set at 0.02 m/s. It was assumed that the scope of changes of the actual unit pressure and the relative speed value should correspond, with certain proximity, to conditions of the briquetting process in the roll press. The counter sample of the frictional pair was made from NC4 steel and was then thermally processed to a hardness of 55HRC.

The experimental verification of the susceptibility to consolidation of calcium fluoride in a roll press was conducted using an experimental installation for compacting and consolidating grain materials, which is located at the AGH Department of Manufacturing Systems. The main element of the installation is a proprietary laboratory roll press marked with the LPW-450 symbol. The press is fitted with a set of exchangeable forming tools which include rings with a 450-mm diameter and 62-mm working width. The rings have different groove shapes on the working surface. Thanks to this, the agglomeration process may be conducted in a symmetrical and asymmetrical compacting system [4,7]. The peripheral speed of rings may be regulated within the scope of 0.05 – 0.6 m/s. The hydraulic support system for the press's slidable roll ensures a unit pressure of 30 kN/cm. The gap between rings assumes values within the scope of 1-6 mm. The press may work with various dosing subassemblies (feeders), i.e. gravitational, worm and tapered-channel ones. Their task is to feed, in a gravitational or forced way, the required amount of material into the compaction area of the roll press.

The mechanical strength of obtained briquettes was determined in a test specified as drop strength test because it best simulates the conditions of reloading as well as transportation which briquettes may be subject to. A triple drop was performed of randomly selected 10 briquettes from each sample, from the height of 2 m onto a steel slab. Next, the crushed mass was sifted through a sieve with a mesh size of 18x18 mm. The size of the sieve was selected so that it would constitute 2/3 of the average value calculated from two maximum briquette sizes, measured in mutual perpendicular directions. The drop strength of briquettes was determined based on the following formula:

$$K = \frac{m_z}{m} \cdot 100 \quad \%$$

where:
K – drop strength of briquettes
m_Z – briquette mass after drop
m – briquette mass before drop

Test results

Some of the determined compaction characteristics of CaF_2 are presented in Figure 2. The level of compaction was specified as the quotient of the material sample's volume before compaction and the compacted material's volume at a given measuring point, i.e. for the given unit pressure.

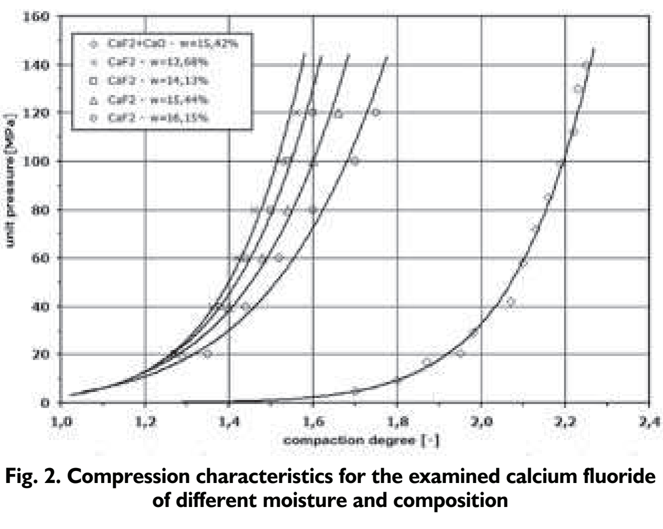

Fig. 2. Compression characteristics for the examined calcium fluoride of different moisture and composition

The maximum compaction level constitutes the quotient of the initial sample's volume and the volume of the obtained briquette; for the tested samples, it falls within the range of 1.55 - 2.25. In the case of calcium fluoride compacted without an additional component it was stated that together with the increase of water content in the sample, compaction curves shift in the direction of higher compaction levels. After adding CaO as component, the initial characteristics segment inclined at a small angle with regard to the level was extended. It is characterized by a quick increment of the compaction level to a value of approximately 1.8, with a relatively small pressure (<10MPa). Such behaviour of material points to the shifting and filling (packing) of free spaces between grains, with a simultaneous possible crushing of those characterized by smaller strength. Crushing may also occur in places of grain contact where the exceeding of allowed unit pressures occurred. Each of the set compaction characteristics has a progressive shape. This leads to the conclusion that a positive effect of CaF_2 compaction and consolidation in roll presses is possible. On the other hand, the high degree of compaction and the initial long segment of density increment with small pressures suggest that in the case of calcium fluoride with an addition of CaO there is a justified assumption that, in order to obtain a suitable quality briquette from this mixture in the roll press, it will be necessary to apply an asymmetric compaction unit and/or initial compaction, e.g. in a worm feeder.

The discharge calcium fluoride has a tendency to create congestions and bridges and to deposit in handling or dosing devices. Its limited capability of free flowing in these devices indicated possible problems in supplying the required amount of material to the press's compaction area and proper filling of the forming rings' grooves. Because of this, it was decided to change its properties. The 0-3 mm fraction was separated from the CaF_2 composition and CaO was used as an additional component. Adding a suitable amount of this component led to the binding of water surplus contained in CaF_2, thanks to which the desired mixture moisture was obtained and the costly drying process was eliminated. A graphic representation of test results of plastic flow and contact friction parameters for such a mixture, with an average humidity equal to 15.42%, was presented in Figure 3. The flow index $ff_c < 2$ (1.53 and 1.58) confirms that the material belongs to the highly cohesive, non-flowing group.

Test results of the changeability of the static and kinetic external friction factor of the steel - fine-grained CaF_2 frictional pairs, with a moisture w = 14.2% in the scope of unit pressures equal to 0 – 70 MPa, are presented in Figure 4. The obtained results lead to the conclusion that, together with the increase of unit pressure, the value of measured friction factors increases, obtaining at 60 MPA the level of 0.5 for the static and 0.45 for the kinetic external friction. Such behaviour of calcium fluoride suggests possible difficulties during briquetting in a roll press.

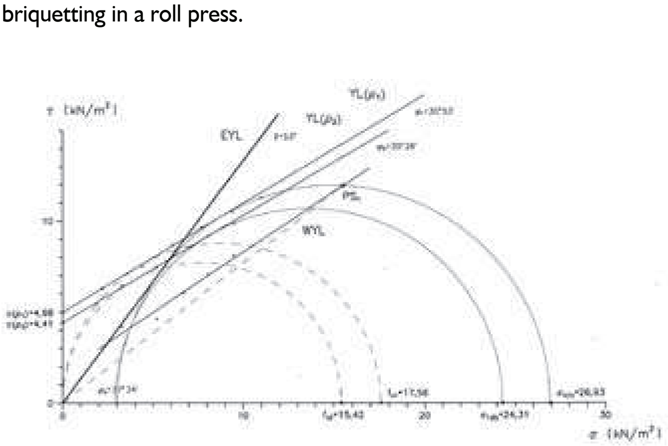

Fig. 3. Graphical comparison of test results for parameters of plastic flow and contact friction of calcium fluoride

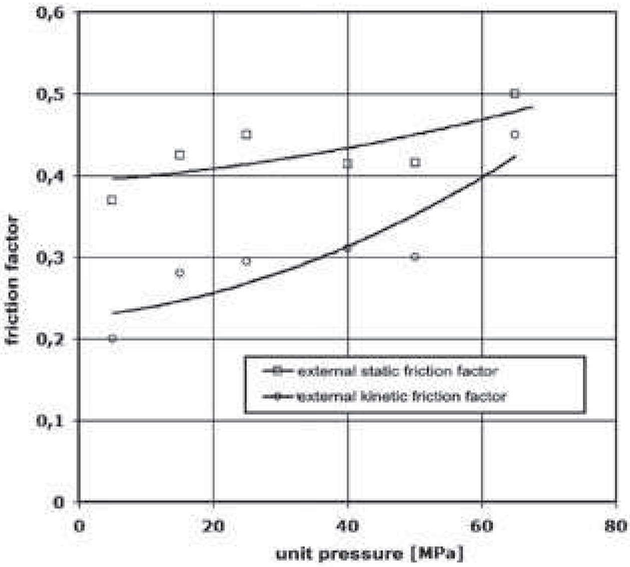

Fig. 4. Influence of unit pressure during briquetting CaF_2 with a moisture content 14.2% on the value coefficients of static and kinetic external

An experimental verification of CaF_2 compaction and consolidation was performed in a laboratory roll press at different configurations of the briquetting machine's compaction area. Due to its properties, calcium fluoride turned out to be a difficult material for briquetting in a roll press. The application of a gravitational dosing container placed under the press's compaction area led to the hampering of the feed flow because of the creation of bridges and congestions. The use of a classical compaction system, i.e. forming rings with grooves allowing for obtaining briquettes with a separation surface, also created many difficulties. During briquetting it was observed that part of the briquettes (briquette halves) remains in forming grooves, undergoing separation in the separation surface. The remaining part of briquettes, even if it left the groove, underwent disintegration in this surface after dropping on the grate below the press's consolidation area. Undoubtedly, CaF_2 properties influence such behaviour of the agglomerate. Especially the value of the external friction kinetic factor, increasing together with unit pressure, hampers the proper leaving of forming grooves by briquettes, causing their disintegration. The bad quality of obtained briquettes influences the decision regarding the change of the type and shape of forming grooves. An asymmetrical compaction system was used with grooves and without a saddle-shaped separation surface [4,7]. This way, the main area and place of unfavourable phenomena causing a destructive effect on the created briquette were eliminated. Because of the change of the working surface of the press's forming rings, no phenomena were observed as in the case of briquettes with a separation surface. With a peripheral speed of rings equal to 0.1 m/s, briquettes were obtained with a density of 1.912 g/cm^3 and drop strength of 86%. The average value of registered unit pressures in the forming groove equalled 78 MPa. Further improvement of briquette quality was obtained by dosing feed into the compaction area and consolidating press with an initial, forced compaction in tapered channels. In this case, with the same peripheral speed of rings as previously, the values of recorded parameters and the briquette quality increased considerably. This time, the unit pressure value in the forming groove equalled 102 MPa, briquettes had a density of 2.151 g/cm^3 and their drop strength reached the level of 97%.

Conclusions

Calcium fluoride turned out to be difficult to briquette in a roll press. The narrow scope of humidity favourable for briquetting, the tendency to suspend and create bridges in dosing containers, as well as the necessity to apply an asymmetrical compaction system and forced dosing into the press's consolidation area cause the material to be weakly susceptible to agglomeration in a roll press, despite positive test results. The main conclusions drawn from the conducted tests may be formulated the following way:

The progressive shape of compaction characteristics confirmed the possibility of briquetting calcium fluoride in a roll press and provided a general outlook on its behaviour during this process, making it possible to assess the required unit pressure needed to obtain a specific compaction degree and a durable briquette.

Values of the static and kinetic external friction factor, growing together with the increase of unit pressure, require the use of a suitable configuration of the press's compaction area, i.e. an asymmetrical compaction and/or dosing system through tapered channels or in a different, forced way in order to obtain durable briquettes from CaF_2 in a roll press.

Testing the plastic flow parameters of calcium fluoride makes it possible to determine the right geometry of the charging container, thanks to which the material dosed into the press's consolidation area fills out the forming grooves in the required amount, allowing for obtaining a durable briquette with high mechanical strength.

Currently preparations are made for testing briquetting with the use of a new shape of the forming rings' working surface. The advantage of the new solution, which is the subject of a patent application, is the intensive and even charge of the feed into the compaction area as well as forming briquettes in favourable conditions. This will contribute to unifying the compaction of briquettes, at the same time causing the increase of their mechanical strength.

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