

Article

Disc Granulation Process of Carbonation Lime Mud as a Method of Post-Production Waste Management

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Abstract: Carbonation lime mud is a by-product formed during the production of sugar in the process of raw beetroot juice purification. On average, during one campaign, over 12,000 tons of carbonation lime mud is obtained in the operation of one sugar production plant. It is stored in prisms, which negatively affects the environment. The chemical properties of carbonation lime mud allow using it as a soil improver. This article presents the results of research into the development of carbonation lime mud disposal technology and its management. The chemical composition and physical properties of waste were determined. It has been proposed to use carbonation lime mud as the basic raw material in the production of mineral–organic fertilizers. Tests were conducted in a disc granulator. The granulated material was wetted with water and aqueous solution of molasses. Carbonation lime mud is a material that is easily subjected to the granulation process, using any wetting liquid. The beds wetted with 33% and 66% solutions of molasses are characterized by a greater homogeneity and smaller size of the obtained product. During experiments in which wetting with water was applied, the product obtained after drying demonstrated low resistance to compression; granules wetted with 33% aqueous solution of molasses demonstrated resistance to compression below 10 N; and granules wetted with 66% aqueous solution of molasses demonstrated resistance to compression above 10 N.

Keywords: carbonation lime mud; disc granulation; mineral–organic fertilizers; waste as fertilizers

1. Introduction

Carbonation lime mud—also called defecosaturation mud—is the by-product formed during the production of sugar in the process of raw beet juice purification.

The process of defecation occurs during liming and saturation of the solution of sucrose with carbon dioxide. In the presence of calcium, gelatinous complexes of calcium saccharate-carbonate are then formed in the form of poorly soluble gels. During saturation, the precipitation of calcium carbonate occurs. It is absorbed by colloidal impurities (non-sugars). During this process, the precipitation of insoluble calcium salts, organic acids, and non-organic pectic substances, and their passage to the deposit occurs. The main component of carbonation lime mud is CaCO_3 , whose share reaches up to 50% [1].

On average, in the operation of one sugar production plant during one campaign, over 12,000 tons of waste carbonation lime mud. It is stored in prisms, which negatively affects the environment.

Carbonation lime mud usually occurs in fine-grained form, forming bigger agglomerates [2,3]. There are basically two possible ways of handling these residues: landfill or recycling as a secondary material [4]. The literature indicates that food waste can be considered as a promising substrate e.g., for bioethanol production [5,6]. Ozkan et al. [7] showed the bio-hydrogen generation potential of sugar industry wastes. Kirby et al. [8] developed the anaerobic digestion of pig carcase with or without sugar beet pulp as a novel on-farm disposal method. Kantiranis [9] explored the possibility of recycling sugar ash (a material rich in calcium carbonate that is produced as a by-product in the Greek sugar industry) for use in the lime industry. In addition, the bioconversion of sugar with other biodegradables into enriched compost [10] and co-ensiling as a technique for the long-term storage of agro-industrial waste with low sugar content prior to anaerobic digestion [11] are an important possibilities in need of research.

The chemical properties of carbonation lime mud allow using it as a soil improver. Thus, adjusting the soil pH value is possible and it reduce acidity. Carbonation lime mud can be a starting material applied in the production of calcium or multi-component fertilizers. Obtained analyses concerning the content of nitrogen, phosphorus, sodium, and magnesium confirm this possibility [12–15]. The chemical composition of mud can be its advantage. The possibility of applying carbonation lime mud as agricultural fertilizer is known for decades; however, it did not receive wide recognition among potential recipients due to the unfavorable physical properties of unprocessed carbonation lime mud. They make transport, storing, and distribution to soil, including uniform dosing, difficult. The solution to the problem related to the unfavorable mechanical properties of mud in the powdered form is its granulation. It is possible to obtain mineral or mineral–organic fertilizer with good practical qualities as a result of this process.

Techniques of pressure [16–18] and non-pressure granulation [19,20] are often applied to process post-production wastes. In the case of fine-grained materials processing, non-pressure granulation, which involves a dumping movement of the bed combining dusts and powders into several millimeter-wide agglomerates, is most often used. The process of granulation of fly ash (e.g., from a combustion of hard coal) [21,22], Municipal Solid Waste Incineration (MSWI) bottom ash, and Air Pollution Control (APC) fly ash or sugarcane bagasse fly ash [23] may significantly reduce the consequences of its storage and transport that are harmful to the environment, mainly secondary dusting.

A factor initiating the granulation process is the provision of combining liquid, most often water or aqueous solutions of various compounds, to the bed. The process can be realized in drum [24–27], disc [28–31], mixing [32], vibrating [33,34] granulators and in multi-stage technologies [35]. Prior to granulation, the initial mixing of dry material is performed in order to obtain the homogeneous distribution of all components. The process comprises the subsequent stages: wetting, nucleation, consolidation, growth, wear, and crushing [36]. Methods of granulated mineral calcium fertilizers' production—among others, from dolomite and limestone flours, chalk, gypsum and mixtures of the above-mentioned components in disc and drum granulators with the use of water or aqueous solutions of molasses and sugars—are well described [37]. These methods described above can be implemented also for the granulation of different kind of biomass such as straws from maize, oat, energy crops such as *Miscanthus*, or woody biomass carbonized to biocarbons in a torrefaction process using superheated steam. Then, these are used as carriers for organic fertilizers production use with mixtures of the above-mentioned components in disc and drum granulators with the use of water or aqueous solutions of molasses and sugars, which can be found in [38–44].

2. Materials and Methods

2.1. Testing Properties and Chemical Composition of Carbonation Lime Mud

Analyses of carbonation lime mud included the determination of particle size distribution (granulometric analysis) conducted by means of a set of sieves and a KAMIKA Instruments AWK 3D

analyzer operating in the infrared spectrum. The obtained results of bulk density, angle of natural repose, and humidity of defecosaturator mud are presented in Table 1.

Table 1. Characteristics of carbonated lime mud.

Property	Value
Range of changes in grain size (mm)	0–0.56
Average grain size (mm)	0.12
Tangent of the angle of natural repose	1.34
Bulk density (kg/m ³)	1131
Humidity	38%

In addition, elemental composition occurring in the raw material was examined. The content of separate elements was analyzed using atomic absorption spectroscopy (AAS). The determination was performed in accordance with PN-ISO-8288:2002. A sample weighing about 100 mg was wet mineralized in the closed system in an Anton Paar Multiwave 3000 V 2.02 microwave extractor. An examination procedure, the so-called blank test, was performed for each series of mineralization. The mineralization of each sample was performed in two repeats. The concentration of metals was determined using atomic absorption spectroscopy (AAS) and electrothermal atomic absorption spectroscopy (ET-AAS) on a Perkin-Elmer 3110 spectrometer equipped with a graphite tray of the HGA 600 type (USA). The chemical composition of carbonation lime mud obtained as a result of the above tests is presented in Table 2.

Table 2. The chemical composition of carbonation lime mud.

Element	Content	Unit
Zinc	15.3	mg/kg dry matter
Nickel	0.89	mg/kg dry matter
Lead	0.19	mg/kg dry matter
Cadmium	0.06	mg/kg dry matter
Copper	4.6	mg/kg dry matter
Magnesium	18.6	mg/kg dry matter
Mercury	0.003	mg/kg dry matter
Chromium	4.11	mg/kg dry matter
Boron	0.03	mg/kg dry matter
Potassium	0.06	% dry matter
Phosphorus	0.15	% dry matter
Calcium	45.10	% dry matter

The obtained test results indicate the possibility of applying mud to produce mineral or organic–mineral fertilizers. The content of heavy metals does not exceed requirements specified in law regulations (the Ordinance of the Minister of Economy on the method of packing mineral fertilizers, listing of fertilizer components on these packages, method of mineral fertilizers' testing, and types of fertilizer calcium (Journal of Laws of 2010, No. 183, item 1229), n.d., the Ordinance of the Minister of Agriculture and Rural Development of 18 June 2008, Journal of Laws, No. 119, item 76 concerning admissible concentrations of heavy metals, n.d., the Act on fertilizers and fertilization of 10 July 2007, Journal of Laws No. 147 item. 1033, n.d.), and additionally, this mud is rich in elements typical of mineral fertilizers commonly used in the agricultural sector.

Despite the beneficial chemical composition, the practical qualities of the material need to be modified—in particular, its granulometric composition—in order to eliminate dusting and agglomeration. They are unfavorable phenomena occurring during transport, storing, and dosing fertilizers to soil. To eliminate the mentioned usability faults, the fertilizer's granulometric composition was initially determined at 1–10 mm. This corresponds to the commercial characteristics of other

agricultural fertilizers. After initial tests, the optimum size fraction of 2–6 mm was assumed. The value of force applied to fertilizer's granules without destroying their structure, for the assumed size, should be no less than 10 N.

2.2. Testing Properties and Chemical Composition of Carbonation Lime Mud

The main part of the granulator is a rotating disc with a diameter $d = 0.5$ m and height of the rim $h = 0.08$ m. The granulator disc is mounted to a shaft and connected to the electric motor by means of belt transmission and a regulator. The rotational speed is determined and controlled by means of an inverter. Wetting liquid is provided from the container mounted at the height of 2.5 m through a hydraulic sprinkler, and its flow intensity is measured by a rotameter.

Carbonation lime mud obtained from waste dumps, due to high initial humidity and the phenomenon of agglomerating, required initial screening. The material was screened on sieves with a mesh size of 1 mm. The granulation process was conducted in the disc granulator Figures 1 and 2 in a periodic way. Each time 1 kg of screened material (carbonation lime mud or the mixture of mud and gypsum) was placed on the disc, then the disc was set in rotational motion so that the material circulated freely inside the disc rotating at the speed of 9.5 rpm. Directly after the commencement of the process, the material was wetted with wetting liquid. At first, water was used for wetting, while in the next trials, solutions of molasses with concentrations of 33% and 66% were applied as well. The material was wetted by the droplet method in the case of trials to which water was added or by continuous stream when an aqueous solution of molasses was added. The wetting time t_h was changed within the range 4–8 min. During wetting, about 200 mL of liquid was provided to the bed each time. This liquid was necessary to initiate the formation of granules. Due to the high initial humidity of the material and over-wetting of the bed at the end of the process necessary in the case of such a specific raw material, the formed granules were powdered by various loose materials, the use of which prevented the agglomeration of granules. Moreover, the additional benefit was that the mineral components contained in the powder improved the quality of the final product (fertilizer) and improved its diversity. Raw materials commonly used in the fertilizer industry—that is, dolomite, chalk, limestone flour, and gypsum—were used as powder. In addition, trials in which no powder was applied and when the over-wetted bed was powdered with carbonation lime mud were performed. In the case of trials with powdering, the total process time t_g depending on the type of liquid used for wetting changed within the range 6–30 min. Granulation was run until the majority of the fine-grained material attached to granules, making sure that the agglomeration of granules did not occur (no coalescence phenomenon), which would cause the excessive increase in the size of formed agglomerates.

After the completion of granulation, the obtained product was weighed and then dried at temperature 96 °C for 24 h. After the drying process, the granules in Figure 3 were weighed again, and then measurements of the particle size distribution, humidity, angle of natural repose, and bulk density of the product were conducted, and the resistance to the compression of selected size fractions was determined. The granulated product was screened using sieves with mesh sizes: 12.5; 10.0; 8.0; 6.3; 5.0; 4.0; 3.0; 2.0; and 1.0 mm, which enabled the determination of the mass share of separate fractions in the obtained granulate. Table 3 presents parameters of the conducted trials of carbonation lime mud granulation.

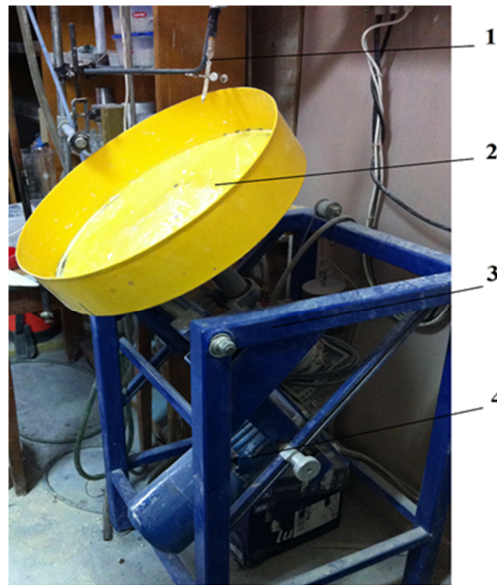


Figure 1. Disc granulator—test stand. 1—Moisturizing system, 2—granulation plate, 3—supporting structure, 4—drive system.

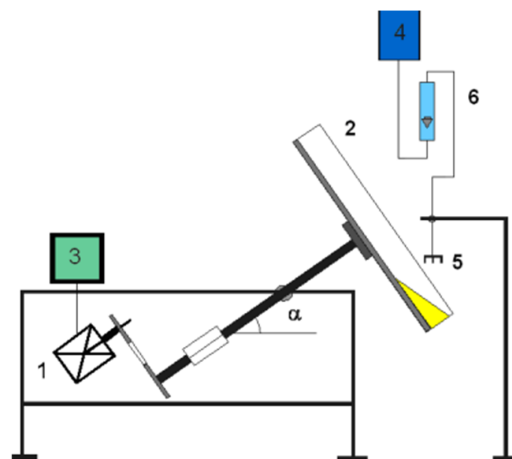


Figure 2. Diagram of apparatus for disc granulation tests: 1. geared motor, 2. granulation plate, 3. inverter, 4. tank for binding liquid, 5. sprinkler, 6. rotameter.



Figure 3. Granulated post-saturation mud.

Table 3. A description of the process parameters for carbonation lime mud granulation.

Trial Number	Raw Material	Wetting Time t_n (min)	Granulation Time t_g (min)	Material Used for Powdering	Type of Wetting Liquid
1	mud	6.0	30.0	gypsum	water
2	mud	4.0	30.0	gypsum	water
3	mud	8.0	17.0	-	water
4	mud, gypsum	6.0	15.0	gypsum	water
5	mud, gypsum	5.0	11.0	gypsum	water
6	mud, gypsum	4.0	8.5	-	water
7	mud	5.0	8.0	gypsum	water
8	mud	5.0	10.0	gypsum	water
9	mud	5.0	8.0	dolomite	water
10	mud	5.5	8.0	dolomite	water
11	mud	6.0	15.0	chalk	water
12	mud	6.0	14.0	chalk	water
13	mud	5.5	12.0	limestone flour	water
14	mud	6.0	12.0	limestone flour	water
15	mud	6.0	12.0	dry mud	water
16	mud	7.0	12.0	dry mud	water
17	mud	7.5	10.0	gypsum	33% solution of molasses
18	mud	7.5	10.0	dolomite	33% solution of molasses
19	mud	5.0	12.0	chalk	33% solution of molasses
20	mud	5.0	10.0	limestone flour	33% solution of molasses
21	mud	7.5	15.0	-	33% solution of molasses
22	mud	3.0	12.0	gypsum	66% solution of molasses
23	mud	4.0	12.0	dolomite	66% solution of molasses
24	mud	4.0	12.0	chalk	66% solution of molasses
25	mud	4.0	10.0	limestone flour	66% solution of molasses
26	mud	3.5	6.0	mud	66% solution of molasses
27	mud	4	6.0	mud	66% solution of molasses
28	mud	4.5	6.0	mud	66% solution of molasses

3. Results and Discussion

Figures 4 and 5 present the exemplary comparison of mass shares of the obtained size fractions.

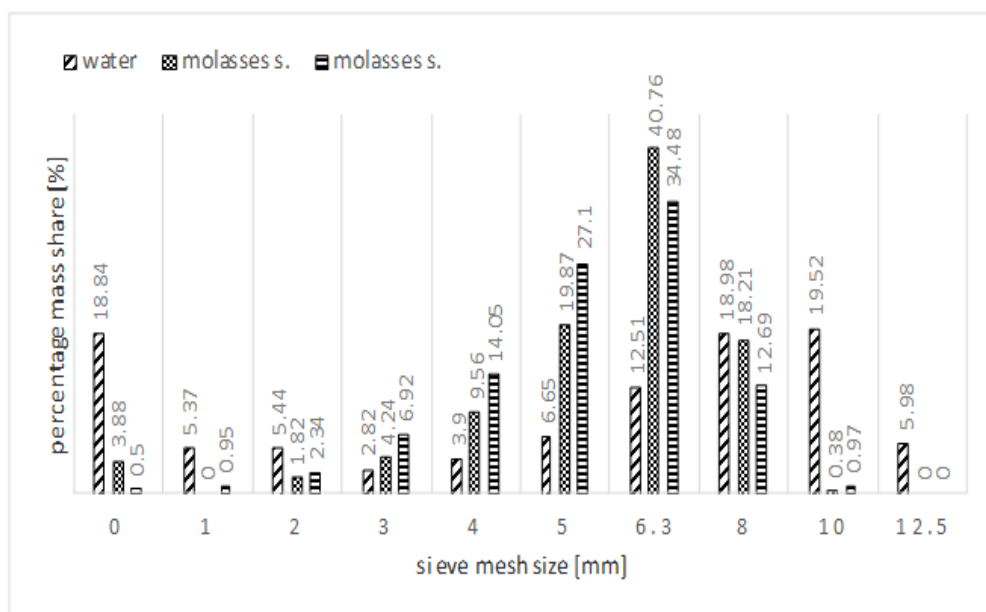


Figure 4. The comparison of mass shares for individual size fractions. Experiments with additional limestone flour powdering.

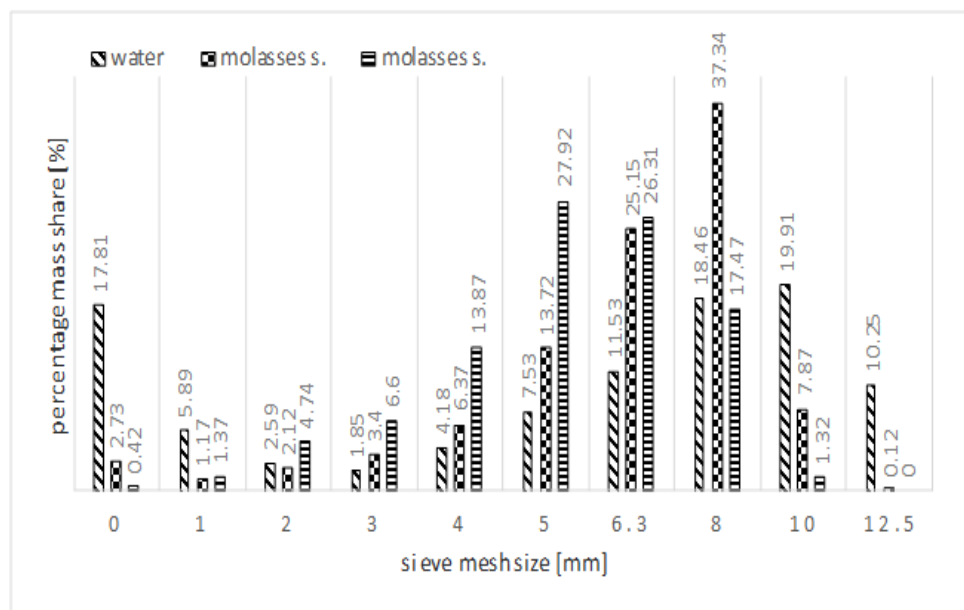


Figure 5. The comparison of mass shares for individual size fractions. Experiments with additional dolomite powdering.

The presented results concern trials of carbonation lime mud granulation in which water, 33% solution of molasses, and 66% solution of molasses were applied as wetting liquid. In all trials, whose results are compared in Figures 4 and 5, the final powdering of the over-wetted granulate by means of fine-grained limestone flour (Figure 4) and dolomite (Figure 5) was applied.

The analysis of the obtained results indicates that the most beneficial sizes of granulated fertilizer are obtained during trials with wetting the bed with 66% aqueous solution of molasses.

The product obtained for these trials is characterized by granulometric composition with particles slightly smaller than during wetting with water and 33% solution of molasses, which is more beneficial during dosing fertilizer to soil. During trials with 66% solution, more than 99.5% of the material mass was granulated, which indicates on one hand better process conditions and on the other the less wear of agglomerates formed earlier. The product's resistance to wear is of major importance during transport, when fertilizer granules slightly change their mutual location, causing friction between their external surfaces. A parameter that is characteristic of the strength properties of the obtained granulate is its compressive strength. This property is very important during storing fertilizer in bags or 1-ton packages, in which it presses on granules that are at the bottom of the package. Initial experiments demonstrated that granules obtained during wetting with water break down under small load; therefore, tests of resistance to compression were conducted only for trials performed during the wetting with solutions of molasses.

The analysis concerning the value of compressive force at which the destruction of granules occurred was conducted for agglomerates with sizes of 4, 5, 6.3, 8, and 10 mm. Tests were conducted on the Instron analyzer—Figure 6, which measured the value of force in the function of the displacement of a head that compressed a granule. The maximum value of destructive force is illustrated in graphs by the local minima of the presented graphic dependencies in relation to the initial value. Each time the resistance of five granules from each size class was examined, and then the arithmetic mean was calculated. The exemplary results of measurements of resistance to compression for granules obtained during granulation with wetting with solutions of molasses are presented in Sections 3 and 3. Graphic dependencies present the value of compressive force at which the breaking down of the examined granule occurs (minimum in the graph). In each figure, the results of compression for five randomly selected granules from a given size fraction are presented. The negative value in the graph for the compressive load results from the assumption adopted by the calculation program in which tension

forces are defined as positive and compressive forces are defined as negative. Figure ?? presents exemplary results of strength tests conducted for granules with sizes 4–5 mm, concerning trials in which gypsum was used for powdering over-wetted agglomerates. It can be seen on their basis that the sufficient resistance of granules to compression (about 20 N) is obtained during granulation with the use of 66% solution of molasses. Analogous results (Figure ??) were obtained for granulates powdered with dolomite. It can be assumed that resistance to compressive force is more affected by the concentration of the used solution than by the type of material used for powdering the granulated bed.

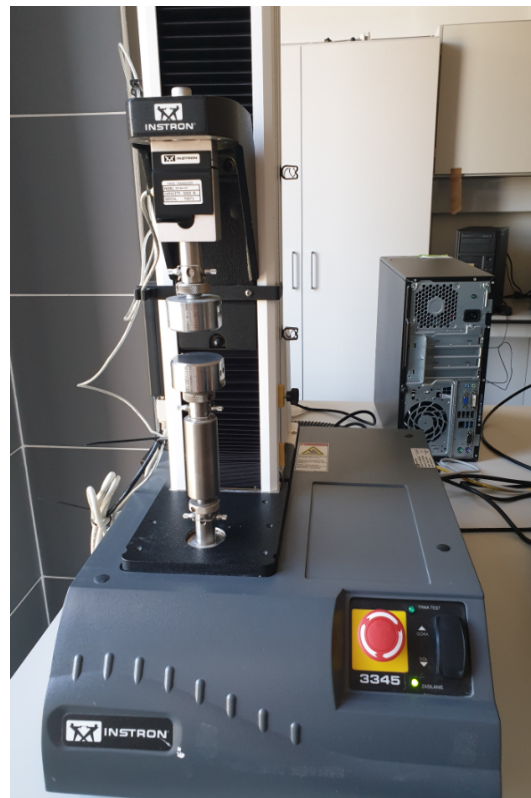


Figure 6. The comparison of mass shares for individual size fractions. Experiments with additional dolomite powdering.

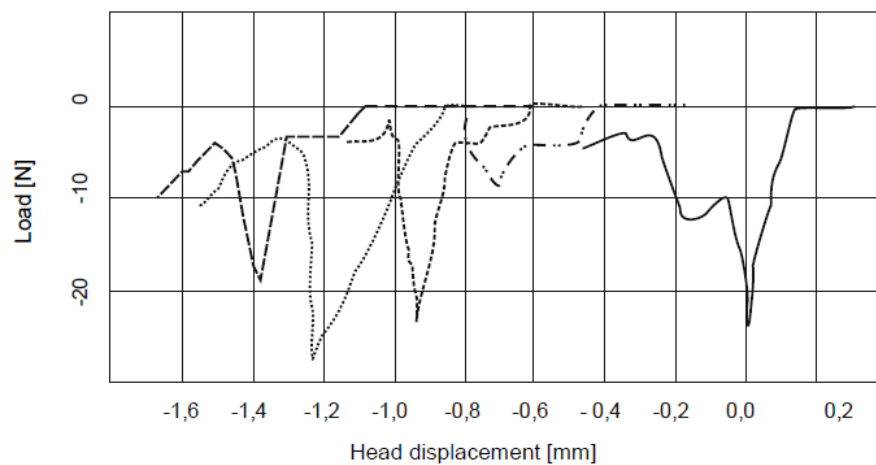


Figure 7. Destructive forces applied to granules formed with 66% solution of molasses, gypsum powdering.

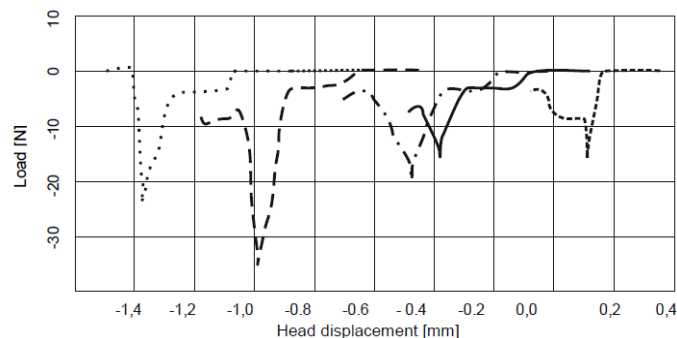


Figure 8. Destructive forces applied to granules formed with 66% solution of molasses, dolomite powdering.

The results of tests concerning the strength of granules Table 4 confirm that granulate obtained during wetting with 66% aqueous solution of molasses meets the strength requirements. However, the technology of disc granulation of carbonation lime mud requires further optimization, which on one hand would concern obtaining desired results with using solutions with less concentration of molasses—lower production costs—and on the other, expanding the commercial offer concerning the final product.

Table 4. The average values of loads destroying granules in individual size fractions.

Trial Number	Material Used for Powdering	Liquid Used for Wetting	Fraction Size (mm)	Average Force Destroying Granules (N)
18	gypsum	33% solution of molasses	4.0	4
			5.0	6
			6.3	4
			8.0	6
			10.0	7
19	dolomite	33% solution of molasses	4.0	4
			5.0	8
			6.3	5
			8.0	9
			10.0	8
20	chalk	33% solution of molasses	4.0	4
			5.0	3
			6.3	5
			8.0	8
			10.0	12
21	limestone flour	33% solution of molasses	4.0	3
			5.0	4
			6.3	4
			8.0	5
			10.0	8
22	-	33% solution of molasses	4.0	4
			5.0	4
			6.3	4
			8.0	4
			10.0	8
23	gypsum	66% solution of molasses	4.0	9
			5.0	11
			6.3	25
			8.0	26
			10.0	25
24	dolomite	66% solution of molasses	4.0	13
			5.0	16
			6.3	21
			8.0	24
			10.0	46
25	chalk	66% solution of molasses	4.0	15
			5.0	13
			6.3	29
			8.0	42
			10.0	12

Table 4. Cont.

Trial Number	Material Used for Powdering	Liquid Used for Wetting	Fraction Size (mm)	Average Force Destroying Granules (N)
26	limestone flour	66% solution of molasses	4.0	11
			5.0	18
			6.3	20
			8.0	28
			10.0	27
27	carbonation lime mud	66% solution of molasses	4.0	11
			5.0	12
			6.3	17
			8.0	22
			10.0	34
28	carbonation lime mud	66% solution of molasses	4.0	11
			5.0	11
			6.3	13
			8.0	14
			10.0	37

4. Conclusions

Carbonation lime mud granulation is a beneficial technology of post-production waste management. Carbonation lime mud due to the content of lime in its composition and due to the low level of heavy metals' concentration is a promising raw material in the production of agricultural fertilizers.

Mud granulation enables obtaining mineral-organic fertilizer with the desired mechanical properties and beneficial chemical composition, meeting the commercial requirements. The proposed granulation technology expanded by the stage of powdering enables enriching fertilizer with other mineral components. Carbonation lime mud is a material that is easily subjected to granulation with the use of any wetting liquid. The bed wetted with 33% and 66% solutions of molasses is characterized by the greater homogeneity and smaller size of the obtained product.

Based on the conducted strength trials, it can be concluded that during experiments in which wetting with water was applied, the product obtained after drying demonstrated low resistance to compression; granules wetted with 33% aqueous solution of molasses demonstrated resistance to compression below 10 N; and granules wetted with 66% aqueous solution of molasses demonstrated resistance to compression above 10 N. Granulation changes the starting material with a grain size of 0–0.5 mm (with a predominance of fine fractions) into a bed with agglomerate sizes in the vast majority above 1 mm. Due to the fact that this waste (mud) is stored in heaps, dusting occurs on windy and dry days of the tiniest fractions. After granulating, such material, due to its chemical composition and the use of additives, can be a soil de-acidifying fertilizer—it can be sold. Molasses solution (also waste) is successfully used as a binding liquid. So, instead of ecological problems with dusty waste, you can generate profits from the sale of fertilizer, the basic components of which are sugar waste. In addition, in one of the variants, sulfogypsum is also used as an additive—which is also waste from flue gas desulfurization. The idea is dedicated to sugar factories that can buy a “know how” and start producing fertilizer instead of paying environmental fees and fines.

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