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ScienceDirect
Procedia Engineering 136 (2016) 269 – 274
Procedia Engineering
www.elsevier.com/locate/procedia

The 20th International Conference: Machine Modeling and Simulations, MMS 2015

Influence of the value of limit densification stress on the quality of the pellets during the agglomeration process of CO_2

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Abstract

The article presents the results of static compression tests of concentrated crystallized carbon dioxide. Included results of test describe relation between the agglomerating stress and density of compressed dry ice. Quality of dry ice pellet is determined by value of density. Results of research allow to determine the effective value of stress during the agglomeration process. Due to the fact that the crystallized carbon dioxide has temperature equal minus 78.5 °C and sublimates under normal conditions of the test was proposed the methodology which allowing performance the tests. In first stage of research has been determined value of bulk density of dry ice snow. This value is initial boundary condition of relation between compression stress and density of agglomerated material. In second stage of the research has been investigated influence of compression stress on to density variation of the agglomerated dry ice. Results of both stage of research allow for formulation of function which approximate relation between density of agglomerated dry ice and compression stress. Results will allow for formulation of assumptions, which will be one of main point to create a more efficient machine for agglomeration process of solidified carbon dioxide.

Peer-review under responsibility of the organizing committee of MMS 2015

Keywords: solidified carbon dioxide; dry ice; agglomeration; design of machines; bulk density; density

1. Introduction

Waste materials obtained during production process can be applied again as a raw material. One of such type of materials is carbon dioxide – a waste material obtained during production of ammonia compounds. Percentage fraction of carbon dioxide emission during this manufacturing process is the highest one in comparison with the

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1877-7058
Peer-review under responsibility of the organizing committee of MMS 2015
doi:10.1016/j.proeng.2016.01.209

other emission sources (Fig. 1). This material can be reclaimed by condensation. It is stored for further applications in a liquid phase. Due to large quantities of this waste material the plants have a problem with waste management. Therefore, carbon dioxide is delivered to the recipients in a liquid phase.

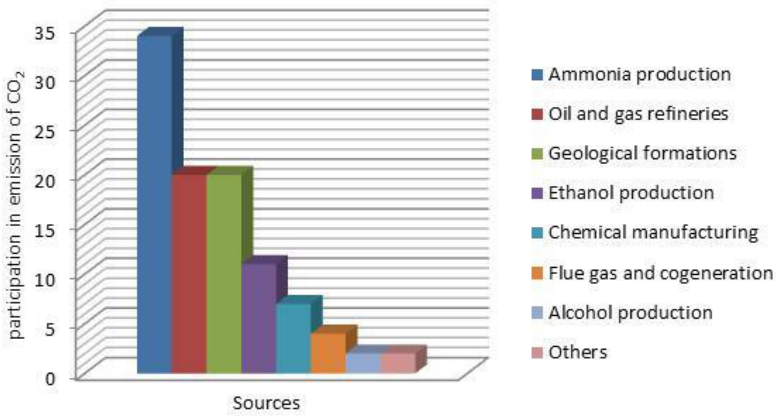


Fig. 1. Percentage fraction of particular sources in emission of carbon dioxide [1].

Liquefied carbon dioxide is subjected to crystallization when it is expended. The obtained product has a temperature -78.5 °C and it sublimates in environmental conditions. Due to the specific properties it is called as dry ice. Both low temperature and sublimation in standard conditions are the main reasons of application of dry ice in food industry (freeze drying, cold room inserts for keeping the products cool etc.).

Agglomeration of crystallized carbon dioxide is done in order to reduce its volume and prolong the time of sublimation. As an effect one can get a consolidated material with higher density than input material. Due to the influence between the material density and its gradient of sublimation it is accepted that the material density is one of basic criteria of quality assessment of process product.

During the review of the scientific literature no information on the description of the relationship between variation of material density and compressive stresses was found. The presented investigations are the supplement of insufficient information on the properties of condensed and crystallized carbon dioxide.

2. The investigations methodology

The investigations on the influence of threshold stress values in agglomeration process on the density of pellets have been divided into two stages i.e. the investigations on bulk density in loose state and investigations on the variation of the agglomerate density in function of compressive stresses.

During first stage the bulk density of dry ice in loose and disintegrated state was elaborated by empirical method. This material was obtained as a result of expansion of liquid carbon dioxide to atmospheric pressure in chamber. The investigation was performed in accordance with standard PN EN 1097-3 concerning the investigation of mechanical and physical properties of aggregates.

A special measuring cylinder with chamber diameter (D) 100 mm and measuring height (h) 127.3 mm was made for the investigations (Fig. 2). This cylinder satisfied the standard requirements for measuring volume of the cylinder (V_C) which is equal 1 dm^3. Measuring chamber of the cylinder satisfied the criterion for diameter-height ratio which should be between 0.5 and 0.8. To reduce the disproportion between the sample mass and cylinder mass, a non-metal material was applied for the cylinder. Thermal conductivity coefficient was taken as a decisive parameter due to the low temperature of the material. Finally, the cylinder was made of PLA.

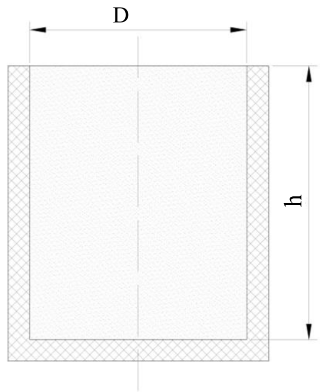


Fig. 2. Measuring cylinder for the measurement of bulk density.

During the investigations the cylinder was poured by freely falling material from conical channel with outlet diameter 5 mm. Pouring process was done until the moment of filling up the cylinder and getting the pile cone on the upper surface of the cylinder. Next the material above the upper edge of the cylinder was removed by a rigid slat. The filled cylinder was weighed with the application of laboratory balance Kern model PCB10000-1. On the basis of the mass measurement and calculated volume of measuring chamber the bulk density was calculated with the following formula [2]:

$$\rho_b = \frac{m_2 - m_1}{V_c} \quad (\mathrm{kg.\,m^{-3}}) \tag{1}$$

where: ρ_b – bulk density in loose state, m_1 – mass of the measuring cylinder, m_2 – mass of the measuring cylinder and sample, V_C – volume of the measuring chamber.

16 denotations were done for the investigation. Estimated value of the bulk density in loose state was calculated by averaging of the results from 10 denotations and eliminating 6 extreme result values, i.e. the highest and lowest ones. Table 1 presents the measurement results.

Table 1. Measurement results of the bulk density.

No.	1	2	3	4	5	6	7	8	10	Avr
ρ_b (kg.m^{-3})	487.1	546.9	496.6	555.8	518.3	516.4	558.8	531.5	539.5	568.8

Estimated value of the bulk density of loose dry ice was equal 568.8(27.11) kg.m^{-3}.

Second stage of the investigations was based on the elaborated methodology [3, 4]. The main aim of the investigations was the determination of characteristics of variation of agglomerate density in function of compressive stresses. Physical quantities for this characteristics have a fundamental meaning during the assessment of the product quality of the agglomeration process. On the basis of data from literature [4] the boundary values have been defined for compressive forces (F_{max}) in range from 6 kN to 40 kN.

Measuring unit i.e. measuring head and testing machine MTS model Insight were applied during the investigations. This unit allowed to manufacture the samples of agglomerate with determined values of the axial forces. Fig. 3 presents the construction of the unit.

At the beginning of every test the compression chamber (1) was filled up with loose dry ice. Next the compressive punch (3) was put into the chamber. Measuring head was placed between the clamps of the testing machine (6) and these clamps were equipped with alignment unit (5). After taring the testing machine the punch was moved with a constant velocity and as a result of this process the pressured agglomeration of dry ice in working area of the head was done. During condensation the signals from the sensors were recorded. The experiment was done for the constant velocity of working point displacement, i.e. 8 mm.s^{-1}. The material was consolidated until the moment of occurrence of the force loading the compressive punch with defined value. After sample consolidation the average of the agglomerate density was calculated. The investigations were done for three repetitions for the same measurement parameters.

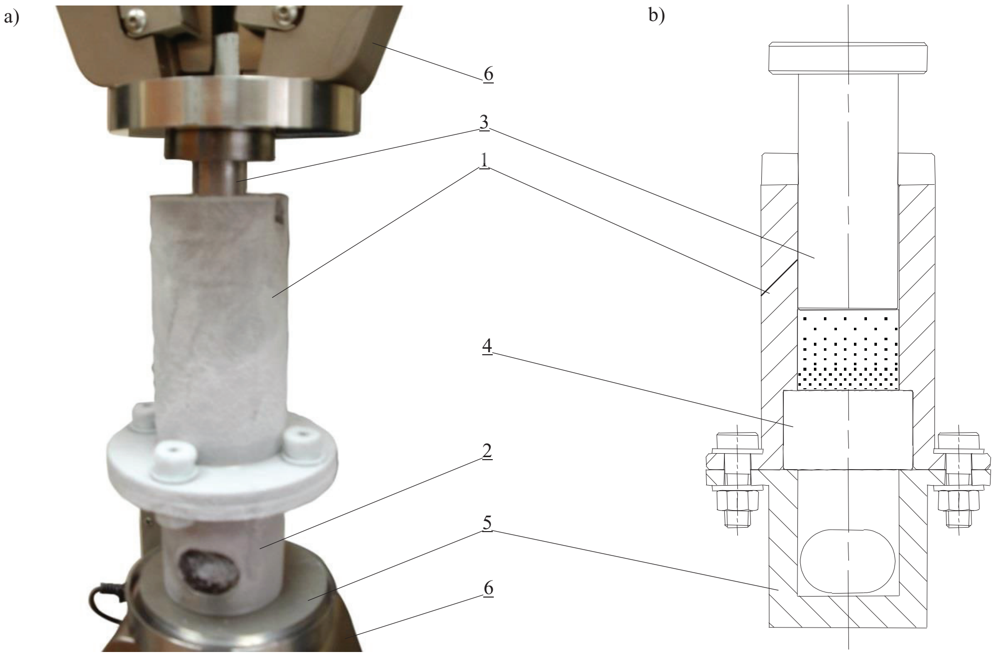


Fig. 3. Measuring unit: a) clamps of testing machine MTS, measuring head and alignment unit, b) cross-section of measuring head: 1 – compression chamber, 2 – head basis, 3 – compressive punch, 4 – closing plate of working area, 5 – alignment unit, 6 – clamps of testing machine.

3. The measurement results

On the basis of the measurement results obtained in second stage of the investigations the density of the consolidated samples with different threshold stresses was estimated. Average value was taken as a result estimator due to the fact that standard deviation did not exceed the value 76.7 kg.m^{-3}. Presented diagram shows the estimated value of the agglomerate density in function of boundary value of compressive stresses.

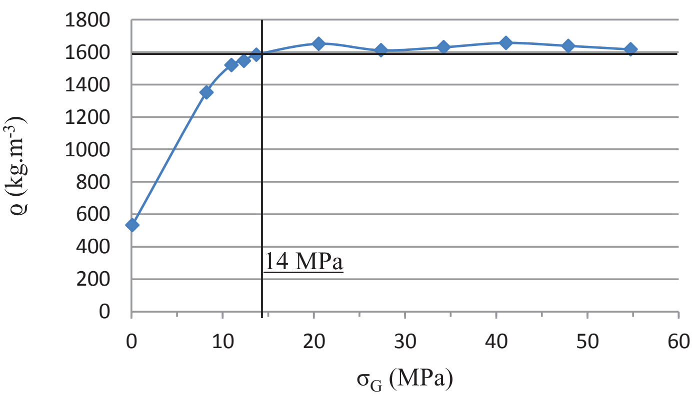


Fig. 4. Variation of agglomerate density in function of threshold stress.

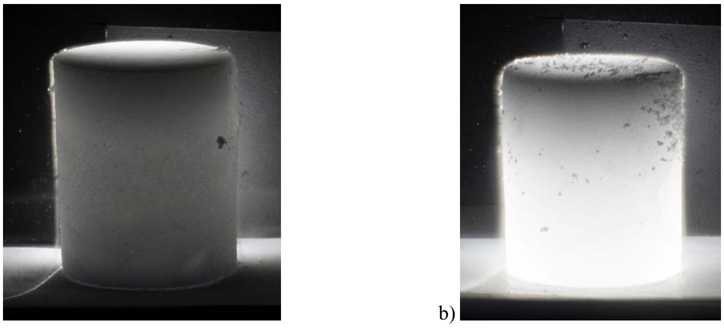
Limiting force F_{max} is a force which has a boundary value and occurs in consolidation process of the sample. Hence, it is possible to determine the equal value for threshold stress with the same denotation. The measurement results are presented on the diagram in relationship between compressive stresses and agglomerate density.

The increase of the agglomerate density is connected with the increase of threshold stresses until the moment of exceeding the threshold stress value 14 MPa. After reaching this value the density is constant, i.e. 1625 (±25) kg.m^{-3} (Fig. 4).

The correctness of the presented characteristics is confirmed by the characteristics of the consolidation of the crystallized carbon dioxide which has a progressive character. Shape of the characteristics is connected with the class of material – carbon dioxide agglomerate belongs to the class of brittle porous materials [3, 5, 6]. The higher the compressive stresses, the lower the volume of the agglomerated sample – this fact is connected with the decrease of percentage fraction of pores in material structure. The increase of the sample density has an effect on the change of the external surface state of the agglomerate – it is connected with the decrease of percentage fraction of pores in material structure.

Gradient of the agglomerate sublimation is connected with the size of the external surface. Thus, it is found that the correlation exists between the sublimation speed and density of the material

State of the external layer of the agglomerate is the main criterion which is applied during the organoleptic assessment of the pellet quality. Lower percentage fraction of the pores is the reason of the decrease of the reflection coefficient of the light wave – this fact can be seen in Fig. 5.



a) b)

Fig. 5. External surface of the sample of dry ice which was agglomerated with different threshold stresses: a) σ_G=11 MPa, b) σ_G=16 MPa.

4. Conclusion

On the basis of the conducted investigations one can formulate the following conclusions:

1. Characteristics of the density variation of the agglomerated material in function of threshold stresses is changed in a non-linear way – this fact is showed by the weak correlation of the results on the level of 0.3 [7].
2. Gradient value of density variation of the sample is equal to zero after exceeding the value of compressive stresses 14 MPa.

On the basis of information from literature and the investigation results we can formulate the following initial boundary condition for the designed machine matrix which can be applied for the agglomeration of the dry ice:

1. Geometrical parameters of the matrix for agglomeration of dry ice should be chosen so as to ensure the static resistance during forcing on the level of 14 MPa.
2. Boundary values of compressive stresses are not dependent on the area of cross-section of the agglomerate (pellet) and cylindrical channel. Hence, forcing resistance should be present due to selection of proper parameters of tapered channel [8].

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