The influence of the limit stress value on the sublimation rate during the dry ice densification process

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Abstract. The article presents the results of research on the influence of limit stress of the densification process on sublimation of dry ice during the forming process in piston technology. The research concerns the process of agglomeration of waste material in fragmented form, obtained as a result of the crystallization process of liquid carbon dioxide. The material is characterized by low temperature and sublimation under ambient conditions. During the research, the focus was on determining the influence of the limit value of densification stress on the value of the material efficiency factor of the process. In previous studies, it was observed that the weight of the product in the form of pellets is significantly smaller than the fragmented dry ice dispensed to the densification chamber. In order to improve the efficiency of the process, tests were carried out to determine the characteristics describing its change in function of the limit value of the densification stress.

1. Introduction

Today’s economy puts more emphasis on the management of waste material from manufacturing processes. One of such materials is carbon dioxide, which is generated in large amounts during the manufacturing of ammonia [1, 2]. However, due to the amount of recovered material, manufacturing plants cannot utilize it entirely. Therefore, it is compressed and liquefied to facilitate storage and transportation. The material is delivered to interested recipients in this form [3, 4].

Due to adiabatic transformation, the liquid material turns to solid. In this form, carbon dioxide is characterized by own temperature of -78.5 °C and sublimates under atmospheric conditions [5–9]. Due to such peculiar characteristics, the material received the common name of dry ice. It is widely employed in abrasive surface cleaning processes [10–14] and in transportation of materials in low temperatures [15, 16].

As a result of crystallization, a fragmented material undergoing intensive sublimation is obtained. In many circumstances, this prevents effective utilization. In order to slow down the sublimation process, the material is agglomerated to reduce the surface of the phase transition. It is possible by utilizing specialized machinery adapted to this task [5, 11].

Dry ice compaction is usually carried out using piston-based technique and single and multi-channel dies. An example working system of the compaction machine is illustrated in figure 1.
The working system comprises of the working chamber (1) inside which the moving compacting piston is placed (2). As a result of motion of the compacting piston at section length L the space taken up by compacted dry ice is reduced. Due to resistance to the forced movement related to the converging shape of the die (3) the material is compressed. The density of deposited material increases until it balances against the force applied to the piston \( F_T \) and resistance force \( F_{OP} \) resulting from forcing the material through the die channels. The limit density value of agglomerated dry ice should be equal to \( 1.6 \text{ g cm}^{-3} \), which is achieved at compaction stress of at least 14 MPa [11]. Higher compaction stress limit value does not affect the density value, therefore subject literature indicates that this is the effective limit value. The process, when implemented under industrial conditions, is characterized by material usage coefficient of approx. 0.5. It is interpreted as the product of weight of the final product and the raw material used in the manufacturing. This parameter value has a material influence on the cost-efficiency of the dry ice pellet manufacturing process, therefore there are devices available on the market used to increase it via repeated compression of CO\(_2\) in gaseous form. The final value of the effectiveness coefficient is influenced, among others, by the parameters of 2 processes performed in the machine, e.g. crystallization of dry ice and its densification.

Based on own studies, it was observed that machines employed in the described process often use dies and technological parameters which significantly exceed the effective limit value of yield stress. No information was found in subject literature regarding the influence of the limit value of densification stress on the efficiency parameter of the dry ice agglomeration process. The results of such studies can be employed for works related to the optimization of the process as well as the geometrical parameters of multi-channel dies.

Available subject literature demonstrates a high degree of interest in works aiming to study and develop knowledge about process parameters which are focused on the application of nonclassical materials and physical parameters of these materials [17–30].

2. The study methodology

Studies on the influence of limit value of densification stress were performed in accordance with the methodology designed in literature [11, 31]. To this end, a special testing station was employed as illustrated in figure 2.

The main goal of the examination was to determine the characteristic of the value change of the process material efficiency indicator \( \delta_m \) as a limit function of densification stress value \( \sigma_z \). To this end, the indicated methodology was supplemented to include dry ice weight measurement before \( m_0 \) and after \( m_1 \) the compaction process, which allows to determine the value of \( \delta_m \) using the formula:

\[
\delta_m = \frac{m_1}{m_0}
\] (1)

At the beginning of each test, a measured amount of fragmented dry ice \( m_0 \) was placed in the densification chamber of testing station 1. Next, the testing head with compression piston 3 was placed
between the grips 7 of the MTS Insight 50 kN durometer. After taring the durometer, the compacting piston 3 was moved at constant speed \(5 \text{ mm s}^{-1}\), and as a result the material was compressed until the value of force applied to the compacting piston \(F_T\) was balanced against the resistance force \(F_{OP}\) resulting from the geometrical parameters of the die and weight of the material \(m_0\) placed in the densification chamber 1. After the force values were balanced, the material was forced through the die channel 4. During compaction of the material, signals from force and displacement sensors were recorded at 100 Hz sampling frequency.

![Figure 2](image)

**Figure 2.** Measuring assembly: a) grips of durometer including the testing head and right angle jig, b) testing head cross-section; 1 – densification chamber, 2 – base, 3 – piston, 4 – multi-channel die, 5 – spacer ring, 6 – right angle jig, 7 – grips [3].

After compacting the material, measurement was taken for the compacted carbon dioxide in the chamber of the head base 2, the value was equal to the material weight after the compaction process \(m_f\).

The examination was performed for 3 multi-channel dies with different parameters, as provided in table 1 with key provided at figure 3.

|   | \(d\) (mm) | \(a\) (mm) | \(b\) (mm) | \(\alpha\) (°) | \(n\) |
|---|-----------|-----------|-----------|--------------|-----|
| MCD1 | 3         | 3         | 15        | 10           | 61  |
| MCD2 | 3         | 6         | 12        | 10           | 37  |
| MCD3 | 4.5       | 3         | 15        | 10           | 37  |
Figure 3. Geometrical parameters of the multi-channel die: a) front view of forming die layout, b) forming channel; 1 – conical section of the forming die, 2 – cylindrical section of the forming die, $\alpha$ – angle of convergence of the conical section, $l_1$ – length of the conical section, $d$ – diameter of the cylindrical section, $l_2$ – length of the cylindrical section [3].

In order to alter the value of densification stress $\sigma_z$ during individual examinations the weight of fragmented dry ice introduced into the densification chamber before compression $m_0$ was changed. Study results available in literature provide the dependence between the stress value and the volume of compacted dry ice in the densification chamber [32]. Based on the above, the described examination was carried out for 4 values of $m_0$, presented in table 2.

The examination was carried out in 3 repetitions of the same measuring parameters of each die.

Table 2. $m_0$ values during examination.

| No. | 1   | 2   | 3   | 4   |
|-----|-----|-----|-----|-----|
| $m_0$ (g) | 30  | 20  | 15  | 10  |

3. Research results

Based on measured results, the parameter $\delta_m$ value change was determined for different values of $\sigma_z$. Because of the fact that standard deviation value for the parameter $\delta_m$ did not exceed the value 0.038, it was assumed that the correct result estimator is the average value. For the purpose of approximation of the function describing the graph line of the studied characteristics, a linear function was used as the correlation coefficient value was not lower than 0.9, in every analyzed case.

The characteristics presented in figure 4 show the change in value $\delta_m^{AVR}$ as a function of limit value of densification stress $\sigma_z^{AVR}$, for 3 multi-channel dies used in the examination, where during the measurement of limit densification stress the initial weight of dry ice $m_0$ was changed.
The illustrated characteristics demonstrate that the average value of the $\delta_m^{AVR}$ coefficient increases linearly together with the increase in the value of limit densification stress as a result of the change in the dosed material in the densification chamber, regardless of the die type used in the examination.

Based on the data registered during the examination, the parameter to describe the variance in $\delta_m^{AVR}$ was established as a function of the value of limit densification stress (figure 5), which varied as a result of use of multi-channel dies with different geometrical properties.

The graph line of the examined characteristics indicates that the average value of the $\delta_m$ coefficient changes linearly together with the increase of the limit value of densification stress. This is caused by the use of dies with different value of resistance force $F_{OP}$, with the same dosed weight of material into the densification chamber of the testing station.

4. Conclusions
The results of the study provided above allow to formulate the following conclusions:

- the decrease in $m_0$ value causes a decrease in the value of $\delta_m$ coefficient, which is disadvantageous from the standpoint of efficiency of utilization of liquid CO$_2$;
- the decrease in limit densification stress $\sigma_z$, as a result of utilizing a die with suitable geometrical parameters causes an increase in the value of the $\delta_m$ coefficient, which is beneficial for the efficiency of utilization of liquid CO$_2$.
The analysis of the formulated conclusions indicates that the value of \( \delta_m \) coefficient can be increased as it is advantageous from the standpoint of efficient utilization of liquid \( \text{CO}_2 \) in the pellet manufacturing process. This requires designing multi-channel dies with geometrical parameters adapted to the parameters of the utilized dry ice agglomeration process. Furthermore, the conclusions indicate that it is not possible to propose a universal design of the multi-channel die which would be adapted to different geometrical parameters of the working systems and initial weight \( m_0 \) of the agglomerated dry ice.

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