

INFLUENCE OF DIE LENGTH ON EXTRUSION PRESSURE AND EXTRUDATE SURFACE QUALITY

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Abstract: The extrusion process is very popular in industry. Nowadays, extrusion is also closely associated with the 3D printing process, but this article is focused on extrusion, as a process that is mainly associated with spheronization. The extrusion product is shaped in such a way that it is suitable for the ongoing spheronization process. With this two-step extrusion-spheronization (E–S) process, it is possible to create pellets with controlled high sphericity, which are desired in tablet and capsule production due to their easily characterized (and controlled) dosage profile and good flow properties. During extrusion, it is important to monitor several parameters. This post is dedicated to one of them and that is the length of the die and its effect on the extrusion pressure as well as the effect on the extrudate surface quality.

KEYWORDS: extrusion, paste, die length, extrusion pressure

1 INTRODUCTION

The quality of the extrudate surface is one of the critical parameters for assessing the extrusion process. If the extrudate is the final product, e.g. for various ceramic products, the extrusion must be carried out in such a way as to achieve a smooth, crack-free surface. If the extrudate is an intermediate product, e.g. for spheronisation, the surface quality is not so critical, because the final product is created only in the spheronisation process and here the shape and surface quality of the granulate is adjusted. The quality of the product is therefore largely dependent on the value of the extrusion pressure and the geometry of the die holes. This paper focuses on the study of these parameters in an extruder of special design, which consists of a screw and a rotor with a special geometry. This configuration allows control of the extrusion pressure without the need to modify the rheological properties of the paste by the addition of various additives [1, 2].

2 THEORETICAL ANALYSIS

The effect of pressure on the extrusion process was already investigated in 1993 by Benbow & Bridgwater. They described the linear form for the total pressure decrease P in equation (1):

$$P = 2(\sigma_0 + \alpha V^m) \ln\left(\frac{D_0}{D}\right) + 4(\tau_0 + \beta V^n) \frac{L}{D} \quad (1)$$

Where the sum of $\sigma_0 + \alpha V$ is taken to be the interfacial tension of the wet powder material when V is the paste velocity; α is a factor characterizing the effect of velocity; and σ_0 is the interfacial tension extrapolated to zero velocity. Then the shear stresses τ_0 together with β

characterize the paste flow in die because β is the paste velocity factor at the wall and τ_0 is the wall shear stress.

The parameter m , the exponent of the bulk velocity, determines the dependence of the interfacial shear stress of the paste with respect to the flow velocity, n , the exponent of the paste velocity at the point of contact of the paste with the die surface, describes the effect of the shear stress in the paste at the wall on the paste flow velocity [3].

This Benbow & Bridgwater model - mentioned in more than 40 papers between 1994 and 2004 - relies primarily on its usefulness: so far, few of the six parameters appearing in the equation (τ_0 , β , n , and σ_0 , α , m) can be predicted a priori. It has been used to develop "pragmatic" models for paste extrusion in non-capillary geometries such as multi-hole dies [4], screw extruders [5], and cylindrical extrusion [6], where the accuracy reflects the uncertainty in the characterization and represents a useful tool for design calculations in the absence of reliable constitutive models [7].

Also, Fekete et al. [8] describe changes in extrusion pressure in an extruder of special design such that as rotor speed increases, the resulting pressure in the extruder decreases. They justify this by the change in the apparent viscosity of the paste, which is a function of the shear strain rate tensor, the components of which are affected just by the rotor effect.

2.1 Effect of die length on the extrusion process

During the extrusion process, imperfections in the quality of the extrudate may result, ranging from an uneven surface to a complete cracking of the extrudate. These defects dependent on factors such as the extruding material or process conditions. The ability to predict these defects is a crucial factor in modern extrusion processes. Although products with observable defects result in a loss of productivity, small or internal defects may also be potential sources of weakness in the final product. Fig. 1 shows two main types of surface cracks.

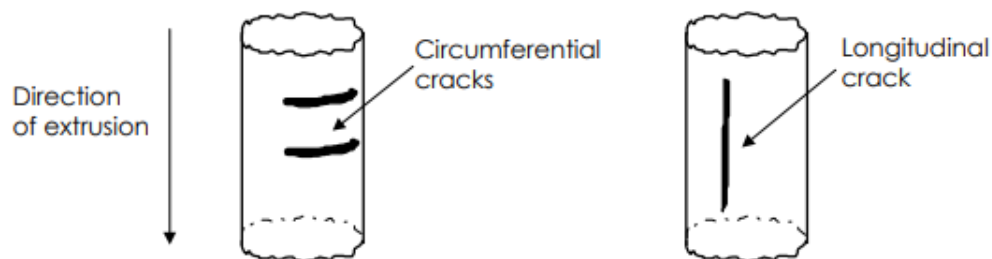


Fig. 1. Two main types of extrudates surface cracks. [5]

Domanti, in her work, has focused on this area. Her experimental results show that significant surface fracture was observed when extruding a matrix with $L/D = 1/6$ ratio. This was typical in that deep cracks often appeared along the extrudate surface. Extrusion at $L/D = 1/3$ ratio also resulted in surface fracture, although the fracture did not extend as deeply into the extrudate. Extrusion through dies with $L/D \geq 1$ ratio resulted in a smoother extrudate surface. Fig. 2 shows surface fracture typical of extrusion through dies with $L/D = 1/3$ ratio for die diameters of 3 mm, 6 mm and 9 mm, at extrusion speeds of $22 \text{ mm} \cdot \text{s}^{-1}$ [9].

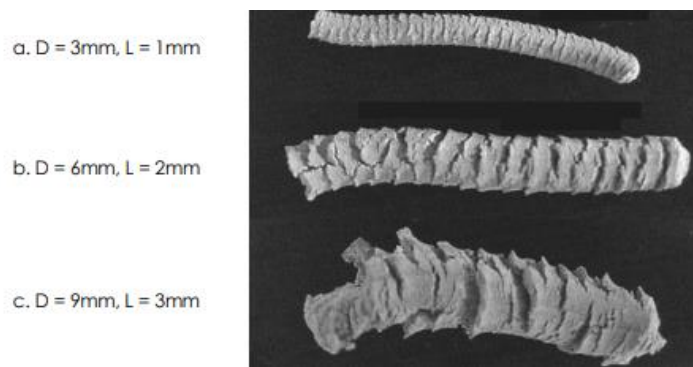
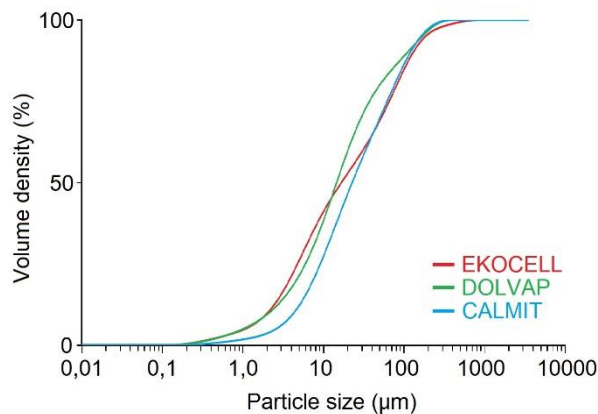


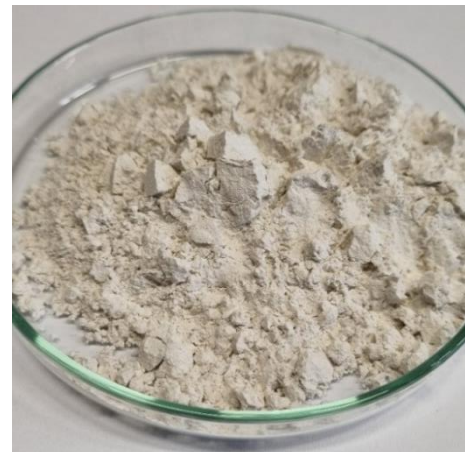
Fig. 2. Surface fracture during paste extrusion; extrusion speed = $22 \text{ mm} \cdot \text{s}^{-1}$ [10].

3 SELECTION OF EXPERIMENTAL MATERIAL

Extrusion uses particulate substances which, together with the liquid, form a paste that has suitable properties for the process. In our experiment, finely ground limestone was used. This material is readily available and hence economically viable. A choice of 3 types of available limestone was made. The first was limestone from EKOCEL, the second was from DOLVAP and the third was from CALMIT. On the basis of laboratory tests and measurements of material properties, we chose limestone from CALMIT. Their particle size distribution was measured on a Malvern Mastersizer 3000 using the Aero Series module and is shown in Fig. 3, along with a sample of the powder [11, 12, 13, 14].



a)



b)

Fig. 3. Particle size distribution of three different ground limestone samples.

a) comparison of the distribution curves for the 3 selected materials

b) selected suitable experimental material (CALMIT)

Table 1: Granulometric composition based on volumetric equivalent particle diameter.

	$d_{10} (\mu\text{m})$	$d_{50} (\mu\text{m})$	$d_{90} (\mu\text{m})$
EKOCEL	2,03	16,7	129
DOLVAP	2,13	14,6	110
CALMIT	4,31	22,7	120

The values d_{10} , d_{50} , d_{90} in Table 1 determine the value of the particle diameter in μm present in the sample from 10 %, 50 % and 90 %. Based on the tests of the paste made from these samples, it was found that the most suitable material is limestone from CALMIT with a characteristic dimension of $d_{50} = 22,7 \mu\text{m}$. The liquid phase consists of demineralised water.

4 THE EXPERIMENT

The paste is formed by a mixture with finely ground limestone and demineralized water in a ratio suitable for extrusion. Thus, extrusion uses particulate matter which, together with the liquid, forms a paste with suitable rheological properties. It is therefore necessary to obtain a homogeneous liquid-powder system.

For this reason, the preparation of the paste was carried out in two steps (Fig. 4). The aim of homogenization No. 1 in the mixer was to form the paste as a mixture of water and limestone. During mixing, due to the rotary motion of the blades in the cylindrical chamber, a spherical agglomerate with dimensions up to 20 mm is formed. Therefore, the aim of homogenisation 2 was to break up these agglomerates and obtain a homogeneous, free-flowing material, which was fed into the extruder.

The extrusion itself was then carried out in a machine of special design. Subsequently, this step prepares the extrudate for the rotating disc to be spheronised. Two different die geometries were used in the experiment (Fig. 5) for each of them, experiments were done with three different die rotation frequencies and five rotor rotation frequencies. Each measurement was repeated three times. Thus, a set of 135 results was obtained.

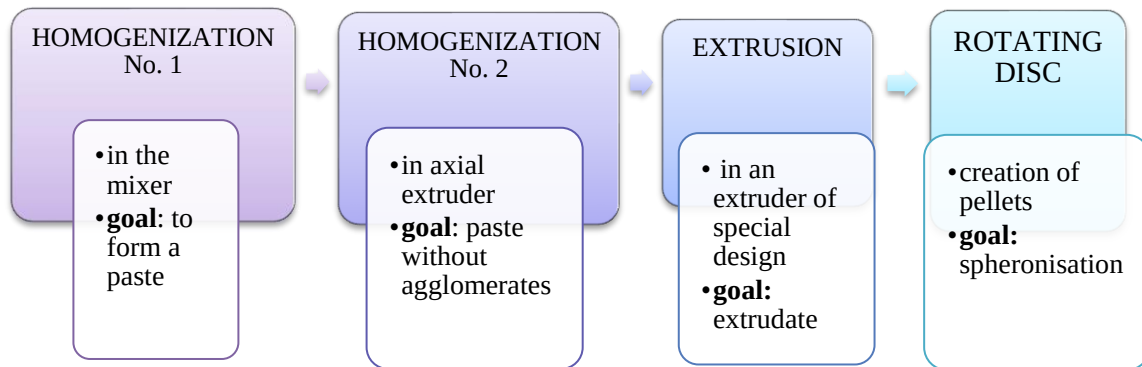


Fig. 4. Stages of the extrusion process - paste preparation as well as stages of the E-S process.

5 RESULTS AND DISCUSSION

The set of 135 results is processed as follows. Figure 5 shows the diagrams of the matrices used. The difference in the matrices is mainly in the L/D ratio and the shape of the hole. For the matrix with a conical hole, the direction of the paste flow is in the direction of the expanding hole. This eliminates the friction of the paste against the surface of the P_w holes and thus allows the pressure required to push the paste into the P_0 hole to be measured. The cylindrical hole die is designed to measure the friction of the paste against the surface of the holes, which corresponds to the pressure P_w .

Since it is not possible to eliminate the pressure required to push the paste into the holes P_0 , the friction must therefore be calculated from the pressure P according to equation (2):

$$P = P_0 + P_w \quad (2)$$

where P is the measured extrusion pressure for the cylindrical hole die and P_0 is the extrusion pressure for the conical hole die.

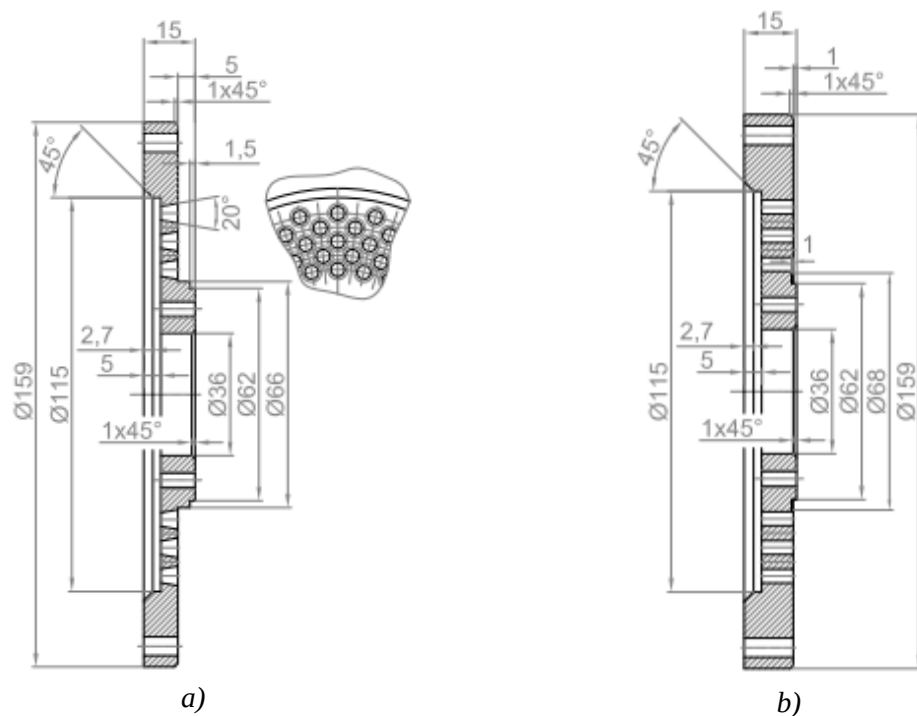


Fig. 5. Geometry of dies.
a) die 1 - conical hole, $L/D = 5 \text{ mm} / 4 \text{ mm}$,
b) die 2 - cylindrical hole, $L/D = 9 \text{ mm} / 4 \text{ mm}$

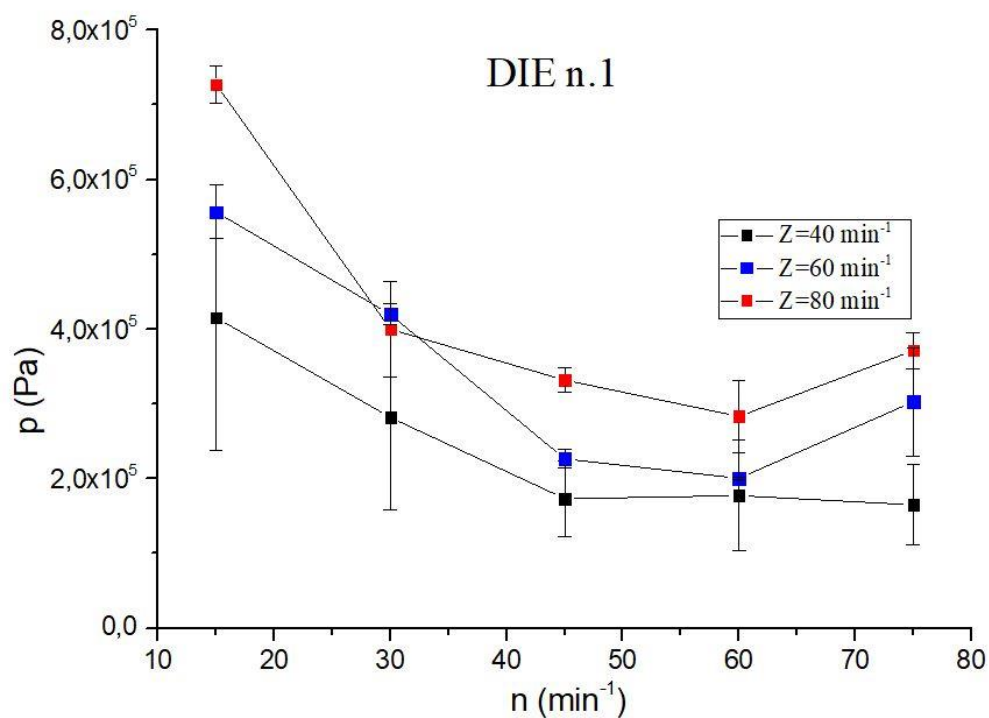


Fig. 6. Monitored extrusion pressure parameter for die 1 and set constant screw rotation frequency ($Z = 40 \text{ min}^{-1}$, $Z = 60 \text{ min}^{-1}$, $Z = 80 \text{ min}^{-1}$) at five ascending rotor frequencies.

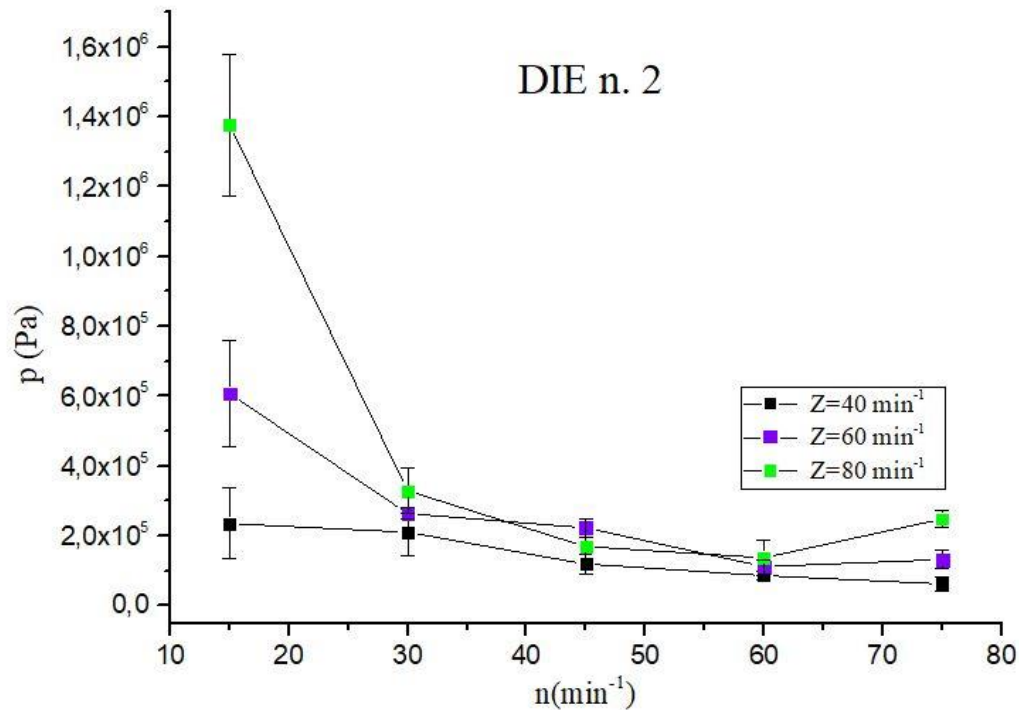


Fig. 7. Monitored extrusion pressure parameter for die 2 and set constant screw rotation frequency ($Z = 40 \text{ min}^{-1}$, $Z = 60 \text{ min}^{-1}$, $Z = 80 \text{ min}^{-1}$) at five ascending rotor frequencies.

Pressure depends on the moisture content of the paste. The paste moisture content in all experiments was $w = 16.76 \%$. In extrusion, proper paste preparation is very important because even small changes will have a significant effect in the extrusion process.

The extrusion pressure, as a monitored parameter, increases with decreasing rotor rotation frequency n_r , while the screw rotation frequency n_z remains constant. This is due to the change in the rheological properties of the paste in terms of an increase in apparent viscosity as the shear strain rate decreases, caused by the decrease in rotor speed. This means that the paste is more compressed, so its density increases and also the migration of the liquid phase occurs [15].

5.1 Extrudate surface quality

We used direct optical methods to evaluate the surface quality of the extrudate. It was analyzed directly by photography and also by microscope. The individual samples were examined in terms of length, the amount of cracks on the surface, and a longitudinal section was also made to investigate the depth of the cracks. At the lowest frequency of rotation of the screw and just the opposite at the highest frequency of rotation of the rotor, the extrudate has only small flakes at the die 1. This is because of the conical hole, which does not allow the extrudate surface to be formed by contact with the cylindrical surface of the hole. The resulting extrudate, does not have the character of a cylinder. However, as the frequency of rotation of the rotor decreases, the extrudate elongates and therefore takes shape. This is due to the increase in pressure. The change in the shape of the extrudate from scales to cylinders can therefore be observed.

After analysing the surface of the extrudate, we have also seen under the microscope that the most significant feature is the presence of regular fractures on the surface, which roughly encircle the extrudate and can therefore be referred to as circumferential cracks. Such surface fractures are an example of an extrusion error that can compromise the strength and performance of the extruded product. Thus, the surface is not smooth. The surface fractures are caused by the absence of the cylindrical surface of the die holes.



a) $Z = 40 \text{ min}^{-1}$



a) $Z = 60 \text{ min}^{-1}$



a) $Z = 80 \text{ min}^{-1}$



b) $Z = 40 \text{ min}^{-1}$



b) $Z = 60 \text{ min}^{-1}$



b) $Z = 80 \text{ min}^{-1}$

Fig. 8. Extremes at constant screw for $Z = 40 \text{ min}^{-1}$, $Z = 60 \text{ min}^{-1}$, $Z = 80 \text{ min}^{-1}$
a) die 1; b) die 2 and the rotor rotation frequency of 15 min^{-1} , the dried sample.



a) $Z = 40 \text{ min}^{-1}$



a) $Z = 60 \text{ min}^{-1}$



a) $Z = 80 \text{ min}^{-1}$



b) $Z = 40 \text{ min}^{-1}$



b) $Z = 60 \text{ min}^{-1}$



b) $Z = 80 \text{ min}^{-1}$

Fig. 9 Extremes at constant screw for $Z = 40 \text{ min}^{-1}$, $Z = 60 \text{ min}^{-1}$, $Z = 80 \text{ min}^{-1}$
a) die 1; b) die 2 and the rotor rotation frequency of 75 min^{-1} , the dried sample.

For die 2, we can see in Fig. 8, the fundamental influence of the matrix length. We observe that at the extrema, namely at the lowest frequency of rotation of the screw and the lowest frequency of rotation of the rotor, the extrudate already has its shape. The flakes are represented in small or no quantity. By elongating the resulting extrudate, it can be stated that the cylindrical surface of the die holes causes both an increase in extrusion pressure by the friction of the paste against the surface of the die holes, but also contributes to forming the shape and quality of the extrudate surface.

CONCLUSION

The aim of this work was to demonstrate how pressure and different die parameters affect the final quality of the extrudate. The experiment shows that extrusion pressure combined with die length have a great influence on the resulting extrudate surface quality. It is clearly demonstrated that the cylindrical surface of the die holes, as opposed to the conical one, causes an increase in extrusion pressure by the friction of the paste against the surface of the die holes, and also contributes to the shape and surface quality of the extrudate. The longer the die (i.e. the L/D parameter) the more the extrudate has a cylindrical shape and the higher the pressure, the smoother the surface quality of the extrudate, i.e. without significant cracks. In the extrusion process, pressure control is very important, therefore the higher the pressure, the more the paste is compressed in the process and thus there is a risk of an undesirable phenomenon, namely the migration of the liquid phase.

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LIST OF SYMBOLS

P	extrusion pressure	Pa
P_0	extrusion pressure for conical hole die (deformation pressure)	Pa
P_w	friction pressure against the surface of the holes	Pa
σ_0	initial stress	Pa
α	paste speed factor ($m = 1$)	$\text{Pa} \cdot \text{s} \cdot \text{m}^{-1}$
V	paste speed	$\text{m} \cdot \text{s}^{-1}$
w	paste moisture content	%
τ_0	initial wall stress	Pa
β	wall velocity factor ($n = 1$)	$\text{Pa} \cdot \text{s} \cdot \text{m}^{-1}$
L	screw length	m
D	screw diameter	m
n_r	rotor rotation frequency	s^{-1}

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