PREDICTING THE FLOW BEHAVIOR OF POWDERS, WITH PARTICULAR REFERENCE TO ROTOMOLDING MATERIALS

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Abstract

The rotomoulding industry uses a simple but crude measure to assess the flowability of powders, which involves timing the discharge of powder through a funnel with a circular bottom orifice.

The flow of granular materials through orifices has been the subject of considerable research elsewhere and equations exist that relate flow to the various parameters involved. The most well accepted formula was assessed for its suitability to typical rotomoulding materials, as well as a variety of other powders and granulates.

The characteristics of many materials (including rotograde micropellets) showed good correlation to theoretical predictions. Polyethylene and polypropylene rotograde powders showed behaviour that could be described by a modified version of the existing equation.

Introduction

The common polyolefin materials used for rotational molding (rotomolding) have traditionally been supplied as fine powders (mean particle size 300 to 350 μm). More recently, micropellets have been proposed as an alternative material format. Typically micropellets have a mean particle size that is larger than roto powders. The distribution of particle sizes is also narrower, with zero fine material (<150μm). This is demonstrated in Fig 1.

It is well understood that a powder with poor flow characteristics can create problems when it is rotomoulded. Typical problems associated with poor powders include:

- the increased incidence of tiny surface defects (pinholes)
- the creation of larger voids (“bridging”) in restricted mold sections
- unacceptable thickness variation in the part
- uneven inside surface of the part

The standard method for evaluating rotograde powders is the Dry Flow test, which measures the time taken for a 100g powder sample to flow out of a standard funnel with a circular bottom orifice of 10mm diameter.

Some of the powder leaving the funnel is collected in a container of known weight and volume, which is weighed and the Bulk Density of the powder can then be calculated.

A recent study by the author, into the accuracy of these tests, concluded that the flow from the funnel orifice (ie the Dry Flow value) appeared to be subject to chaotic fluctuations, which created a wide variation in values, even for the same sample, measuring equipment and test conditions. In contrast, it was possible to make Bulk Density measurements on a much more consistent and reproducible basis.

This previous study prompted the question of whether a mathematical model already existed that would link Dry Flow and Bulk Density measurements together. Whilst this does not appear to be available within the literature on rotomolding, a substantial body of relevant research has been conducted on the design of the bottom outlets of silos.

Equations have been proposed and extensively verified to link together the flow of granular material through a circular orifice, the orifice diameter, the bulk density of the material and the particle size of the granulate. The original equation of this type is credited to Beverloo et al:

\[ W = C \rho_B g^{0.5} (D_0 - kd_p)^{2.5} \]

Fig 1: Typical Powder & Micropellet Particle Size Distributions
Where:  
C is the discharge coefficient (typically ≈ 0.58)  
W is the mass flow of the material, in kg/sec  
ρ₀ is the bulk density of the material, in kg/m³  
g is the acceleration due to gravity, in m/sec²  
D₀ is the diameter of the outlet orifice, in m  
k is the shape coefficient (typically ≈ 1.4)  
d₀ is the particle size, in m

In terms of applying the Beverloo equation to roto grade powder flowing through a Dry Flow funnel, a number of questions arise:

a. Mankoc et al⁴ report that the validity of the Beverloo equation has only been established for mono-sized granular samples with a d₀ larger than 500µm. Rotograde powders fall outside this definition.

b. D₀ must be large enough to prevent particle jamming and consequent interruptions to powder flow; this means that D₀ ≫ d₀.

c. Typical roto powders have a relatively wide distribution of particle sizes. Should the mean of the distribution be used? Fortunately, it may be possible to ignore this problem, because d₀ is extremely small relative to D₀ and it may be possible to ignore the term k d₀, term, at least as a first order approximation.

d. Flow from a silo or hopper has been characterized⁵ as either core flow (where the flow of powder is from the top of the bed through a preferential flow channel or “rat hole”) or mass flow (where all the powder is in motion during discharge). Which of these types of flow predominate in the case of a Dry Flow funnel?

e. It was recognized that the mass flow of the material (W) could be calculated directly from a Dry Flow measurement by the simple relationship shown in Equation (2). However, this would depend on the material flowing out of the funnel at a constant rate; is this what actually happens in practice?

\[ W = \frac{M_{DF}}{DF} \]  

Where:  
M₁ is the mass of powder used in the test (actually 100g)  
DF is the time taken for the Dry Flow funnel to empty (sec)

f. The dependence of W on D₀²⁵ has been widely discussed and Mankoc et al⁴ argue that the relationship appears to be plausible if (just above the outlet) there is a free-fall zone limited by an arch; the size of this arch would be somehow proportional to the radius of the orifice.

g. The empirical constant C may be dependent on various factors related to the funnel upstream of the discharge (eg frictional forces, funnel angle) and factors related to the material (eg angle of repose and percentage of fines)⁶.

The goal of the study described in this paper was to evaluate whether a simple relationship could describe the link between Dry Flow time and Bulk Density. A simplified Beverloo-type equation could offer such a relationship.

Using the simplifications discussed above, a modified formula was proposed:

\[ W = 0.01817 \, \rho₀ \]  

Assuming:  
C = 0.58 
W in g/sec (NB: not kg/sec)  
ρ₀ in kg/m³  
g = 9.81 m/sec²  
D₀ = 0.01m  
Ignore kd₀ term

**Experimental**

**Materials**

Five different rotomolding powders were tested, plus polypropylene micropellets; see Table 1. All these materials were samples of commercially available products, sold to the European rotomolding industry. The powders had a typical roto grade size specification (ie average particle diameter 300-350 μm).

Product grade names have been omitted for reasons of confidentiality. It is understood that the LMDPE NP, LMDPE CP and XLPE had all been produced by ambient grinding, whereas the PP and POP powders had been ground cryogenically.

<table>
<thead>
<tr>
<th>Material</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>LMDPE NP</td>
<td>Linear medium density polyethylene, natural powder</td>
</tr>
<tr>
<td>LMDPE CP</td>
<td>Linear medium density polyethylene, compounded color powder (grey)</td>
</tr>
<tr>
<td>XLPE NP</td>
<td>Crosslinkable polyethylene, natural powder</td>
</tr>
<tr>
<td>PP NP</td>
<td>Polypropylene copolymer, natural powder</td>
</tr>
<tr>
<td>POP NP</td>
<td>Polyolefin plastomer, natural powder</td>
</tr>
<tr>
<td>PP MPLLT</td>
<td>Polypropylene copolymer, natural micropellets, 500 μm approx. diameter</td>
</tr>
</tbody>
</table>

Table 1: Rotomoulding Materials used in Study
In addition to rotomoulding materials, 14 other powders or granulates were sourced for testing. These are detailed in Table 2.

<table>
<thead>
<tr>
<th>Material</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>PSF Sand</td>
<td>Graded fine brown sand, 120μm Used in golf bunkers</td>
</tr>
<tr>
<td>Garnet</td>
<td>Graded medium red sand, 250μm Used in water jet cutting machines</td>
</tr>
<tr>
<td>Cenospheres</td>
<td>Hollow glass spheres, 150μm By-product from fly ash</td>
</tr>
<tr>
<td>Ground rice</td>
<td>Very small evenly sized particles Food product</td>
</tr>
<tr>
<td>Tapioca</td>
<td>Coarse spheres, 2.0-2.4 mm Food product</td>
</tr>
<tr>
<td>Demerara Sugar</td>
<td>Coarse Demerara sugar, sticky Food product</td>
</tr>
<tr>
<td>Granulated Sugar</td>
<td>Medium refined brown sugar Food product</td>
</tr>
<tr>
<td>Caster Sugar</td>
<td>Fine white sugar Food product</td>
</tr>
<tr>
<td>Table Salt</td>
<td>Fine table salt Food product</td>
</tr>
<tr>
<td>Linseed</td>
<td>Seeds, oblate spheroids approx. 3mm Food product</td>
</tr>
<tr>
<td>Oatmeal</td>
<td>Stoneground powder, wide distribution Food product</td>
</tr>
<tr>
<td>Pepper</td>
<td>Fine powder, very dusty Food product</td>
</tr>
<tr>
<td>Poppy Seeds</td>
<td>Even spherical particles, approx. 650μm Food product</td>
</tr>
<tr>
<td>Soap Powder</td>
<td>Coarse soap powder Household product</td>
</tr>
</tbody>
</table>

Table 2: Non-rotomoulding Materials used in Study

**Dry Flow / Bulk Density Apparatus**

The funnel used to determine Dry Flow was machined from aluminium grade 6082, using a Protturn XYZ lathe, according to the drawings in the ARM Test Method and was blue anodised. The orifice diameter of the funnel was measured by micrometer as 9.995mm.

A test temperature of 22.5°C was maintained for all tests, by locating equipment and materials in a controlled environment room and conditioning them for a minimum of 24 hr before testing commenced.

For each material tested, 10 Dry Flow test runs were conducted. Each test run comprised 3 repeat observations, which were averaged.

The Dry Flow results (measured in sec) were converted to discharge rate (measured in g/sec) using Equation (2).

Bulk Density measurements were made, also according to the ARM Test Method.

As well as standard Dry Flow and Bulk Density tests, initial experiments were run to determine whether the material flowed out of the funnel at a constant rate and to assess whether core flow or mass flow was the predominant mechanism by which the funnel emptied.

For the initial experiments, the Dry Flow funnel was suspended over a container (volume approx. 1 litre), which was standing on a set of digital scales. For rotograde powders, a standard 100g powder sample was poured gently into the top of the funnel, while the funnel outlet orifice was temporarily closed. The orifice was then opened, allowing the powder to flow out into the container. The increase in container weight was noted at time intervals as the funnel emptied and the container filled.

For other materials, especially those with a significantly higher bulk density, an initial sample weight was selected that approximately filled the funnel.

**Results and Discussion**

Fig 2 shows the results of the initial experiments to determine whether, during a standard Dry Flow test, the material flowed out of the funnel at a constant rate. The particular example shown is for LMDPE NP, but the tests were repeated for all the other polymer powders, as well as most of the non-rotograde materials. The behaviour was similar in all cases.

A linear regression with a forced zero intercept yielded a gradient of 3.744 g/sec, which would translate (via equation (2)) to a Dry Flow (100g sample) of 26.7 sec. The correlation coefficient, across all data, was 0.9972.
It was also noted that there was no evidence of core flow in any of the tests, whatever the materials used. The flow from the funnel was definitely by mass flow, i.e., all the powder was in motion during discharge. There was some evidence of a depression forming in the upper surface of the descending powder, which would be the precursor to the formation of a preferential channel, but this did not develop into a “rat hole”.

The domination of mass flow is of particular interest when viewed against the conflicting evidence regarding the effect of surface roughness (of the inside surface of the funnel) on Dry Flow. Laws reported that Dry Flow time decreased significantly as the surface roughness of a flow funnel was increased. This time decrease was attributed to the powder being held on the sides of the funnel and “rat-holing” being promoted.

Conversely, recent research by the author showed that surface roughness caused only marginal changes in Dry Flow. It should be noted that surface roughness values were not given for the Laws data, so it is difficult to reach a clear conclusion.

Fig 3 shows a plot of $W$ vs. $\rho_B$, for some of the non-roto materials used.

These were: PSF Sand, Garnet, Table Salt, Ground Rice, Poppy Seeds, Caster Sugar and Granulated Sugar.

Fig 3: Mass Flow vs. Bulk Density for “Beverloo” Materials

It will be observed that the positions of the clusters of results for each of the selected materials conform closely to the predictions of the modified Beverloo formula – Equation (3). A linear regression with a forced zero intercept yielded a gradient of 0.0177, compared to a theoretical slope of 0.0182. The correlation coefficient, across all data, was 0.9883.

The degree of fit of the data with theory is good and is especially impressive, considering the wide range of bulk density for the different materials. This ranged from approx. 580 kg/m$^3$ (for Poppy Seeds) to approx. 2200 kg/m$^3$ (for Garnet).

Fig 4 shows a plot of $W$ vs. $\rho_B$, for the other non-roto materials used.

These were: Cenospheres, Tapioca, Demerara Sugar, Soap Powder, Linseed, Oatmeal and Black Pepper.

Fig 4: Mass Flow vs. Bulk Density for “Non-Beverloo” Materials

The reasons for the non-compliance of these materials to the Beverloo predictions have not been fully analyzed at the time of writing.

However, one common feature of many of the materials was a high level of fines (<150 μm); this was certainly true of the Cenospheres, Soap Powder, Ground Oatmeal and Black Pepper.

The Demerara Sugar and Oatmeal both felt “sticky” to the touch, which was assumed to be due to the presence of some type of additive. The deleterious effect of this on the friction between particles moving over each other (and therefore mass flow) can be readily envisaged.

The poor flow of the Tapioca was, on first consideration, surprising. The particles were smooth and perfectly spherical and the particle size distribution was narrow. However, the average particle size (approx. 2.2 mm) was large compared to the diameter of the orifice through which they were flowing and the assumption $D_0 \gg d_\ell$ could not be considered as valid.

Mankoc et al., in their investigation into the jamming behavior of particles flowing through orifices, identified a ratio, $R$, between the diameter of the orifice and the particle diameter flowing through it:

$$R = \frac{D_0}{d_\ell}$$

At a critical value of $R$, identified as $R_c$, the probability of particles jamming the orifice would rise...
from negligible to near-certain. \( R_c \) was stated as a value of approx. 5; a value of \( R \) less than this would be an indication of a high probability of jamming.

In the case of the Tapioca sample, \( d_p \) was 2.0-2.4 mm and \( D_0 \) was 9.995 mm, so the value of \( R \) was be approx. 4.5, which is less than \( R_c \). The probability of jamming would therefore be high; this was confirmed by the observation that, on all runs of tapioca through the test funnel, multiple taps were required to maintain a steady flow.

The reasons for the poor flow of the Linseed are similar, in that two dimensions of the particle were large (>4mm and >2mm).

Fig 5 shows a plot of \( W \) vs. \( \rho_B \), for the polyethylene-based and polypropylene powders that were tested, plus the polypropylene micropellets.

![Fig 5: Mass Flow vs. Bulk Density for all PE and PP powders](image)

It will be observed that the data for all PE-based and PP powders does not conform to the Beverloo prediction, although the PP micropellets do conform closely.

Interestingly, a line of best fit through the data points for PE-based and PP powders had a slope that was very close to the Beverloo exponent: 0.0184 compared to 0.0182. It should be noted that the correlation coefficient for the line of best fit was respectable, at 0.9255 and that the linear correlation was executed on 30 separate data points, which represents a reasonable sample.

Although of correct slope, the line had an intercept of -2.7294, indicating that, at a certain Bulk Density, the Dry Flow would be zero. This value was calculated to be 148 kg/m³. Although this is a theoretical figure, it has been observed that, for LMDPE powders that are badly ground and have unacceptably low Bulk Density, no flow can be established through the funnel. Therefore the idea of a minimum Bulk Density to achieve any powder flow has a strong basis in reality.

The very different performance of micropellets compared to powder (for PP) could be explained by a combination of two factors.

Firstly, the micropellets have a tighter size distribution than the powder (see Fig 1). In this respect, they perform similarly to Poppy Seeds, which are also uniform and have a similar mean particle size.

Secondly (and of greater significance) all the materials that conformed to “Beverloo behavior” had a low level of fine (<150μm) material. It can readily be envisaged that the presence of fines will increase the internal friction and retard the flow of particles across one another.

It is generally recognized that the surface condition of polymer particles ground under ambient conditions can be deleterious to Dry Flow, if micro-strands (aka “tails”) are present on the particle surface.

In this respect it is interesting to note that all the rotograde powders behaved similarly. Two of the rotograde materials that were tested (POPP NP and PP NP) had been cryogenically ground and were substantially free of such “tails”. This indicates that the presence of fines may be a more significant factor than either wide size distribution or particle condition.

**Conclusions**

The flow of powders from a small funnel, of the type recommended by the ARM procedure, appears to be by mass flow only. There is no evidence of core flow through the funnel and this suggests that the internal roughness of the funnel will have little effect. This conclusion has been corroborated by other work, recently published by the author.

The validity of the commonly accepted Beverloo equation has been demonstrated for a range of materials from different origins and with widely varying Bulk Density.

The non-conformance of some materials to Beverloo can be explained by factors such as fines level, surface stickiness and particle over-size compared to funnel orifice diameter.

Rotograde powders appear to conform to a modified Beverloo behaviour, in that their flow-density dependence shows the same linear rate of increase. However, internal friction effects (thought to be mostly caused by the presence of fines) retard flow, seemingly in a consistent manner.
The results of this study, combined with the findings from other work recently carried, indicate that the current understanding of powder flow within the rotomoulding industry would benefit from expansion and improvement.

It is intended to continue the study of flow behaviour of powders, especially rotograde polymer powders, so as to provide new insights that will be of practical use to both producers and processors.

References

8. R.D. Laws, Rotation (Jan-Feb 2004).