LIMITATIONS & LEVEL OF ACCURACY OF TESTS FOR ROTOMOLDING POWDERS

Dr Nick Henwood, Rotomotive Limited, Northampton, UK

Abstract

The rotomolding industry commonly uses two connected tests to assess the quality of plastic powders: Dry Flow and Bulk Density. Industry-specific recommended test methods are now available for both parameters.

Repeated measurements were carried out using five different rotomolding powders, in order to assess the influence of the various equipment and environmental parameters that are thought to affect the test. This enabled estimates to be made of the limits for the accuracy and repeatability that are achievable practically.

The results obtained from the Dry Flow test suffer from significantly higher variance than those obtained from the Bulk Density test.

Introduction

The common plastic materials used for rotational molding (rotomolding) are normally supplied to the mold in the format of a fine powder. This powder typically has a mean particle size of approx. 350 μm. There is no such thing as a "standard" specification for rotomoulding powder, but some typical criteria are shown in Table 1.

Property	**Units**	**Value**
Powder sizes >600μ	%	0
Powder sizes >500μ	%	<5
Powder sizes <150μ	%	<15>5
Flowability	sec	<30
Bulk Density	g/cm^3	>30

Table 1: Typical Powder Specification

Until quite recently, the test methods for Dry Flow and Bulk Density were generally well understood, but equipment limits and test procedures were not specified in precise detail. This was believed to result in considerable variability during general usage, increasing the possibility of disputes between molders and material suppliers.

In an attempt to rectify this situation, a technical committee from the Association of Rotational Molders (ARM) drafted a recommended Test Method[1] which, after various modifications, was adopted worldwide by all regional trade bodies belonging to the Affiliation of Rotomoulding Organisations (ARMO).

The test uses a conical funnel, which has been machined to precise dimensions, suspended (at a defined height) over a container of known volume. 100g of the test powder is poured gently into the top of the funnel, while the funnel outlet orifice is temporarily closed. The orifice is then opened, allowing the powder to flow out into the container. The time taken for the funnel to completely empty is recorded as the Dry Flow value, expressed in sec.

The container, which is sized to hold considerably less than 100g of powder, has now been fully filled by the powder exiting the funnel. Excess powder is scraped off the top of the funnel, which is then weighed, emptied and weighed again. The Bulk Density of the powder can then be calculated from the weight of powder in the container divided by the volume of the container. Typically, Bulk Density is recorded as g/100cm^3.

The ARM Test Method draws on ASTM D1895-96 for its basic methodology, but seeks to improve the reproducibility of results by tightening the ASTM funnel specification in a number of areas:

- The specification limits for orifice diameter are tightened considerably, to 10.00mm ± 0.01mm.
- Other funnel dimensions are specified and a detailed drawing is provided; the funnel material is specified as "aluminium alloy" and the complexity of design means that funnels must be machined, from a block of metal, rather than fabricated from flat sheet.
- The roughness of the funnel surface is specified, as "400 microns, 16 microinches". This stipulation draws on a previous study which reported that flow from a funnel would increase if the surface was rough rather than smooth[4].
- A "sulphuric anodised – blue" finish is specified.
- The range of powder temperatures at which the test should be conducted is specified as 20-25°C.
- The common practice of administering a tap to the funnel, if necessary, to initiate flow, is stated as not in accordance with the Test method.

The goal of the study described in this paper was to measure how accurate and representative the new test methods would be; personal anecdotal experience

indicated that, despite the refinements of the new method, there could be other factors at work and that the test results might still exhibit considerable scatter.

Other industries that use powder in their manufacturing processes have alternative measurement methods and different ways of expressing powder characteristics. In particular, it is common to measure the bulk density of a powder in both its freely settled form and in a more consolidated form, which is achieved by vibrating the sample until its bulk density reaches a maximum value.

The Hausner Ratio[2], *H*, is calculated by the following formula:

$$H = \rho_T / \rho_B \quad (1)$$

Where ρ_T and ρ_B are the respective densities of the tapped sample and the freely settled sample.

The Carr Index[3], C, is an alternative indication of the same properties and is calculated by the following formula:

$$C = 100 \times (1 - \rho_T / \rho_B) \quad (2)$$

Carr Index is related to the Hausner ratio as follows:

$$C = 100 \times (1 - 1 / H) \quad (3)$$

A Hausner Ratio greater than 1.25 is considered to be an indication of poor flowability. A Carr Index greater than 25 (equivalent to H = 1.33) is considered to be an indication of poor flowability and a Carr Index less than 15 (equivalent to H = 1.18) is considered to be an indication of good flowability.

Both *H* and *C* have been criticized as not having a strong theoretical basis. However, they have been found to be useful empirical measures and the equipment and test methods used are relatively simple.

Experimental

Materials

Five different rotomolding powders were tested, as described in Table 2. All these materials were samples of commercially available products, sold to the European rotomolding industry.

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.

LMDPE NP	Linear medium density polyethylene, natural powder
LMDPE CP	Linear medium density polyethylene, compounded color powder (grey)
XLPE NP	Crosslinkable polyethylene, natural powder
PP NP	Polypropylene copolymer, natural powder
POP NP	Polyolefin plastomer, natural powder

Table 2: Materials used in Study

The key particle size distribution parameters for each material are given in Table 3. The only unusual powder was the PP NP, which was much more coarsely ground than the other materials.

Material	**>600µ**	**>500µ**	**<150µ**
LMDPE NP	0.5%	2.1%	11.0%
LMDPE CP	1.0%	7.1%	14.0%
XLPE NP	0.5%	5.6%	7.8%
PP NP	28.7%	11.8%	7.8%
POP NP	1.1%	6.6%	6.0%

Table 2: Key Particle Size Distribution Parameters for Materials used in Study

Dry Flow / Bulk Density Apparatus

Ten identical aluminium funnels were machined from aluminium grade 6082, using a Proturn XYZ lathe, according to the drawings in the ARM Test Method.

Finished funnels were blue anodised, again as per the ARM Method. Post-manufacturing measurements of the funnel orifice sizes are shown in Table 3. It will be seen that all funnels conformed to the orifice size specification contained in the ARM Procedure, with a mean orifice size of 9.994mm and a standard deviation of sizes of 0.005mm

Funnel Number	**Hole Diameter (mm)**
#1	9.994
#2	10.003
#3	9.990
#4	9.992
#5	9.995
#6	9.995
#7	9.985
#8	9.995
#9	9.993
#10	9.995

Table 3: Test Funnels

For each standard test, a sample of 300g of powder was selected and three repeat Dry Flow and Bulk Density measurements were carried out.

When a tap on the funnel was required to initiate powder flow, this was noted. For a batch of results a Tap Probability, *T*, was calculated as follows:

$$T = 100 \times (N_{tap} / N_{total}) \quad (4)$$

Where N_{tap} is the number samples in a batch requiring a tap and N_{total} is the total number of samples in the batch.

For the tests on funnel surface roughness, four different sized funnels were used; these items were surplus to requirements and could be sacrificed for the test.

First, the funnels were mechanically buffed to a very high polish (ie a very low surface roughness) and Dry Flow tests were carried out. The roughness of the surface was then progressively increased, using a CNC controlled milling device. After each increase in roughness, Dry Flow measurements were carried out.

Environmental Chamber

In order to investigate the effects of test temperature, all Dry Flow and Bulk Density tests were carried out inside an environmental chamber, where the ambient temperature could be controlled to ±1°C.

The chamber, which measured 2.70m x 3.13m, used floor-mounted on/off tubular electric heaters which were modulated by a PID controller using input from two compensating thermocouples placed strategically within the chamber. This chamber has been shown to be capable of delivering reliable ambient temperatures between 15°C and 45°C. Materials and equipment were all conditioned at test temperature for at least 24 hr before tests were carried out.

Consolidation Device

In order to calculate Hausner Ratio and Carr Index, it was necessary to measure bulk density of the powder in a fully consolidated form.

The necessary consolidation force was delivered using a Rotap sieve shaker; calibration tests were carried out to determine a tap frequency and total tap time that would deliver full consolidation to the powder sample. A modified container was made (see Fig 1), which could be split after consolidation had taken place, so as to provide a known volume of fully consolidated powder, ready for weight measurement.

Fig 1: Split Container for Consolidated Bulk Density Determination

Results and Discussion

Fig 2 shows the results of an initial "robustness test" that was carried out on the Dry Flow method. The same sample of LMDPE natural powder was tested, on 15 consecutive occasions. After each individual test, the material was carefully collected and re-tested through the funnel. The total loss of material, across the 15 tests was less than 0.5g.

It can be seen that, despite all the care and precautions taken, there was a surprising variation in measured values; Dry Flows varied from a minimum of 25.8 sec to a maximum of 27.9 sec. The mean Dry Flow time was 26.6 sec and the standard deviation across all measurements was 0.46 sec.

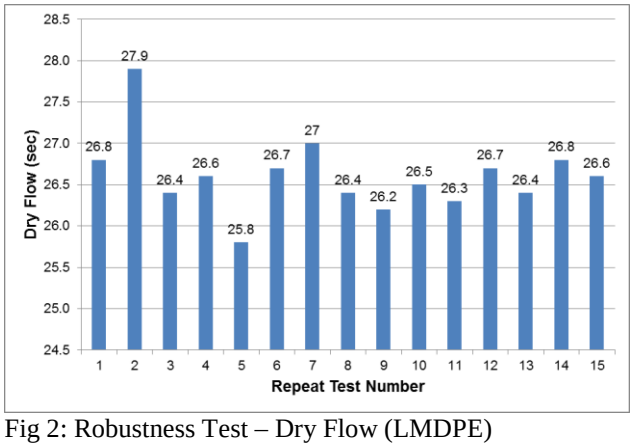

Fig 2: Robustness Test – Dry Flow (LMDPE)

Fig 3 illustrates the variation in measured Dry Flows (average of 3 repeats each) at an ambient temperature of 25°C; this is shown as an example, the behavior was

similar for all ambient temperatures. The Dry Flow results appear to vary slightly from funnel to funnel, with funnel #5 appearing to give marginally lower numbers with most types of material.

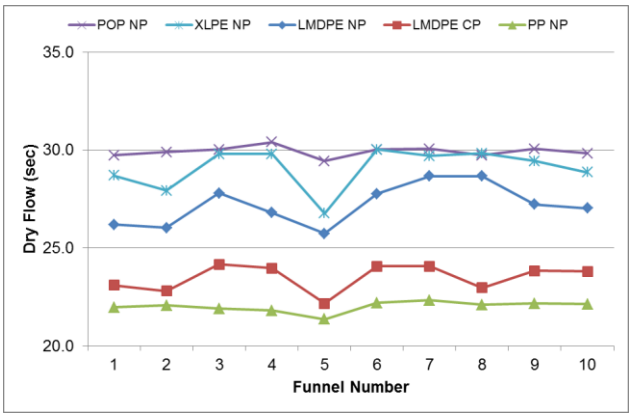

Fig 3: Funnel to Funnel Variation – Dry Flow

Fig 4 shows how measured Dry Flow (average of 3 repeats each) varied with ambient temperature of the material and test equipment. Whilst there are some minor variations, it is difficult to discern any trend, either up or down. The same absence of trend can be seen in the variation of Bulk Density with temperature and the variation of Tap Probability with ambient temperature (Fig 5 and 6 respectively).

Having regard for the apparent insensitivity of both Dry Flow and Bulk Density to ambient temperature variation, together with the lack of major deviations from funnel to funnel, it was considered reasonable to combine all data for each material together, to increase sample data size and enable some meaningful statistical analysis to be carried out.

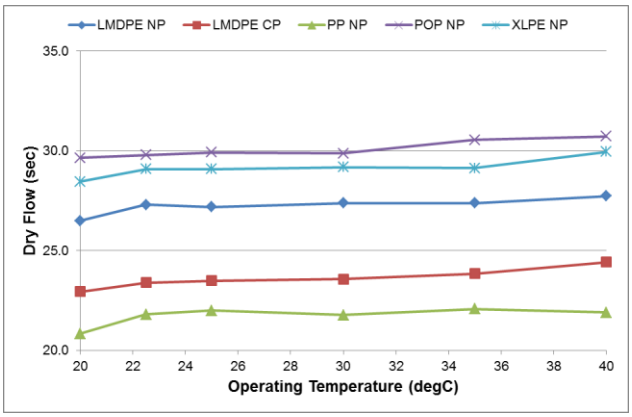

Fig 4: Dry Flow Variation with Temperature

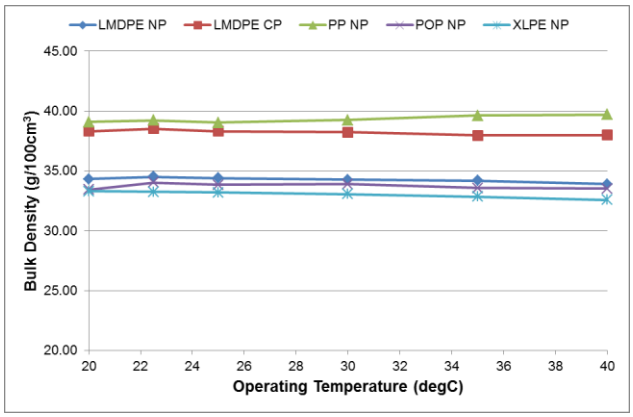

Fig 5: Bulk Density Variation with Temperature

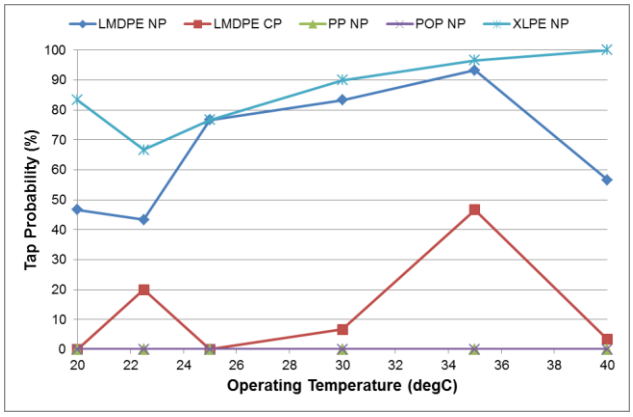

Fig 6: Tap Probability Variation with Temperature

Fig 7 (left hand portion, solid line) shows the probability distribution of the entire data set for LMDPE, comprising measured Dry Flows, 3 repeats each, for all 10 funnels and all 6 ambient temperature ranges; this means there were 180 data points included in the statistical analysis. It will be observed that the distribution is not normal, but appears to have two peaks, suggesting a bifurcation of the results. In a bifurcated system, uncontrolled factors will cause the data to swing from one state to another.

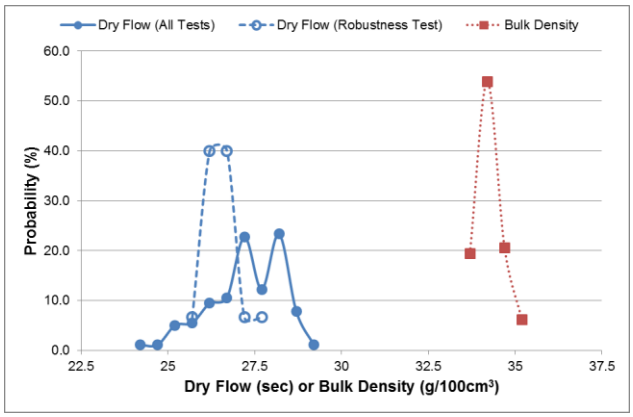

Fig 7: Distribution of Dry Flow and Bulk Density Results (for LMDPE)

The maximum probability of measurements in a particular range is low: only 22.8% probability for the first peak and 23.3% probability for the second peak. Note that, for this analysis, a data grouping range of 0.5 sec was selected.

In addition to the apparent bifurcation, the distribution spreads over a wide range of measured times; Dry Flow measurements from 24.1sec to 29.6 sec were recorded, a range of 5.5 sec.

The mean Dry Flow from this data set was 27.3 sec and the standard deviation was 1.06 sec.

If the distribution were normal, the three-sigma rule would predict that nearly all (ie over 99%) of values would lie within three standard deviations on each side of the mean, suggesting a range of 6.4 sec. For a lower level of certainty (95% degree of confidence) a two-sigma rule would be applicable, equivalent to a range of 4.2 sec.

An additional observation is that, unlike a normal distribution, there is no near-coincidence between the peak of the distribution and the mean value. Actually, the mean value coincides with the trough between the two bifurcated peaks of the distribution. It is considered that the data set is sufficiently large for this to be a significant finding and not the result of an insufficiently large data sample.

As a comparison, Fig 7 (left hand portion, dashed line) also shows the spread of data from the robustness test, which represents a total of 10 data points only. From this (limited and inadequate) data set, one might draw the conclusion that the data clustered around a mean and that it was approximately normally distributed. However, note that even this (more narrow) distribution shows a surprisingly large range (2.1 sec).

As a further comparison, Fig 7 (right hand portion, dotted line) shows the probability distribution of the entire data set for LMDPE, comprising measured Bulk Densities for the full 180 data points included in the statistical analysis. In this case, the distribution is much tighter and the distribution appears near-normal. The distribution peaks around the mean value (34.3 g/cm^3), the standard deviation is low (0.35 /cm^3), as is the range (1.7 g/cm^3).

It is difficult to believe that the significant ranges found for the Dry Flow data can be explained by experimental error. Considerable care and effort was expended in producing near-identical funnels and the technician conducting the test was both experienced and diligent. These factors argue against an explanation based on sloppy procedures, as does the consistency of the Bulk density measurements, which were made using the same apparatus, by the same operator, at the same time.

Another way of illustrating the erratic dispersion of the data system is by using a Poincaré Map, see Fig 8.

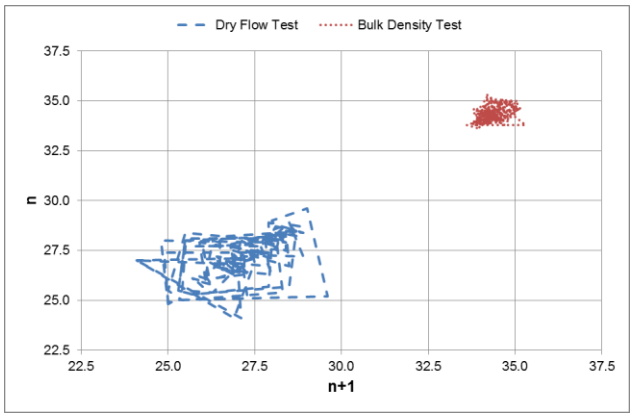

Fig 8: Poincaré Map for Full Data Sets (for LMDPE)

Each member of the data set has been plotted using its value as the x co-ordinate and the succeeding value as the y co-ordinate. Successive points are joined together by a straight line. In this way, the spread of the data can be readily visualized.

From this representation, it can be appreciated how much data spread has been encountered in the Dry Flow measurements, compared to the Bulk Density measurements.

The inexplicably wide range of Dry Flow measurements, combined with the apparent bifurcation of the peaks of the distribution, strongly suggests that the Dry Flow test has the characteristics of a *chaotic* system. In the mathematical sense, this is defined by the results of the tests being strongly dependent on small alterations in the boundary conditions, which cannot be controlled.

An example of the behavior of a chaotic system, in a completely different field, would be the way that very minor variations in atmospheric conditions can have a major effect on weather systems.

In the present case, the cause of the chaotic behavior may be due to a number of factors. It is possible that there may be a random build-up and discharge of static charge in the funnel, due to the motion of highly insulating particles (polymer powder) as the funnel is filled.

Variation in humidity might also have an effect, although this is unlikely, given the relatively stable conditions in the controlled temperature chamber.

It is believed that the most likely factor is an inconsistency in the particle size distribution of the powder, at a micro scale, in different parts of the powder mass contained in the funnel. It is known from other

work that the presence of fines can have a marked effect on Dry Flow. In addition, it has also been observed that the onset of powder Dry Flow deterioration can be sudden, at a critical percentage of fines.

Therefore it is proposed that minor variations in the forces resultant from filling the funnel can create areas of relatively high fines in parts of the powder mass, together with areas of depleted fines in other parts of the powder mass. The interplay of these areas, as the funnel empties, results in variations in the flow that are unexpectedly large and unpredictable.

The Tap Probability data provides a degree of corroboration to this hypothesis. There was a need to apply taps, on occasion, to the three powders (LMDPE NP, LMDPE CP and XLPE) that were not cryogenically ground.

Fig 9 illustrates a series of 180 tests, using the data for LMDPE NP. The data for LMDPE CP and XLPE shows a similar trend. The need for a tap on a particular test run is indicated by a vertical line. There is no obvious pattern apparent and the need to tap appears to be a random event.

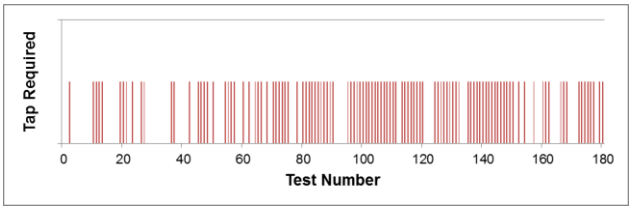

Fig 9: Incidence of Taps across 180 Consecutive Tests (LMDPE)

On further detailed examination of the data, it was felt that there was an outside possibility that the bifurcated distribution might be explained by some sort of funnel-to-funnel effect, even though a significant variation, in terms of average Dry Flow results, had not previously been apparent.

As a double-check, a completely new series of tests was carried out, using another batch of a selected powder (LMDPE), a single funnel (#7) and a single test temperature (22.5°C). The tests were carried out in batches of 12 (4 tests, 3 repeats) over a period of 5 days; in all, 180 tests were carried out. The results of this test are shown in Fig 10.

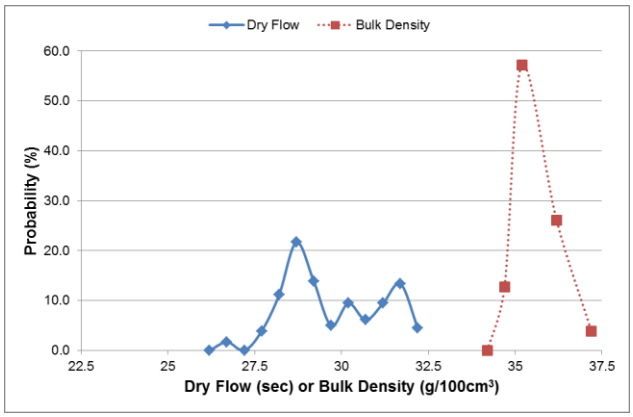

Fig 10: Distribution of Dry Flow and Bulk Density Results (for LMDPE)

It will be observed that, as in the previous case, the distribution is bifurcated and that the maximum probability of measurements is low: only 21.7% probability for the first peak and 13.3% probability for the second peak.

Once again, the distribution spreads over a wide range of measured times; Dry Flow measurements from 26.7 sec to 32.4 sec were recorded, a range of 5.7 sec.

The mean Dry Flow from this data set was 29.7 sec and the standard deviation was 1.40 sec.

These results, for an even more controlled system than previously, show the same (actually slightly worse) spread of data and chaotic behavior.

Figs 11 and 12 show the results from the tests made to assess whether funnel roughness had a significant effect on Dry Flow. A previous study[4], made by others, had indicated that this was an important variable and that the Dry Flow would be expected to be lower in a funnel with a rough inside surface, compared to a highly polished funnel. The results of this previous study are what led to the ARM method specifying a surface roughness for the funnel of 400µ.

In these tests, the roughness of the surface was progressively increased (from almost zero roughness to approx. 400µm) between batches of Dry Flow measurements. All five test materials were used for this work.

In all cases, the average Dry Flow measured for a specific funnel roughness was compared to the Dry Flow measured with the funnels when highly polished. The Dry Flow values were normalized as follows:

$$\text{Normalized DF} = DF_{rough} / DF_{polished} \qquad (5)$$

Fig 11 shows the variation in normalized Dry Flow measured on four funnels, against the surface roughness of the funnel.

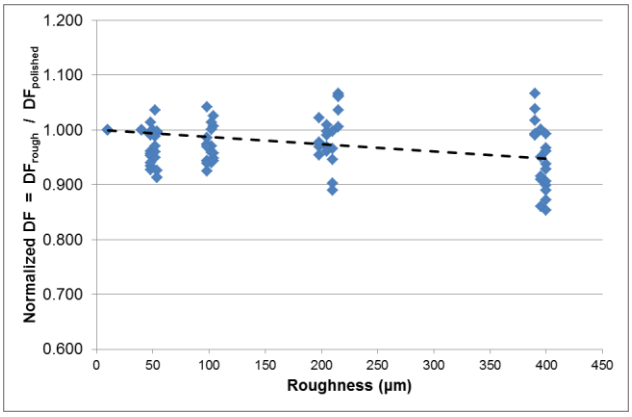

Fig 11: Variation of Normalized Dry Flow with Funnel Surface Roughness (all materials)

A trendline drawn through all data indicates that Dry Flow decreases slightly as funnel roughness increases. However, it will be observed that the correlation of the trendline is extremely poor and that there is a high degree of data scatter. This is unsurprising, given the high degree of scatter generally experienced in Dry Flow testing.

Fig 12 shows the same data (for all values of roughness), in terms of its distribution around the mean. There appears to be a near-normal distribution and a close coincidence between the distribution peak and the mean value (0.975). The range of acceptable data values (according to the three-sigma rule) would be 0.844 to 1.105.

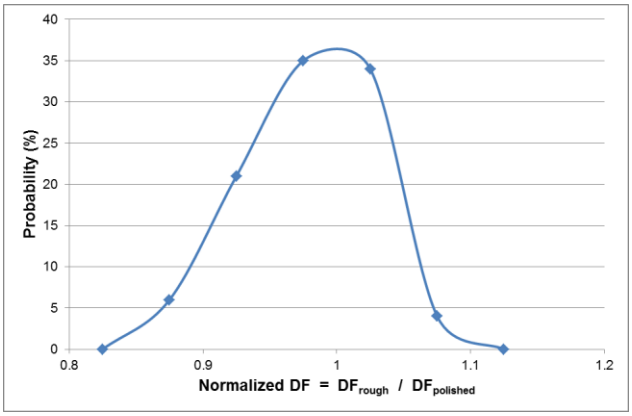

Fig 12: Distribution of Normalized Dry Flow Results (for all materials and all roughness values)

Given the level of data scatter, the claim of a strong dependence of Dry Flow on surface roughness is not supported by the results of this study.

Given the significant additional costs of machining to a high tolerance for surface roughness, it seems unreasonable for this to feature so prominently in the ARM method.

Calculations were made for the Hausner Ratio and the Carr Index, for the LMDPE NP and the PP NP.

The Hausner Ratios were 1.41 for LMDPE NP (ambient ground) and 1.32 for the PP NP (cryogenically ground). According to commonly accepted criteria, (H<1.25), both these powders would be considered to have poor flowability.

The Carr Indices for these two powders were calculated as 29.3 and 24.3 respectively; under normal criteria (C<25), the LMDPE NP would be considered to have poor flowability and the PP NP would be considered to have marginally good flowability.

On first consideration, it would not appear that Hausner Ratio and Carr Index give strong indications of flowability for rotomoulding powders. With an average Dry Flow of 22 sec, the PP NP powder would be considered to have significantly better flowability than the LMDPE NP (average Dry Flow 27 sec).

Conclusions

It is clear that, from all the data that was collected during this study, measurements obtained from the Dry Flow test will be significantly less reliable than measurements obtained from the Bulk Density test.

The care and precision taken during this study, as well as the large size of data sets, make it extremely unlikely that variations can be explained by experimental or operator error.

There are clear indications that general conditions within the Dry Flow test create an environment where chaotic behaviour is present and that this is the most likely cause of the significant data scatter experienced.

Test measurements from apparently identical funnels show a data spread which is wide enough to call into question the use of this test as a quality assurance method.

The variation measured, ie a data spread of 6.4 sec for an above 99% certainty, is probably "as good as it gets" with the current equipment and methodology. In reality, the accuracy that was achieved in these tests is likely to be considerably *better* than that achieved in practice, because the additional factors of operator-to-operator, funnel-to-funnel and inter-laboratory variations were not present in the current study.

We would postulate a maximum variation of approx. 10 sec might be possible, between measurements of the same batch of material. This being so, the use of the Dry Flow test, as a quality assurance measure, would appear to be highly questionable. It seems very unjust that a batch of powder could be rejected by a customer for non-conformance to a Dry Flow specification, when the test measurement error is apparently so extreme.

Using a value for Dry Flow calculated from the results of only three repeats would seem to be highly inadvisable. In the absence of any other modifications to the technique, it would seem appropriate to immediately raise the number of repeats to, for example, 15. In addition to the average Dry Flow, the standard deviation of results should always be calculated. This would enable the reproducibility from batch to batch to be assessed.

Two of the test variables that were tightened in the ARM method were test temperature and funnel roughness. There is no evidence, from the current study, that these variables have any major effect, between the ranges tested.

Bulk Density appears to yield much more reliable and reproducible results than Dry Flow. Setting a powder specification on this parameter would seem to be justifiable. However, there is a danger that just measuring Bulk Density may not allow poor flowing materials to be identified.

From a brief, and admittedly superficial, examination, Hausner Ratio and Carr Index may not be useful characterization parameters for rotomoulding powders. It would seem that most roto powders will fail or marginally pass the currently accepted criteria of $H<1.25$ and $C<15$. Given the relative simplicity of this test, plus the fact that it uses equipment (the Rotap) likely to be readily available, it might be worth considering whether more applicable limits of H and C can be suggested for rotomoulding powders.

It seems clear that, despite efforts to improve the Dry Flow test, the test methods commonly used by the rotomolding industry are not yet sufficiently reliable and reproducible. More work is required to establish the causes of the considerable data scatter that seems, for the moment, inevitable. It would also be beneficial to examine methods used by other industries to characterise powders.

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