ON-LINE MONITORING OF PASTES UNDERGOING EXTRUSION

Russell BD, Wilson DI, Lasenby J* and Blackburn S[†]
Department of Chemical Engineering, University of Cambridge, UK.
*Department of Engineering, University of Cambridge, UK.
[†]IRC in Materials Processing, University of Birmingham, UK.

INTRODUCTION

PASTE EXTRUSION

The manufacture of semi-solid products via paste extrusion is becoming an increasingly important technology in industry. Pastes can be most conveniently defined as a concentrated suspension of solid particulates in liquid (1), where there is enough liquid that it can be moulded readily, but still stiff enough that it retains its shape if there is no applied deformation. Pastes usually feature a solids volume fraction greater than 60%. The two main modes of extrusion are ram extrusion, where the material is presented to the die by a moving piston; and auger extrusion, where the material is fed by rotating screws. Figure 1 shows the configuration of a ram extruder similar to that used in this work.

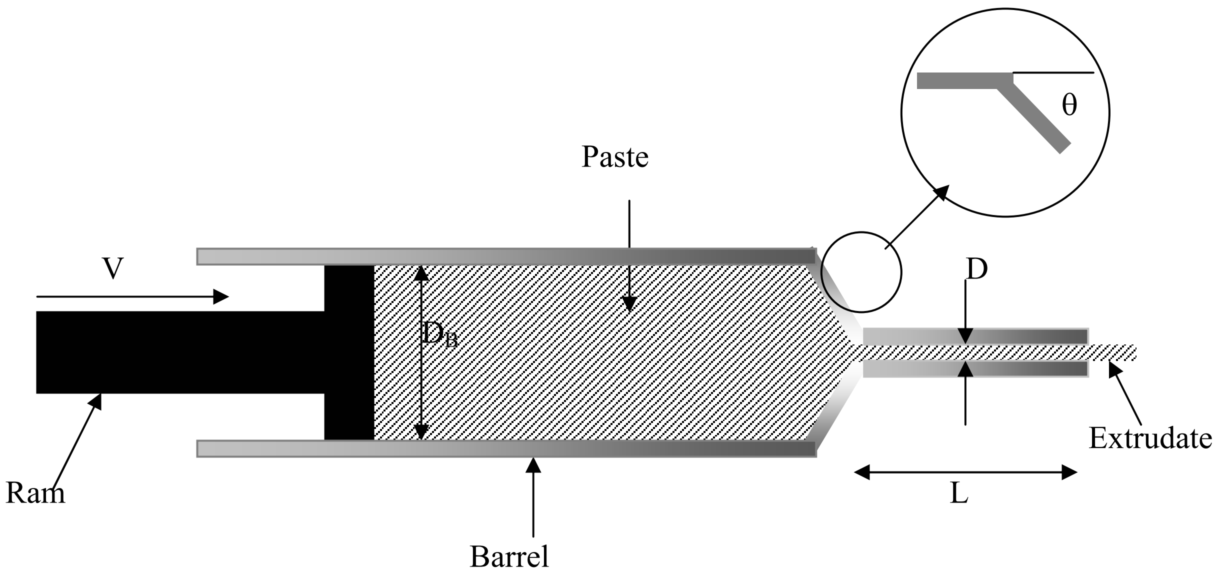

Figure 1 Schematic diagram of a ram extruder. A motor drives the ram at constant velocity, V, into the barrel, extruding the paste through the die land, length L. D_B = barrel diameter, D = die diameter, θ = die entry angle.

The aim of this research is to develop on-line, *in-situ* monitoring techniques for paste extrusion processes, based on analysis of pressure signal data, which are often collected by operators. The central hypothesis is that fluctuations in the observed pressure signal are indicative of the quality of the paste (2), being generated by force events linked to various defect phenomena in the paste. It has been shown that different defects give rise to different trends in the signal (3). The ability to relate a particular trend to a particular defect in the paste forms the basis for monitoring, and ultimately controlling, the process.

EXPERIMENTAL

Pressure transducer data were generated in batch ram extrusion experiments performed on a modified strain frame (Dartec, Stourbridge, UK) fitted with various axisymmetric, circular dies (D_B = 25 mm, D = 3 - 12 mm, θ = 15-90°) fabricated from stainless steel. Pressures were measured by normal stress transducers (Kulite, Basingstoke, UK) recorded at 100 Hz on a PC data-logging system and analysed afterwards using MATLAB™ routines. Results are reported for three different pastes: (*i*) an α-alumina based ceramic catalyst paste mimicking a catalyst formulation, termed Mix 25; (*ii*) a starch-based food paste, provided by United Biscuits; and (*iii*) a detergent paste, provided by Procter & Gamble containing bentonite clay, LAS powder and sodium citrate. The extrusion behaviour of these materials exhibits noticeable yield stress and wall slip, and can be characterised by the Benbow-Bridgwater approach (1). A detailed description of their gross extrusion behaviour is given elsewhere (2),(4).

ANALYTICAL TECHNIQUES

OUTLIER ANALYSIS

Agglomerates are one of the major sources of extrusion defects in pastes. These have been shown to cause the pressures measured at the die to fluctuate strongly (5). Böhm & Blackburn (6) reported that pastes containing large numbers of agglomerates exhibited many sharp peaks in the pressure signal. Entrapped air is also an important source of defects, and Amarasinghe (2) showed that bubble collapse was accompanied by sharp decreases in extrusion pressure, or 'pits'. Outlier analysis can therefore be a useful technique for quantifying both pits and spikes. Defect content can be correlated with counts of outlier events.

SPECTRAL ANALYSIS

Surface fracture is also a common problem in paste extrusion. One of the most common types is circumferential fracture, where the break runs around the circumference of the extrudate normal to the axis, as shown in Figure 2.

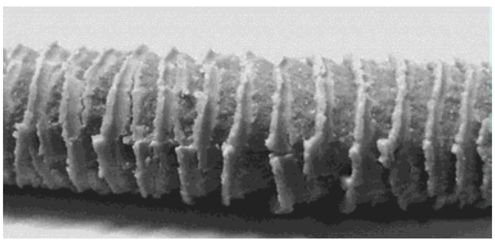

Figure 2: Circumferential surface fracture of detergent paste. D = 9 mm, V = 0.5 mm/s.

It can be seen in Figure 2 that the distance between these fracture lines is approximately constant. Domanti (7) found that the period of fracture is approximately equal to half the die diameter D. For steady extrusion, the frequency at which fracture will occur, ω_F, is expected to be given by:

$$\omega_F\,(Hz) = V\left({D_B}/{D} \right)^2 (S)^{-1} \tag{1}$$

where S is the distance between the fracture lines. Because the fracture lines are periodic, it is believed that a periodic fracture mechanism is responsible (8). A useful technique for detecting signals of a given frequency is the Fourier transform (9), which transforms a time-based signal to a frequency-based one.

FRACTAL ANALYSIS

A fractal-based approach was used by Fan *et al.* (10) to quantify the highly stochastic characteristics of three-phase fluidised beds. Hurst's rescaled range analysis (11) was used to yield an estimate of the Hurst Parameter, H. H lies between 0 and 1. For $H < 0.5$, the process is said to be anti-persistent (the local magnitude of the signal is comparable with total magnitude). For $H > 0.5$, the process is said to be persistent: purely Brownian motion yields an H value of 0.5. In the context of paste extrusion, Brownian motion is expected to indicate a perfectly homogenous paste. A paste that gives rise to a noisy signal, due to local inhomogeneities, will yield a Hurst Parameter less than 0.5, while $H > 0.5$ will signify a relatively smooth pressure profile, usually due to a low signal-to-noise ratio, sometimes associated with high mean extrusion pressures.

STANDARD ERROR ANALYSIS

A complementary technique to fractal analysis is standard error analysis, which quantifies deviations from an average value and therefore also indicates homogeneity. This is expressed here in two forms: *global deviations*, which quantify the deviations in the pressure signal from the mean extrusion pressure of the sample; and *local deviations*, which quantify the deviations from a moving average. Both are expressed as a percentage of the mean extrusion pressure, to give a form of coefficient of variation. This is illustrated in Figure 3 and equation 2.

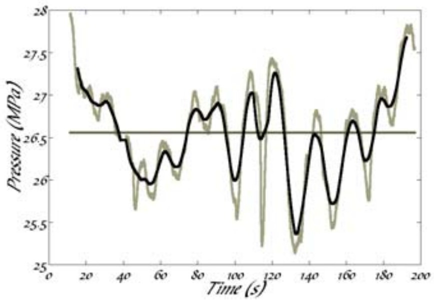

Figure 3: Basis for calculating local and global deviations for extrusion sample. Source: detergent paste, L/D = 36/3 mm/mm, V = 0.5 mm/s. Pressure signal, mean extrusion pressure, moving average.

$$global\ deviations = \left(\frac{pressure\ signal - mean\ extrusion\ pressure}{mean\ extrusion\ pressure} \right) \times 100\%$$
$$local\ deviations = \left(\frac{pressure\ signal - moving\ average}{mean\ extrusion\ pressure} \right) \times 100\% \qquad (2)$$

As with fractal analysis, one would expect that different pastes should feature different standard errors. When calculating the local deviations, an important factor is the selection of an appropriate time period. In the work reported here, the period is based on the time taken by the paste to pass through the die entry region, which has proven to be a suitably characteristic space time.

RESULTS

OUTLIER ANALYSIS

A series of experiments performed using different operating conditions and die geometries showed little variation in outlier density, except when small die diameters were used. At diameters less than 4 mm, the number of spikes for Mix 25 increased with decreasing diameter, as shown in Figure 4. Böhm & Blackburn (6) carried out similar experiments over a similar range of die diameters, and reported that the agglomerates were most efficiently broken down using the smallest diameter, (1 mm in their work). This result is consistent with the data shown in Figure 4. The low spike densities for the detergent and starch pastes in Figure 4 indicates that these materials do not contain as many stiff agglomerates as Mix 25, which is consistent with their formulations. It should be noted that the largest average particle size present in the component powders was 35 µm, so that bridging or other structural effects were unlikely to arise in dies of diameter > 1 mm.

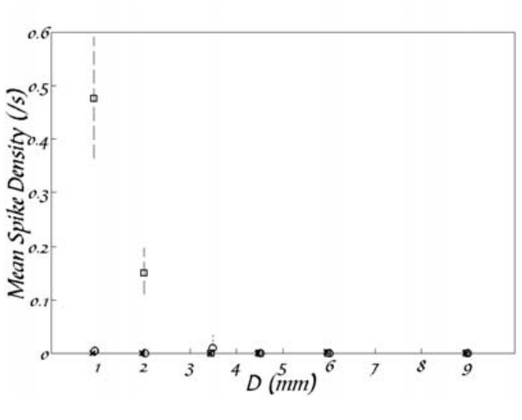

Figure 4: Mean spike density as a function of die diameter for the three different pastes. V = 1 mm/s, angle = 90º, L = 12 mm. × - starch, □ - Mix 25 (ceramic), o – detergent.

STANDARD ERROR ANALYSIS

Figure 5 shows the variation in the global deviation parameter with four process or geometrical parameters. The local deviations showed similar trends. It can be seen that the starch paste generally displayed the 'smoothest' signal, with the smallest standard error. The detergent and Mix 25 paste were both 'noisier', with Mix 25 usually having the greatest standard error.

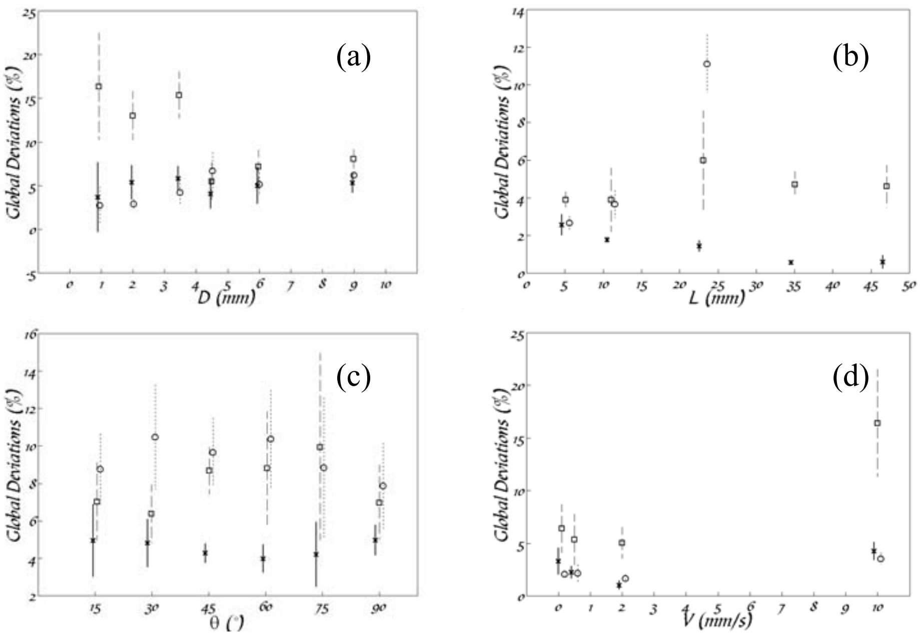

Figure 5: Variation in global deviations with (*a*) die diameter, *D,* (*b*) die land length, *L*; (*c*) die entry angle, θ, (*d*) extrusion rate, *V*.
× - starch, □ - Mix 25 (ceramic), o – detergent.

There are no trends evident in each of the Mix 25 and starch parameters presented in Figure 5. The high standard errors for Mix 25 with low diameters in Figure 5(*a*) are linked to the spikes generated by agglomerate breakdown discussed previously. With the detergent paste in Figure 5(*b*), the deviations increased with increasing die land length. In this case, the extrusion pressures were very large, and it was not possible to extrude the material at larger *L/D* values in the apparatus. Very unusual behaviour was observed during extrusion at the larger *L/D* values tested, giving rise to increased global deviations. These effects are linked to changes in temperature and paste density.

Comparison of the three materials indicates that the standard error is paste-dependent and relatively process-independent, suggesting that this type of analysis may be useful as a control reference in industrial processes.

SPECTRAL ANALYSIS

The extrudate shown in Figure 2 featured fracture lines with a periodic spacing of approximately 2 mm, giving an expected fracture frequency from Equation (1) of 3.9 Hz. The frequency spectrum of the signal in Figure 6 shows a peak around 3.9 Hz, which indicates that there are periodic stresses present in the signal corresponding to the occurrence of circumferential fracture.

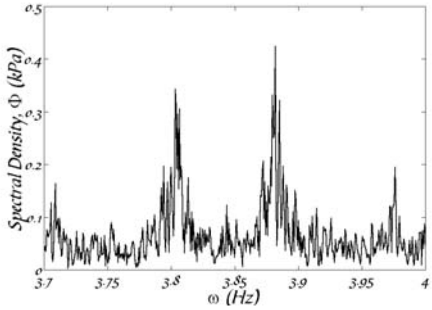

Figure 6: Spectral density of detergent paste with an expected fracture frequency of 3.9 Hz.

One possible way of further investigating surface fracture using spectral analysis may be to map the time-frequency behaviour of the signal. The Fourier Transform, used to produce Figure 6, assumes that the forces at the different frequencies are always present, which is not necessarily the case in paste extrusion. For example, the circumferential fracture lines observed are often next to smooth areas of the extrudate, leading to the possibility that the peaks in the frequency spectrum change with time. Wavelets (12) offer the best method for analysing the time-frequency behaviour and are currently being considered.

FRACTAL ANALYSIS

A fundamental challenge with fractal analysis of the paste extrusion data collected to date has been the presence of sinusoidal components in the signal, which are known to be non-fractal (13). One method (Crossover Method) proposed by Van der Walle & Ausloos (14) is based on a superposition algorithm, and has been shown to be effective in estimating fractal exponents when different trends, including periodic trends, are present. However, this has been found to present problems with applied to these pressure signals, as the superposition technique is only feasible if the data do not display spectral decay, which has been found to be the case with our data.

An alternative method of estimating the fractal characteristics of data featuring spectral decay is to relate a decay exponent to a parameter such as the Hurst Parameter, as discussed by Heneghan and McDarby (15). For such data, the spectral density, Φ, is proportional to the frequency, ω, to the power of some exponent, α, termed the decay exponent. The Hurst Parameter is then directly proportional to the decay exponent, but contains an intrinsic error (14) which may render it too coarse for single-variable control. However, it is thought that it can be used in conjunction with other variables, such as the local deviations, as a basis for multi-variable control. This is discussed in the next section.

BENCHMARKING

For effective control, it is important to have an understanding of the expected characteristics of the pastes, how these will vary between different states or behaviours of a single material, and between different materials. For each of the pastes studied in this work, the quantitative parameters described above have been found to exhibit a degree of variation, even when the process has been behaving satisfactorily. Poorly behaved systems are therefore more readily identified by comparing cross-plots of data, and looking for data lying outside the 'confidence cluster'. This approach is illustrated in Figure 7, where two of the parameters expected to indicate homogeneity are plotted (in dimensionless form) for the three pastes.

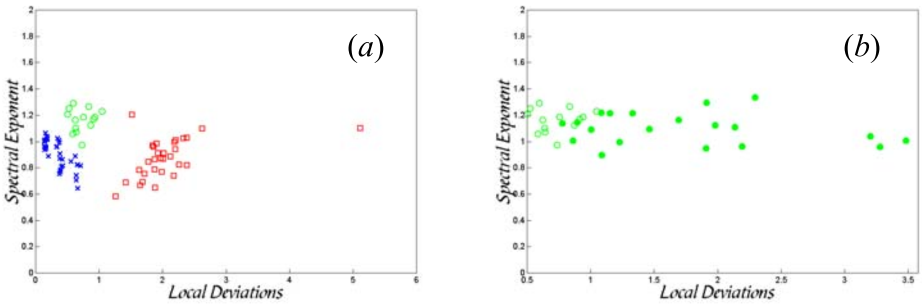

Figure 7: Spectral exponent and local deviation correlation, with variation in die length: *(a)* well-behaved pastes ; *(b)* detergent, with outliers.
× - starch, □ - Mix 25 (ceramic), o – detergent: solid symbols - outliers

It can be seen that the spectral exponents for each paste data set lie in a band, which varies with each paste, although there is some overlap. The (dimensionless) local deviations also lie in similar bands, despite the absolute values of the pressure fluctuations varying by over one order of magnitude. Figure 7(*a*) shows evident cluster behaviour for 'well-behaved' pastes. The effect of poor processing in shown in Figure 7(*b*), where data from poorly behaved tests have been added in as outliers. The change in detergent paste behaviour is evident, although the spectral exponents all lie in the same band while the local deviations have definitely changed. This indicates that as well as relatively poor resolution of the fractal exponent, it may not be sensitive to changes in paste properties. It is believed that wavelets, as well an alternative to spectral analysis, will enable estimation of a fractal exponent more sensitive to the paste homogeneity. This has been the case in other areas of work (16).

CONCLUSIONS

Signal processing techniques have been developed to characterise quantitatively the pressure fluctuations observed in ram extrusion data collected from three different paste materials. Outlier analysis has been shown to be a useful tool for monitoring the presence of agglomerates in the paste, as well as detecting the bursting of pockets of entrapped air. Standard errors, in both the local and global deviations, were found to depend strongly on paste condition and process phenomena, indicating that these techniques present opportunities for control applications.

Spectral analysis can be used to detect the presence of forces involved with circumferential fracture, but more work, such as analysing the time-frequency behaviour, is required for it to be applicable to paste extrusion. Fractal analysis has been found to be insensitive as a single measure of paste inhomogeneity, but has potential as one component in multi-variate analyses, particularly if wavelets are used as the basis for estimation.

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