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Single screw extrusion of mullite-based tubes containing Al-rich anodising sludge

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Abstract

An Al-rich anodising sludge has been used as the feedstock to produce mullite-based tubes by single screw extrusion. Extrudability and plasticity of the pastes was shown to be strongly dependent on the amount and type of used additives. The pastes developed from the sludge could be characterised by the Benbow–Bridgwater relationship using six paste parameters and this characterisation was extended to predict the behaviour of the screw extrusion process.

In this work, the effects of the screw speed and partial pressures in relevant zones of a single screw extruder on the processing behaviour of sludge-based pastes were predicted using the Benbow–Bridgwater model. It is shown that the model can predict the pressure drop through the die-entry and the die-land regions (with similar design as used to produce ceramic hole-tubes) and the pressure development along the barrel of the extruder with reasonable accuracy. The paste parameters calculated from simple ram extrusion do not match the experimental behaviour of the screw extruder and this was proved to be due to the paste being worked to a stiffer consistency in the barrels of the extruder and to being subjected to vacuum during the process, with consequent water segregation. This was confirmed by re-examination of the paste properties immediately after the screw extrusion.

Keywords: D. Mullite; Al-rich sludge; Screw extrusion; Modelling

1. Introduction

Mullite ($3Al_2O_3 \cdot 2SiO_2$) is one of the most important property-determining phases of traditional clay-based ceramics. Often mullite is the main constituent of pottery, porcelain, and sanitary-ware products [1]. Additionally it is also commonly present in advanced or technical ceramics, particularly those produced for structural applications. It exhibits low thermal conductivity, good creep resistance, reasonable chemical stability, and high-temperature resistance, which are prerequisites for refractories and electronic and optical devices [1–4].

Several authors show that the strength and toughness of mullite ceramics depends on the amount of glassy phase in the composite matrix, the exact relationship is dependent on the composition and green density of the body. Density is primarily affected by the grain size distribution and by the selected ceramic shaping process. There is evidence in the literature [5,6] that screw extrusion assures high green-packing density and good microstructural homogeneity which, in turn leads to enhanced densification and final proprieties such as improved bending strength and reduce water absorption.

Benbow and Bridgwater demonstrated that the extrusion of particulate pastes, comprising fine particles suspended in a liquid continuous phase, through dies with circular cross section and having a square entry, can be described by Eq. (1) [7]:

$$P = P_e + P_1 = 2(\sigma_0 + \alpha V^n)\ln\left(\frac{D_0}{D}\right) + (\tau_0 + \beta V^m)4\left(\frac{L}{D}\right) \quad (1)$$

where α is a velocity-dependent factor for the convergent flow, β is the velocity-dependent factor for parallel flow, n and m are

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exponents, σ_0 is the paste bulk yield value, τ_0 is the paste characteristic initial wall shear stress, D_0 and D are the diameters of the barrel and of the die, respectively, L is the die-land length and V is the extrudate velocity [7]. In this equation, die-entry (P_e) and die-land (P_l) pressures are separated.

A few years later, Burbidge and Bridgwater developed from the equations of Benbow–Bridgwater a model for the prediction of pressure generation in a single screw extruder [8,9]. In their model it was assumed that the screw has constant pitch and channel depth, and perfectly fits the barrel. This means that there is no leakage between the edges of flights and the constraining barrel wall. The model considers the flow by combining two aspects: a geometric relationship between the barrel and paste plug velocities and a force balance around the paste plug [9,10]. The model was developed further by Engländer et al. [10] to account for variable pitch and channel depth.

The adaptation of Benbow–Bridgwater equations [9,10] to describe the balance of forces for a measured pressure drop along the screw extruder channel Ps results in:

$$\mathrm{d}P_s = \frac{C_b(\tau_0 + \beta V_r^m)\cos(\varphi + \phi) - C_s(\tau_0 + \beta V_p^m)}{A \sin\phi}\,\mathrm{d}x \qquad (2)$$

where C_b is the barrel contact area and C_s is the screw contact area, both per unit length, V_p is the paste velocity vector, V_r is the vector that results from the difference between the barrel velocity and the paste velocity vectors, β is a wall velocity factor, ϕ is the helix angle of the screw, τ_0 is the wall shear yield stress, m is an index associated with the flow along the die land, A denotes the cross-sectional area of the channel, and φ is the flow rate angle made by the movement of the paste in relation to the rotation plane, i.e. the angle between V_r and V_p [9,10].

In the present work, mullite-based tubes were extruded using a ceramic paste containing Al-sludge (generated in the wastewater treatment of aluminium anodising units) as the main component. Diatomite (a by-product of sand mining, commonly found in Portugal) and natural clay-based raw materials (kaolin and ball clay) were used to adjust the composition and the plasticity level of the pastes. Processing conditions were optimised by adjusting the operation parameters (tube-die size and shape), and by using rheological modifiers (plasticizers and lubricants) with the idea to simulate the industrial ceramic tubes production. The applicability of the Burbidge–Bridgwater model to this type of extrusion was verified from ram extrusion experiments [11]. Prior to the extrusion tests, the plastic behaviour of the pastes was characterised by interpreting stress–deformation curves and comparing that data with standard pastes prepared industrially [12].

2. Experimental

The mullite paste (MP) was prepared from a premixed powdered material batch containing 42 wt.% of pre-calcined (at 1400 °C) Al-anodising sludge (Extrusal, S.A., Aveiro, PT), 15 wt.% ball clay (BM-8, Leiria, PT), 15 wt.% kaolin (Mibal-B, Barqueiros, PT), and 28 wt.% diatomite (Anglo-Portuguese Society of Diatomite, Óbidos, PT). The full characterisation of the raw materials and some details of preparation and characterisation of the sludge have already been reported [13,14]. In order to adjust and generally increase the plasticity level, commercial additives were used: two plasticizers, Zusoplast PS1 and Zusoplast C28 and a lubricant, Zusoplast O59, all from Zschimmer & Schwarz, D, were added to give six pastes, one containing just the powder blend and 31.0 wt.% water, one with 1.0 wt.% C28, 0.5 wt.% O59 and 32 wt.% water, three; where 2 wt.% PS1, 2 wt.% O59 were fixed and the water set at 29.4. 30.5 and 32 wt.% and finally 4 wt.% PS1, 2 wt.% O59 and 32 wt.% water.

The yield value and plasticity level of the paste was measured from stress–deformation tests carried out by plastic compression (Lloyd Instruments LR 30 K) in metal moulds, using a method fully reported in the literature [12]. Thermogravimetric analysis (TGA) of fine powders was performed in air (Netzsch 402 EP, Germany, heating rate 10 °C/min).

Tube-die extrusion was performed in a pilot-scale screw extruder with two single screws in series separated by a vacuum chamber (see Fig. 1). Tests were made at different screw rotational velocities and the pressure was measured in the die-entry zone. Fig. 2 details the die region and shows the location of the pressure sensor and all the contributing partial pressure drops. Pressure values in the die-entry region (P_{de}) were measured through a digital sensor specially developed for the

(A) (B)

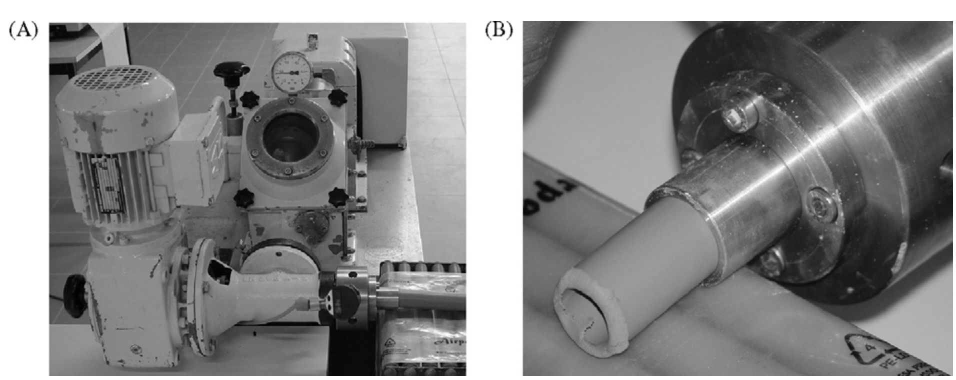

Fig. 1. Photographs of the pilot-scale screw extruder (A) and extrudate exit (B).

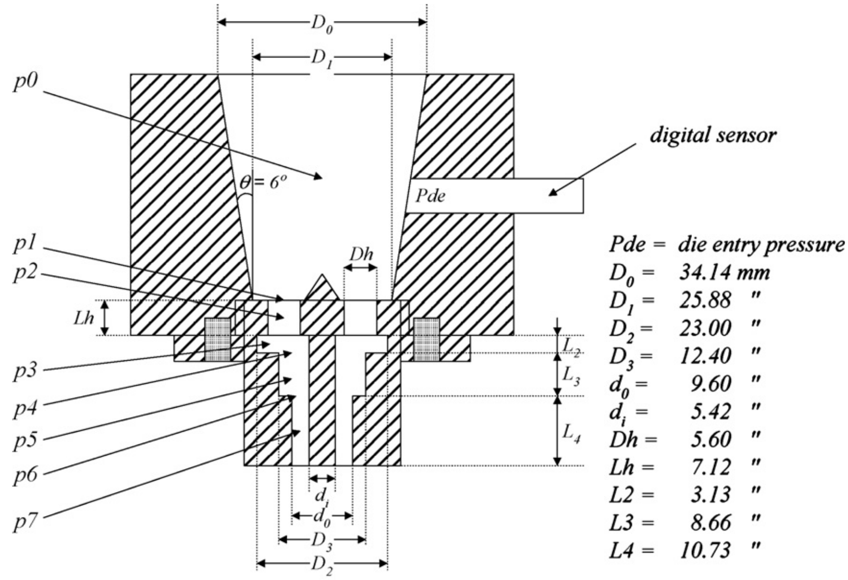

Fig. 2. Detailed representation of the die used for ram and screw extrusion of hole-tubes.

project, which was connected with a registration system. The pressure sensor was calibrated by IFM-electronic (Germany).

In order to check the paste properties the Benbow–Bridgwater parameters were measured on the feed paste and on the resultant extrudate in a ram driven capillary system. The methodology for the determination is given in the literature [11].

3. Results and discussion

3.1. Stress–deformation behaviour

The plastic deformation of MP pastes, with or without lubrication and plasticizer additions were obtained by confined compression. The resulting curves are shown in Fig. 3. The paste without additives exhibited low levels of plasticity [12]. Paste formulations exhibiting this curve shape tended to develop problems during extrusion with unacceptably high pressures and wear of the equipment. The plastic deformation region was very narrow and yield stress values rather high (≈0.26 MPa).

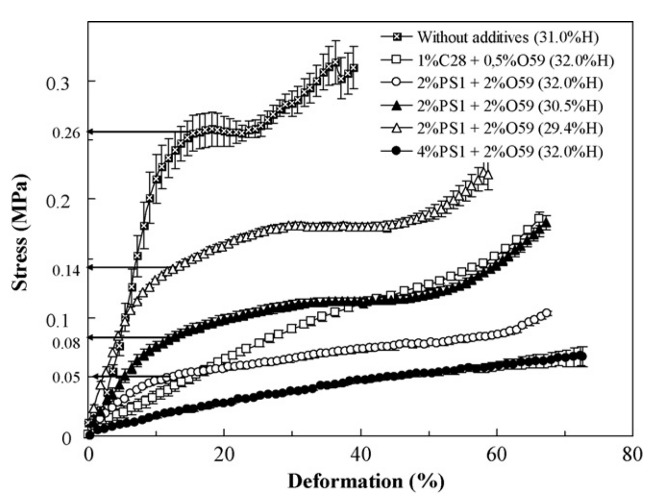

Fig. 3. Stress–deformation curves of tested formulations, obtained by plastic compression.

As expected, the use of commercial additives modified the plastic behaviour of the pastes significantly, by decreasing the yield stress and enlarging the plastic region. Fig. 3 shows that acceptable plastic behaviour for the MP pastes was reached by adding 2 wt.% PS1 + 2 wt.% O59, for a moisture content around 32 wt.%. This paste is herein referred as MP/2P2L32H. The moisture value is still rather high when compared with those normally used for processing common industrial pastes (e.g., around 23% for porcelain) [12], and thus careful control in the subsequent drying step was required to avoid the formation of defects (deformation or cracks) in the extruded tubes. However, the leaner character of actual formulations did not permit extrusion at moisture contents below 30%. Small variations in the moisture content caused significant changes in the plasticity level and yield stress values, as can be observed for the pastes prepared with 2% of plasticizer and 2% of lubricant. This is illustrated for example, by a 2.6% variation in the moisture content inducing a change from 0.05 to 0.14 MPa in the yield stress value. The yield stress values shown in Fig. 3 were then used as an input parameter for extrusion modelling to fix σ_0.

Fig. 4 shows TGA curves of paste with and without organic extrusion-add additives (plasticizer and lubricant agents). The elimination of those organics additives induces an extra weight loss (3%) between 250 and 480 °C, deserving a careful control of the heating rate in order to prevent the generation of defects. Typically, the pre-heating and heating zones of the industrial kiln should be re-designed to facilitate degassing reactions. The other two weight losses observed in TGA curves, at 100–200 °C and 500–580 °C, are due to the typical removal of free water and to the dehydroxylation of clay minerals, respectively.

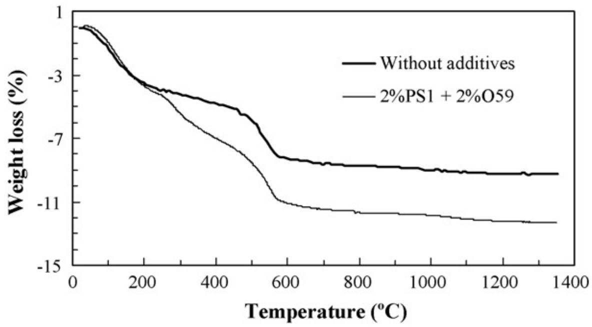

Fig. 4. TGA curves of dried samples of MP extruded bodies without and with organic additives (2% plasticizer PS1 and 2% lubricant O59).

Table 1
Relevant extrusion parameters of the MP/2P2L paste obtained in a ram extruder, for the pastes before and after screw extrusion

Parameter	Before extrusion MP/2P2L32H	After extrusion MP/2P2L30.5H
α [MPa (s m^{-1})^n]	1.061	0.428
n	0.77	0.03
β [MPa (s m^{-1})^m]	0.099	0.101
m	0.217	0.213
σ_0 (MPa)	0.05	0.08
τ_0 (MPa)	0.00005	0.00136
μ	0.001	0.017

3.2. Extrusion characterisation

Table 1 lists the six extrusion parameters determined from tests conducted with the same paste immediately before and after screw extrusion. This procedure promotes a decrease in the moisture level of the extrudate (from 32.0% to 30.5%), probably due to heating and water segregation, which will alter the paste rheology. It can be seen in Fig. 3 that reducing the water content by 1.5 wt.% increases the yield by 37% (0.05–0.08 MPa). The parameters most influenced by this change are τ_0, and σ_0 and to a lesser extent α, inducing a significant increase of the *static friction coefficient*, μ, for the extrudate ($\mu = \tau_0/\sigma_0$). A full description of the procedure and results refinement is described elsewhere [11].

Eq. (2) gives the pressure drop along the extruder barrel for a single screw extruder. In practice, the pressure drop builds to a maximum near the end of the screw, summing all the contributions along dx. Fig. 5 shows the geometrical characteristics of this screw, and shows the helicoidal shape of the channel. For screws with this configuration C_b is given by [9]:

$$C_b = 2h\left(1 + \frac{h}{D_s}\right) + \pi D_s\left(1 - \frac{2h}{D_s}\right)\sin\phi \quad (3)$$

where h is the channel depth (h = 6 mm) and D_s is the diameter of the screw (D_s = 26 mm) at the flight tip. The paste velocity vector, V_p, is defined by [9]:

$$|V_p| = \frac{\pi D_s N_s \tan\varphi}{(\tan\varphi + \tan\phi)\cos\phi} \quad (4)$$

where N_s is the screw rotation speed. This velocity can be related with the extrusion flow (Q) by [9]:

$$Q = \frac{\pi}{4}[D_s^2 - (D_s - 2h)^2]\frac{\pi D_s N_s \tan\varphi\tan\phi}{\tan\varphi + \tan\phi} \quad (5)$$

The vector that gives the difference between the barrel velocity and the paste velocity vectors V_r, is then given by:

$$|V_r| = \frac{\pi D_s N_s \tan\phi}{(\tan\varphi + \tan\phi)\cos\varphi} \quad (6)$$

By combining the last equations with Eq. (2), we can obtain the expression that gives the pressure drop variation (dP_s) along the length of the screw channel (x):

$$\mathrm{d}P_s = \frac{\begin{array}{c}[2h((1)+(h/D_s)) + \pi D_s((1)-(2h/D_s))\sin\phi(\tau_0 + \beta \\ ((\pi D_s N_s \tan\phi/((\tan\varphi+\tan\phi)\cos\varphi)))^m)\cos(\varphi+\phi)] \\ - C_s[\tau_0 + \beta((\pi D_s N_s \tan\varphi)/((\tan\varphi+\tan\phi)\cos\phi))^m]\end{array}}{A\sin\phi}\,\mathrm{d}x \quad (7)$$

In an ideal paste, the extrusion parameters defined by Benbow–Bridgwater (β, m and τ_0) are independent of the geometry through which the paste passes and thus reflect the intrinsic behaviour of the material. It is therefore legitimate to transpose the parameters determined by ram extrusion [11] into the geometrical situation presented here for the screw and die assembly. Fig. 6 shows a plot of P_s for various rotational speeds plotted as a function of the volumetric flow rate for paste MP/2P2L32H, obtained by applying Eq. (7). The pressure drop for the conical entry section of the die assembly can be calculated from [15]:

$$p_0 = \left[2(\sigma_0 + \alpha V^n + \tau_0\cot\theta)\ln\left(\frac{D_0}{D_1}\right) + \beta V^m\cot\theta\right] \quad (8)$$

where θ is the angle of die-entry region. The results from Eq. (8) can be converted to plot P as a function of Q, these results are plotted onto Fig. 6. Any values of screw performance above the

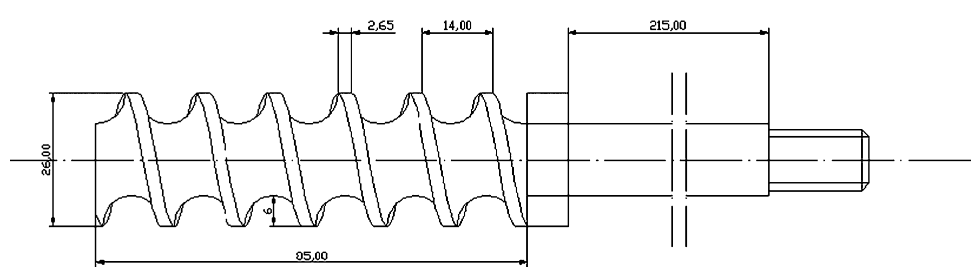

Fig. 5. Detailed view of the screw placed before the die for single screw extrusion of tubes with vacuum (helix angle of the screw, Φ = 10°).

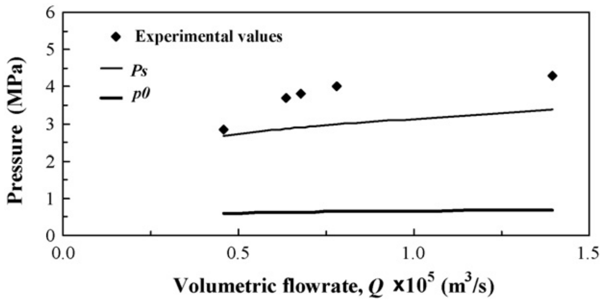

Fig. 6. Predicted along the screw pressure generation (P_s) and pressure drop through a conical die entry (p_0) and the full die during the extrusion of MP/2P2L32H (calculated).

plotted line for the die entry pressure drop will allow extrusion when the tube die is not present. The screw will self regulate to match the feed rate to the extrusion requirements. Adding a die to the end of the conical section will increase the pressure required to force the paste through the system. According a previous work [11], it was demonstrated that a recycled paste can be extruded trough complex die geometry for the production of ceramic hole-tubes, where the pressure drop for the full die system is given by:

$$\begin{aligned} P_{\mathrm{dl}} &= p_1 + \cdots + p_7 \\ &= \left[2\left(\sigma_0 + \alpha\left(\frac{4Q}{\pi Dh^2 N}\right)^n\right)\ln\left(\frac{D_1}{Dh\sqrt{N}}\right)\right] \\ &\quad + \left[4\left(\tau_0 + \beta\left(\frac{4Q}{\pi Dh^2 N}\right)^m\right)\left(\frac{Lh}{Dh}\right)\right] \\ &\quad + \left[(\tau_0 + \beta V^m)\left(\frac{L_2 M_2}{A_2}\right)\right] + \left[\ln\left(\frac{A_2}{A_3}\right)(\sigma_0 + \alpha V^n)\right] \\ &\quad + \left[(\tau_0 + \beta V^m)\left(\frac{4L_3}{D_3 - d_i}\right)\right] + \left[\ln\left(\frac{A_3}{A_4}\right)(\sigma_0 + \alpha V^n)\right] \\ &\quad + \left[(\tau_0 + \beta V^m)\left(\frac{4L_4}{d_0 - d_i}\right)\right] \end{aligned} \tag{9}$$

where N is the number of internal holes with diameter Dh, A_x is the area at location x, M_x is the perimeter length at the location x and L_x is the die-land length at location x.

The pressure drops associated with each section of the die can be calculated to show their individual contribution to the

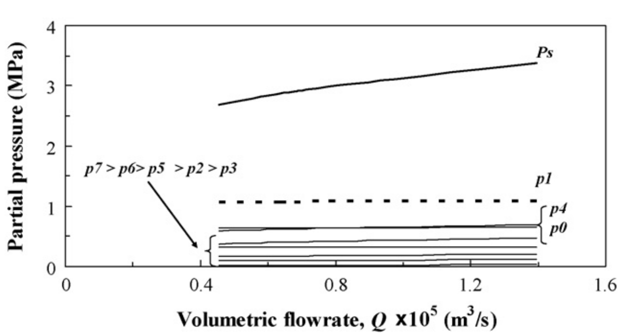

Fig. 7. The component pressures through the die as predicted by each part of Eq. (9) for paste MP/2P2L32H.

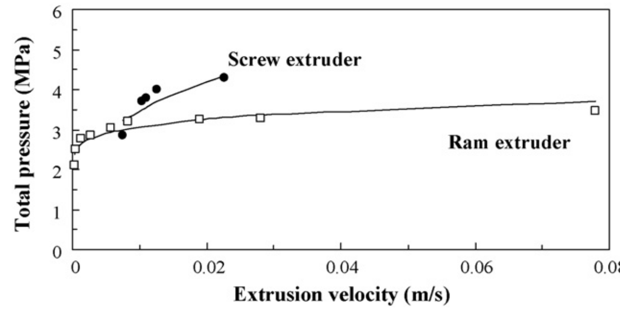

Fig. 8. Measured pressure for ram extrusion and screw extrusion through the same die, pressure being measured at the same location in the die. Measured (points) and fitted (lines) results are given.

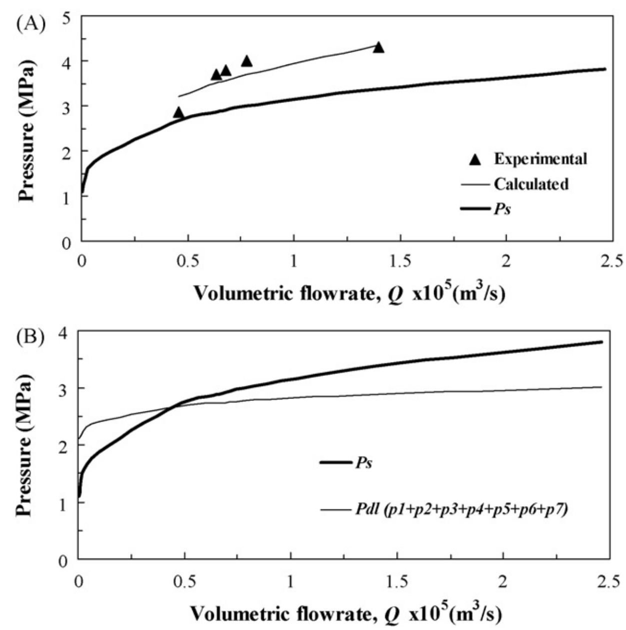

Fig. 9. (A) Predicted pressure generation in the screw and pressure loss through the die calculated using the post extrusion paste parameters. (B) Comparison between the screw pressure (P_s) and the pressure in the die length (P_{dl}).

overall die pressure drop. These relationships are shown in Fig. 7. It can clearly be seen that each sections contribution is different. The major contributors in the die are the partial pressures p_1, p_4 and p_0 which are the entrances to the constriction zones on the tube forming die. The resulting values have been plotted in terms of volumetric flow rate as in Fig. 6. As can be seen the screw will have to work harder to force the material from the die. For example to extrude the material at

Table 2
Fitting of screw extrusion results of the MP/2P2L32 paste according to Eqs. (8) and (9)

$Q \times 10^5$ (m^3/s)	P experimental (MPa)	Fitting (Eqs. (8) + (9))	
		P (MPa)	Error (%)
0.45647	2.85	3.21	12.9
0.63575	3.70	3.50	5.4
0.67698	3.80	3.55	6.4
0.77958	4.00	3.69	7.6
1.39520	4.30	4.34	1.0

Table 3
Comparison of relevant properties of sintered MP-bodies processed by extrusion and dry pressing [13]

Process	Sintering temperature (°C)	Bending strength (MPa)	Linear shrinkage (%)	Water absorption (%)	Apparent density (g/cm^3)
Extrusion	1450	121.5 ± 12.7	12.0 ± 0.1	7.3 ± 0.6	2.20
	1550	143.8 ± 11.8	13.9 ± 0.4	3.3 ± 0.3	2.37
	1650	154.0 ± 16.7	14.9 ± 0.6	0.04 ± 0.0	2.57
Dry pressing	1450	35.4 ± 2.3	15.4 ± 0.2	9.7 ± 0.7	2.13
	1550	37.9 ± 2.6	16.4 ± 0.3	7.5 ± 0.9	2.23
	1650	58.5 ± 2.6	19.0 ± 0.8	0.7 ± 0.3	2.50

4.56×10^{-6} m^3 s^{-1} and have a total pressure developed round 3 MPa, it will be necessary to run the screw at a relatively high rotation rate (≈130 rpm).

The pressure drops measured for both the screw extruder under examination here and for the die being fed by the ram extruder are shown in Fig. 8. For any given extrudate velocity the pressure required for flow through the die must be the same in both cases for the same paste. Fig. 8 plots the predicted pressure drop for the die using Eqs. (8) and (9). The results show that it fits the ram extrusion data well but not the screw extrusion data. It can be seen that there has been a significant increase in the extrusion pressure required for the screw extruder and this confirmed that there is some difference in the rheological properties of the material after work has been input by the screw. The data for the screw is limited due to the available operating conditions. This pressure rise could be induced by batch-to-batch inconsistency, by water segregation during the extrusion process or due to further mixing of the paste by work being applied during screw conveyance. To evaluate these possibilities the paste rheology was measured before extrusion through the screw and after passing through the extruder. The results are shown in Table 1. It can be seen that there has been a stiffening of the paste through the process, with a significant alteration on the σ_0 and τ_0 values. If these post-extrusion parameters are used to create a plot similar to Fig. 6 (see Fig. 9A) the predicted pressure developments and die pressure drops lie closer to the pressure values measured during screw extrusion. Thus the hypothesis that the paste properties were modified by the screw extrusion process is upheld. Table 2 shows the improved level of agreement between experimental values and predicted pressures calculated trough the Eqs. (8) and (9). The pressure predicted to be generated by the screw and the pressure predicted to be lost in the flow through the die assembly are shown in Fig. 9B. It can be seen that they show similar trends and cross at an acceptable operating condition. This is encouraging for screw design. p_0 can also be added to P_{dl} and this simply moves the line upwards and the intercept to a slightly higher value.

It is suggested that the screw model underpredicts the pressure development in this system and this is not surprising as the system is in fact rather more complicated than the geometry input into the model. There are some errors associated with the equation fitting particularly at low velocities and the velocities in the screw are relatively low compared to the velocities used in the capillary measurements used to determine the rheological parameters. These errors may also systematically contribute to the discrepancy between the die pressure drops and the screws pressure generation at any given volumetric flow rate. However, for such a simple approach to a complex situation, both geometrically and rheologically, the predictions are reasonable.

Table 3 shows some relevant characteristics of optimised extruded and sintered MP-bodies. For comparison, results of pressed samples are also given. Extruded bodies show higher densification in the as-shaped or green condition and, as a consequence, the sintering process is more effective, as clearly denoted by comparing the bending strength evolution. Accordingly, linear shrinkage and water absorption values tend to be lower, while the apparent density is higher.

4. Conclusions

Al-sludge containing mullite formulations were studied and optimised to process tubes by extrusion through a pilot-scale screw extruder. The control of the plastic behaviour was reached by adding commercial additives (plasticizer and lubricants) and assessed by performing stress–deformation curves under compression.

The Benbow–Bridgwater model was addapted to fit the pressure evolution through a single screw extruder. The water segregation detected during the screw extrusion changes the paste rheology (mostly the τ_0 and σ_0 parameters), this was confirmed by re-examination of the paste properties immediately after extrusion. The model shows that individual contributions to the overall die pressure drops were significantly different, and that the major contributors are the entrances to the constriction zones.

It is shown that by using the corrected paste rheological data the pressure geration and pressure loss through the system are in balance. Thus the model can be used in a predictive capacity.

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