

Rheological studies on cordierite honeycomb extrusion

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Abstract

The rheological parameters for a cordierite honeycomb extrusion were investigated using the Benbow–Bridgwater model. The extrusion pastes were formulated using typical binders, plasticizers, lubricants and pore-forming agents normally used for such extrusions. Pastes were found to have good pseudo-plasticity, low bulk yield and very low die-land shear stress, compared with the velocity dependent components of the respective pressure drops. In some formulation, reduced velocity contribution to the extrusion pressure with increased binder content found useful for better honeycomb extrusion. These characteristics of the honeycomb extrusion batches were analyzed by using dynamic bulk and shear stress components derived from the Benbow–Bridgwater six-parameter model and compared with the results of honeycomb extrusion trials. Plasticizing agents like polyethylenglycol (PEG) and glycerin were found to decrease the die entry pressure but showed no external lubricating effect leading to higher die-land pressure. Pore-forming agent, graphite was found to reduce both die entry and die land pressures, whereas, carbon increases the die entry pressure. © 2002 Elsevier Science Ltd. All rights reserved.

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1. Introduction

A cordierite-based ceramic honeycomb is one of the ceramic products, which are produced, in considerably large numbers.¹ The mass production of the intricate honeycomb product was possible because of the invention of the honeycomb extrusion die² and the extrusion process.^{3,4}

Benbow and Bridgwater proposed a relationship giving the pressure required to extrude a particulate paste made with a liquid phase.⁵ They suggested the extrusion mechanics of pastes and the influence of paste formulations on extrusion parameters^{6,7} using an extruder schematically shown in Fig. 1. According to the model, the pressure drop, P , in pastes during extrusion through circular dies with die entry angle of 90° can be represented by the following equation, which is referred to here as six-parameter model;

$$P = P_e + P_l \\ = 2(\sigma + \alpha V^m) \ln(D_o/D) + 4(\tau + \beta V^n)(L/D) \quad (1)$$

where, P_e is the die entry pressure drop, P_l is the die land pressure drop, D_o is the extruder barrel diameter, D is the die diameter, L is the die land length, σ is the bulk yield stress of the paste, τ is the die land wall shear stress, α is the velocity sensitivity factor of the bulk yield stress, β is the velocity sensitivity factor for the wall flow, m is the bulk velocity exponent, n is the wall velocity exponent and V is the velocity of the extrudate. The six extrusion parameters which characterize the paste rheology are σ , τ , α , β , m and n .

The above extrusion parameters can be obtained by laboratory ram extrusion experiments in which extrusion pressures are measured for square entry, circular cross-section dies of different lengths at a range of extrudate velocities. These data can then be used to predict the relationship between pressure and extrudate velocity for other dies and to relate the behavior of pastes to their compositions.

The Benbow–Bridgwater model is advantageously used in ceramic fabrication for prediction of extrusion pressure.^{5,8} This model along with finite element analy-

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sis⁹ and artificial neural network (ANN) model¹⁰ have also been employed for obtaining solutions and estimation of the extrusion pressure. Using the Benbow–Bridgwater model, Blackburn and Bohm have developed a method for calculating the pressure drop across a honeycomb die and demonstrated for alumina extrusion.¹¹ The model is also applied to predict the pressure generation in a screw extruder¹² and also for designing paste and extruder for co-extrusion.¹³ The Benbow–Bridgwater model is also used to study the effect of binders on extrusion rheology.^{6,14} The effect of methylcellulose binders of different molecular weight on paste rheology and cordierite honeycomb extrusion was also studied using a laboratory ram extruder.¹⁵

Other approaches were reported by employing capillary rheometer for a slit capillary die¹⁶ and a model honeycomb die.¹⁷ The later die, consisting of only one feed hole and the outlet cross-slit, was used in order to study the extrusion pressure drops across the length of the die particularly when the cross-sectional geometry changes from round to cross slit. Various die lengths including dies formed only with individual round and slit shapes were used in combination. Pressure losses at various die regions as a function of extrudate velocity were successfully resolved and compared with honeycomb extrusion and drying results. However, more quantitative information on paste characteristics, particularly the material dependent properties, which are influenced by the paste formulation control correlating various binders, binder combinations and liquid levels was not obtained. Most of these studies were carried out on alumina or catalyst extrusion and we could not find much scientific analysis on the rheological parameters for cordierite honeycomb extrusion paste. Studies on rheological properties of cordierite extrusion and quantitative analysis of their parameters responsible for good honeycomb extrusions are thus useful for understanding the industrial mass-fabrication of cordierite honeycombs by extrusion method.

In this work, rheological properties of cordierite extrusion pastes under various extrusion conditions were analyzed using six parameters based on the Benbow–

Bridgwater model. Influence of various additives on the extrusion rheology of honeycomb was also studied.

2. Experimental

Extrusion paste for forming a single phase cordierite was prepared by mixing raw powders adjusting cordierite forming batch formula as follows; 39 mass% of talc from Rajasthan, India, 46 mass% of kaolin clay from Kundra, India and 15 mass% of alumina (HTM-30, Indalco, Belgaum, India). After thorough mixing of these constituents in dry ball mill, the batch was mixed with 2 and 3 mass% of methylcellulose (K4m from Dow Chemicals, USA), in a close gap high-shear zigma-blade mixer (Fabdecon, Mumbai, India) for 45 min using de-ionized water. These batches are designated as B1 and B2, respectively. One more batch, designated as B3, was made with 2 mass% of methylcellulose and 1 mass% of polyvinyl alcohol (SD Fine Chem., India), as secondary binder. Two paste batches were prepared from each of B1, B2 and B3 by adding two levels of water at 19 and at 23 mass%, respectively while mixing in zigma-blade mixer. Thus, six mixed paste batches, i.e. B1(19), B1(23), B2(19), B2(23), B3(19), and B3(23) were obtained as summarized in Table 1. These paste batches were then de-aired and extruded into a bar of 24 mm diameter using a screw extruder 55 mm in barrel diameter. This extruded bar was inserted in the barrel of the laboratory ram extruder, fabricated as per Benbow et al.⁵ and fitted to a universal testing machine (AG 5000E, Shimadzu, Japan) for subsequent rheological characterization.

Cylindrical dies (4 mm Φ) with L/D of 1, 2, 4 and 8 were used for the rheological characterization test. Each die was progressively taken one at a time from the set and assembled in one end of the barrel. From the other end of the barrel, a piston type ram was introduced after inserting appropriate length of the 24 mm diameter test blank from the prepared paste batches. The ram was programmed to descend at various pre-decided ram speeds (e.g. 0.2, 1, 2, 5, 10, 20, and 40 mm/min) and the load caused by the extrusion was recorded.

The measured total extrusion pressure was separated into die entry pressure and die land pressure after obtaining pressure at $L/D=0$ by extrapolating extrusion pressure vs. L/D curve using the Benbow–Bridgwater model (Eq. 1). The results were then fitted by the six-

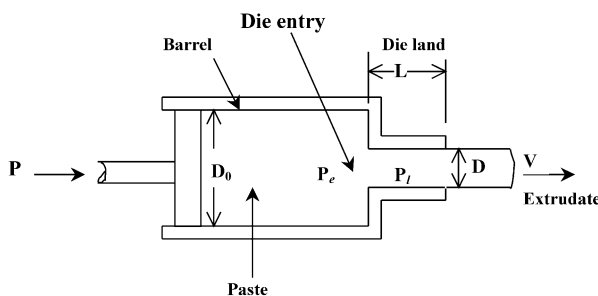


Fig. 1. Total extrusion pressure (P), die entry pressure (P_e) and die land pressure (P_l) of paste through square entry die in a ram extruder.

Table 1
Binder and water content (mass%) of the extrusion batches

	B1(19)	B1(23)	B2(19)	B2(23)	B3(19)	B3(23)
Methylcellulose	2	2	3	3	2	2
PVA	–	–	–	–	1	1
Water	19	23	19	23	19	23

parameter model, assuming plug flow in the die. These calculations were done for pastes with various binder and water contents. Pastes added PEG (molecular weight 3500–4000) and glycerin in amounts of 0.4, 0.8 and 1.2 mass% in B1(20) batch were also examined to elucidate the effect of plasticizer. Pastes added carbon (C) and graphite (G) powders (99.5% carbon, Graphite India, Bangalore, India) in amounts of 5 and 10 mass% in B3(22) batch were prepared to study the influence of these pore forming agents to extrusion.

The prepared pastes were then extruded using a honeycomb die 90 mm in diameter with cell density of 400 cpsi (cells per square inch) in a hydraulic ram extruder. The results of the honeycomb extrusions for each set of pastes were correlated with their rheological parameters.

3. Results and discussion

3.1. Effect of binder content and water content

Changes of total extrusion pressures for pastes with various binder and water contents as a function of extrudate velocity are shown in Fig. 2(a). At the same water content, extrusion pressure increased with increasing binder content in the pastes. This is because more binder requires more water to have the same viscosity. When water content increased, extrusion pressure reduced as expected. Addition of PVA as secondary binder decreased the extrusion pressure because PVA works as a good plasticizer for the cellulose binder.

Changes of die entry pressure and die land pressure as a function of extrudate velocity are shown in Fig. 2(b and c), respectively. The effect of water content is more distinct both on die entry and die land pressures than binder content. This means that viscosity of binder film between particles varies according to the amount of water for the tested batch. The influence of binder contents on die land parameters has less effect at higher water content indicating that only the die entry pressure has major contribution to the total extrusion pressure. On the other hand the influence of binder contents (for B1 and B2) on die entry become insignificant at lower water content and the die land pressure shows the more influence of binder levels. Less drop of die land pressure and more drop of die entry pressure from B2(23) to B1(23) pastes may be related to the higher water content of the paste with reference to the time scale of the experiment. Behavior of liquid excess inter-particle volume is very critical in extrudability of paste in complex shapes like honeycomb. Uniform distribution of excess liquid provides improved lubrication for paste flow whereas liquid migration causes lubrication failure and problems during extrusion. The behavior of the liquid depends on the paste formulations, particularly the presence of good rheology modifier.¹⁸

Six extrusion parameters obtained by mathematical analysis are listed in Table 2. The yield strength (σ) of the paste increases with increase in binder content, while decreases with increase in water content. These changes are thought to be the main reason for change in die entry pressure by variation of binder and moisture content in the extrusion paste. In the die entry region, shear occurs in the paste owing to the flow of the particles and the particles are diverted into die land. The shear resistance of the particle changes depending on its yield strength. It was found that when the σ of the paste was between 0.15 and 0.3 MPa, good honeycomb extrusion with higher output was obtained. When the σ was lower than that range, monolithic honeycomb structure could not be retained because of low yield strength of the paste. When the σ was larger than 0.3 MPa, the pressure requirement for extrusion became very high. This

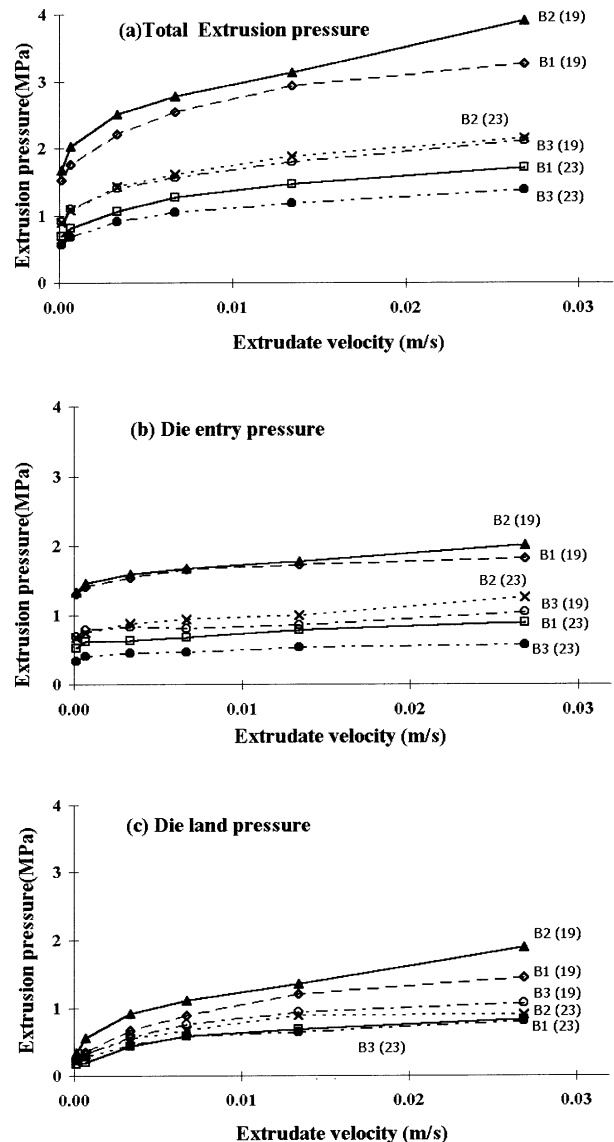


Fig. 2. Effect of binder content on extrusion pressure.

requires higher capacity extruder and thicker and/or stronger honeycomb die.

The α value, the velocity sensitivity factor of yield strength increased with increase of binder. This parameter is thus a relative measure for the dependence of flow resistance to extrudate velocity. The larger α value generally means increase in flow resistance for a given incremental increase in the extrusion velocity. However, this may give wrong interpretation if we analyze using only this parameter in the present case of honeycomb extrusion paste. This is therefore further examined by using a dynamic bulk stress component in the next section.

The m values are in the range of 0.18–0.59 indicating the pastes tested are significantly pseudo-plastic. Higher yield of the honeycomb extrusion is found with this range of m values, preferably <0.4 . Higher pseudo-plasticity is required in honeycomb extrusion paste because it is preferable to be low viscosity when the paste passes through the die, but it attains high viscosity when comes out of the die to retain the honeycomb shape.

The wall velocity factor β decides the dynamics of the sliding stresses and is significant for the present honeycomb extrusion paste because the value is effectively large compared with the wall shear stress τ . The achieved τ value, which is the yield strength of the thin lubricating film between the paste and the die land, is remarkably low. They were less than 1/100 of the yield strength of the bulk paste for B1(23) and B2(23) batches. However, it was found beneficial to have low τ value of less than 0.02 MPa to get better honeycomb extrusion. This suggests that there may be a compositional difference between the material sheared in the die land interface and that in the die entry region. Reed and Price¹⁹ showed that die land film was composed of water admixed with very fine clay and feldspar particles in case of porcelain extrusion. A thin layer of paste, which is depleted of particles owing to local packing con-

straints is also likely to be sheared²⁰ in addition to the thin layer of liquid between particles and the die wall. Similar phenomenon may be happening in the present case, where lesser amount of ceramic particles is dispersed in the higher binder and water concentrations at the die land interface.

3.2. Dynamic stress components

The honeycomb extrusion batches studied require a six-parameter model for reliable fit. Also their velocity dependent component of the die-entry pressure is quite comparable with the bulk yield stress of the paste. At the same time, it is not accurate to interpret the results only from α or β , because it is related through the unit dimensions to the respective velocity exponent. We found it useful to interpret the pressure results with the help of (αV^m) parameter which may be referred as “dynamic bulk stress component” in line with the “dynamic surface shear stress component” presented by Chen et al.¹⁸ The calculated values of these dynamic components are listed for a typical velocity in Table 2. These dynamic components give many useful interpretations those are earlier not so clearly obtained from the values of individual parameters.

The effect of increasing water has been clearly reflected by the drop of dynamic bulk stress component (αV^m) in batches B1, B2, and B3. On the contrary, the α values always showed increase with increasing water for each of these formulations. It will be therefore very complex to interpret the drop of actual die entry pressure from comparing each set of α and m values when water is increasing and yield stress levels are varying. The values of αV^m directly indicate the trend that the increase of water decreases the velocity contribution on entry pressure and the mobility of the paste has increased. This is more beneficial in honeycomb extrusion where the ranges of operating velocities can be broadly defined.

The decrease of αV^m with increase of binder content [between B1(19) and B2(19)] indicates that velocity contribution of the pressure actually reduces even though yield strength increases. This result is significant and useful for designing particular extrusion process to achieve higher output with good handling strength of the honeycombs without going for higher extrusion pressure. This also explains why a very stiff paste with high yield extrudes easily through honeycomb die at high output rate. When the difference between the yield stress and the dynamic stress is less, αV^m becomes more significant near the operating extrusion pressure. The αV^m parameter gives a convenient indication when calculated with the average of the desired velocity range.

It is necessary to have a simple tool to resolve, estimate and locate the cause of the die land pressure drop for assessing changes in paste formulations for the

Table 2
Extrusion parameters for various binder concentration and moisture content

Extrusion parameters	Paste batches					
	B1(19)	B1(23)	B2(19)	B2(23)	B3(19)	B3(23)
σ [MPa]	0.26	0.14	0.35	0.18	0.18	0.11
α [MPa(sm ⁻¹) ^{m}]	0.45	0.79	0.90	1.20	0.34	0.40
m (exponent)	0.18	0.59	0.43	0.57	0.27	0.43
τ (MPa)	0.008	0.001	0.012	0.002	0.003	0.004
β [MPa(sm ⁻¹) ^{n}]	0.40	0.16	0.49	0.15	0.23	0.20
n (exponent)	0.43	0.31	0.43	0.26	0.33	0.40
αV^m [MPa] ^a	0.18	0.04	0.10	0.07	0.09	0.04
βV^n [MPa] ^a	0.04	0.03	0.05	0.04	0.04	0.03
σ/τ ratio	31	145	29	113	62	32

^a For $V=0.006$ ms⁻¹.

honeycomb production shop. Often liquid migration and other problems are influenced by the time taken for the paste to flow through the deformation region. The settings of operating conditions which sometimes achieve excellent yield of honeycomb extrusion, also need to be assessed by measurable parameter. The dynamic surface shear stress component (βV^n) could be helpful for such situations. It is more required for designing a honeycomb extrusion process, since in honeycomb die manufacturing technology, the entry channels are possible to design with many options, but the land regions are freeze by the product specification and strength requirements. This problem has attracted attentions of many workers and the use of suitable die material is also suggested.²¹

The βV^n and σ/τ values of these pastes are listed in Table 2. The σ/τ is related to liquid mobility and the more mobile the liquid, the better the shear lubrication leading to higher values of the σ/τ ratio. The βV^n decreases and the σ/τ increases when water content is increased for binders B1 and B2. These observations indicate that the die land pressure reduction is predominantly caused by the increase in liquid phase and its mobility. This significantly appears in honeycomb extrusion, because die land pressure dominates the honeycomb extrusion pressure¹⁷ due to its thin long slots on the die. It is very important to reduce die land pressure at the same time suppress the liquid migration, which disrupts continuous extrusion of defect-free honeycombs.

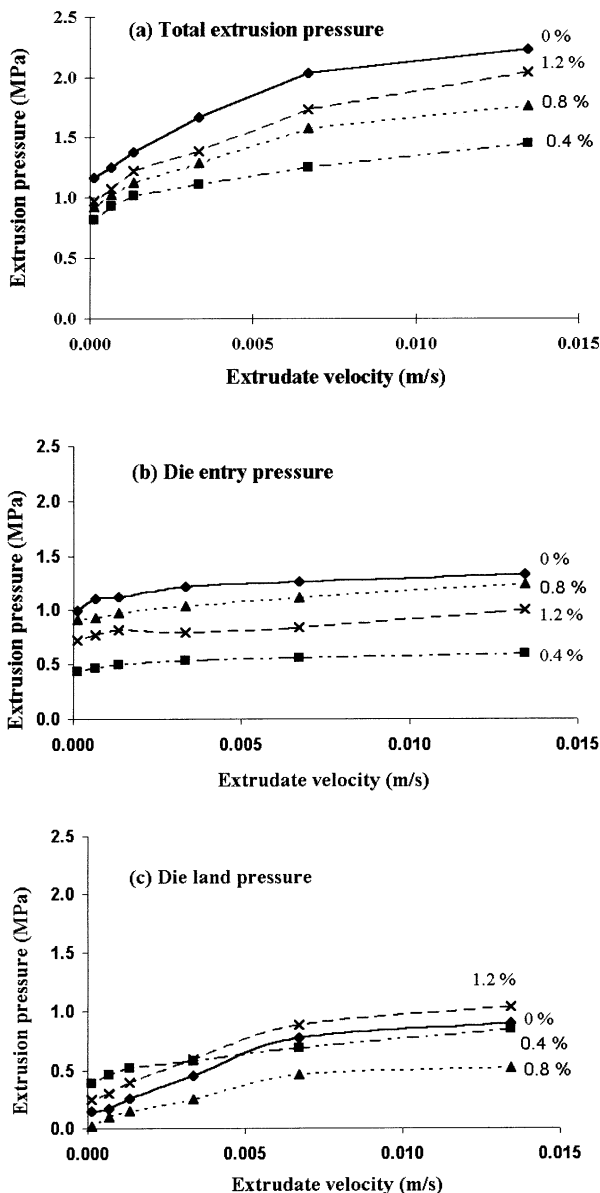


Fig 3. Effect of PEG addition on extrusion pressure.

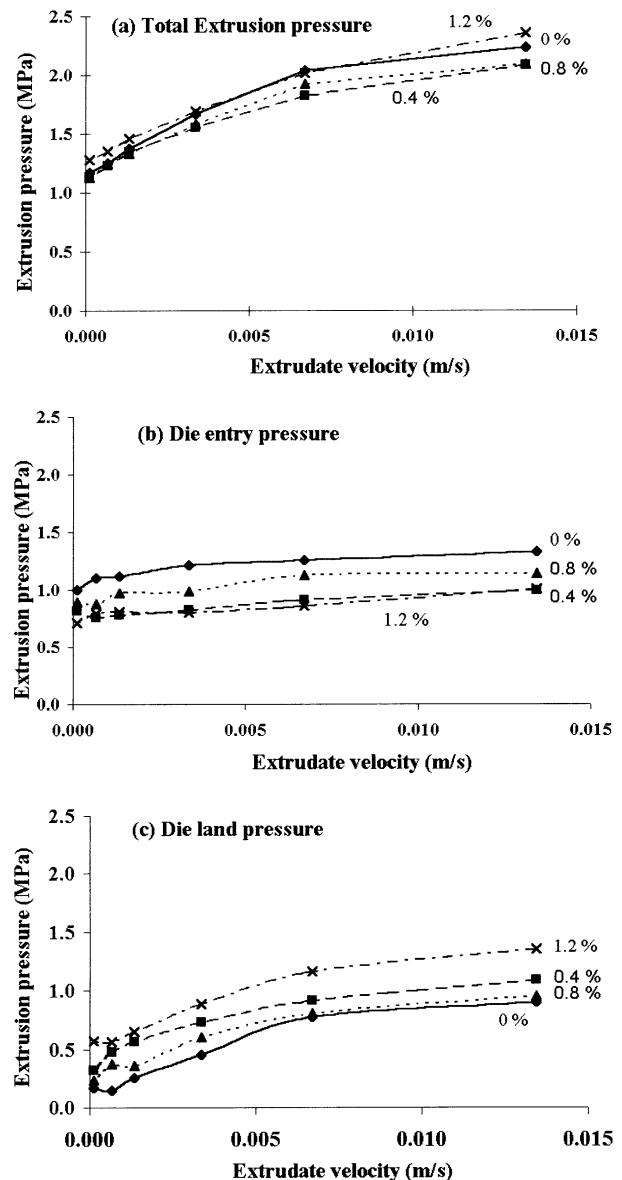


Fig 4. Effect of glycerin addition on extrusion pressure.

3.3. Effect of polyethyleneglycol (PEG) and glycerin addition

As appears from Fig. 3(a), the addition of PEG in the paste reduced total extrusion pressure. The maximum reduction of the extrusion pressure was observed at 0.4 mass% addition. The major contribution of the reduction is attributed to the decrease of die entry pressure [Fig. 3(b)]. On the other hand, the die land pressure increases with the addition of PEG [Fig. 3(c)]. Since PEG addition worked to decrease the die entry pressure, total pressure reduced appreciably. These results may indicate that PEG reduces the internal friction between particles in the paste by reducing viscosity of binder water film between the particles while increases the friction between paste and die wall. Thus, PEG is a plasticizing agent as is known for but not a good lubricant for extrusion.

As apparent from Fig. 4 (a), addition of glycerin did not reduce extrusion pressure. A small reduction is observed up to 0.4 mass% addition but no effect is seen over this addition. This indicates that plasticizing effect of glycerin is lower than that of PEG in the present extrusion batch. The decrease of die entry pressure [Fig. 4(b)] and increase of die land pressure [Fig. 4(c)] indicate that glycerin can act as internal lubricant or plasticizing agent to reduce die entry pressure similar to PEG, but the contribution is less and mainly effective up to 0.4 mass% addition.

The six extrusion parameters obtained by mathematical analysis for the above pastes are listed in Table 3. This also shows that 0.4 mass% PEG addition is optimum to lower yield strength (σ) and velocity sensitivity factor (α) with increase of bulk velocity exponent (m). On the other hand, glycerin addition increases σ and α , which could not be compensated by increase of m value. The honeycomb extrusion also could not produce continuous defect-free extrusion with higher than 0.4 mass% of glycerin. The influence of the increase in m value on pressure for the velocity range of interest can be demonstrated by (αV^m) factor.

Table 3
Extrusion parameters for various PEG and glycerin additions

Extrusion parameters	PEG addition (mass%)				Glycerin addition (mass%)			
	0.0	0.4	0.8	1.2	0.0	0.4	0.8	1.2
σ [MPa]	0.18	0.10	0.20	0.19	0.18	0.20	0.12	0.18
α [MPa(sm ⁻¹) ^m]	0.35	0.18	0.59	0.44	0.35	1.50	0.40	0.41
m (exponent)	0.15	0.28	0.34	0.39	0.15	0.68	0.16	0.37
τ [MPa]	0.007	0.018	0.001	0.011	0.007	0.020	0.015	0.031
β [MPa(sm ⁻¹) ⁿ]	2.49	0.39	1.05	0.77	2.49	0.34	0.51	1.77
n (exponent)	0.86	0.56	0.87	0.59	0.86	0.34	0.52	0.75
αV^m [MPa] ^a	0.16	0.04	0.11	0.06	0.16	0.05	0.17	0.06
βV^n [MPa] ^a	0.03	0.02	0.01	0.04	0.03	0.06	0.04	0.04

^a For $V=0.006$ ms⁻¹.

3.4. Effect of carbon and graphite addition

To make ceramic honeycomb with high porosity, particularly for filtration application, pore-forming agents such as carbon, graphite, wood powder, etc. are considered as the candidates. These additions are suggested to affect rheology of ceramic paste and resulting effects for honeycomb extrusion, however, no data are available on optimum amount of addition to reduce extrusion pressure and give uniform pores. The effects of two pore-forming agents namely carbon (C) and graphite (G) were investigated in this work. The effects of carbon and graphite on the total extrusion pressure are shown in Fig. 5(a). The extrusion pressure increases only a little at 5 mass% of carbon addition but becomes apparently higher at 10 mass% of carbon addition. On the other hand, graphite decreases the extrusion pressure at 5 and 10 mass% addition.

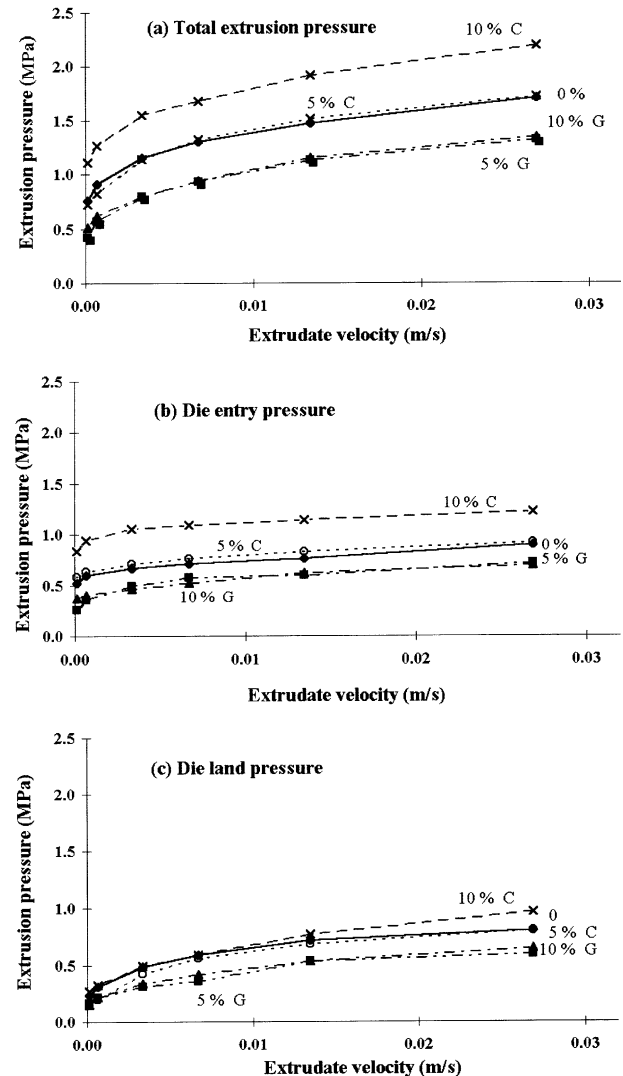


Fig. 5. Effect of graphite and carbon addition on extrusion pressure.

Table 4
Extrusion parameters with and without addition of graphite and carbon

Extrusion parameters	No additive	Graphite (mass%)		Carbon (mass%)	
		5	10	5	10
σ [MPa]	0.14	0.00	0.09	0.14	0.08
α [MPa(sm ⁻¹) ^m]	0.52	0.37	0.55	0.39	0.36
m (exponent)	0.45	0.19	0.48	0.36	0.10
τ [MPa]	0.001	0.007	0.004	0.001	0.013
β [MPa(sm ⁻¹) ⁿ]	0.12	0.15	0.14	0.16	0.28
n (exponent)	0.25	0.42	0.36	0.32	0.48
αV^m [MPa] ^a	0.06	0.15	0.05	0.06	0.21
βV^n [MPa] ^a	0.04	0.02	0.02	0.03	0.02

^a For $V=0.006$ ms⁻¹.

Major contribution of increase in the total extrusion pressure by addition of 10 mass% carbon is attributed to the increase in die entry pressure [Fig. 5(b)]. In this sample, both the dynamic bulk stress and the wall flow yield stress components increased largely but bulk yield stress and dynamic wall flow shear stress did not increase. The carbon addition showed a little effect for die land pressure [Fig. 5(c)]. These results indicate that carbon increases friction between particles and increases yield strength of the paste and that the effect becomes distinct when the content of carbon is more than 5 mass%. On the other hand, addition of graphite decreases both die entry and die land pressures. These results indicate that graphite works both as internal and external lubricant for extrusion because flaky graphite particles reduce the friction between clay and talc particles, and reduces the yield strength σ (Table 4). This corresponds to decrease bulk yield stress and dynamic wall flow shear stress.

Thus, the present study indicates that 5 mass% of carbon can be used to introduce lower level of porosity in the honeycombs because it gives uniform and controlled porosity without affecting extrusion. It is however necessary to use graphite to introduce higher level of porosity because it is effective to reduce extrusion pressures even adding large amount.

4. Conclusions

Rheological parameters in cordierite honeycomb extrusion were analyzed by the Benbow–Bridgwater model.

1. The pastes used showed low bulk yield stress, good pseudo-plasticity and very low die-land wall stress, lower than the die-land velocity contribution to the pressure.
2. With increasing binder content, the major contribution to the total extrusion pressure was (1)

by the die land pressure at lower water content and (2) by the die entry pressure at higher water content.

3. The dynamic bulk and shear stress components, which correspond to the velocity contributions to the respective pressure drops, were found useful in interpreting honeycomb extrusions. The dynamic bulk stress term helped in designing pastes with specific advantage to honeycomb extrusion by identifying pastes with high yield and low velocity contribution
4. Addition of PEG and glycerin showed plasticizing effect and reduced the die entry pressure, but no external lubricating effect leading to higher die-land pressure. An optimum addition of PEG in decreasing the total extrusion pressure was 0.4 mass%.
5. Pore-forming agent, graphite was found to reduce both die entry and die land pressures, whereas, carbon increased the die entry pressure.
6. Desirable values of the extrusion parameters are thus evaluated, using the Benbow–Bridgwater model, which are helpful for achieving optimized continuous honeycomb extrusion. The rheological parameters for each paste formulation can be used as a process control tool in industrial extrusion of complex ceramic structures such as honeycomb monoliths.

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