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A simple model for viscosity of powder injection moulding mixes with binder content above powder critical binder volume concentration

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Abstract

A simple model is proposed for the variation of viscosity of powder injection moulding mixes with binder content above Critical Binder Volume Concentration (CBVC), a characteristic of powder. Coarse silica and fine alumina powders are mixed with different types of low viscosity binders at different binder proportions. The viscosities of these mixes are measured by capillary rheometry with a special die at different shear rates. The CBVC values of the powders estimated by the model from the viscosity data of the mixes are matching with those values arrived from the experimental torque rheometry and temperature measurement methods. The proposed model has shown excellent correlation between the calculated and experimentally measured viscosity data at different shear rates, binder contents with different binders and powders. The model also gave precise CBVC values with the viscosity data of the previous authors validating the model to other powders and binders. The model is useful for the prediction of viscosity of PIM mix with known binder content, viscosity of binder and CBVC of the powder.

Keywords: Al_2O_3; Injection moulding; SiO_2; Viscosity

1. Introduction

Powder Injection Moulding (PIM) is one of the prime processes for manufacturing complicated shapes of metals and ceramics.[1,2] In this process, a large volume of metal/ceramic powder is mixed with small amount of sacrificial binder to give sufficient fluidity during injection moulding. To produce consistent products with the process, it is essential to understand the flow characteristics of PIM mixes. Since the rheology is a complex subject with a number of parameters like properties of powders and binders, their contents, operational and experimental conditions, etc., it was investigated in terms of the effect of individual parameters (like shear rate or temperature) on the viscosity by few authors[3–7] ignoring the effect of binder content for all practical purposes.

But various theories have been proposed to predict the viscosity of concentrated solid-liquid suspensions with solids/liquid content modifying the Einstein[8] dilute suspension model as power series[9,10] or exponential functions or differential form[11] depending on particle interactions. These empirical models[12–19] correct the variation of viscosity with liquid content accounting the particle-particle interactions by introducing various particle/binder characteristics like particle shape, distributions, powder content, packing ability, non-Newtonian, viscous fluids, etc., and have shown good fits up to a maximum solid phase content of 55%. The validity of these empirical models of solid-liquid suspensions for highly filled PIM mixes is not thoroughly verified except for limited claims of good fits by few authors at narrow shear rates and binder contents. If these models are really valid with their accountability of particle interactions, the particle characteristics should be verified with the model from the mixes experimental data. But such systematic efforts incorporating the experimental data of mixes viscosity with the models as a function of powder characteristics are even more limited in literature. Hence, efforts are made in the paper to validate the models by

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verifying one critical powder characteristic, critical binder volume concentration (CBVC) independently, with that obtained from the models with experimental viscosity data. The CBVC, is the minimum amount of binder content necessary to impart fluidity to given powder and its complementary term Critical Particle Volume Concentration (CPVC) denotes the maximum attainable packing density for a given powder. These terms and methods of their estimation for the powders were reported earlier.[20,21]

Mutsuddy[22] fitted the viscosity data on alumina with ethylene vinyl acetate copolymer binder mix upto 63% powder volume fraction and concluded that the data was in good agreement with Mooney's[13] model at shear rate 100 s^{-1}. But his data showed higher CBVC (obtained from CPVC data) for the powder and this was attributed to poor sphericity and agglomerations of powder particles. Similarly, in experiments of Steadman[23] viscosity data of silicon nitride with poly propylene–wax binder mix, use of Mooney's model estimated an unrealistic CBVC of −20%. Wright[24] viscosity data with high alumina powder loading (64%) with polypropylene, microcrystalline wax binder also arrived at similar unrealistic CBVC value of −19%. Here the Eiler,[12] Chongs[16] models estimated CBVC values to be 24 and 27%, respectively, in comparison to the experimental value of 35% obtained from shrinkage measurements and 35% obtained by Markhoff[25] from the torque rheometer method for the same powder. Thus, these models are not realistic for high powder loading PIM mixes. So the authors propose a new model for the variation of viscosity of powder injection moulding mixes with binder content and powder characteristic, CBVC.

2. Theory of new model

The PIM mix consists of constituents, powder and binder. The fluidity of the mix is given by rule of mixtures, as sum of fluidity of its constituents, i.e.

$$1/\eta_{\mathrm{m}} = 1/\eta_{\mathrm{b}}^{*}\mathrm{BVC} + 1/\eta_{\mathrm{p}}^{*}\mathrm{PVC} \tag{1}$$

where $1/\eta_{\mathrm{m}}$ is PIM mix fluidity, $1/\eta_{\mathrm{b}}$ is the binder fluidity, $1/\eta_{\mathrm{p}}$ is the powder fluidity and BVC, PVC are the binder and powder volume contents, respectively. But in a powder–binder mixture, the flow is essentially due to the binder only and the powder does not flow under shear due to inter-particulate friction. Hence, the fluidity of the mix is proportional to binder fluidity, $1/\eta_{\mathrm{b}}$ and its content, BVC, i.e.

$$1/\eta = 1/\eta_{\mathrm{b}}^{*}\mathrm{BVC} \tag{2}$$

This is subject to two constraints. The mix does not flow up to a minimum amount of liquid called the CBVC as all the liquid present in the mix is utilized for filling up the particle structure in close packing. Therefore no free liquid is available below CBVC in the system to provide flow to the mix, i.e. for $\mathrm{BVC} \leqslant \mathrm{CBVC}$, $(1/\eta) = 0$ and $\eta = \infty$ and also the viscosity of the mix obviously equal to that of binder when there is no powder loading in it. Incorporating these two conditions,

$$\eta \rightarrow \infty, \text{ as BVC} \rightarrow \text{CBVC and } \eta \rightarrow \eta_{\mathrm{b}} \text{ as BVC} \rightarrow 1.$$

Eq. (2) can be modified as,

$$1/\eta = 1/\eta_{\mathrm{b}} * (\mathrm{BVC} - \mathrm{CBVC})/(1 - \mathrm{CBVC}) \tag{3}$$

Simplifying the present model, Eq. (3) can be linearized as,

$$\eta \cdot \mathrm{BVC} = \eta \cdot \mathrm{CBVC} + \eta_{\mathrm{b}}(1 - \mathrm{CBVC}) \tag{4}$$

Now, if a graph of product of mix viscosity and binder content is plotted with mix viscosity, the CBVC of the powder can easily be evaluated from the model with the viscosity data. To compare the present model with those from solid–liquid suspensions and validate, the authors measured the CBVC of different powders with torque rheometry and temperature measurement methods and measured the viscosity of PIM mixes at different shear rates, binder contents with capillary rheometry.

3. Experimental

3.1. Powder characterisation

Silica powder (coarse) of average particle size 35 μm, with broad distribution (6 to 100 μm) from Remet Corp., USA, alumina powder (fine) of particle size 1.9 μm, narrow distribution (1 to 5 μm) from Alcan, Canada, were used in the present study. The powders were characterised for their particle size (ESD), distributions by light diffraction method using particle size analyser (NEC SKC-2000, Japan) and shapes by Scanning electron microscope (ISI 100A). The particle size, distributions and shapes of silica and alumina powders are shown in Figs. 1 and 2, respectively. The surface areas of the powder particles were measured by BET technique using Micromeritics Gemini 2375 V3.03 surface area analyzer. The powders apparent and tap densities were measured with appropriate methods. The CBVC values of the powders were estimated by torque and temperature measurement methods by monitoring the torque/temperature variation during mixing with linseed oil addition to powders, using the mixer of Brabender torque rheometer.[20,21] The CBVC value of the silica powder was estimated as 26.7 (±2.7) vol.% as shown in Fig. 3 and that of alumina powder was estimated as 42.5 (±1.6) and 43 vol.% with torque rheometry and temperature measurement methods respectively as shown in Fig. 4. The silica and alumina powders characteristics are summarized in Table 1.

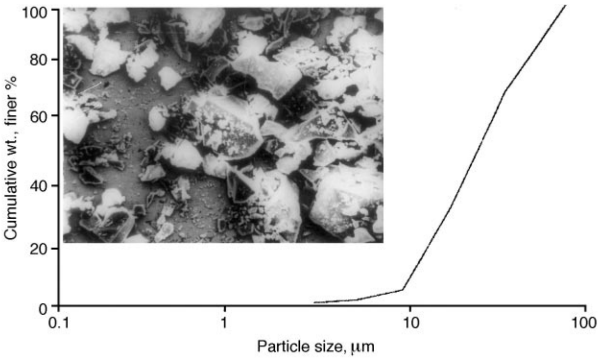

Fig. 1. The particle size and shape distribution of coarse silica powder.

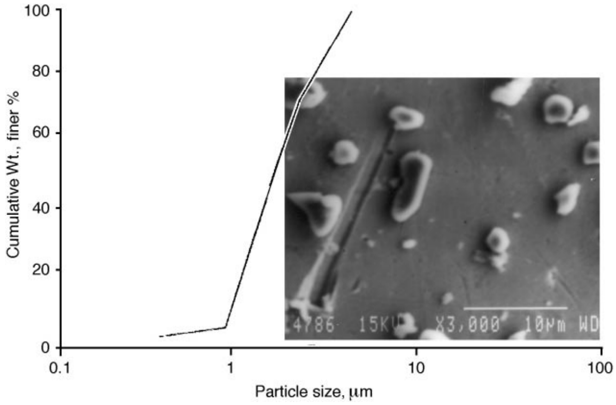

Fig. 2. The particle size and shape distribution of fine alumina powder.

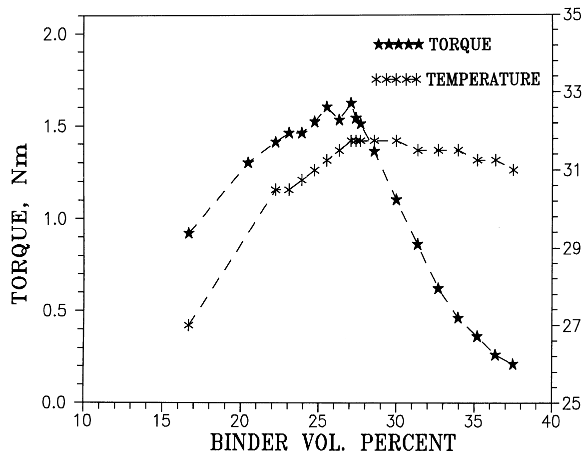

Fig. 3. The torque or temperature variation with binder volume during mixing of silica powder with linseed oil in torque rheometry and temperature measurement methods in Brabender mixer.

3.2. Binder systems preparations

Combinations of polymer–wax binder systems were selected for viscosity measurements as PIM mixes as thermoplastic systems were reported to cause excessive wear because of high shear during processing[26] and the lower viscosity wax binder systems were highly sensitive to temperature. Three types of binder systems with two constituents (3:1 ratio) were selected for the experiments. The major constituent of binder is to give the required fluidity and minor constituent is to modify the flow and to help during debindering. First binder system is complete wax-based system. Second binder system is having wax as major binder constituent. Third system is having polymer as major constituent. The binder systems compositions and mixing times, temperatures are given at Table 2.

The wax based binder system #10 was prepared by mixing paraffin wax and microcrystalline wax. Paraffin wax was of food grade type fully refined with melting point 61°C from China and microcrystalline wax was of amber type with melting point of 67°C from Japan. The polyethylene granules were having a flow rate FR-190/2.16 = 21.4 g and were procured locally. The fourth binder system, 30P was prepared with addition of polymethyl methaacrylate (PMMA) to #30 binder system to study the effect of the surfactant. The binder systems were prepared by mixing the appropriate constituents at suitable temperatures with stirrer till the uniformity of mix was observed.

3.3. Powder–binder mixes preparation

Powder–binder mixes were prepared for each binder/powder in five different proportions of binder contents by mixing the appropriate weights of powder, binder mixes in Brabender rheometer mixer at specified temperatures

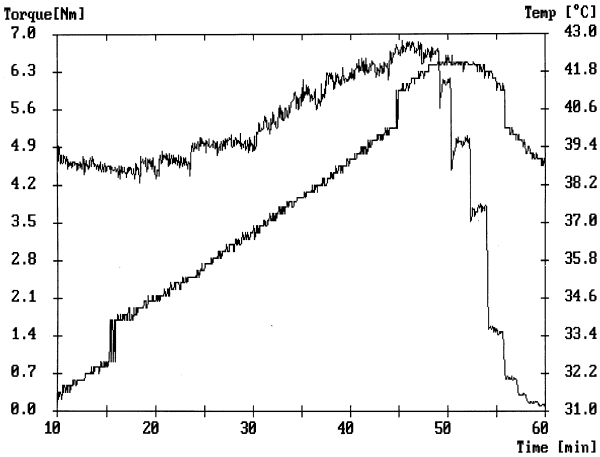

Fig. 4. The torque or temperature variation with time on linseed oil addition to alumina powder during mixing in torque rheometry and temperature measurement methods in the Brabender mixer.

Table 1
The silica and alumina powders characteristics

Powders	Average particle size (μm)	Surface area (m^2/g)	App. density (%Th.Den.)	Tap density (%Th.Den)	CBVC torque rheo. (%)	CBVC temperature method (%)
Silica	35	0.539	38	58.5	26.7 (± 2.7)	26
Alumina	1.9	0.935	25	30	42.5 (± 1.6)	43

Table 2
The binder systems compositions, mixing temperatures and times

Notation	Binder composition (vol.%)	Mixing temperature (°C)	Mixing time (h)
#10	75% Paraffin wax + 25% microcrystalline wax	70	2
#20	75% Microcrystalline wax + 25% polyethylene	120	3
#30	75% Polyethylene + 25% microcrystalline wax	150	3
#30P	71.5% Polyethylene + 23.5% microcrystalline wax + 5% PMMA	150	3

for about 45 min at 50 rpm. The mix compositions were selected to have binder contents greater than CBVC. The maximum binder fraction was selected at a volume beyond where the mix torque equals to that of oil in rheometer curve. The silica powder–binder mixes 1S, 2S, 3S, 4S, 5S were having binder contents 50, 36, 32, 30 and 28 vol.%, respectively. Similarly the alumina powder–binder mixes 1A, 2A, 3A, 4A were having binder contents 50, 46, 44 and 43 vol.%, respectively. The mixes notation for any composition can be arrived by adding the binder and powder codes. For example a mix 31S will have Silica powder and #30 binder with binder content of 50 vol.%. The mixing temperatures were selected to be above the softening temperatures of the binders which were measured through Melt flow index extrusion die with a capillary of 2 mm under load of 2.15 kg. The mixing time was selected after observing the torque stabilisation during mixing. The powder binder mixes were crushed into fine globules to facilitate their filling in the capillary die for viscosity testing. The PIM mixes were verified for their homogeneity and binder content, by monitoring the actual weight loss of the mixes after debindering at a temperature of 900°C for 1 h.

3.4. Viscosity measurements

A die having dimensions similar to that of standards ASTM D 1238-86 and ISO R1133 of polymers flow rate testing, was designed and fabricated with auto plunger ejection mechanism for the determination of viscosity of PIM mixes. A detachable capillary with a hole of 1.5 mm and length to diameter ratio > 30 was incorporated in the die to give the mix a smooth and lamellar flow and minimum entrance corrections (Bagley entrance correction for stress due to funnelling effect at the entrance). A thermal gradient from the bottom to top of die was provided to facilitate the extrusion of the mix by reducing heat loss at bottom and incorporating a heat sink at top. A view port was incorporated at the bottom of the die to prevent its damage if the mix was not extruding. A photograph and sketch of different parts of the die are shown in Figs. 5 and 6.

The die was fixed on our Instron machine, model 4483 with proper alignment and heated to required temperature with a strip heater. The processing temperature, above the softening point (20% excess over softening

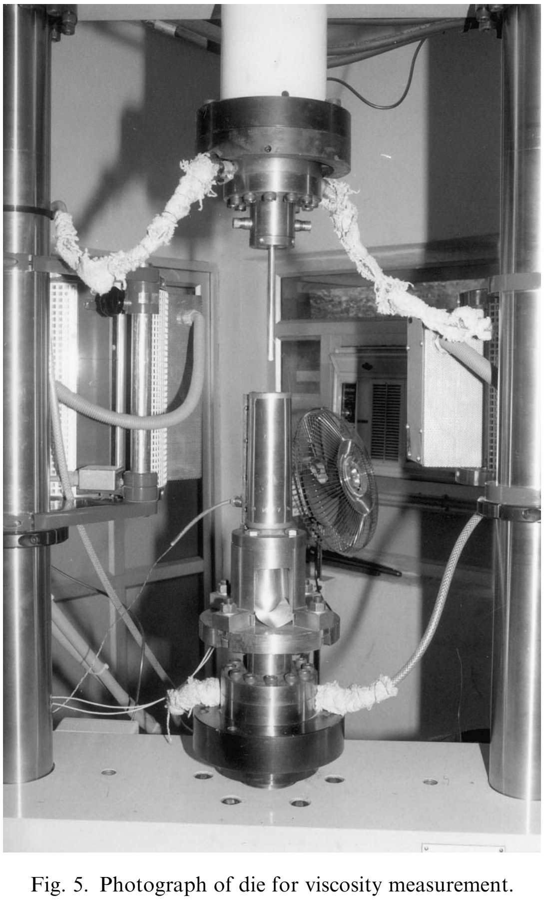

Fig. 5. Photograph of die for viscosity measurement.

point of the binder), but below the melting point was selected for viscosity measurement of these mixes. It was selected such that both binders, powder–binder mix could be processed at same temperature for comparative studies. A lower temperature of processing was possible with binder, but the mix viscosity would be very high with high powder loadings. At higher temperatures mixes could be processed with lower loads, but the binder required very low loads, this would result in high errors in viscosity measurements. Temperatures of 60, 90°C for #10, #20 and 125°C for #30 and #30P were selected for binder systems and powder–binder mixes viscosity measurements.

The material was loaded into the die and a homogenisation time of 5 minutes was allowed for temperature equilibration and the material was extruded with Instron in compression mode at desired ram rate by setting proper piston speed for a length of 50 mm travel. This ensured the load stabilization during the material injection through the capillary at the desired shear rate. The piston speeds were selected to give shear rates from 10 to 1000 s^{-1}. With the load and the piston speed data, the viscosities of binder systems and powder-binder mixes were calculated using standard equations[27] at different shear rates and temperatures. The viscosities of various binders at different shear rates from 10 to 1000 s^{-1} were measured with different powders, loadings and binders. The Rabinowitch correction[28] for the non-Newtonian character of the binders was calculated and the viscosity data was corrected.

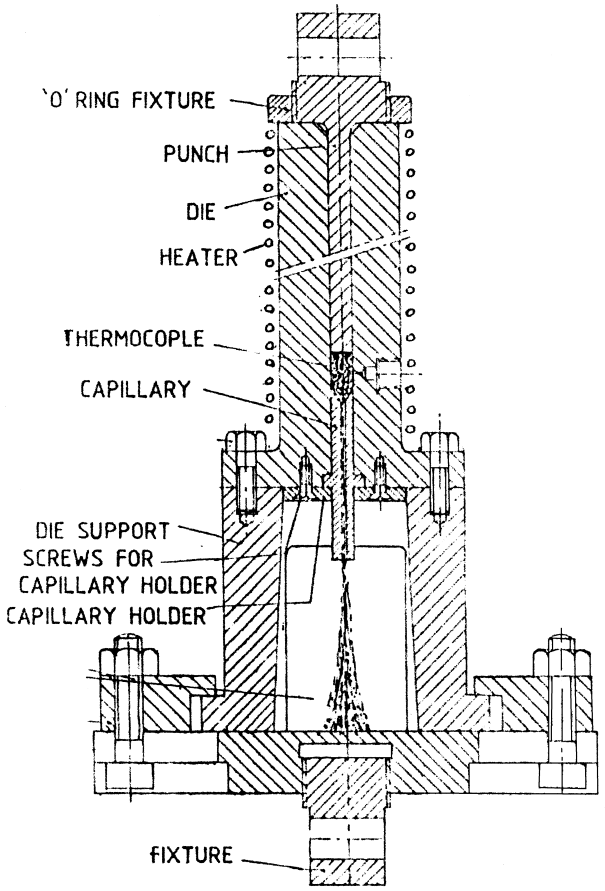

Fig. 6. Sketch showing the various parts of viscosity die.

4. Results and discussion

The PIM mixes of binder #10 were getting segregated during the viscosity testing by even minor temperature fluctuations due to the binder's low viscosity and high sensitivity to temperature. The squeezing of low viscosity binder on shear stress from the highly viscous PIM mix during viscosity test escalated the mix viscosity and resulted in seizure of extrusion number of times. Hence, the data of these mixes were excluded from the analysis.

4.1. Experimental viscosity data — existing models

The existing solid–liquid models were used to calculate the relative viscosity (the ratio of mix to binder viscosity) values for various PIM mixes for different binder contents using maximum volume fraction of packing as 0.74 (as suggested by Eilers[12] or Mooney's[13] model for spherical particles) and the constant B in Mooney's equation is assumed to be 2.5 (as the degree of agglomeration will be negligible with coarse silica particles) and compared with those of experimental values from the capillary rheometry. Figs. 7 and 8 show the relative viscosity variation with powder/binder volume fraction for PIM mixes of silica and alumina powders with #30 binder with different models and the experiments. Most of the models overestimated the viscosity raise of the PIM mixes with the powder loading beyond 50 vol.%. Chongs[16] model is the nearest model to the experimental results among all the models.

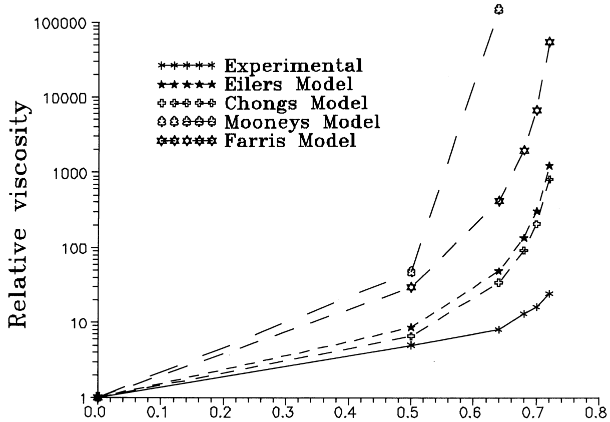

Fig. 7. The change in viscosity of #30S PIM mixes with powder volume fraction from experimental data and various models of solid–liquid suspensions.

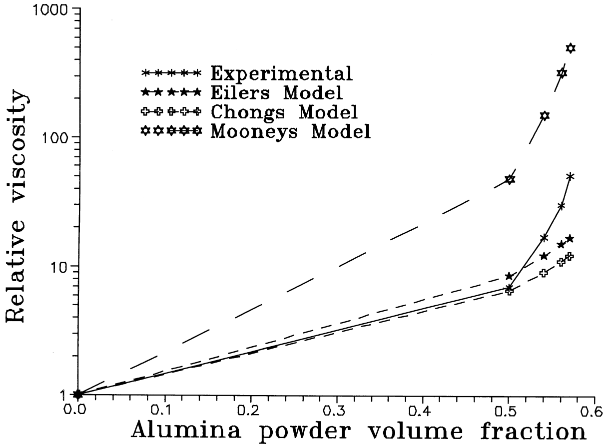

Fig. 8. The change in viscosity of #30A PIM mixes with powder volume fraction from experimental data and various models of solid–liquid suspensions.

Table 3
The CBVC values arrived from different models by calculation with the viscosity data

Model	Shear rate (s^{-1})	CBVC (%) silica	Correlation coefficient	CBVC (%) alumina	Correlation coefficient
Eiler					
#20	100	12	0.99	−5	0.67
	200	12	1.00	−17	0.70
#30	100	13	0.99	40	1.00
	200	13	0.99	40	1.00
Chong					
#20	100	−20	0.96	−57	0.98
	200	−10	0.99	−42	0.99
#30	100	−4	0.92	69	0.99
	200	−3	0.95	66	1.00
Mooney					
#20	100	−25	0.89	12	1.00
	200	−28	0.97	−5	1.00
#30	100	−16	0.90	35	1.00
	200	−16	0.94	31	0.99
Torque rheometer		26.7 (±2.7)		42.5 (±1.6)	

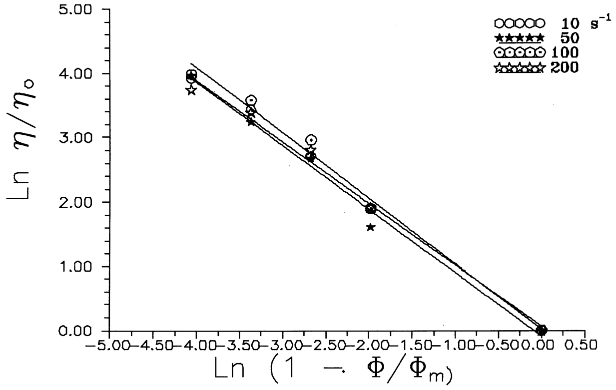

Fig. 9. The variation of relative viscosity with (1−ϕ_r) for #30A powder–binder mixes at different shear rates.

4.2. CBVC estimations with existing models

Then attempts were made to calculate the powder characteristic, CBVC of the powders independently from the single fit constant models using the measured viscosity data and binder content values of PIM mixes. The Eilers'[12] model was linearized as, η_r–1 = ϕ_m (η_r–1)/ϕ–1.25 ϕ_m to plot variation of (η_r–1)/ϕ with η_r–1, so that the slope of the curves will give ϕ_m, the CPVC value of the powder. Similarly the Chong's[16] equation was linearized as, (η_r–1)/(η_r–0.25) = ϕ/ϕ_m to plot the variation of (η_r–1)/(η_r–0.25) with ϕ, powder volume content in the PIM mix. The slope of the curves gave 1/ϕ_m value. The Mooney's[13] model was linearized as, Ln η_r = ϕ_m(Ln η_r–2.5ϕ)/ϕ to plot the variation of Ln η_r with (Ln η_r–2.5ϕ)/ϕ, the slope of the curves gave value ϕ_m. The CBVC values calculated from ϕ_m are shown for different mixes, shear rates in Table 3. The values from the models are unrealistic, have poor correlation coefficients and do not match the experimental values obtained from torque rheometry and temperature measurements. The viscosity data when fitted to double fit constant equations like Farris[15] model, taking CBVC from the estimations of previous gave unacceptable slope k values from 0.97 to 1.2, as shown in Fig. 9, at different shear rates while the model had k values as 21, 5.8, 3.6 and 3 for mono, bi, tri and infinite model particle size distributions. Other models by Quemada[29] and Kitano et al.[19] had k value of 2, Krieger and Dougherty[14] the intrinsic viscosity [η_i] value 5/2 for spherical particles and Barne[30] gave empiricisms for discs and rods with different axial ratios, between 1.31 and 3.77. But the data from the present experiments showed the value to be near 1 irrespective of particle shape and size. Thus the viscosity data of alumina and silica powder mixes did not follow both single and double fit constant models.[12–16,19,29,30] These results are also showing similar trend as that of the previous investigations[22–24] invalidating the models for correlating PIM mixes viscosity with the powder characteristics and powder/binder contents.

4.3. Use of new model to the viscosity data

The new model has a complementary approach to earlier models. It considers the problem of PIM mix viscosity in terms of amount of binder content available for flow instead of concept of earlier models of viscosity raise with solids loading in the binder. Though the approach appears deceptively similar, a closer examination of the model will reveal the differences between the complementary terms (powder/binder content) and reciprocal terms (viscosity and fluidity).

The variation of the product of viscosity and binder content with viscosity for different powder–binder mixes

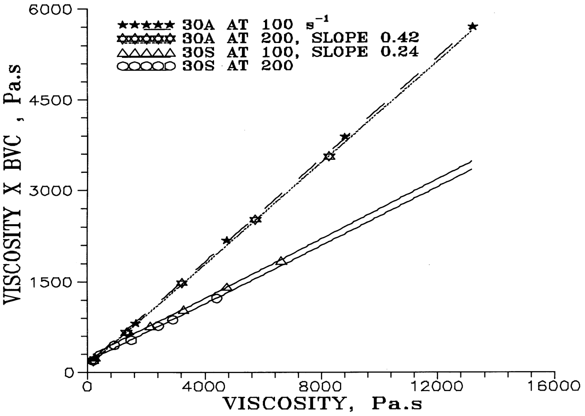

Fig. 10. The variation of the product of viscosity and binder content with viscosity for alumina and silica powders with #30 binder system at rates shear 100 and 200 s^{-1}.

is plotted using the viscosity data of capillary rheometry measurements at various shear rates. The new model fits are simple and precise as they are in linear relation rather than exponential or logarithmic like the previous models. Fig. 10 shows the variation of the product of viscosity and binder content with viscosity for PIM mixes with #30 binder system and alumina/silica powder at 100, 200 s^{-1} shear rates. These plots not only show very good straight line fits, in conformity with the model, but their slopes gave CBVC values of the powders from the viscosity data which are coinciding with the values of torque rheometry and temperature measurement methods. The viscosity data for different powder–binder mixes is fitted to linearized model and the correlation coefficients and CBVC values obtained are summarized in Table. 4. They have excellent correlation coefficients (>98%) at all shear rates. The CBVC values from all shear rates are in close agreement with those experimentally determined by torque rheometry and temperature measurement methods for both coarse silica and fine alumina powders in four binder systems, thus validating the new model.

4.4. Validation of new model with literature data

The capillary viscosity data of Mutsuddy[20] on A 16-SG alumina powder and ethylene vinyl acetate binder mixes with different shear rates, temperatures and three loadings (55, 60, 65 vol.%) were digitized and fitted to the model to see its validity with different other powders and binders. The data gave excellent fit correlation coefficient (0.9993). The CBVC (34.5%) value of alumina A 16 SG obtained from the digitized viscosity data with the model by plotting the variation of product of η. BVC with η, is matching with that obtained from torque rheometer CBVC (35%) by him in other experiments,[25] thus validating the model for other PIM mixes with different powders and binders.

Table 4
The CBVC values obtained from present model with experimental viscosity data for silica and alumina powders with different binder systems and shear rates

Powder	Binder	Shear rate (s^{-1})	CBVC from model (vol.%)	Correlation coefficient	Av. CBVC (S.D.)	CBVC by torque method
Silica	#20	10	24.64	0.9809		
		20	22.38	0.9781		
		50	24.36	0.9995	24.04	26.7
		100	24.45	0.9994	(0.92)	(±2.7)
		200	23.59	0.9990		
		500	25.22	0.9967		
		1000	23.65	0.9969		
Silica	#30	10	25.54	0.9967		
		20	23.33	0.9870		
		50	23.21	0.9890	23.35	26.7
		100	22.99	0.9944	(1.00)	(±2.7)
		200	22.69	0.9960		
		500	23.13	0.9959		
		1000	22.58	0.9972		
Silica	#30P	10	26.32	0.9948		
		20	23.53	0.9972		
		50	25.48	0.9994	25.43	26.7
		100	25.57	1.0000	(0.90)	(±2.7)
		200	25.90	0.9998		
		500	25.91	0.9995		
		1000	25.27	0.9993		
Alumina	#20	10	41.46	0.9998		
		20	41.58	0.9970	41.28	42.5
		50	41.72	0.9936	(0.62)	(±1.6)
		1000	40.36	0.9967		
Alumina	#30	10	42.27	0.9998		
		20	42.34	0.9999		
		50	42.14	0.9999	41.88	42.5
		100	41.91	0.9998	(0.60)	(±1.6)
		200	41.59	0.9999		
		500	40.66	0.9998		
		1000	42.28	1.0000		

It may be noted that the CBVC values obtained from the viscosity data for the powders are slightly lower (within the error limits) than that of torque rheometry and temperature measurement methods in all the cases. This may be due to inhibition of particle packing to its closest level during the CBVC measurement with torque rheometry or temperature measurement method as the particles are always covered with fine polymer film (may be equivalent to few molecular layers). But the new model CBVC predictions seem to be more rational as they are estimating the value at closest packing of particles from a large viscosity data (with an error allowance for polymer film in other estimates).

5. Conclusions

A simple model based on rule of mixtures is proposed for variation of viscosity of powder injection moulding mixes with binder content, that is in excess of CBVC of powder. The proposed model has shown excellent correlation between calculated and measured viscosity data at different shear rates, binder contents, binders and powders. The model is also validating the viscosity data of other authors with different powders and binders. CBVC values of the powder can be determined from the model with the viscosity data and values are matching with that of torque rheometer method and temperature measurement method.

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