# The Effect of Temperature, Heating Method and State of Dispersion on the Vacuum Filter Casting of Alumina Suspensions

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#### *Abstract*

*It is well established that with advanced ceramic powders the use of dispersed slips can result in superior microstructures compared to those obtained ,from partially or fully flocculated slips but at the cost of a signtjicantly slower casting rate. Recent published research has also indicated that bodies produced from coagulated slips have similar properties to those.from dispersed slips due to the lubricating nature of the hydrated layer surrounding the powder particles. However, little attention has been given to the possibility of accelerating the slow casting rate obtained with dispersed or coagulated advanced ceramic slips by the use of heating the slip. This paper investigates the effect of elevated temperature on the rheological properties and casting rates of four slips: one dispersed electrostatically, one dispersed electrosterically and two which are based on electrostatic dispersion followed by the addition of direrent salt levels to achieve coagulated slips. It is observed that the use of elevated temperatures results in increases in the casting rates in*  all cases, however the coagulated slips behave differ*ently to dispersed slips. The latter display casting rates which increase linearly with increasing temperature whilst coagulated slips display an initial rapid increase after which the casting rate decreases*  significantly. This is believed to be due to the inter*action between the permeability of the green body and the state of dispersion of the slip. A further observation arising from the work is that there is generally an enhanced casting rate when microwave energy is used to heat the slips compared to that achieved with a convection oven. The mechanism of acceleration is believed to be due to the rate of water* 

*movement through the cast being increased more than the rate of water uptake by the cast.*  $\odot$  *1998 Elsevier Science Limited. All rights reserved* 

#### **Introduction**

Colloidal processing of sub-micron ceramic powders is generally accepted as a method of producing components containing a more homogeneous microstructure with fewer and smaller defects than those produced by pressing routes. However they are usually slow processes and as such often avoided except for the production of large or relatively complex shaped articles such as hollow ware. In the advanced ceramics industry reliability is essential for structural components and the use of a fast near net shape colloidal processing route would be of great benefit. Various methods have been suggested to reduce the time required for colloidal filtration, these include vacuum, pressure and centrifugal casting. For the standard slip casting approach, methods include the use of partially or fully flocculated slips and also elevating the temperature. The final body produced by colloidal filtration has similar characteristics to the initial slip used, provided agglomeration can be avoided.' This was confirmed by Boschi<sup>2</sup> who determined that bodies cast by dispersed slips produced superior bodies to those from partially and fully flocculated slips despite casting being significantly slower.

Electrostatic control of aqueous suspensions is the simplest dispersion method available however additives are needed to improve both the slip and the green handling strength of the cast bodies and this method of dispersion is not ideal for all systems. Polyelectrolytes which provide electrosteric control are now widely used as deflocculating agents for advanced ceramic slips as they overcome

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some of the problems inherent with electrostatic control. Unfortunately they are sensitive to the properties of the medium and any alteration of the slip condition can result in changes in the absorption properties of the polyelectrolyte onto the powder surface. $3$  A further approach to casting slips involves the addition of salt to electrostatically stabilised suspensions; there are two possible effects that can be seen dependant on the concentration added. Firstly, a compression of the electric double layer occurs:4

$$
\kappa = \sqrt{\frac{F^2 \sum C_1 Z_1^2}{\varepsilon \varepsilon_0 RT}}
$$
 (1)

where the reciprocal of the Debye length,<sup>4</sup>  $\kappa$ , provides the thickness of the double layer. *F* is the Faraday constant, *R* the universal gas constant, C is the concentration of electrolyte, z the ion valence,  $\varepsilon$  and  $\varepsilon_0$  are the vacuum and relative permittivity, respectively, and *T* the temperature. When the salt addition reaches a critical level coagulation of the slip can occur. This is believed to be due to the formation of a hydrated layer around the powder particles which results in strong, short-ranged repulsive forces. These prevent flocculation of the particles by holding them in short-range equilibrium positions.<sup>5</sup> Recent work by Velamakanni<sup>6,7</sup> and Chang<sup>8,9</sup> has investigated the effect of coagulation on the rheology, pressure casting, centrifugal casting and storage of aqueous alumina slips. Their results indicate that the coagulated slips act in a manner similar to dispersed slips; on storage no settling occurred and on casting the bodies had similar green densities. However, coagulated slips have the advantage that, like flocculated slips, they cast with a significantly faster rate than dispersed slips but without the appearance of the density gradients typical of flocculated slips.

The use of elevated temperature during slip For electrostatic control, the slips were dispersed casting has been investigated by various authors at  $pH$  3-4 using concentrated nitric acid at a solid using both clay<sup>10,11</sup> and advanced ceramic slips.<sup>12,13</sup> content of 72 wt% (39 vol%) alumina. These slips The results have indicated that an increase in the are termed acid slips and form slip A in the ensuing casting rate can be achieved compared to ambient figures and tables. For electrosteric control, the temperature. The use of microwave energy to raise slips were dispersed using either Dispex A40, a the temperature of clay slips has been investigated 40% ammonium polyacrylate solution from Allied by Tobin<sup>14</sup> and Ontario Hydro.<sup>15</sup> Both found that Colloids, UK, or Bevaloid 211, a 40% sodium not only an increase in casting rate could be polyacrylate from Rhone-Poulenc in France. achieved but that the time required for drying of However, the former was found to alter the rheothe cast and mould could also be significantly logical characteristics of the suspension at elevated reduced. More recently, the present authors have temperatures in such a manner that a skin was compared the effect of using a convectional heat formed which prevented casting. Thus this paper oven and microwave energy on slip casting.<sup>16</sup> It focuses on the use of the sodium polyacrylate as was determined that increasing the slip casting the dispersing agent. The polyacrylate was used at

ing rate and that the use of microwaves caused a greater degree of acceleration than conventional heating. In addition, measurements indicated that the rate of water movement through the plaster of paris mould was increased more than the rate of water uptake by the mould, particularly during the microwave accelerated casting process. This suggested that at least part of the acceleration mechanism is due to faster moisture movement within the mould. A final, and surprising, result was that the permeability of the cast decreased with increasing casting temperature. This was interpreted as meaning that a more close packed microstructure was being produced, even though there was little apparent change in the density. However, little previous work has been undertaken into the effect that an elevated temperature could have on the slips themselves beyond an inference that a temperature change could modify the state of aggregation of the particles in the slip.14

This paper examines the vacuum filter casting of three different alumina-based slips; an electrostatically dispersed slip, an electrosterically dispersed slip and a coagulated slip obtained by adding salt to the electrostatically dispersed slip. It also investigates the effect of elevating the casting temperature on both the casting rate and state of dispersion for each slip, using both convectional heat energy and microwave energy.

## **Experimental**

#### **Slip preparation and characterisation**

For each slip, the required mass of Alcoa A16SG alumina powder was mixed with a known volume of deionised water containing the required dispersing/coagulating agents. After mixing the slurry was subjected to ultrasonic agitation and subsequently cooled and deaired prior to the pH, viscosity and solid content being determined.

temperature resulted in an acceleration of the cast- a concentration of  $1.6$  mg g<sup>-1</sup> which previous work

had found to result in the lowest viscosity<sup>17</sup> and once again the solid content was  $72 \text{ wt\%}$  (39  $\text{vol\%}$ ) alumina. These slips are termed polyacrylate slips and form slip B. For coagulated slips, both ammonium and sodium chloride were investigated as a potential coagulating salt. However, once again the ammonium salt was found to result in skin formation at elevated temperatures and thus work with this salt was discontinued. The sodium chloride was added to a  $60 \text{ wt}$ % (27  $\text{vol}$ %) slurry dispersed at pH 4 with nitric acid using two concentrations of salt,  $0.05 \text{ mol dm}^{-3}$  and  $0.075$  mol dm<sup>-3</sup>, and form slips C1 and C2. The use of sodium salts for slips B, Cl and C2 was not ideal since sodium is known to be detrimental to the final properties for alumina ceramics however since this work focuses only on the rheological characteristics of the slips under different conditions it was deemed acceptable. It should also be noted that slips C1 and C2 were based on  $60 \text{ wt\%}$  solids content rather than 72 wt% because otherwise the viscosity rose to above 1 Pa s and this was found to be unsuitable for slip casting experiments.

The rheological characteristics of the slips were determined using a Bohlin Visco 88 BV viscometer with a concentric cylinder arrangement. The shear stress-shear rate curves were obtained over a shear rate range of  $18.5-1122 s^{-1}$  for both increasing and decreasing shear sweeps. The viscosity results were obtained using a continuous shear rate of  $18.5 \text{ s}^{-1}$ until equilibrium was attained. The rheological characteristics of the slips were also monitored in the same manner at elevated temperature, with the slips being heated via either a water bath or microwave oven to the required temperature, and after cooling back to 20°C.

Settling tests were undertaken on acid dispersed slips in a fridge/convection oven using sealed perspex tubes at temperatures ranging from 7 to 65°C. As the particles in the suspension settled, the height of the lower, cloudy part of the suspension was monitored until it was constant for 48 h.

## **Casting and characterisation of green ceramics**

Castings were carried out using  $1 \mu m$  glass microfibre filters 0.68 mm thick using a Buchner funnel apparatus, (Fig. 1) at a vacuum pressure of 260mmHg. Castings were performed at 20, 45 and 65°C for pre-determined time intervals with the thickness of the as-cast body being used for casting rate determinations. The elevated temperature castings were carried out using both a microwave oven and convection oven. The electric field distribution in the microwave chamber was determined to ensure that casting was carried out under conditions of uniform heating. The solid contents of the slips were also measured prior to casting, dur-



**Fig. 1.** Schematic of equipment used for vacuum filter casting.

ing casting and after cooling back to 20°C to allow comparison to allow any changes to be noted.

The densities of the dried green bodies were determined by the Archimedes technique using mercury. The cast microstructures were examined using a Joel 6400 scanning electron microscope on bodies 'biscuit' fired for 1 h at 1100°C in an attempt to provide information on the particle structures formed during casting.

## **Results and Discussion**

#### **Slip characterisation**

The viscosities of the slips at ambient temperature  $(20^{\circ}C)$  are given in Table 1. They show that slips A, B and Cl had similar viscosities and were essentially dispersed whilst slip C2 displayed the higher viscosity of a coagulated suspension. Figure 2 shows shear stress versus shear rate curves for slip A as a function of temperature using a water bath to obtain the higher temperatures. It indicates that although there is some evidence for a small amount of thixotropic behaviour due to incomplete dispersion, the shear stress is lowest for the intermediate slip temperature of 45°C. Almost identical results were obtained when the slips were heated using microwave energy. This result is supported by the relative viscosity values in Fig. 3 which suggest that the slip viscosity is independent of heating method used. It can also be seen that the relative viscosity of the slip compared to that of water remains approximately constant until about 40°C after which an increase is obtained, suggesting a change in state of dispersion in the slip to a more flocculate state. On cooling the slip back to  $20^{\circ}$ C it was noted that the viscosity did not return to its original value but increased to a considerably higher value. Specifically, it decreased from its initial value of 145 mPa s at 20°C to 120 mPa s on

**Table 1.** Viscosities of slips at ambient temperature (20°C)

Slip	Solid content	Dispersal coagulant agent	Nature of slip	Viscosity range $(mPa s)$
A	$72 \,\mathrm{wt}$ %	Acid	Dispersed	120–140
B	$72 \,\mathrm{wt}$ %	Polyacrylate	<b>Dispersed</b>	$95 - 105$
C <sub>1</sub>	$60 \,\mathrm{wt}\%$	$0.050$ mol dm <sup>-3</sup> salt	Dispersed	$70 - 90$
C <sub>2</sub>	$60 \,\mathrm{wt}$ %	$0.075$ mol dm <sup>-3</sup> salt	Coagulated	$290 - 320$

heating to  $65^{\circ}$ C, passing through a minimum of about 105 mPas at 45"C, but then increased back to 210 mPa s on cooling back to 20°C. Experiments showed that this could be only partially accounted for by water evaporation, hence it is further evidence for a change in the state of dispersion on heating.

The study of the settling behaviour of slip A (Fig. 4) suggests that the lower the temperature the slower the degree of settling, i.e. the greater the degree of dispersion. This supports the idea that a degree of partial flocculation or coagulation occurs



**Fig. 2.** Shear stress/shear rate curves for slip A at different temperatures and heated using a water bath.



**Fig. 3.** Relative viscosities of slip A as a function of temperature.

with increasing temperature. Although the degree of settling appears less at  $65^{\circ}$ C than at  $20-45^{\circ}$ C, the slip had a virtually clear supernatant (the supernatant remained slightly cloudy for the tests at  $7-45^{\circ}$ C) implying that a larger, denser sediment was being formed.

The slips containing sodium polyacrylate (slip B) also displayed no significant difference in their behaviour whether they were heated with microwave or conventional energy. Both have the same trends of decreasing viscosity and shear stress (Fig. 5) with an increase in temperature to 45°C. The curves produced are rheologically very similar to those obtained with slip A.



**Fig. 4.** Settling behaviour for slip A as a function of time at various temperatures.



**Fig. 5.** Shear stress/shear rate curves for slip B at different temperatures and heated using two different techniques.

The shear stress curves for slips containing  $0.05$  mol dm<sup>-3</sup> sodium chloride, slip C1, showed similar behaviour again, Fig. 6, with the minimum shear stress occurring at 45"C, particularly at higher shear rates. By 65°C the shear stress has increased and a yield stress has appeared with the dilatancy point moving to higher shear rates. This indicates that the slip is probably in a coagulated state by this temperature. Figure 7 displays the rheological data for slip C2, containing  $0.075$  moldm<sup>-3</sup> sodium chloride, as a function of temperature. In this case a yield stress occurred at all temperatures and the shear stress increased with temperature rather than decreasing to a minimum around 45°C. In addition, the dilatancy point remained the same which is believed to indicate that there is no fundamental change in the state of dispersion of the slip. Plots of viscosity versus shear rate also showed an increase in viscosity with temperature and the slip became significantly pseudoplastic, an observation which has also been reported by Velamakanni et *al.'* for coagulated slips. Figure 8 shows the relative viscosity values for slips Cl and C2 compared to water at a steady



**Fig. 6.** Shear stress/shear rate curves for slip Cl at different temperatures and heated using a water bath.



**Fig. 7.** Shear stress/shear rate curves for slip C2 at different temperatures and heated using a water bath.



**Fig. 8.** Relative viscosities of slips Cl and C2 as a function of temperature.

shear rate of  $18.5 s<sup>-1</sup>$  and where the heating was achieved using a waterbath. As noted for slip A, the viscosity of the lower salt concentration slip is relatively stable until  $40-45^{\circ}$ C when an increase is observed which is consistent with the reduction in the degree of dispersion. Slip C2 shows a steady increase in viscosity at all temperatures which is consistent with its coagulated nature.

# **Evaluation of casting data**

Figures 9–11 display the casting rates achieved for slips A, B and C, respectively, as a function of



**Fig. 9.** Vacuum filter casting rates for slip A at different temperatures.



**Fig. 10.** Vacuum filter casting rates for slip B at different temperatures.



**Fig. 11.** Vacuum filter casting rates for slips Cl and C2 at different temperatures.

temperature, with the numerical values being reported in Table 2. Table 3 shows the ratio of the casting rate at elevated temperature to that at 20°C. The ranges of the green densities obtained for the cast bodies produced by each slip are provided in Table 4. There was no discernable trend with either increased casting time/thickness or temperature for either slips A or B. However, as observed in Fig. 12, slip Cl displayed a steady increase in density with casting time at 20°C and a two stage density curve at 45°C. Up to a thickness of about 2.5mm (which corresponds to a casting time of about lOmin), the density increased with casting time. However after this point there was no further increase in density. No differences were noted in the densities of the microwave cast and convection oven cast bodies. Slip C2 also showed a two stage density curve. However this was found at both 20 and 45°C and so there was no observable variation in density with casting temperature or heating method. Finally, it should be noted that none of the slips displayed a significant change in its solids content as a result of the casting process. In general the solids content increased by  $\lt 1\%$  even when casting at a temperature of 65°C indicating that moisture loss was not a significant problem.

**Table 2.** Casting rates for vacuum filter cast bodies as a function of slip type and temperature

Casting temperature	Casting rate ( $mm2 min-1$ )			
$(^{\circ}C)$	Slip A		Slip B Slip C1	Slip C <sub>2</sub>
20	0.48	0.74	0.38	$0.77^a$ $0.46^{b}$
$45 \text{ mW}$	0.73	1.33	1.13 <sup>a</sup> $0.36^{b}$	$2.15^a$ 0.91 <sup>b</sup>
oven	0.61	$1-18$	1.05 <sup>a</sup> $0.34^{b}$	2.15 <sup>a</sup> $0.88^{b}$
$65 \text{ m}$ w	0.99			
oven	0.92			

<sup>a</sup>lst linear portion.

b2nd linear portion.

**Table 3.** Ratio of casting rates at elevated temperatures to  $20^{\circ}$ C

Casting temperature $(^{\circ}C)$	Ratio of casting rates at elevated temperature to $20^{\circ}$ C			
			Slip A Slip B Slip C1	Slip C <sub>2</sub>
$45 \text{ m}$ w	1.5	1.8	3.0 <sup>a</sup> 0.9 <sup>b</sup>	2.8 <sup>a</sup> 2.0 <sup>b</sup>
oven	1.3	1·6	2.8 <sup>a</sup> 0.9 <sup>b</sup>	2.8 <sup>a</sup> 1.9 <sup>b</sup>
$65 \text{ m}$ w	2.1			
oven	1.9			

<sup>a</sup>1st linear portion.

<sup>b</sup>2nd linear portion.

The examination using the SEM of the microstructures of the cast bodies heat treated to 1100°C failed to show any difference in the particle packing regardless of the slip, casting time or casting temperature used. This can be attributed to the fact that once the bodies were removed from the filtration equipment the drying process caused particle rearrangement, masking any differences that there might have been in the microstructures of the bodies.

Three observations may be drawn from Figs  $9-12$  and Tables 2-4. Firstly, in all the cases examined there is an increase in casting rate with an increase in casting temperature. Secondly, it may be noted that coagulated slips behave differently to dispersed slips. Whilst thirdly, there is generally a slightly enhanced casting rate when using microwave energy compared to that achieved with the convection oven.

Considering the second observation. The plots for slips A and B (Figs 9 and 10) are linear at all temperatures measured and that for Cl (Fig. 11) is linear at 20°C whilst the plots for slip Cl at 45°C and for slip C2 at both 20 and 45°C show changes in gradient after about 10 min. Thus dispersed slips display linear increases in casting rate with increasing temperature whilst coagulated slips display a rapid initial increase after which the casting rate increase is smaller. The rheological data discussed earlier provided evidence for a degree of partial flocculation appearing in slip A at temperatures above about  $40-45^{\circ}$ C and yet no step change in casting rate was observed. To attempt to explain this result it is necessary to consider results obtained in a related study by the same authors where the work was based on the slip casting of slip A as a function of temperature.<sup>17</sup> In this study it was observed that whilst the permeability of the cast decreased with increasing casting time as would be expected, it also decreased with increasing casting temperature-which was not expected. It is postulated that the effect of the change in state of dispersion of the slip at elevated temperatures is

**Table 4.** Green densities of bodies vacuum filter cast from slips

Slip	Density range		Comments	
	$(g \, cm^{-3})$	$\frac{\gamma}{6}$ )		
A	$2.38 - 2.45$	$60.0 - 62.3$	Natural scatter; no trend with casting time or temperature	
B	$2.29 - 2.34$	$58.3 - 59.5$	Natural scatter; no trend with casting time or temperature	
C1	$2.15^{a} - 2.33$	$54.79 - 59.3$	Casting temperature of $20^{\circ}$ C: steady increase in density with casting time	
	$2.16^{a}-2.28$	$55.0^{a} - 58.0$	Casting temperature of $45^{\circ}$ C: increasing density with time over first 10 min	
	$2.25 - 2.31$	$57.3 - 58.8$	No trend with further increases in casting time	
C <sub>2</sub>	$2.21^{a} - 2.23$	$53.9^{\circ} - 56.7$	No trend with temperature: increasing density with time for first 10 min	
	$2.19 - 2.25$	$55.7 - 57.3$	No trend with further increases in casting time	

"Based on extrapolation.

offset by the decrease in permeability of the cast, resulting in the linear plots observed in Fig. 9. This explanation does not seem particularly satisfactory and it is believed that there are more factors still to be determined. It is assumed that the same explanation, if true, would account for slip B, which forming a lower density green body than slip A, retained a higher casting rate.

With slip C2 an initially low density body formed, possibly due to particle bridging occurring. This permitted a high degree of permeability for the water in the slip to pass through the body which in turn resulted in a fast casting rate. However, this time the density of the body increased with casting time and casting thickness resulting in a decrease in casting rate once a critical value had been reached. This increase in particle packing has been attributed to particle rearrangement and the movement of particles within the fluid through the cast to remove the area of voidage formed during the first minutes of casting.18 From Table 4, it would appear that the critical value is in the region of 55-56% of theoretical for the  $60 \text{ wt}$ % solids content slip C2. Slip Cl is more complicated. At 20°C the slip rheologically behaves as if it is dispersed, however the density of the green body can be seen to increase steadily with casting time/



Fig. 12. Green densities of bodies vacuum filter cast from slip Cl as a function of cast thickness for different casting temperatures

thickness, more in line with a coagulated body. However there is no critical value of density, and hence permeability, when the casting rate changes. At  $45^{\circ}$ C, the rheological behaviour of the slip is more akin to that of a coagulated suspension and we see the same type of casting behaviour as observed for slip C2 although the casting rates are approximately half those of slip C2 even though the green densities are not much different. As a final comment, care should be taken when comparing the numerical values for the casting rates achieved for slips Cl and C2 against slips A and B since different solids contents were used.

Considering the third observation noted above, that there is generally a slightly enhanced casting rate when using microwave energy compared to that achieved with the convection oven, a similar result was found during the slip casting experiments with slip  $A^{17}$  although the effect was larger. On the basis of mould permeability measurements, the mechanism of acceleration was believed to be due to the rate of water movement through the mould being increased more than the rate of water uptake by the mould. This suggested that during microwave-assisted slip casting the water movement within the mould was faster than with conventional heating. This was attributed to the volumetric nature of heating when using microwaves, ie the energy reaches the water directly rather than relying on conduction via the mould. This has the effect of keeping the mould slightly dryer and so accelerates the process. Since the movement of moisture through the 0.68 mm thick glass microfibre filters will not be affected in the same way as a plaster of paris mould, it follows that the smaller effect observed in this study is primarily caused by an increased moisture movement through the cast itself.

#### **Conclusions**

Increasing the temperature of an alumina-based, dispersed slip results in an increase in the flocculated nature of the slip, the effect becoming increasingly notable above about 45°C. By 65°C the slips display significantly flocculated behaviour. However, for slips which display coagulated behaviour at room temperature, there appears to be no fundamental change in the state of dispersion of the slip on heating. The work performed to date would not permit anything other than purely speculative theories to be put forward to account for this phenomenon, hence this issue will be addressed elsewhere when further work has been performed.

The use of elevated temperatures during the vacuum filter casting of a range of alumina slips has resulted in increases in the casting rates in all cases. However it has been determined that coagulated slips behave differently to dispersed slips. The latter display casting rates which increase linearly with increasing temperature whilst coagulated slips display a rapid initial increase after which the casting rate increase is smaller. This is believed to be due to the interaction between the permeability of the green body and the state of dispersion of the slip.

For dispersed slips the increasing permeability of the body being formed with increasing temperature appears to have offset the increasingly flocculated nature of the slip, even though this is a most surprising result. However, the coagulated slips formed an initially low density green body, possibly due to particle bridging occurring, which resulted in a highly permeable cast structure. This in turn resulted in a fast casting rate. However due to particle rearrangement and the reduction in the area of voidage formed by subsequent particle deposition, the density of the cast increased with casting time resulting in a decrease in casting rate once a critical density, and hence permeability, had been reached.

A further observation arising from the work is that there is generally a slightly enhanced casting rate when microwave energy is used to heat the slips compared to that achieved with a convection oven. On the basis of the results presented here, and those obtained in a related study, $17$  the mechanism of acceleration is believed to be due to the rate of water movement *through* the cast being increased more than the rate of water uptake  $by$  the cast. This suggests that during microwave-assisted vacuum filter casting the water movement within the cast is faster than with conventional heating. This is attributed to the volumetric nature of heating when using microwaves, i.e. the energy reaches the water directly rather than relying on conduction via the cast.

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