Journal of the European Ceramic Society 22 (2002) 135–142

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Enhanced casting rate by dynamic heating during slip casting

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Received 15 June 2000; received in revised form 18 February 2001; accepted 4 March 2001

Abstract

The slip casting process is widely used to consolidate ceramic particles from aqueous suspensions to form green compacts, particularly those with complicated shapes. Raising the slip temperature during slip casting is known to increase the casting rate, this is believed to be via a decrease in water viscosity. However, differences have been observed when using convection and microwave heating to raise the temperature. In the present work, it has been found that the use of short-pulses of microwave energy to heat the casting system dynamically causes a greater degree of acceleration than when using conventional radiant heating. The increased uptake of water from the slip by the porous mould is believed to be indicative of a vaporisation–condensation cycle mechanism. A negative pressure would be created during the condensation stage of the cycle, acting as an additional suction force to the capillary action and hence accelerating the casting process. © 2001 Elsevier Science Ltd. All rights reserved.

Keywords: Clays; Microwave processing; Slip casting; Suspensions

1. Introduction

Slip casting is the process by which a porous mould, typically made of plaster of Paris, is filled with a slip consisting of a suspension of a fine ceramic powder in liquid. The capillary action due to the pores in the mould withdraws the liquid medium from the slip. As the liquid penetrates the mould, a cast layer is simultaneously formed on the plaster surface. The porous structure of the mould provides a suction pressure ranging from 0.1 to 0.2 MPa (1–2 atm).[1]

The quality and strength of the final product depends on the microstructure of the cast layer. Highly porous bodies formed by a flocculated slip will usually shrink unevenly during drying and sintering, resulting in cracks and reduced strength whilst dispersed slips result in less particle aggregation and better structural uniformity. In general, aggregated slurries lead to compressible bodies that are characterised by a variation in porosity with respect to distance. The structure is less consolidated at the cast surface and becomes more compact as the plaster is approached. The cast layer structure as reflected by the porosity variation is intimately tied to particle size, shape, degree of aggregation, and slurry concentration.[2]

A major disadvantage of slip casting is that it can be time intensive with the speed of casting ranging from hours to days for large objects. Hence, technologies are desired which can increase the casting rate without introducing heterogeneities into the component produced. Methods of accelerating the consolidation of ceramic particles have been studied by many researchers.[2–6] The basis behind most of the methods is manipulation of the parameters that control casting; these can be expressed generically by Eq. (1):

$$L^2 \propto \frac{P.t}{\eta} \tag{1}$$

where L is the thickness of the body, P the differential pressure across the system, t the casting time and η the viscosity of the suspension. Most previous work has examined the potential for changing the differential pressure by either applying increased pressure to the slip or vacuum to the mould.[7] However, a number of studies have been performed where the slip temperature is increased to achieve a decrease in its viscosity.[8–10] The

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PII: S0955-2219(01)00243-6

use of microwave energy to increase the temperature of the slip has been studied by several authors [11–14] and a significant increase of casting rate demonstrated, often higher than that achieved through conventional radiant heating of the slip to the same temperature. However, the precise mechanism for the increased casting rate has not yet been satisfactorily explained.

2. Experimental

Slip casting experiments were performed using a clay slip supplied by Sanindusa, Portugal, which had a density of 1.8 g/cm^3. The mould used was made of plaster-of-paris (produced with a plaster: water ratio of 3:2) and consisted of a right cylindrical hollow measuring $\phi 60 \times 27$ mm in a plaster block measuring $\phi 85 \times 52$ mm. Castings were carried out under three different sets of conditions: (i) at 20°C, 60% humidity in the open laboratory, (ii) at 45 and 65°C in a standard convection oven (oven), and (iii) at the same temperatures in a 1.3 kW microwave oven (MW). With the elevated temperature castings using the convection oven, the slip and mould were heated separately to the required temperature prior to casting, this typically took about 2 h, whilst with the microwave oven the slip and mould were heated rapidly from cold. The rheological behaviour of the slips heated with convection oven and microwave oven were measured and it was found that there was no difference. During the measurement, the cylinder sample holder was immersed in a water bath adjusted to the temperature required.

After the slip was poured into the mould, the mould top was covered with a polymeric film (NESCOFILM, Bando Chemical Industrial. Ltd, Japan) to ensure that all the water removed from the casting system came from the mould and there was no direct evaporation from the slip surface. The output of the microwave oven was controlled via a temperature controller connected to a thermocouple immersed in the suspension. The accuracy of the temperature measurement was verified by heating water to known temperatures before the first casting was performed. The microwave oven was turned off and on (pulsed) automatically once the temperature of the slip had reached its pre-set value, typically requiring only a few seconds of power at a time to maintain the slip temperature. After casting for a predetermined time, the excess slip was poured away from the mould and the cast body was removed from the mould 10 min later. The cast body was then allowed to dry at room temperature, about 20°C, for one week. At least 6 measurements of cast thickness and three of cast density (by mercury immersion) were made on each slip cast body. The green microstructure was also examined using scanning electron microscopy.

To investigate the effect of slip viscosity, the original slip was also modified using sodium silicate. It was found that the slip exhibited its lowest viscosity at a sodium silicate concentration of 0.1 wt.%. The viscosity of the original and modified slips are shown in Fig. 1. It can be clearly seen that the original slip displayed both shear thinning and thixotropic behaviour. The latter suggested the presence of a weakly flocculated structure within the slip. When the slip was modified with the addition of 0.1 wt.% sodium silicate the thixotropy disappeared. These results suggest a higher state of dispersion for the slip.

In order to investigate if the casting mechanism varied with heating method, the water movement within the casting system was determined. This involved obtaining the following measurements: the mass of the dry mould before casting, W_m; the mass of slip poured into the mould, W_s; the mass of the mould plus slip on completion of casting, W_{ms}; the mass of the wet mould after draining the surplus slip and removal of the cast body, W_w; and the mass of the cast body, W_c.

The water evaporation from the mould, W_e, was calculated from Eq. (2) and the water content within the mould after casting, W_h, from Eq. (3). The total content of water removal from the slip (W_t) was calculated from Eq. (4).

$$W_e = W_m + W_s - W_{ms} \tag{2}$$

$$W_h = W_w - W_m \tag{3}$$

$$W_t = W_e + W_h \tag{4}$$

3. Slip casting model

The filtration of a suspension through a porous media can be described by Darcy's law:[16]

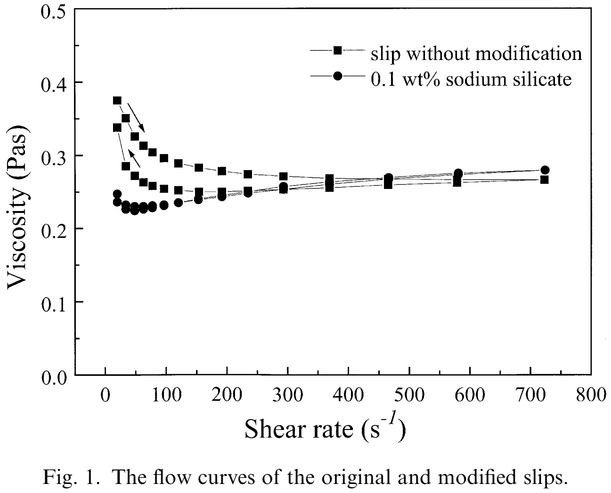


Fig. 1. The flow curves of the original and modified slips.

$$dP/dL = \eta q/K \quad (5)$$

where dP/dL is the pressure gradient, η is the viscosity of filtrate medium, q is the apparent flow rate of the filtrate, and K is the permeability of the porous media, i.e. the cast layer. Rearranging Eq. (5) we get:

$$q = KdP/(dL\eta) \quad (6)$$

The thickness of the cast layer, L, will be proportional to the volume of slip to have been filtered, Q, the solid content of the slip, k, and the area of filtration, s:

$$L = kQ/s \quad (7)$$

and the flow rate q will be represented by Eq. (8).

$$q = (1-k)dQ/dt = KdP/(dL\eta) \quad (8)$$

$$Q(1-k) = KtdP/(dL\eta) \quad (9)$$

$$Ls(1-k)/k = KtdP/(dL\eta) \quad (10)$$

$$LdL = KktdP/[s\eta(1-k)] \quad (11)$$

Hence the cast layer thickness can be expressed as a function of casting time t by:

$$L^2 = 2KktP/[s\eta(1-k)] \quad (12)$$

$$L^2 = \varepsilon Pt/\eta \quad (13)$$

For an ideal system in which k, s, K, P and η are constant, then L^2 will increase linearly with casting time, t. ε Is referred to be the system parameter, which is a constant for an ideal system.

For a real casting system, the suction pressure, P, will be determined by the mould and the filtrate involved.[15]

$$P = 4\delta/(D\rho) \quad (14)$$

Where δ is the surface tension of the liquid and ρ is the density of the liquid. D is the diameter of the capillary. If the porous structure of the mould is considered to consist of many horizontal capillaries then there will be no gravitational force to balance the pressure and, hence, it will remain constant throughout the casting process until the mould becomes fully saturated. It should be noted that this simplification opposes the view of Hampton et al.[16] who suggested that the absorption of water within mould will cause the pressure to drop. It should be remembered that when using Darcy's law that P is the pressure applied by the mould to the casting layer, not the pressure of the casting front.

The thickness of cast layer for similar suspensions but with different dispersion states will be determined by the packing of the particles in the layer, which in turn determines the permeability of the layer. For a dispersed suspension, the particles will be densely packed in the cast layer during filtration and there will be limited secondary rearrangement of the particles after they have landed on the cast layer surface, as shown in Fig. 2A. However, when the suspension is weakly flocculated, clusters of particles exist in the suspension. Therefore, the cast layer will have a greater degree of porosity but will be highly compressible. As the layer builds up (much more rapidly than with the dispersed slip), the drag force will accumulate from layer to layer through points of particle contact. Local porosity will decrease in the direction of flow as the accumulated drag force increases. Therefore, the innermost region or layer (L_1) will be the most compact because it sustains all of the accumulated drag force as shown in Fig. 2B. The outmost layer (L_2) will be very loose. This layer has high porosity and may slip away after de-moulding. N.B. It should be noted that there is unlikely to be a hard interface between the different regions for the flocculated suspension shown in (Fig. 2B), this is considered for simplicity.

4. Results and discussion

4.1. Casting rate and cast structure

The comparison between theoretical and experimental changes of cast layer thickness with casting time is shown in Fig. 3. As has been indicated earlier, the theoretical treatment of the slip casting process considered that the permeability of the cast layer remained unchanged during casting. It can be seen from Fig. 3A–C that when the original, weakly flocculated slip was used, the casting rate was not consistent with the theory for castings produced at room temperature or at elevated temperatures when heated by the convection oven. The deviation from Darcy's law occurred after the cast layer exceeded a critical thickness of about 6 mm and is believed to have been due to the increased resistance to water movement through this layer.[17] As has been explained above, the layer itself will have been formed because the suction force on the particle aggregates will have been too weak to form a relatively dense cast layer.

The cast bodies from the flocculated suspensions were soft and easily deformable for all castings including at 20°C. However, it was particularly noticeable that the outer layer of the cast bodies produced at elevated temperatures using convection heating was of high moisture content and hence, very low density and consequently broke away easily from the cast body when draining the excess slip out of the mould. This observation supports the model proposed in Fig. 2B and may have led to the deviation from theory observed in Fig. 3. However,

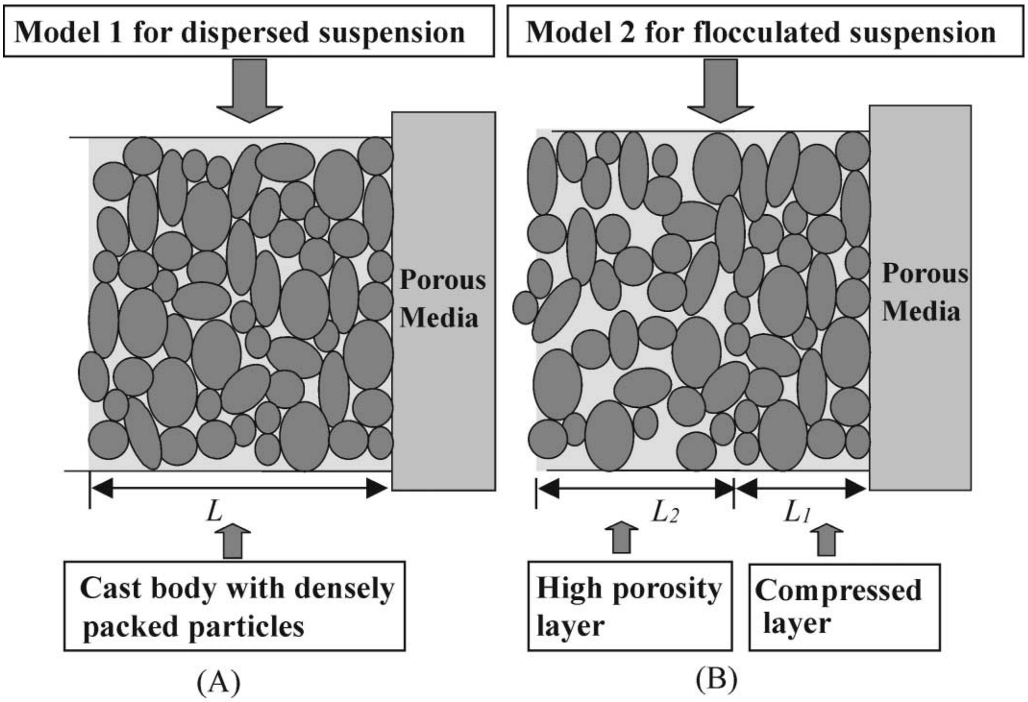


Fig. 2. Schematic representation of the slip casting process for a well-dispersed suspension (A) and a weakly flocculated suspension (B).

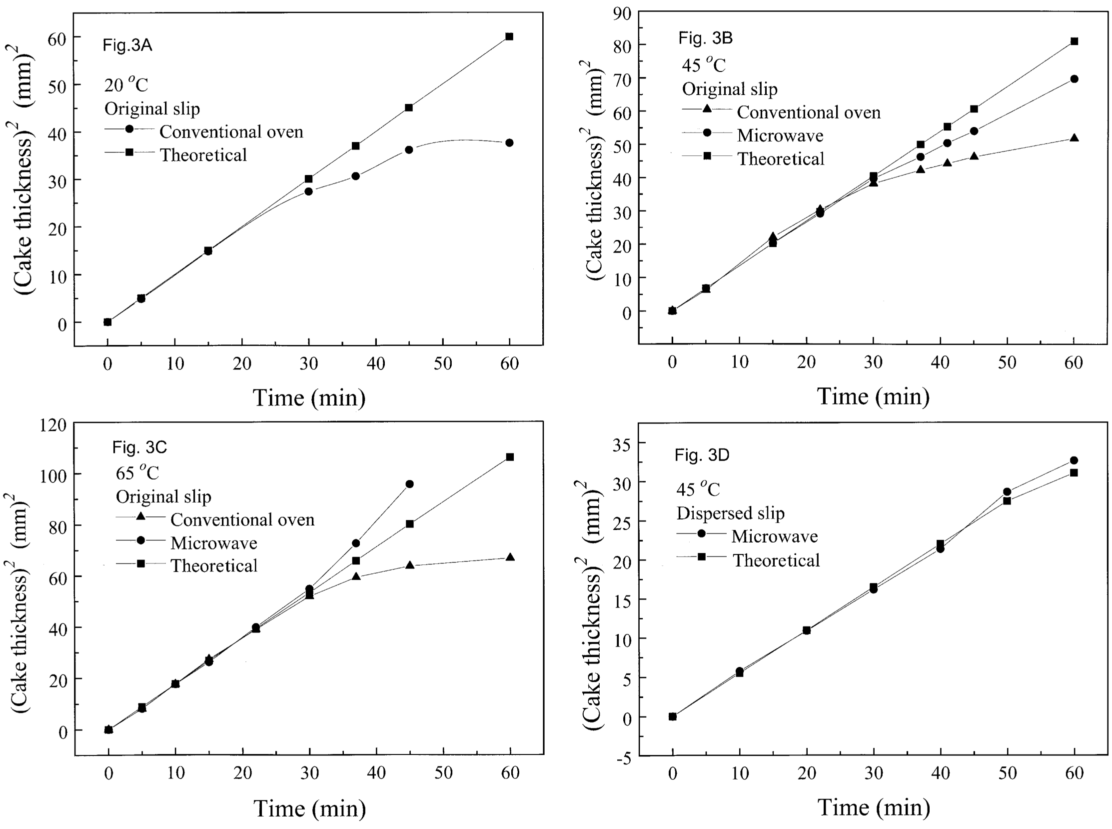


Fig. 3. The theoretical and experimental changes of cake thickness with casting time. (A) 20°C original (flocculated) slip, (B) 45°C original (flocculated) slip, (C) 65°C original (flocculated) slip, (D) 45°C modified (dispersed) slip.

when casting a slip heated by pulsed microwave energy, although a slightly excessive thickness of cast was achieved when casting at 65°C a good fit to the theoretical prediction was achieved at 45°C with both the weakly flocculated and dispersed suspensions (Fig. 3B–D). These bodies proved to be more rigid, suggesting a lower water content within the bodies. The casting process deviated very much from theoretical prediction when the casting carried out at 45 and 65°C heated by convection oven for weakly flocculated slips.

When slip casting the dispersed (modified) slip, the casting rates observed were consistent with the theoretical prediction at both room temperature (not shown in Fig. 3) and elevated temperatures when microwave heating was utilised. The cast bodies produced were observed to be much stiffer and hence contained a much lower water content than the bodies produced from the original, weakly flocculated slip. The casting rate was also significantly reduced. This was due to the highly dispersed nature of the particles in the slip[18,19] which consolidated to a denser structure as indicated in Fig. 2A.

4.2. Water movement within the casting system

Water movement within the casting system under different casting conditions is illustrated in Figs. 4–6. It is believed that at room temperature, the water movement within the mould was purely due to the capillary forces from the mould and there was negligible evaporation. From Fig. 4 it can be seen that the water movement from cast layer to mould was increased by the use of microwave energy whilst the evaporation process from the mould was enhanced by the convection heating. The process of water movement as a function of casting time can be clearly seen in Fig. 5. As expected, at room temperature the evaporation of water from the mould was negligible even after 60 min casting. However, when the slip was heated by microwave energy, noticeable water started to evaporate from the mould after about 20 min casting. These phenomena suggested that the water in the mould moved faster toward outside of the mould when the casting system was under microwave heating than at room temperature.

The changes in water content within the mould under different casting conditions are clearly shown in Fig. 6. When casting was performed at room temperature, the water content within the mould became constant after about 45 min. This probably indicates that the mould reached its saturation point since it is known that negligible amounts of water evaporated from the mould at room temperature. Similar behaviour was observed with microwave heating. The water content within the mould increased with casting time until after about 45–55 min, depending on the temperature, the mould became saturated and the water content began to level off (or even decrease due to evaporation). The contribution of the capillary force to the overall suction force should be low after this point as a result of saturation of the mould.

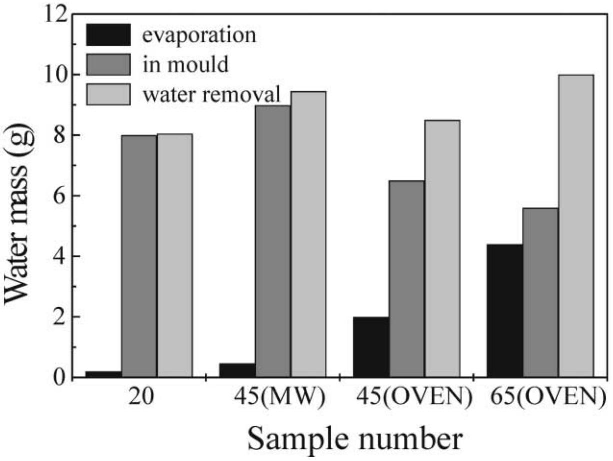


Fig. 4. The effect of heating method on water movement (after 50 min casting).

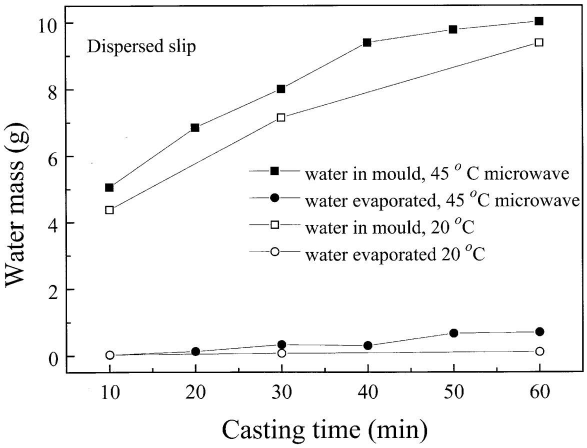


Fig. 5. Water movement as a function of casting time for casting performed at room temperature and with microwave heating.

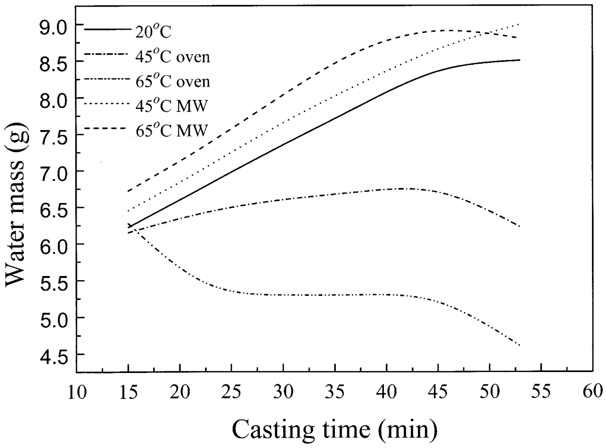


Fig. 6. Changes in water content within the mould as a function of casting time for different casting conditions.

However, the casting rate shown in Fig. 3 does not show a matching decrease.

The data for casting in the convection oven is quite different to that obtained either at room temperature or using microwaves. At 45°C, after an initial increase at the onset of casting, the water content increased only slightly for the period from about 15–40 min, prior to decreasing again. This suggests that over this period the rate of water evaporation from the mould approximately balanced the rate of up-take by the mould. Then the evaporation rate began to dominate, presumably because the water was nearer the surface of the mould and thus easier to evaporate. When casting at 65°C, for a 10 min period from 15–25 min the water content in the mould actually decreased prior to becoming constant. Presumably this was due to a shift in the permeability of the cast layer being deposited. The near constant water content within the mould from about 25 to 40 min casting indicates the rate of up-take by the mould is equal to that of evaporation. These reveals that the mould was kept at low level of water content during casting by convection heating and hence, dryer mould. Consequently, it may be reasonable to assume that the enhanced casting rate and better samples produced when pulse microwave energy was used for heating was attributed to the dynamic effect using pulse microwave.

As the slip and mould was at room temperature prior casting, the temperature of the water and slip would be increased dramatically as microwave energy was applied, whilst the mould was cooler because of its transparent to microwaves. Therefore, a significant amount of water vapour would be created within the mould, and hence cause a positive pressure to push the water within the capillary towards the outer skirt of the mould. As a result, a faster movement of water within the mould was observed. However, when the microwave energy was off, the water vapour at the outer part of the mould would condense first to seal the capillary in the mould. As a result, a low pressure phase would be created which could then act as an extra suction force besides the capillary force. The above predictions and explanations may help to explain the observed higher casting rate and stiffer cast bodies.

4.3. Characterisation of the cast body

The green bodies produced under the different heating methods were examined by measuring their density and through scanning electron microscopy of their microstructures. To allow equivalence, all the samples examined had been cast for 42 min and the SEM photos were sampled from the middle part of the cross-sections. The densities are shown in Fig. 7 whilst the microstructures are reproduced in Fig. 8.

It can be clearly seen from Fig. 7 that the density of the bodies cast using original slip at different conditions was virtually identical. The SEM images display that there are no significant differences in the microstructures for samples cast either at room temperature or elevated temperature heated by convection oven and microwave oven. However, it can still be observed from the SEM image of green body cast with dispersed slip that the structure is relative denser and finer than the rest. The green body also exhibited apparently higher density. These differences in density and microstructure are attributed to higher degree of dispersion status of

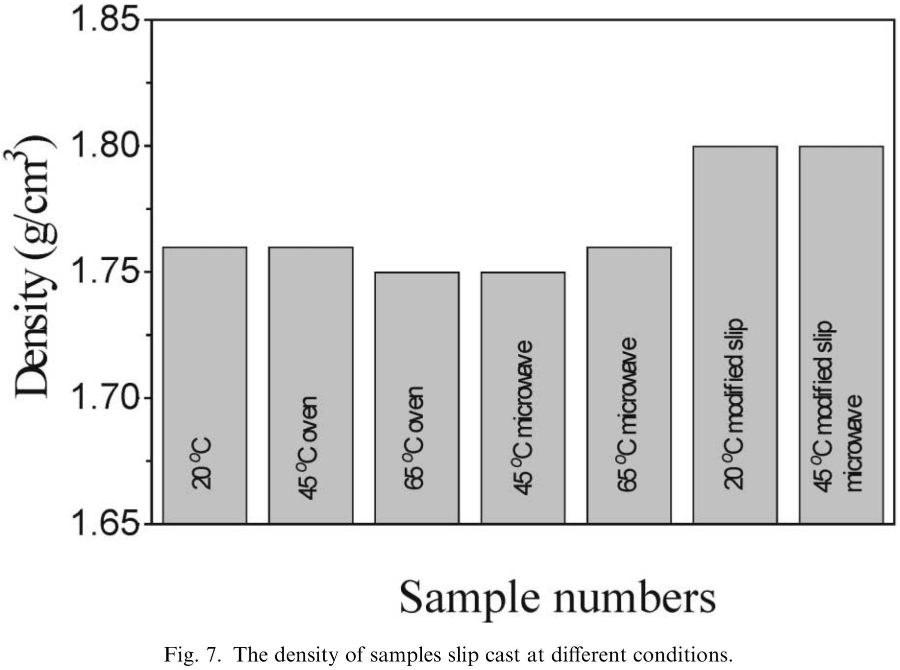
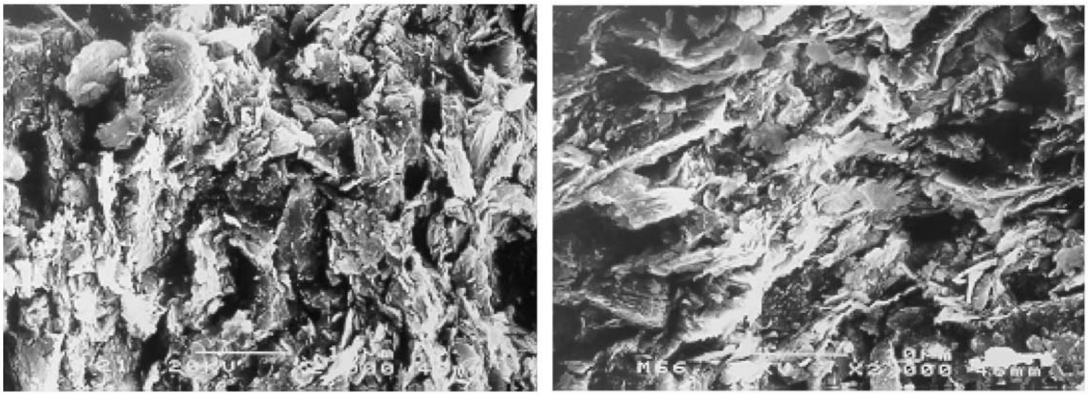
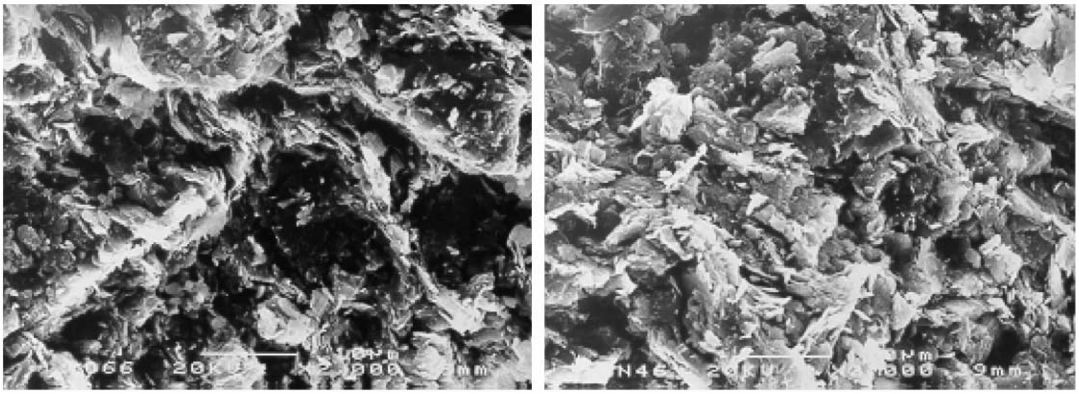


Fig. 7. The density of samples slip cast at different conditions.



(A) 20°C original slip (B) 65°C MW, original slip



(C) 65°C Convection Oven, original slip (D) 45°C MW, dispersed slip

Fig. 8. The microstructure of green bodies cast at different conditions.

particles in the slip. It was also found that there were no significant differences in the microstructure across the fracture section of cast body. It is very likely that the drying process caused particle rearrangement with the removal of the residual water. This would mask significant difference in the original microstructure of the body, especially the inside layer of the cast body before dried up. Nevertheless, the green body cast using dispersed slip has higher density and this is consistent with the observation of green body which had less content of water than others.

5. Conclusions

Casting rate can be increased at higher casting temperature. When the casting rate was counted, the dispersion state of slips should be comparable. A sustained high casting rate could be achieved by dynamic heating with pulse microwave energy. Water movement through the mould depended on the heating source and it was found that pulse microwave heating removed water from the slip more efficiently. Pulse microwave heating produced a better green body for handling and there were no significant differences in microstructures for green bodies cast. It is also possible that due to the pulsed nature (on–off cycles) of the microwave energy used, a low pressure phase will have been created within the mould, most possible at the inner part of the mould due to the vaporisation and condensation of water and hence, the water would move dynamically within the mould. The vaporisation–condensation cycles would result in a high-pressure and a low-pressure short period leading to faster movement of water within the mould, higher casting rate and better green body. The use of conventional radiant heating increases the casting rate by decreasing the viscosity of water and the maintenance of dryer mould could not assist the casting process.

References

1. Aksay, I. A. and Schilling, C. H., *Processing of Ceramics, Glasses, and Composites*, ed. L. L. Hench and D. R. Ulrich. Wiley, New York, 1984, pp. 483–491.
2. Tiller, F. M. and Tsai, C.-d., Theory of filtration of ceramics: I, Slip casting. *J. Am. Ceram. Soc.*, 1986, **69**(12), 882–887.

3. Adcock, D. S. and McDowall, I. C., Mechanism of filter pressing and slip casting. *J. Am. Ceram. Soc.*, 1957, **40**, 355–362.
4. Aksay, I. A. and Schilling, S. H., *Advances in Ceramics*, vol. 9, ed. J. Mangels. American Ceramic Society, 1984, pp. 85–93.
5. Hampton, J. H. D., *Experimental and Theoretical Studies of Filtration of Ceramic Suspensions*. PhD thesis, McMasters University, Montreal, Canada 1991.
6. Kostic, B., Kicevic, D. and Gasic, M., Mathematical modelling of ceramic slip casting. *Ceram. Int.*, 1990, **16**, 281–284.
7. Ferreira, J. M. F. and Diz, H. M. M., Pressure slip casting of bimodal silicon carbide powder suspensions. *Ceram. Int.*, 1999, **25**, 491–495.
8. Schramm, E. and Hall, F. P., Notes on casting slips. *J. Am. Ceram. Soc.*, 1934, **11**, 262–267.
9. Gastic, M. and Kostic, B., Fused silica slip casting at elevated temperatures. In *11th Riso International Symposium on Metallurgy and Materials*. Science Publisher Riso National Lab, Denmark 1991, pp. 291–296.
10. Tobin, L. W., Ceramic material processing. US patent 4292262, 1981.
11. Ferrari, C., Castlefranco, A., Zannini, P. and Mori, M., Fast automatic casting of sanitaryware. *CI News*, 1986, **6**(1), 7–12.
12. Binner, J. G. P., The effect of raising temperature using microwaves on the slip casting of alumina based ceramics. *Br. Ceram. Trans.*, 1992, **91**, 48–51.
13. Klieb, J., Microwave application to ceramic processing. *Canadian Ceram.,* October 1988, 18–19.
14. Deich, S. M. and Kaplan, E. D., Kinetics of sherd (body) formation during slip casting. *Glass and Ceramics*, 1997, **44**(5–6), 210–212.
15. Hampton, J. H. D., Savage, S. B. and Drew, R. A. L., Experimental analysis and modelling of slip casting. *J. Am. Ceram. Soc.*, 1988, **71**(12), 1040–1045.
16. Sobue, M., Sakai, J. and Nakamura, K., Analysis of the dehydration rate in slip casting process. *Journal of The Ceramic Society of Japan*, 1987, **95**, 309–315.
17. Haerle, A. G. and Haber, R. A., Real-time monitoring of cake thickness during slip casting. *J. Mater. Sci.*, 1993, **28**, 5678–5683.
18. Phelps, G. W., Particle size and permeability in slip casting. *Mater. Sci. Res.*, 1978, **11**, 57–65.