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Particle segregation phenomena occurring during the slip casting process

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Abstract

Homogenous microstructures are important to improve the reliability of the final ceramic products. This target is usually hindered by particle segregation phenomena that occur during colloidal processing when the suspended powders present significant differences in average particle size. In the present work binary suspensions have been prepared by mixing fumed silica ($D_{50}=0.07$ μm), with coarser silica powders (P600, $D_{50}=2.2$ μm and P10, $D_{50}=19$ μm) in different proportions. The effects of milling time, average particle size, proportion of the components in the mixtures and total solids volume fraction on the extent of particle segregation that occurs during unidirectional slip casting were evaluated. Particle size distribution analysis and scanning electron microscopy revealed that successive layers of fine/coarse/fine particles have been successively deposited during casting. The extension of segregation was dependent upon particles' size ratio, total solids volume fraction and milling time. It could be concluded that the clogging effect of the cake by the fine particles was the main mechanism responsible for particle segregation.

Keywords: A. Slip casting; A. Mixing; A. Suspensions; D. SiO_2; Clogging effect

1. Introduction

The obtaining of homogeneous green microstructures is an important target of ceramic processing, namely, for the sintering behaviour and the ultimate materials properties. In recent years the quality of the starting powders has been identified as an area in which considerable processing improvements are still possible [1,2]. By gaining better control over particle size, particle size distribution and particle shape, it may be possible to achieve a reduction in the number of defects in the ceramic product [3]. One of the most important features of any ceramic powder is the manner by which individual particles pack together in the powder compact. It is known that the packing ability of a given powder is improved by using colloidal shaping techniques, which enable to control the forces between particles within a liquid [4]. They also enable to take advantage of using mixtures of two or more powders with different particle sizes to further improve the green density, with the fine ones occupying the interstitial pores among the coarse ones [5–9].

However, if the suspending particles present significant size differences, such as in the case of traditional ceramics, particles' segregation can be a problem. Segregation phenomena usually occur during consolidation processes involving liquid removal, such as slip casting. These phenomena cause inhomogeneous green microstructures and, consequently, sintered bodies with low mechanical resistance. Several methods have been proposed to prevent particle segregation [10–14]: (1) increasing the solids loading, the consolidation rate, and suspensions' viscosity; and (2) controlling the size and particle size distribution, particle density and particle interaction forces.

The mechanisms responsible for particle segregation remained obscured and confusing in the past. The gravity force was often pointed out as the main cause of particle segregation. The clogging of the cake by the fine particles, which form a close packing layer adjacent to the mould surface, has been seldom referred to in the literature [15–18]. In some cases, the term "clogging effect" has been improperly used [15], while in other cases, the observed particle segregation has been erroneously attributed to the gravity force [16–18]. Hampton et al.

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[19,20] proposed a mathematical model to predict the growth thickness of a clogged cake. They consider that the fine particles were transported by the filtrate through an inner cake region, where the proportion of coarse and fine particles was about the same as in the slip, and deposited in the voids near the bottom of the cake. However, this model fails when all the slip inside the mould is going to be consumed, like in solid casting. Recently, Ferreira [21] demonstrated that the clogging effect was the main segregation mechanism acting during slip casting of SiC bimodal particles. A more plausible model was proposed, which could better explain not only its own experimental data but also other results already reported by other authors [16–18]. It could be concluded that two particle segregation mechanisms operate simultaneously. The first one occurs in the vertical direction due to the gravity force, which importance, for a given system, obviously decreases with increasing solids loading and the magnitude of attractive forces between the suspended particles. While the clogging effect occurs in any direction parallel to liquid flow and is mainly affected by the solids loading, the particle size ratio, the nature and magnitude of interaction forces between particles (usually controlled by adding dispersing agents), and driving force responsible for the deposition process.

The aim of the present work is to study the applicability of this model to describe the consolidation behaviour of bimodal silica suspensions. The extension of the segregation phenomena occurring during slip casting was evaluated as a function of particle size ratio, solids volume fraction and milling time of the powder mixtures. To reduce the number of experimental variables, no dispersing agent was used to stabilise the suspensions. Powders were just dispersed in distilled water at the natural pH (pH 5.5–6).

2. Experimental procedure

2.1. Slurries preparation and characterisation

A colloidal fumed silica (FS) (D_{50} = 0.07 μm—Aldrich Chemical Company), and two silica powders with different average particle sizes, P10 (D_{50} = 19 μm) and P600 (D_{50} = 2.2 μm), from Sibelco Portuguesa, SA, were selected as starting raw materials. Suspensions containing 5, 10 and 15 wt.% of FS and total solids volume fractions of 40, 46 and 50 vol.% were prepared with each of the different silica powders, P10 and P600. The suspensions were prepared by first adding the FS particles to distilled water under ultrasonic and mechanical stirring. Then, the other silica powder P10 (coarse) or P600 (fine) was progressively added and the final suspension kept under stirring for more 30 min. A portion of these suspensions was used for rheological characterisation and slip casting experiments. The remaining suspension was deagglomerated by ball milling for 20 h in a plastic container, using silicon nitride balls as grinding media (diameter = 15 mm) and then also used for rheological characterisation and slip casting experiments.

Rheological measurements were carried out with a rotational controlled stress rheometer (Carri Med 100 CLS, UK). The measuring configuration adopted was a cone and plate (Ø = 4 cm, 2°, gap = 53 μm) and flow measurements were performed in the shear rates range from ≈0.1 s^{-1} until 1200 s^{-1}. Before starting a measurement, pre-shearing was performed at high shear rate for 1 min followed by a rest of 1 min, in order to transmit the same rheological history to whole suspension being tested.

2.2. Preparation and characterisation of green bodies

Three cylindrical samples (Ø = 23 mm and thickness of about 8 mm) were prepared at RT from each suspension by pouring it into plastic rings placed onto a plaster plate to study the effects of total solids loading, particles size distribution, and milling time on the extent of particle segregation. After demoulding, the bodies were dried at RT for 6 h and then placed into an oven at 120 °C for 24 h for complete drying. Green densities were measured by the mercury immersion method using the Archimedes principle.

To evaluate the extent of segregation during the deposition process, powder samples were collected at three different height levels (bottom, middle and top) of each slip cast cylinder for particle size distribution analysis (Coulter LS 230, UK). For this, the particles were re-dispersed in water under ultrasonic stirring for 15 min before measurements.

The microstructural characteristics of the cylindrical cast samples at the same different levels (bottom, middle and top) were also analysed by scanning electron microscopy (SEM; Hitachi, Tokyo, Japan).

3. Results and discussion

3.1. Effects of solids loading and particle size on rheology of suspensions

The effects of total solids volume fraction and average particle size of the silica powders on rheological behaviour of suspensions prepared from mixtures of the coarser or the finer silica powders with 10 wt.% of FS are presented in Figs. 1 and 2, respectively. These plots show that viscosity increases with increasing solids loading, as expected, because an increasing number of particles per unit volume will offer a higher resistance to flow. The most salient (and apparently unexpected) observed features concern the effect of particle size of silica powders on rheology. In fact, the slips containing the coarser silica powder exhibit shear-thinning behaviours, while the slips containing the finer one show shear thickening behaviours. Further, the shear thinning or the shear thickening characteristics of these systems are accentuated by increasing solids loading. These results are somewhat surprising, since it is usually accepted that the presence of coarse particles in a suspension tends to impart a shear thickening effect, especially for high solid volume fraction [22,23]. This means that the flow behaviour is determined not only by the average particle size but also by particle size distribution [24].

The effect of milling time on rheology was studied for the suspensions containing 10% FS and a total solids concentration of 46 vol.%. Fig. 3 shows that viscosity of the suspensions decrease after ball milling for 20 h and that both the shear thinning and the shear thickening characteristics became less evident. These results can be attributed to the deagglomerating/milling effect, leading to a more favourable particle size distribution. The less resistance offered by the suspensions to flow means that the packing ability of the systems was improved in both systems. The sand P10 has a broader particle size distribution and would pack better in suspension compared with the sand P600 which presents a narrow particle size distribution [24].

The influence of the amount (5, 10 and 15%) of FS on the flow behaviour of the suspensions containing a total of 40 and 50 vol.% solids is presented in Fig. 4 (P10) and Fig. 5 (P600). It can be seen that adding increasing amounts of FS to the P10 suspensions enhance their shear thinning characteristics, while a contrary, i.e. the accentuation of the shear thickening character is observed in the case of P600 suspensions. Once again, these results reveal that particle size distribution is playing the most important role in the rheological

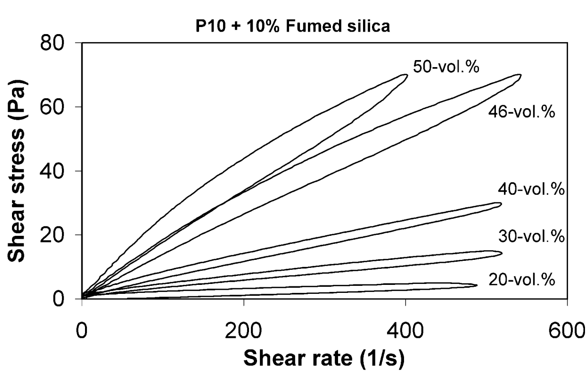

Fig. 1. Flow curves of suspensions prepared with silica powder P10 and 10% of FS at different solids loading.

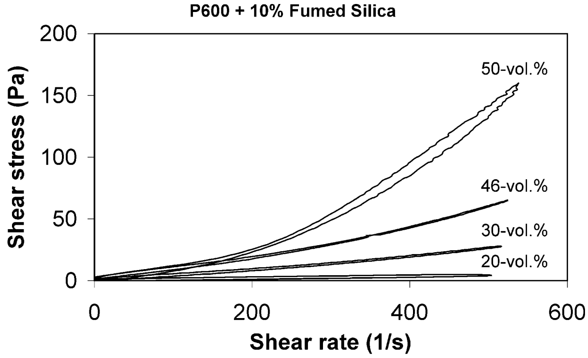

Fig. 2. Flow curves of suspensions prepared with silica powder P600 and 10% of FS at different solids loading.

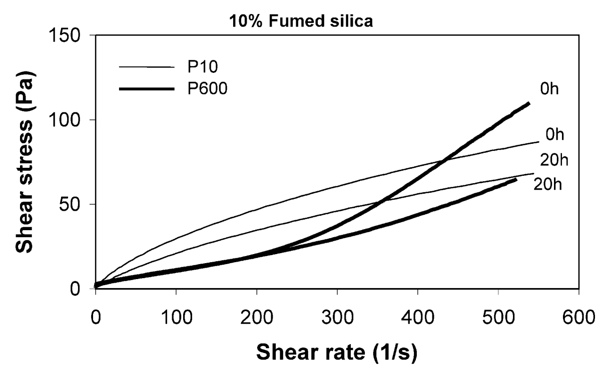

Fig. 3. Flow curves of suspensions containing silica powder P10 or P600 and 10% of FS, after stirring (0 h) and after milling (20 h).

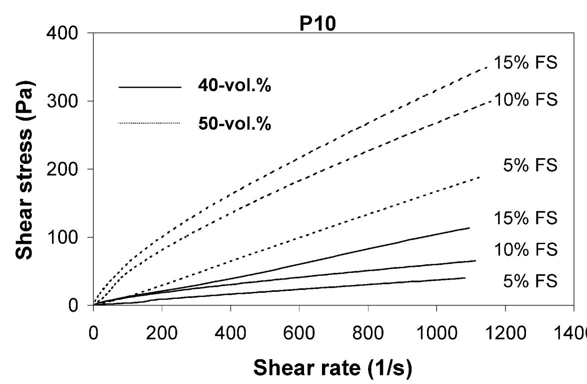

Fig. 4. Flow curves of suspensions containing total solids loading of 40 and 50 vol.% and different proportions of P10 and FS.

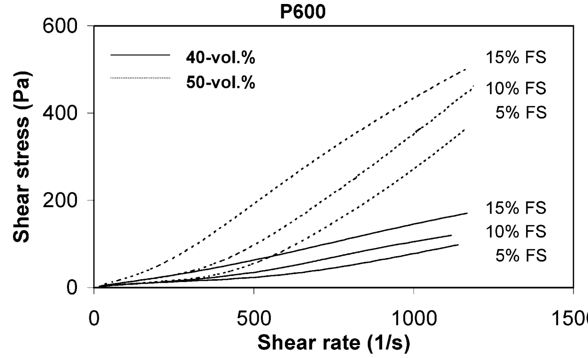

Fig. 5. Flow curves of suspensions containing total solids loading of 40 and 50 vol.% and different proportions of P600 and FS.

behaviour of the suspensions. The strong interaction between water and FS should also account for the observed results. In fact, it was observed that a gel-like structure was formed when FS was added to distilled water, which was accentuated with increasing amounts of FS. Adding coarser sand powders tended to destroy the gel-like structure, dividing the starting gel into small portions [25,26]. For the mixtures containing 15% FS it was even impossible to add the whole amount of FS due to the strong gel features of the suspensions. Further addition of FS was only possible after adding some of the coarser component, which improved the fluidity of the system. The presence of a gel-like structure would enhance the shear thinning character of the system. The portions in which a gel structure was divided would depend on the size of the particles introduced, being larger in the case of P10. This might contribute to the shear thinning behaviour observed with the coarser powder mixtures. In the case of P600 the FS gel is divided into smaller portions so that the intrinsic shear thinning character do not become appreciable. Instead, the lower particle size ratio and the narrowing of particle size distribution seem to play the main role in the case of P600 suspensions.

3.2. Effects of solids loading and particle size on green packing density and extent of particle segregation

The values of relative density of unidirectional slip cast samples obtained from different suspensions are reported in Table 1. It can be observed that the green density increases with increasing solids loading for the most part of the prepared mixtures, except in the case of P600 + 15% FS and P600 + 10% FS. Further, at given solids loading, the green density is higher for suspensions prepared with the finer silica powder (P600) at all FS contents, except in the case of P600 + 15% FS at 50 vol.%. For this silica powder, the green density increases with increasing amounts of FS from 5 to 15%, at the lowest solids loading (40 vol.%), but this trend is reversed for the suspensions with 46 and 50 vol.%. For the coarser silica powder, the relative density increases with increasing amounts of FS at 40 and 46 vol.%, attaining a maximum value of 64 at 50 vol.%, which is independent of the added amount of FS.

The lower green densities obtained from the mixtures containing the coarser silica powder can be understood since particle segregation due mainly to the clogging effect, but also to the gravity force is expected to occur

Table 1
Relative green densities of slip cast bodies obtained from suspensions with mixtures of silica powders and FS at different milling times and total solids loadings

	40 vol.%						46 vol.%						50 vol.%					
	P10			P600			P10			P600			P10			P600		
FS (wt%)	5	10	15	5	10	15	5	10	15	5	10	15	5	10	15	5	10	15
0 h	56	58	60	63	64	65	62	62	62	66	67	63	63	63	62	64	63	62
20 h	58	60	61	64	65	67	62	63	63	69	70	64	64	64	64	69	65	64

Table 2
Average particle sizes measured for powders collected at different height levels of the green cast samples (top, middle and bottom) from suspensions at 40, 46 and 50 vol.% total solid loading, before and after milling

Vol.(%)			FS (wt%)											
			5%				10%				15%			
			Total	Top	Mid.	Bot.	Total	Top	Mid.	Bot.	Total	Top	Mid.	Bot.
40	P10	0 h	17.9	4.88	18.7	15.1	14.6	7.22	15.9	11.2	12.2	6.86	12.6	9.96
		20 h	13.0	4.37	17.7	10.2	12.2	4.34	13.4	9.24	11.4	5.93	11.9	9.35
	P600	0 h	1.86	1.84	1.97	1.89	1.83	1.80	1.89	1.85	1.82	1.79	1.91	1.80
		20 h	1.82	1.74	2.01	1.76	1.81	1.77	1.86	1.79	1.77	1.73	1.85	1.75
46	P10	0 h	14.2	4.47	15.3	10.4	13.7	7.71	17.2	13.3	12.3	9.68	12.9	10.2
		20 h	11.7	3.68	11.9	9.38	10.8	5.27	11.5	10.4	9.52	6.54	10.2	7.23
	P600	0 h	2.27	2.07	2.39	2.29	2.17	2.22	2.35	2.28	2.05	1.76	2.15	1.98
		20 h	1.82	1.75	1.79	1.78	1.83	1.67	1.89	1.85	1.77	1.76	1.89	1.87
50	P10	0 h	14.1	4.36	14.6	10.1	11.7	7.75	14.7	12.8	13.9	12.1	13.4	13.2
		20 h	13.0	4.08	13.9	6.97	12.4	7.25	12.3	9.31	11.4	10.7	13.2	12.6
	P600	0 h	1.83	1.75	1.86	1.82	1.83	1.67	1.81	2.00	1.79	1.87	2.22	1.92
		20 h	1.81	1.75	1.78	1.77	1.79	1.64	1.83	1.88	1.77	1.87	2.20	1.76

more extensively. In fact, coarser particles are heavier and, consequently, more sluggish in moving towards the mould in the liquid flow driven by the capillary forces. The extension of segregation phenomena is expected to decrease with increasing solids volume fraction, as reported in Table 2 and Figs. 6 and 7. This explains why the green density increases with solids loading. The only exception observed for the mixture P600 + 10% and P600 + 15% FS means that other experimental variables such as the proportion between coarse and fine particles, size ratio, and dispersion degree are also playing a role. It is known from the literature [27] that, for a given system, the degree of particle packing should increase with increasing solids loading until a maximum value is attained, followed by a decreasing tendency. Such overall trend depends on the extension of segregation phenomena, entrapment of air bubbles in the suspension and in the cake with increasing viscosity, and a non-efficient dispersing of the powders at very high solids loading.

From the rheological results (Figs. 4 and 5), it is also expected that the increase of the proportion of FS will

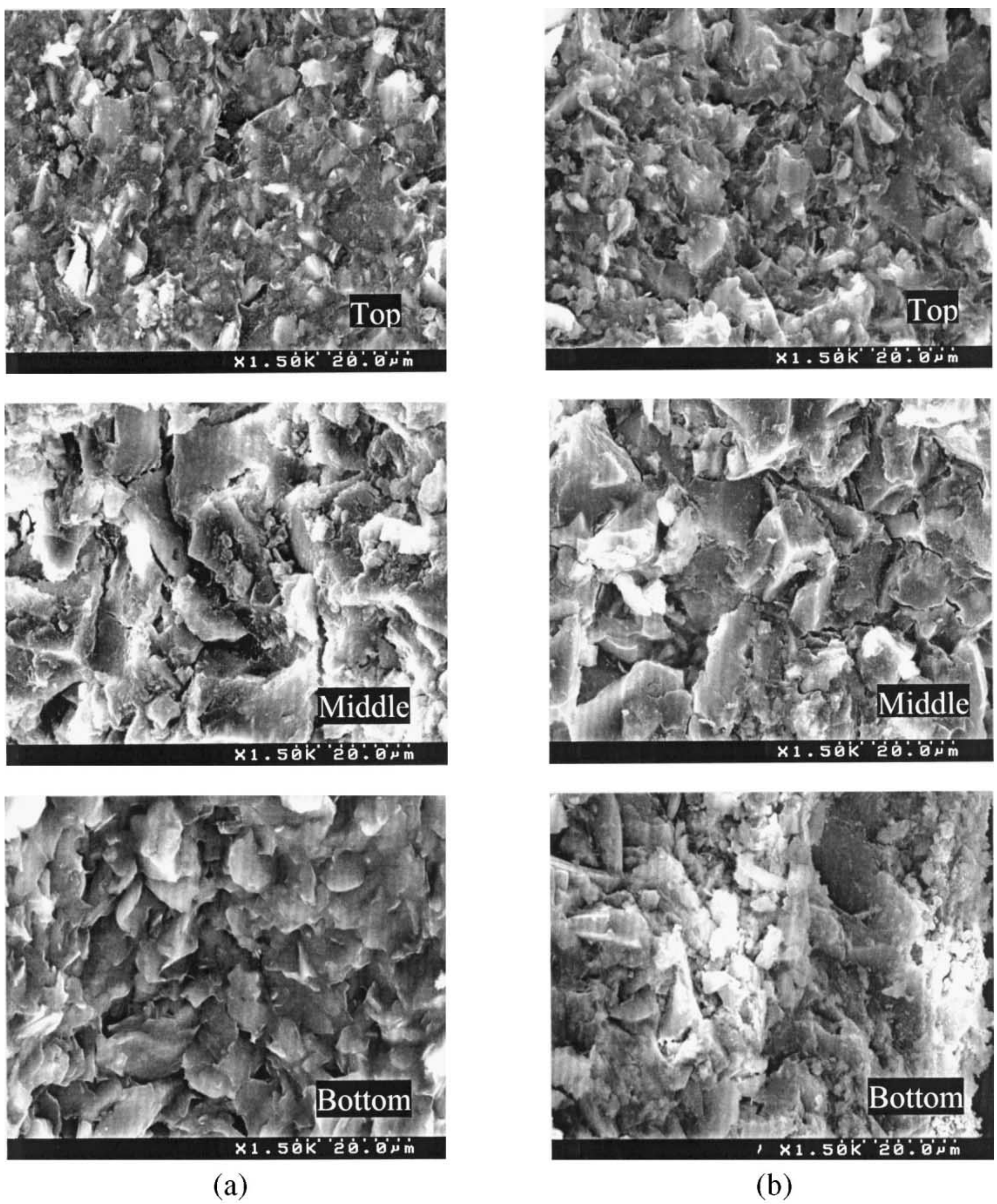

Fig. 6. SEM micrographs taken at different height levels of the green cast samples (top, middle and bottom) for P10 with 10% FS at different total solids loading: (a) 40 vol.%; (b) 50 vol.%.

originate a lower degree of particle segregation and, consequently, more homogeneous green bodies. This is confirmed by the average particle sizes measured at the different height levels in the cast samples (Table 2) and by the SEM microstructures (Fig. 8).

Table 1 also shows that for all mixtures prepared by stirring led to lower relative green densities compared with the ball milled suspensions. These results suggest that the extension of segregation phenomena should be more accentuated for all the suspensions prepared by stirring. This can be understood since the coarser particles are more prone to be broken during milling, changing the particle size distribution of the mixtures [24]. Table 2 shows that for all cast samples, the coarser particle fractions are located in the intermediate regions (middle) between the top and the bottom, while the finer ones concentrated preferentially at the top. At the bottom, the average particle sizes always present intermediate

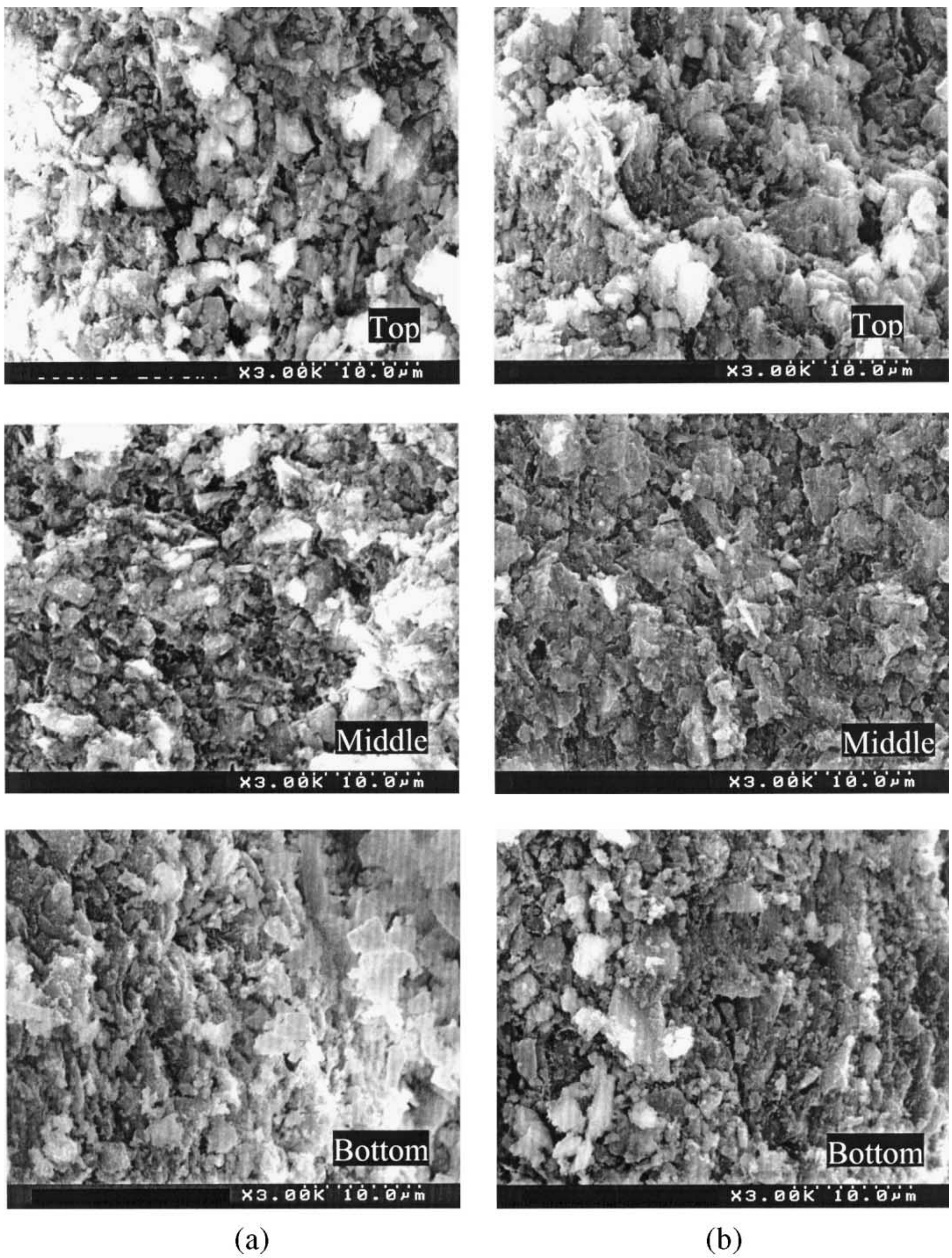

Fig. 7. SEM micrographs taken at different height levels of the green cast samples (top, middle and bottom) for P600 with 10% FS at different total solids loading: (a) 40 vol.%; (b) 50 vol.%.

values relative to the top and middle regions. Differences among average sizes measured at the three different levels decrease with increasing solids loading and with narrowing particle size distribution, as can be seen in Fig. 9. These results clearly show that gravity force plays a secondary role in particle segregation at all solids volume fractions tested, since the coarser fraction do not appear at the bottom but was preferentially concentrated in the middle region.

These results can be explained as follows. At the lower solid contents particles can flow and pack as individuals due to the higher fluidity of the suspension. The number of particles that reach the interface between the slip and the consolidated layer, per unit time, is

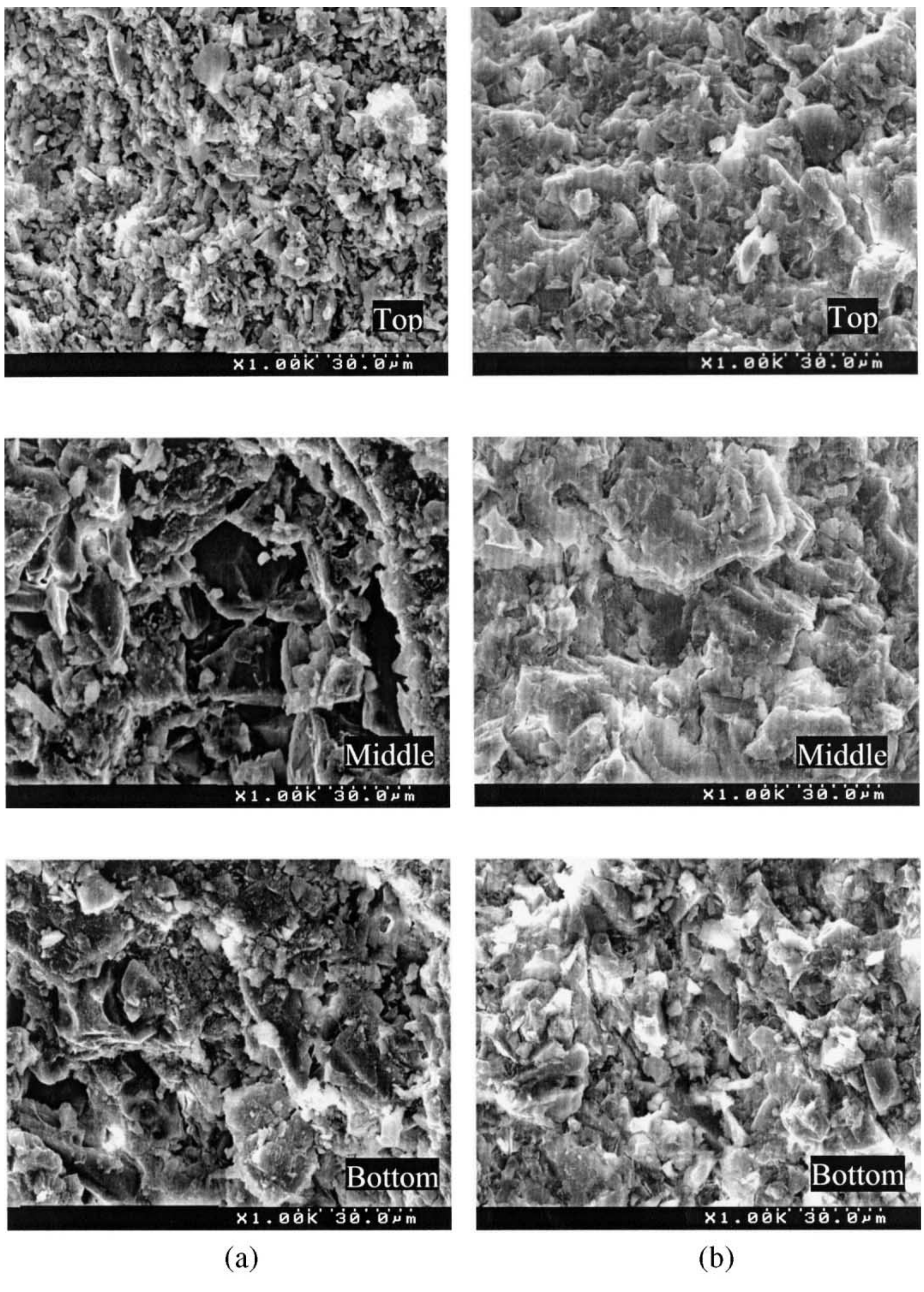

Fig. 8. SEM micrographs taken at different height levels of the green cast samples (top, middle and bottom) from suspensions containing a total solid loading of 46 vol.% and different proportions of P10 and FS: (a) 5%; (b) 15%.

lower and more time is allowed for each particle to be deposited. The liquid flow originated by capillary suction of the mould will first push the fine particles against the mould wall because of their lower inertia. These particles can form closed packed domains if the rate of deposition is relatively slow, as in slip casting, and if the resultant of the interaction forces is repulsive as can be expected from the natural pH (pH 5.5–6) of the suspensions, at which the zeta potential values of about −25 mV [24] are enough to electrostatically stabilise the systems [28].

The formation of closed packed arrays implies that the particles of a second layer will be deposited within the triangular pores of the first layer. It was already

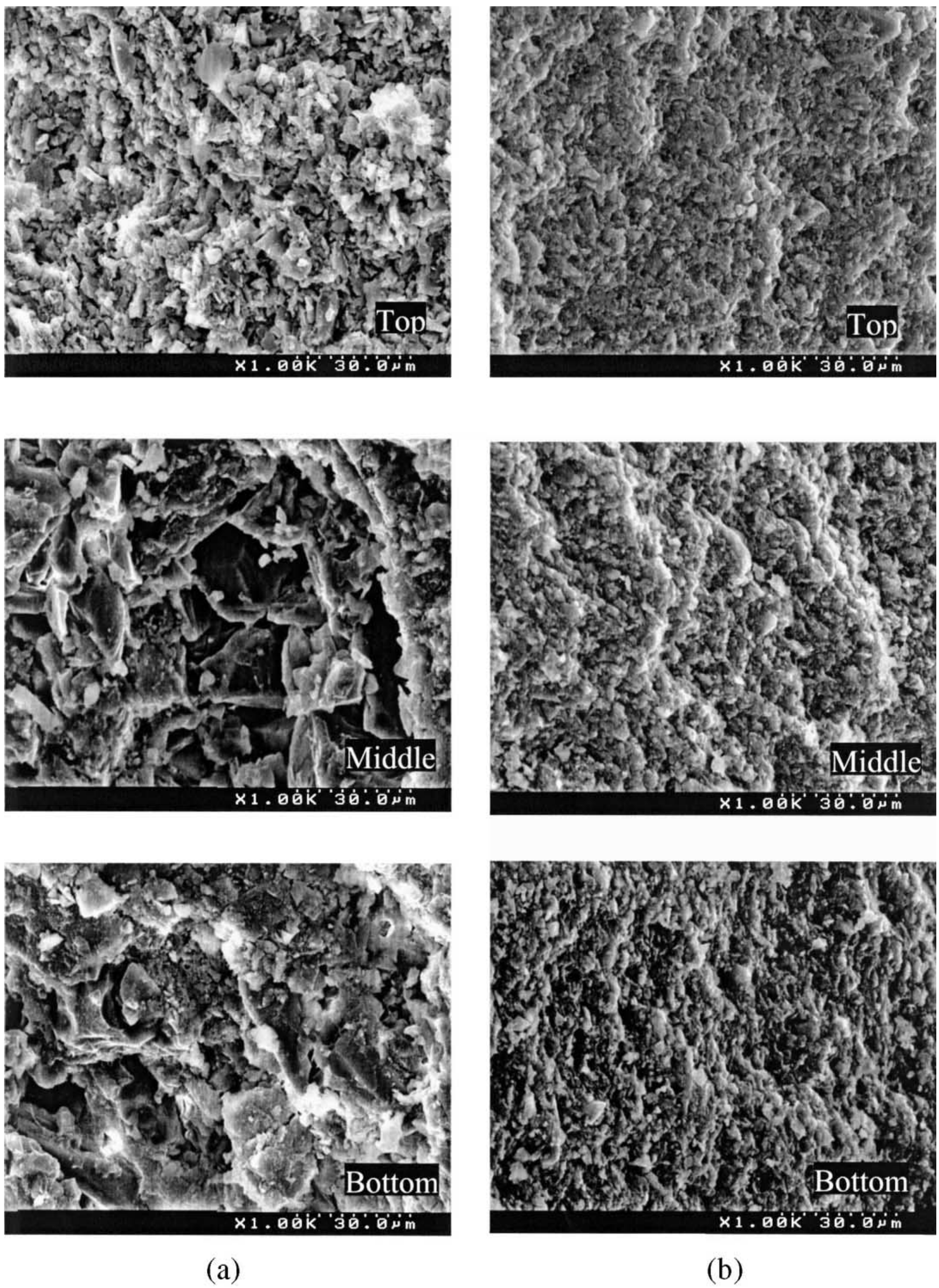

Fig. 9. SEM micrographs taken at different height levels of the green cast samples (top, middle and bottom) from suspensions at a total solid loading of 46 vol.%, containing 5% FS and different silica powders: (a) P10; (b) P600.

demonstrated that a fine spherical particle of the second layer can immerse a significant fraction (0.367) of its own radius in the top plan defined by the first layer, parallel to the mould wall, while a particle 10 times larger can only immerse a very small fraction (0.0016) of its own radius in the same plan [29]. In the present work, the size ratios between P10/FS and P600/FS are about 270 and 14, respectively. This means that from the coarse particles' point of view, the first layer formed appears too smooth. Therefore, when a coarse particle reaches the interface cake/suspension it is not laterally involved and its immediate fixation at the wall is uncertain. Under these conditions, is conceivable that the faster moving fine particles might pass underneath and push the coarser ones upwards enhancing the segregation by the clogging mechanism, according to a model proposed before [21]. The interface cake/suspensions can thus be regarded as a sieve.

As the cake thickness increases, the available driving force for the deposition process gradually decreases. At a given cake thickness the pressure drop along deposited wall might be such that the magnitude of the remaining driving force is no more enough to continue promoting the clogging of the cake. From this moment the gravity force will dominate the deposition (segregation) process. This explains why the coarser particles concentrate in the middle of the samples and the finer ones in the top. On the other hand, the possibility for some particles of the coarser component (probably the less coarser ones) to be fixed at the interface cake/suspension explains why the intermediate average size fractions are find at the bottom.

In summary, the segregation phenomena by the clogging effect is favoured by the difference in the inertia momentum (for example, a particle size ratio of 10 is equivalent to a weight ratio 1000, for a given material), by the freedom of the particles in suspension (repulsive forces, high fluidity) and by a moderate solids loading. At low solids loading, the number of particles that reach the interface cake/suspension per unit volume of absorbed liquid is small and the mould saturation is faster. Under these conditions the clogging effect would not play the dominant role. On the other hand, at high solids loading, the number of particles reaching the interface cake/suspension per unit volume of absorbed liquid is high and the rearranging movements of one particle on deposition are hindered by the new incoming particles, therefore decreasing the trend for particle segregation both by clogging effect and due to the gravity force.

In contrast, in a moderate range of solids concentration, the fine particles might have enough time for searching for the most favourable positions at the cake/suspension interface to enhance their packing degree. The repulsive forces existing between the fixed particles at the wall and those being deposited favour the rearranging movements. Under these conditions, segregation of particles is dominated by the clogging effect, as demonstrated in the present work.

4. Conclusions

The clogging of the cake by the fine particles is the predominant segregation mechanism, confirming the model that was proposed before. It depends upon the interplay between different factors, such as particle size ratio, solids loading and proportion between coarse and fine components in the mixtures.

The particle packing has been improved with the increasing of total solids loading and amounts of fine particles (FS) in the mixture. For all concentrations tested, it was possible to verify that the coarse particles are in the middle of the cast samples and the finer ones on the top and bottom. The suspensions that have a broader particle size distribution (P10 + FS) present a more visible particle size separation when compared with the ones with a narrower distribution (P600 + FS).

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