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Effect of Dispersants on the Rheology of Aqueous Silicon Carbide Suspensions

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Abstract

The effect of cationic and anionic dispersants on aqueous suspensions of as-received and surface-modified silicon carbide particles has been studied via observation of the rheological behaviour. Only the cationic dispersants were effective for the as-received SiC, with polyethyleneimine being superior to Hyamine 2389 probably as a result of a greater electrosteric interaction. SiC particles modified using $\text{Al}(\text{NO}_3)_3$ behaved like alumina and so could be dispersed using the anionic dispersants ammonium polyacrylate and polymethacrylate. Such dispersions displayed no heteroaggregation when alumina was added, although the order of mixing could significantly affect the rheological behavior of the suspension. Nevertheless, the suspensions appeared robust to slight fluctuations in pH.

Keywords: A: suspension; A: Rheology; D: SiC

I Introduction

Achieving adequate dispersion of ceramic powders is important for suspension-based ceramic green forming; it is generally necessary to prepare the suspensions with as high solid content and low viscosity as possible. Due to the wide use of alumina ceramics, the dispersion of these powders into water has been extensively studied and the dispersants used are commonly polyelectrolytes such as polyacrylic acid (PAA), polymethylacrylic acid (PMAA) and their (usually ammonium) salts [1,2,3,4,5]. However, there are very few reported papers on the dispersion of silicon carbide, SiC, powders into water.

A small number of researchers have used ammonia or sodium hydroxide solutions to adjust the pH of their suspensions [6,7,8], whilst Si *et al.* [9] used tetramethylammonium hydroxide (TMAH). In both cases the aim was to maximise the zeta potential of the SiC particles in aqueous suspension. In contrast, Sano [10] used styrene-maleic acid copolymers, the resulting 33 vol% solid content suspension exhibited a viscosity of only 190 mPa s at a shear rate of 6.9 s^{-1} , whilst other promising dispersants are the weak cationic polyelectrolytes polyethyleneimine (PEI) and polyvinylimidazole (PVI). The former has been used for the flocculation of silica[§], SiO₂, suspensions [11] whilst the latter, which has similar properties to

[§]The surface charge behaviour of SiO₂ and SiC is fundamentally similar because there is always a very thin layer of SiO₂ on the surface of SiC [12,13,14]. In both cases, therefore, the zeta potential is negative when the pH is higher than the point of zero charge (PZC), typically $\text{pH } 3 \pm 1$. The polyelectrolytes adsorb onto the surface of the particles due to the electrostatic attraction force between the particles and the ionised groups on the polymer segments, the level of adsorption also being dependent on the molecular weight of the polymer.

PEI and shows good adsorption onto the surfaces of the particles, has been studied for dispersing SiC powder [15]. However, in neither case was a rheological study of the resulting suspensions reported.

A further complicating factor when producing SiC ceramics is that sintering additives are usually required to generate liquid phases and hence lower the sintering temperature. The additives commonly used are alumina and yttria, however these have different surface charge behaviours compared to SiC. The PZC of SiC lies in the range pH 2 – 4, depending on the nature of the powder used [16,17], whilst that of Al₂O₃ is generally pH 8 – 9 [1,2,18]. Mixing these two powders in aqueous media therefore causes heteroaggregation due to the electrostatic attraction of the differently charged particles.

In order to make nonoxide powders such as SiC and Si₃N₄ compatible with Al₂O₃ in aqueous media, a number of surface modification techniques have been studied. For example, Hruschka *et al.* [19] has investigated the combined use of Al(OH)₃ and citric acid diammonium to modify Si₃N₄ particles in the pH range 9 – 10.5. Pei *et al.* [20] and Shih *et al.* [21] examined the effect of directly coating fine alumina particles onto the SiC and Si₃N₄ surfaces. In contrast Lidén *et al.* [22] coated aluminium tri-isopropoxide onto the surface of a SiC powder via mixing the two materials in acetone in a N₂ atmosphere; deionized water was then added to react with the alkoxide and the suspension dried to yield an Al₂O₃-surface coated powder. A similar method has been studied by Luther [23] to coat Si₃N₄ powder. In previous work by the present authors [24], SiC particles have been modified *in-situ* using Al³⁺ complexes in aqueous suspension via additions of Al(NO₃)₃ and control of the pH. The Al³⁺ formed hydrolyzed complexes that

adsorbed onto the charged sites on the SiC particle surfaces, as a result the SiC particles behaved in an alumina-like manner in the approximate pH range 5 - 8. The modified SiC particles were then further treated with two different polyelectrolytes that sequentially adsorbed onto the particle surfaces yielding a maximum surface charge. As a result of this surface modification process, the SiC could be co-dispersed with Al_2O_3 in aqueous suspensions without heteroaggregation occurring. However, the rheology of the suspensions was not studied.

In the present work, the effect of different dispersants on the rheology of aqueous SiC suspensions has been evaluated for both an as-received SiC powder and the Al^{3+} complex-based, surface-modified version described above with a view to developing a practical co-dispersion system for SiC and alumina.

II Experimental Materials and Methods

1. Materials

The ceramic powders used were silicon carbide, SiC (Ultrafein; ESK, Kempton, Germany) and aluminium oxide, $\alpha\text{-Al}_2\text{O}_3$ (99.9%; Alfa Products, Danvers, MA, USA). Both powders were measured, using a Mastersizer (Malvern Instruments Ltd, Malvern, UK), to have average diameters of $\sim 3\ \mu\text{m}$. Aluminium nitrate, $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (99.9%; Fluka Chemical AG, Buchs, Switzerland) was used for the initial surface modification work. Three chemicals were used as dispersants, viz.: Hyamine 2389 [HY], a 50 wt% solution of

methyldodecylbenzyltrimethylammonium chloride and methyldodecylxylenebistrimethylammonium chloride (Merck Chemicals Ltd, Poole, UK); polyethyleneimine [PEI], a 50 wt% solution with a molecular weight (mw) of 50,000 (Fluka Chemical AG, Buchs, Switzerland); and Dispex A40 [A40], an ammonium polyacrylate solution with a mw of 2500 (Allied Colloids Ltd, Bradford, UK). The latter dispersant was used in combination with KA11, an ammonium polymethacrylate solution with a mw of 10,000 (Allied Colloids Ltd, Bradford, UK). Analytical grade KNO_3 powder, NH_4OH and HCl solutions (Aldrich Chemicals Ltd, Gillingham, UK) were also used; the water was deionised.

2. Zeta potential measurements

The zeta potential of the as-received SiC particles in the presence of the HY, PEI and A40 and SiC particles surface modified with varying amounts of $\text{Al}(\text{NO}_3)_3$ in the presence of different concentrations of A40 were measured (Zetasizer4; Malvern Instruments Ltd, Malvern, UK) to evaluate the effects of the dispersants on the surface charges present. The procedure involved preparing 0.3 vol% suspensions using ultrasonic agitation (200 W; Kerry Ultrasonics Ltd, Herts, UK) for 3 minutes followed by a 15 minute period to allow the suspension to settle. 0.5 ml aliquots taken from the upper region of the suspension containing the finest particles were then added to extremely clean glass beakers containing 20 ml of 0.01M KNO_3 solution. The pH values (Jenway 3051; Jenway Co. Ltd., Essex, UK) of the dilute suspensions were adjusted within the range ~2.5 to 10 using 0.1/1 M HCl or NaOH solution. Four separate zeta potential measurements were then obtained on each sample to determine the consistency of the results.

3. Suspension preparation and viscosity measurements

The effect of the different dispersants on the rheological behaviour was evaluated by measuring the viscosities of both the as-received SiC and surface-modified SiC aqueous suspensions as a function of shear rate using a Visco 88 viscometer with 14, 25 and 30 mm diameter concentric cylinders (Bohlin UK Ltd, Cirencester, UK). For the as-received SiC, the procedure involved preparing suspensions containing a predetermined amount of powder, water and, separately, the HY, PEI and A40 dispersants; these were mixed by ball milling for 8 hours before the viscosity measurements were made.

The surface modification procedure for the SiC particles has been described in detail elsewhere [24]. Briefly, it involved mixing $\text{Al}(\text{NO}_3)_3$ solution into deionised water followed by addition of the SiC powder and pH adjustment to 5.8. After stirring for two hours, A40 was added followed by mixing in a ball mill for 4 hours and then KA11 followed by a further 4 hours of ball mixing. The effect of $\text{Al}(\text{NO}_3)_3$ concentration on suspension viscosity was investigated using 6.8, 13.2 and 21.3 mg of $\text{Al}(\text{NO}_3)_3$ per gram of SiC (referred to henceforth as mg g^{-1}) with levels of A40 from 0 to 24 mg g^{-1} . Subsequently, the effect of A40 concentration was studied using 2, 6.7 and 10 mg g^{-1} of A40 at concentrations of KA11 ranging from 0 to 16 mg g^{-1} . The effect of mixing sequence on viscosity was also investigated by using three experimental procedures: (1) SiC particles initially modified with $\text{Al}(\text{NO}_3)_3$, then A40 added and finally KA11, (2) SiC particles initially modified with $\text{Al}(\text{NO}_3)_3$, then KA11 added and finally A40, (3) $\text{Al}(\text{NO}_3)_3$, A40 and KA11 all added simultaneously to the SiC particle suspension.

The effect of suspension pH was investigated by adjusting it to 5.1 or 6.0 using either HCl or NH₄OH solution for 32 vol% solid content suspensions prepared with 13.2 mg g⁻¹ of Al(NO₃)₃ and 14 mg g⁻¹ of A40. Finally, a 32 vol% suspension made up of equal proportions of SiC and Al₂O₃ was prepared using 13.2 mg g⁻¹ of Al(NO₃)₃, 6.7 mg g⁻¹ of A40 and 8 mg g⁻¹ of KA11. The Al₂O₃ was added after the Al(NO₃)₃ but before the A40 to determine the possibility of preparing co-dispersions of SiC and Al₂O₃ using the surface modification process.

III Results and Discussion

1. Rheology of as-received SiC particle suspensions

The changes of zeta potential with increasing concentrations of HY, PEI and A40 are shown in figure 1. Since the HY and PEI were both cationic in nature, they carried a positive charge and so could be adsorbed onto the negatively charged SiC particle surfaces by electrostatic attraction. The isoelectric point (IEP) for the PEI occurred at ~3 mg g⁻¹ and its effect on zeta potential was significant for concentrations up to ~7 mg g⁻¹, whilst the HY, being less positive, was less effective. The IEP was reached at ~4 mg g⁻¹ and the zeta potential was lower at any given concentration. Since the A40 was anionic, its effect was to increase the negative zeta potential and the electrostatic interactions will have been repulsive. Similar behaviour was reported by Hruschka *et al.* [19] for the adsorption of citric acid diammonium onto negatively charged Si₃N₄ particles.

Viscosities for the as-received SiC suspensions as a function of shear rate and PEI

concentration are shown in figure 2a and b for solids contents of 40 and 47 vol% respectively. Figure 2a shows that a maximum occurred at a PEI concentration of $\sim 2.5 \text{ mg g}^{-1}$, matching closely the value for surface charge neutralisation indicated by figure 1. At concentrations of $\text{PEI} \geq 6.5 \text{ mg g}^{-1}$, when the zeta potential was nearing its maximum positive value, the viscosity was as low as 0.2 Pa s at 18 s^{-1} , even with a solid content of 40 vol%, showing that the degree of dispersion was high. Note, however, that when the solid contents and PEI concentration were high the suspensions became shear thickening at high shear rates, figure 2b. Since this behaviour was not observed with any of the other dispersants it is not believed to have been an artefact of the measurement system. Rather, it may have been due to the breakdown of the entanglement of the polymer chains, which, at a given shear rate, can result in shear thickening behaviour [25].

Figure 3 shows the viscosities for the as-received SiC suspensions as a function of shear rate and HY concentration. The suspensions were shear thinning at all shear rates, though the solids content investigated was only 30 vol%, and, at 18 s^{-1} , the viscosity of the suspension was a maximum at an HY concentration of 1.9 mg g^{-1} . This was about half what would have been expected based on charge neutralisation from figure 1. There are a number of reasons why this might have occurred. For example, it might be due to the physical nature of the surfactant; it has a long hydrophobic tail with a positive charge at the end that could give rise to a high steric effect. Alternatively, the explanation could simply be that the zeta potential measurements were obtained on low solids content suspensions of fine particles. This could have biased the data if the surface chemistry varied with particle size. Nevertheless, the best dispersion prepared using HY had a viscosity of about 0.6 Pa s at 18 s^{-1} , three times higher than for the PEI

despite the lower solid content used. Hence the HY was not as effective a dispersant as the PEI for preparing as-received SiC aqueous suspensions.

The viscosity curves of the suspensions prepared using A40 dispersant are shown in Figure 4. The lowest viscosity achieved was 0.6 Pa s at 18 s^{-1} for a solid content of 30 vol%, i.e. similar to that observed for the HY dispersant. The viscosity showed negligible change until the highest level of A40 used, 42 mg g^{-1} . Given that the change in zeta potential with increasing A40 concentration was extremely limited, figure 1, this suggests that saturation absorption was reached at less than $\sim 7 \text{ mg g}^{-1}$. Hence the very limited decrease in viscosity at the higher A40 concentration might be due to the weak depletion effect caused by free A40 in solution.

2. Rheology of surface-modified SiC particle suspensions

2.1 Effect of $\text{Al}(\text{NO}_3)_3$ concentration on viscosity

The viscosity results at 18 s^{-1} for the surface modified SiC suspensions as a function of A40 content for three different $\text{Al}(\text{NO}_3)_3$ concentrations are shown in figure 5a. It can be seen that all the suspensions behaved in a similar manner at low A40 concentrations, the viscosity decreasing with increasing A40 content because of the increased electrosteric force present. However, above about 8 mg g^{-1} the lowest and highest $\text{Al}(\text{NO}_3)_3$ concentration samples displayed a maximum in their viscosities. It is not certain why this behaviour was observed, though a similar phenomenon has been observed before when adding ammonium polyacrylate to alumina suspensions [26,27]. Then, it was attributed to the occurrence of different adsorbed configurations of the ammonium polyacrylate at the different concentrations.

It is known that the configurations of absorbed polyelectrolytes depend on the charge density ratio between the particle surface and polyelectrolyte, and that they affect the interparticle forces present [1,26]. A high charge density ratio results in a low-affinity adsorption and hence high interparticle forces whilst a low charge ratio results in the opposite behaviour [1]. In the present work, whilst the charge density of the particle surface will have changed with increasing A40 concentration, as indicated by the different isoelectric points observed in figure 5b for each of the 3 suspensions, that of the A40 will have been determined by the level of excess Al^{3+} species in solution. Hence, the charge density ratio will have been different at different A40 concentrations. As a result of the relatively high ionic strength of the suspension, the effects of any changes in the adsorption configuration on viscosity will have been potentially quite significant. Whilst this can be offered as a potential explanation of the results in figure 5a, a more definitive version will have to await further research.

The lack of a maximum in the curve for the suspension containing 13.2 mg g^{-1} of $Al(NO_3)_3$ is assumed to indicate that this was close to the ideal addition level. The hypothesis offered is that there were less excess Al^{3+} complexes present in the liquid phase and hence the configuration of the A40 did not change as a function of A40 content. It is also interesting to note how similar the zeta potential curve was for this suspension, at A40 concentrations greater than about 5 mg g^{-1} , compared to that for alumina dispersed with the same ammonium polyacrylate dispersant, figure 5b.

2.2 Dispersion via the combination of A40 and KA11 dispersants

Following the above results, the surface modification of the SiC particles was standardised by

using $13.2 \text{ mg g}^{-1} \text{ Al(NO}_3)_3$ for all further suspensions. The effect of varying the levels of the A40 and KA11 dispersants on the dispersion of the SiC particles, and hence on the viscosity, is shown in figure 6. When the A40 concentration was as low as 2 mg g^{-1} the SiC particle surfaces will have only been partially covered by A40 molecules and thus the KA11 will have been able to adsorb onto the remaining uncovered sites on the particles [24]. Although the total adsorption of the two polyelectrolytes was probably relatively low, yielding weak repulsion and hence high viscosities, as expected the viscosity decreased as more of the KA11 adsorbed. At higher concentrations of A40, more of the particle surfaces will have been covered by a layer of short chain ammonium polyacrylate, increasing the degree of negative charge on the surface and so restricting further adsorption of the longer chain KA11 [24]. At the highest A40 level, 10 mg g^{-1} , most of the particle surfaces will have been covered by a layer of the polymer preventing much KA11 from adsorbing. Overall this resulted in a viscosity that was low but changed little with increasing KA11 concentration. At the intermediate A40 concentration of 6.7 mg g^{-1} , the particle surfaces will have been only moderately covered and so the long chain KA11 polymer could still adsorb [24]. The long tail of the latter would be free to extend out into the liquid phase yielding a higher steric force, and hence a lower viscosity, than present with either 2 or 10 mg g^{-1} of A40, at higher KA11 concentrations.

2.3 Effect of mixing procedures on viscosity

The mechanism by which it is believed the $\text{Al(NO}_3)_3$, A40 and KA11 controlled the dispersion of the SiC particles in suspension was discussed in the previous sections; it follows that varying the order of mixing was expected to have a significant effect. To this end, two variations on the original procedure were evaluated; these were outlined in table 1 and the results are shown in

figure 7. As expected, it may be seen that procedure 3, where the $\text{Al}(\text{NO}_3)_3$, A40 and KA11 were all added simultaneously to the SiC particle suspension, showed the highest resultant viscosity indicating that unless the SiC particles were initially modified via the Al^{3+} complex mechanism the anionic polyelectrolytes were unable to adsorb onto the particle surface and the resulting behaviour was very similar to that for the as-received SiC particles with no dispersants added, figures 3 and 4. Once the SiC particles had been modified, it made only a small difference in terms of the resultant viscosity which of the two dispersants was added first, though it was clearly preferable at low shear rates when it was the A40. If the KA11 was added first, flocculation initially occurred and although the subsequent addition of the A40 helped obtain dispersion it was not to the same level.

2.4 Effect of pH on viscosity

All of the work reported above was performed at a pH of 5.8 since previous work had shown this to be the optimal value [24]. In order to understand the effects of slight pH changes on the rheological behaviour of the $\text{Al}(\text{NO}_3)_3$ -A40-KA11 dispersion system, NH_4OH and HCl solutions were used to modify the pH of the dispersion to 5.1 and 6.0 for the suspension prepared using procedure 1 (see table 1). The resultant viscosities were observed to be 0.3, 0.27 and 0.34 Pa s respectively at 18 s^{-1} . Despite the slight increases in viscosity when the pH was changed, the result implies that precise control of the pH is not essential for this process.

2.5 Effect of addition of alumina on viscosity

As indicated earlier, the purpose behind the surface modification of the SiC particles was to

develop a system that permitted the co-dispersion of SiC and Al₂O₃. To this end, even though such large quantities of alumina would not be used in practice, a suspension containing equal quantities of the two ceramic powders was prepared following procedure 1 (see table 1), with the alumina powder being added after the Al(NO₃)₃ and before the A40 and KA11. The viscosity as a function of shear rate is shown in figure 8 together with a similar curve for an alumina suspension of approximately similar solid content. Although the viscosity of the co-dispersion was slightly higher, probably due to the significant amount of NO₃⁻ present, the curves show that the surface modification process can be used to produce a SiC – Al₂O₃ co-dispersion with workable rheological properties.

IV. Conclusions

The overall aim of this work has been to investigate the potential for achieving stable SiC – Al₂O₃ co-dispersions. As-received SiC particles may be dispersed using Hyamine 2389 (HY) and polyethyleneimine (PEI), with the latter being the superior dispersing agent as a result of generating a stronger electrosteric interaction. As expected, the anionic dispersant Dispex A40 was not effective in this respect. When the SiC particles were surface-modified using Al(NO₃)₃, the concentration of the latter was found to be important in that it significantly influenced the rheological behaviour. When the Al(NO₃)₃ level was too low or too high, a maximum was observed in the viscosity / dispersant concentration plots at A40 concentrations >8 mg g⁻¹, however when 13.2 mg g⁻¹ of A40 was used a smooth curve was observed and the zeta potential plot was very similar to that for alumina at A40 concentrations ≥5 mg g⁻¹. A combination of A40 and KA11 was found to yield better dispersion. As with the Al(NO₃)₃ results, there was an

optimum level for both polyelectrolytes. If the initial A40 level was too low or too high it affected the viscosity by restricting the performance of the KA11, which is believed to have provided a strong steric dispersion effect. As a result, the order of mixing the three surfactants was also important; the SiC particles needed to be initially modified with the $\text{Al}(\text{NO}_3)_3$, then the A40 added and finally the KA11. However, slight variations in the suspension pH were found to have little effect on the rheological behaviour. As a result of the surface-modification approach, it has been possible to prepare SiC – Al_2O_3 co-dispersions that display no heteroaggregation.

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Table Captions

Table 1. Mixing procedures for investigating the effect of the order of surfactant additions.

Figure Captions

1. Changes in zeta potential with dispersant concentration for as-received SiC suspensions at a pH of 5.8.
2. Viscosity as a function of shear rate and PEI concentrations for a) 40 vol% and b) 47 vol% as-received SiC suspensions at a pH of 5.8.
3. Viscosity as a function of shear rate and HY concentrations for 30 vol%, as-received SiC suspensions at a pH of 5.8.
4. Viscosity as a function of shear rate and A40 concentrations for 30 vol%, as-received SiC suspensions at a pH of 5.8.
5. Viscosity (a) and zeta potential (b) as a function of A40 concentration for SiC suspensions modified with different concentrations of $\text{Al}(\text{NO}_3)_3$.
6. Viscosity as a function of KA11 concentration for $\text{Al}(\text{NO}_3)_3$ complex-modified SiC suspensions at different levels of A40.
7. Viscosity of SiC suspensions prepared with different mixing procedures. All suspensions 30 vol% solids content, $13.2 \text{ mg g}^{-1} \text{Al}(\text{NO}_3)_3$, $6.7 \text{ mg g}^{-1} \text{A40}$ and $8 \text{ mg g}^{-1} \text{KA11}$ at pH 5.8.
8. Viscosity as a function of shear rate for SiC – Al_2O_3 and Al_2O_3 suspensions.

Procedure	Description
1	SiC particles initially modified with Al(NO ₃) ₃ , then A40 added and finally KA11
2	SiC particles initially modified with Al(NO ₃) ₃ , then KA11 added and finally A40
3	Al(NO ₃) ₃ , A40 and KA11 all added simultaneously to the SiC particle suspension

Table 1. Mixing procedures for investigating the effect of the order of surfactant additions.

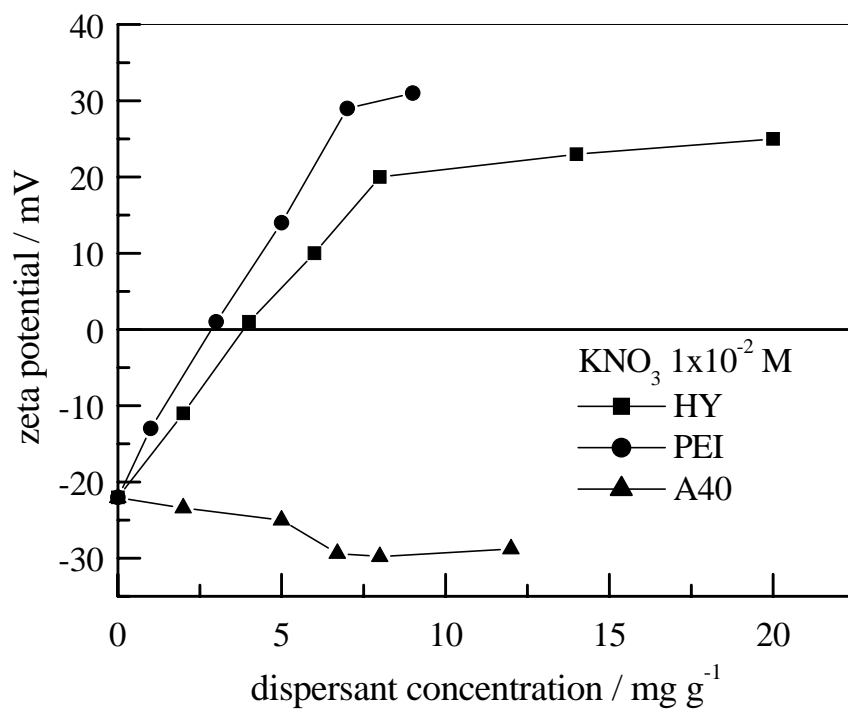
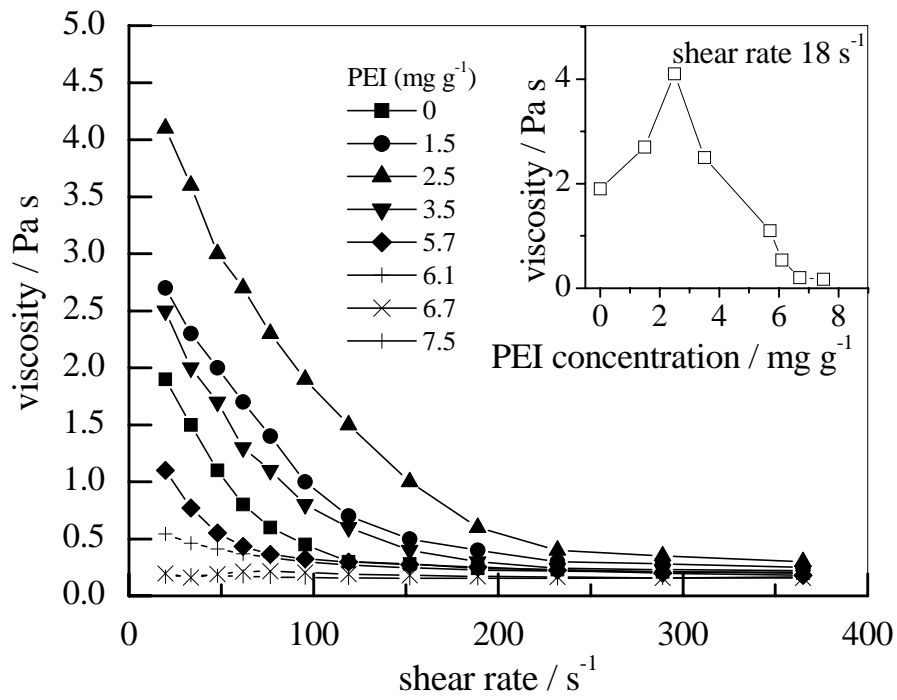
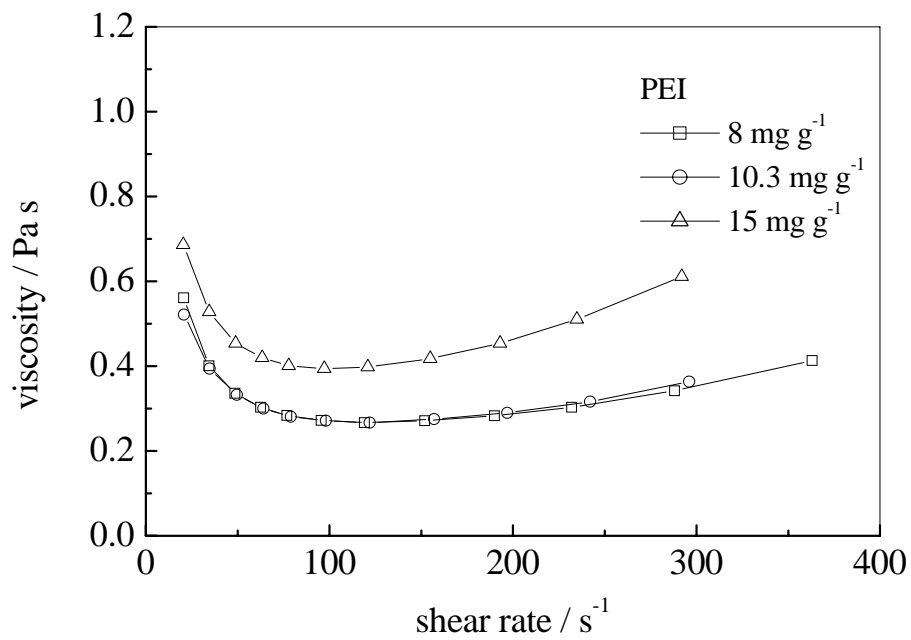


Figure 1. Changes in zeta potential with dispersant concentration for as-received SiC suspensions at a pH of 5.8.



(a)



(b)

Figure 2. Viscosity as a function of shear rate and PEI concentrations for a) 40 vol% and b) 47 vol% as-received SiC suspensions at a pH of 5.8.

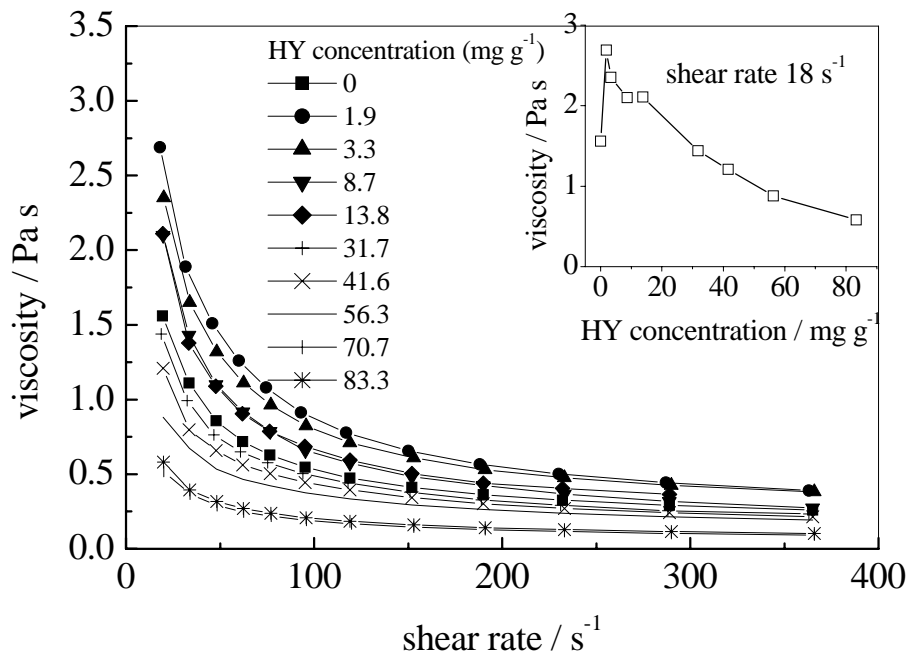


Figure 3. Viscosity as a function of shear rate and HY concentrations for 30 vol% as-received SiC suspensions at a pH of 5.8.

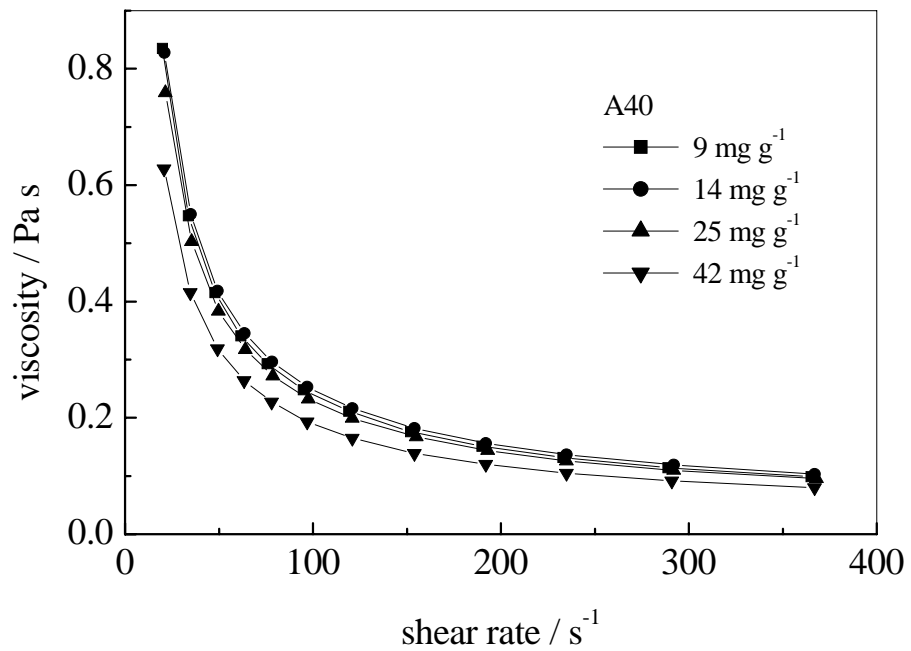
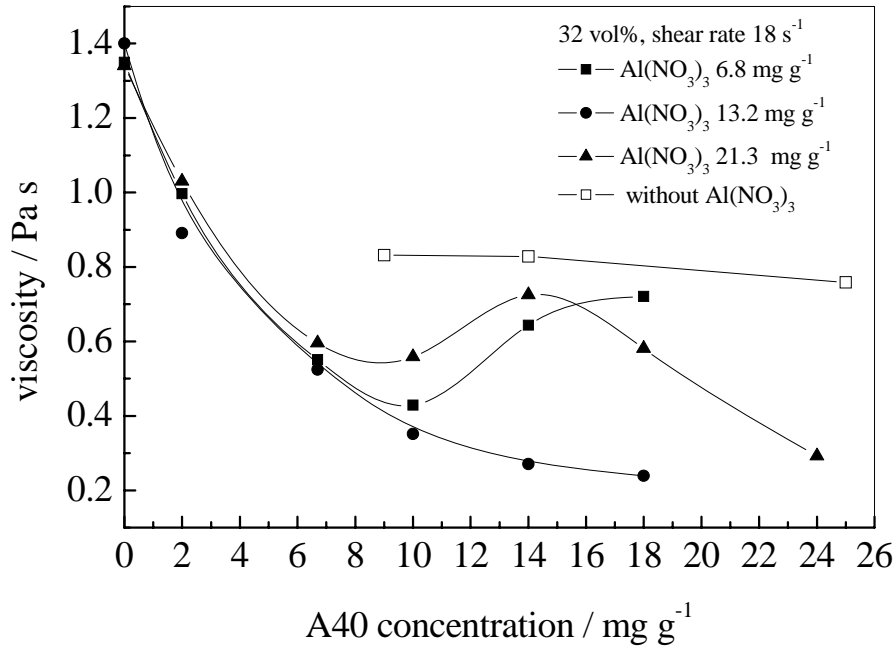
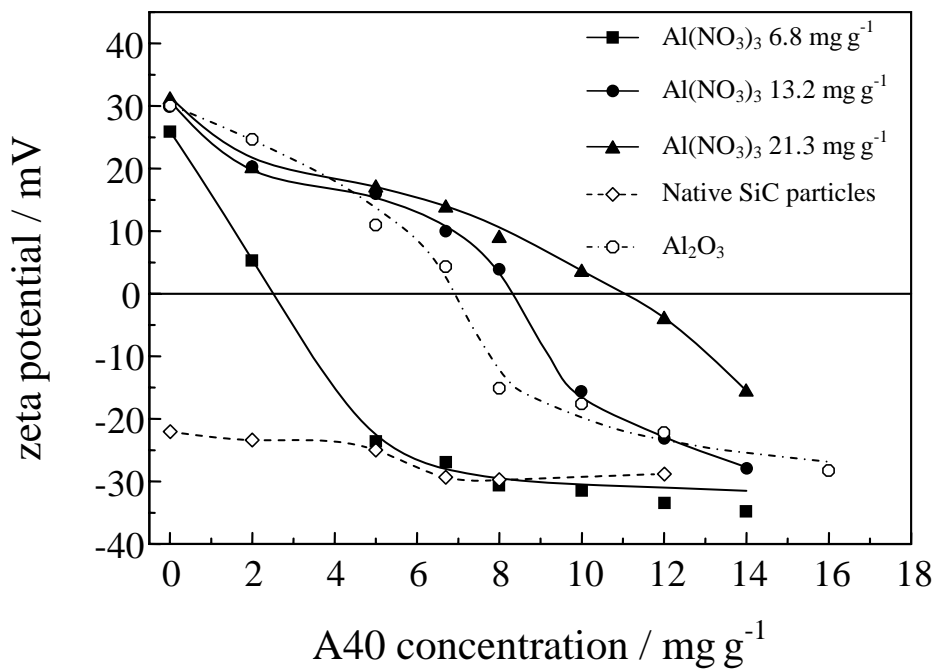


Figure 4. Viscosity as a function of shear rate and A40 concentrations for 30 vol% as-received SiC suspensions at a pH of 5.8.



(a)



(b)

Figure 5. (a) Viscosity and (b) zeta potential as a function of A40 concentration for SiC suspensions modified with different concentrations of $\text{Al}(\text{NO}_3)_3$.

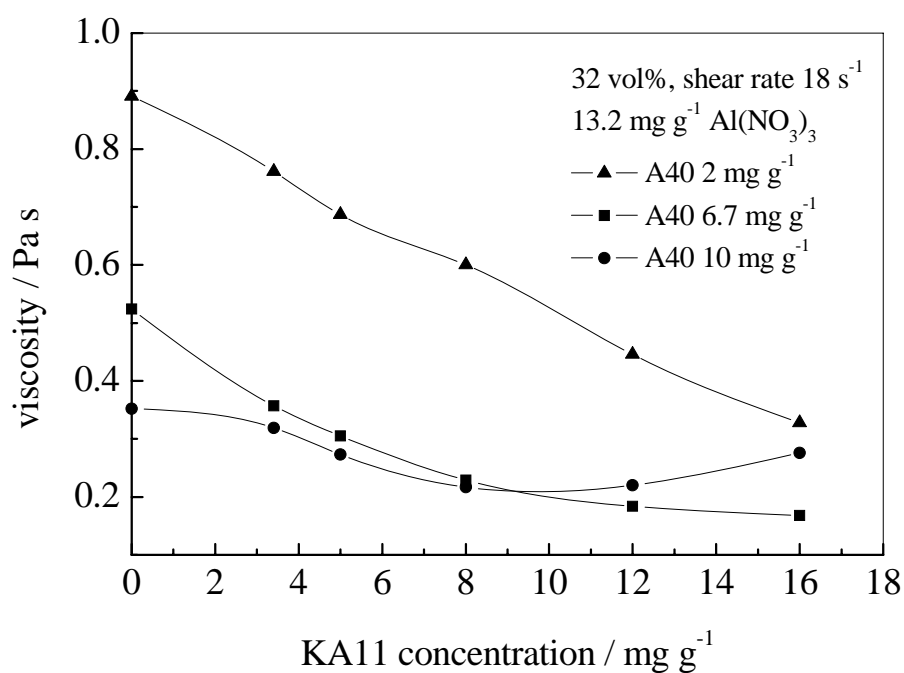


Figure 6. Viscosity as a function of KA11 concentration for $\text{Al}(\text{NO}_3)_3$ complex-modified SiC suspensions at different levels of A40.

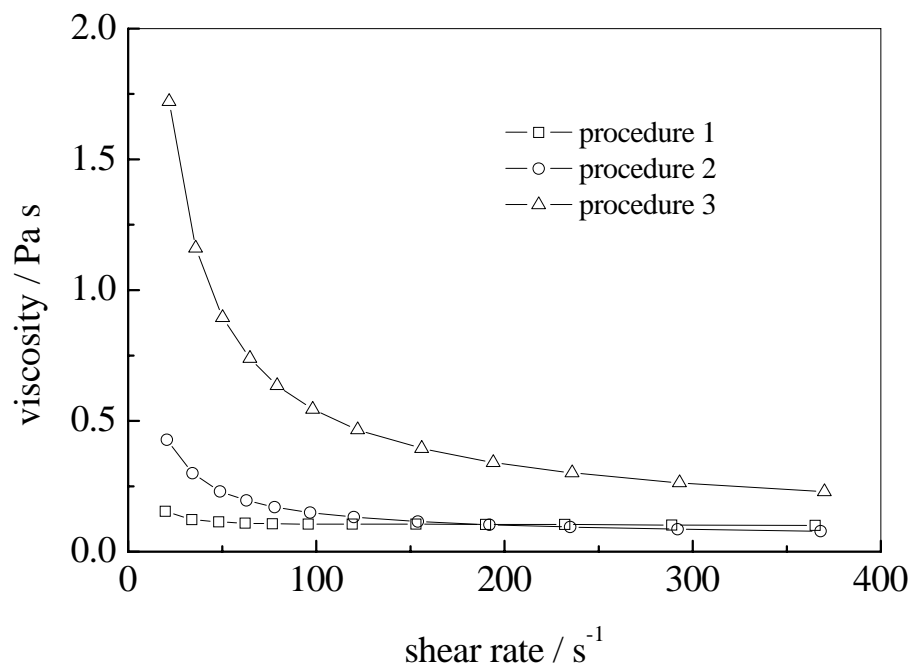


Figure 7. Viscosity of SiC suspensions prepared with different mixing procedures. All suspensions 30 vol% solids content, $13.2 \text{ mg g}^{-1} \text{ Al}(\text{NO}_3)_3$, $6.7 \text{ mg g}^{-1} \text{ A40}$ and $8 \text{ mg g}^{-1} \text{ KA11}$ at pH 5.8.

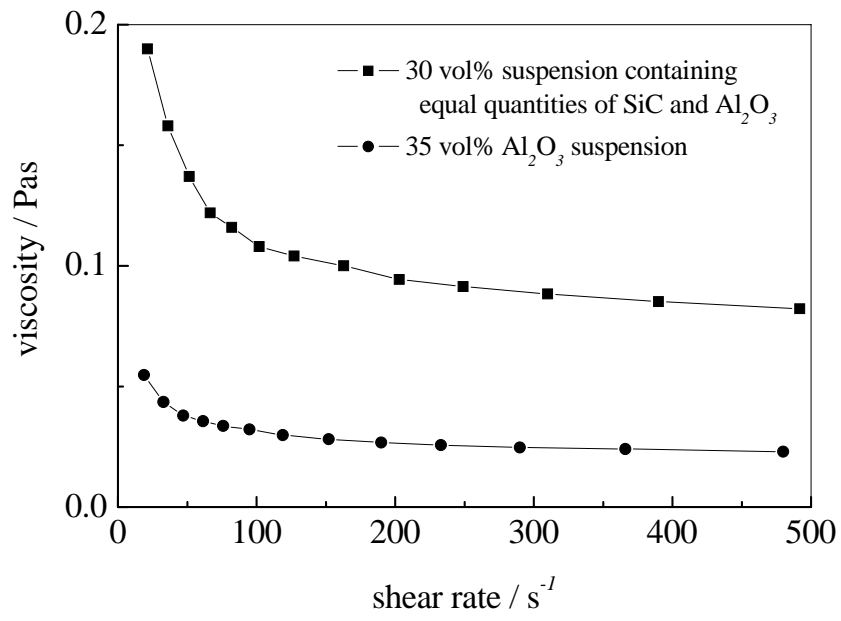


Figure 8. Viscosity as a function of shear rate for SiC – Al₂O₃ and Al₂O₃ suspensions.