

Effects of sintering additives on dispersion properties of Al₂O₃ slurry containing polyacrylic acid dispersant

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Abstract

The effects of sintering aid adsorption on the dispersion properties of aluminum oxide slurries were investigated. We considered Al₂O₃ slurry without additives and Al₂O₃ slurry with a Mg additive with 0.1 mass% in oxide equivalent as a sintering aid. In this study, we evaluated the adsorption isotherm of polyacrylic acid (PAA) onto Al₂O₃ and the dispersion degree of Al₂O₃ slurries in sedimentation tests under gravity. The adsorption isotherm featured a characteristic adsorption isothermal line with a maximum value when Mg additive was present in Al₂O₃. In addition, the packing fractions did not correspond to the apparent viscosity. However, in slurry that was allowed to settle for several days, both of them agreed. Therefore, the disagreement between the packing fraction and the apparent viscosity immediately after preparation arose from changes of the dispersion state, such as the decrease of the distance between particles with time.

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1. Introduction

The density and structure of ceramic green bodies produced by slip casting or tape casting, as well as the density and structure of spray-dried granules, are affected by slurry properties. Accurate evaluation and control of slurry properties is critical because slurries play an important role in ceramic fabrication processes, and thus affect the properties of the final products.^{1–3}

In ceramic manufacturing processes, various materials are commonly added to the main material as sintering aids. One example is the addition of a small amount of MgO, which is used to suppress abnormal grain growth of Al₂O₃.⁴ A significant decrease in zeta potential is known to occur when MgO is present in an Al₂O₃ aqueous suspension, owing to the rapid dissolution and ionization of MgO. This destabilizing effect of multivalent ions has also been confirmed by rheological measurements, which showed an increase in the viscosity and pseudoplastic behavior of Al₂O₃ slurry.⁵

In aqueous suspensions, the particle dispersion can be controlled by adding dispersants, such as poly(acrylic acid) (PAA). However, the adsorption of PAA also can be strongly affected by the presence of multivalent ions.^{6,7} Vormöhlen et al.⁸ have found that due to divalent cations, the repulsion of negatively charged polyelectrolyte segments is reduced. Sun et al.⁹ have observed that a significant increase in PAA adsorption occurs in the presence of Mg²⁺ ions. The effect is small in acidic media; however, Kirby et al.¹⁰ have reported that in alkaline media, a PAA dispersant system is less effective for suspensions with high ionic strength, especially those containing multivalent ions. This can be attributed to the interference of multivalent ions with the electrostatic repulsive barrier, which leads to flocculation.

The effects of multivalent ions on PAA have been discussed in many works regarding the adsorption of dispersant on particles, as described above. However, few works have investigated the dispersion behavior or the effects of dispersants on the structure of particle dispersion in the presence of sintering additives.

In this paper, the effects of sintering additives on Al₂O₃ slurries containing PAA dispersant are investigated. We evaluated the optimal amount of PAA for obtaining well-dispersed Al₂O₃ slurry, the adsorption isotherm of PAA onto Al₂O₃, and the dis-

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Table 1
Chemical analysis reported by manufacturer of Al₂O₃ powder.

mass%	Al ₂ O ₃	SiO ₂	MgO	Fe ₂ O ₃	Na ₂ O	L.O.I	H ₂ O
AES12	99.9	0.06	–	0.01	0.04	0.01	0.01
AES11E	99.8	0.06	0.1	0.01	0.04	0.01	0.02

persion degree of Al₂O₃ slurry in terms of the sediment packing volume fraction.

2. Experiments

2.1. Materials

Two types of low-soda alumina powders (0.5 μm, AES12 and AES11E, Sumitomo Chemical Co., Ltd.) were used as samples. The composition of the powders, according to information from the supplier, is shown in Table 1. AES11E contains a Mg additive with 0.1 mass% in oxide equivalent, which can be used to suppress abnormal grain growth of Al₂O₃ during sintering. Table 2 shows the dissolved ion concentrations in the supernatant of 20 vol.% Al₂O₃ slurries, as measured by inductively coupled plasma (IPC) atomic emission spectroscopy. In the supernatant of the AES11E slurry, 0.1 mM Mg²⁺ was observed. The ammonium salt of PAA (Seruna D305, Chukyo Yushi Co., Ltd.) and distilled water were used as the dispersant and solvent, respectively. The zeta potential of these powders was measured by acoustic and electroacoustic spectroscopy (Dispersion Technology Co., Ltd., DT-1200).

2.2. Rheological measurements

Slurries were prepared from above two Al₂O₃ powders by ball milling for 1 h with 0.7 kg Al₂O₃ balls of 5 mm in diameter in 0.7 L plastic vessels. The solid concentration was 40 vol.%. The amount of added dispersant was varied in the range of 0.1–8.0 mg g⁻¹-Al₂O₃ for AES12 and 1.0–8.0 mg g⁻¹-Al₂O₃ for AES11E.

Rheological measurements were carried out on a rotational controlled stress rheometer (Anton Paar Co., Ltd., Rheolab QC/Rotor, DG42), immediately after preparation and after the samples were left at rest for 7 days at a controlled temperature of 25 °C. The measurement configuration was a concentric coaxial cylinder with shear rate starting at 0 s⁻¹ and increasing linearly to 200 s⁻¹ over 180 s and then decreasing linearly to 0 s⁻¹ over 180 s. The apparent viscosity was taken as the value at a shear rate of 17 s⁻¹. Before starting a measurement, the same pre-shearing protocol was carried out, in order to transmit the same rheological history to all suspensions being tested.

Table 2
Elemental analysis of supernatant of 20 vol.% Al₂O₃ slurry.

mmol L ⁻¹	Mg	Na	K
AES12	0.001	8.5	0.3
AES11E	0.1	8.5	0.5

2.3. Adsorption isotherms

The slurry was centrifuged (Hettich Co., Ltd., EBA20) and then the supernatant was sampled. The dispersant concentration in the supernatant was measured by a total organic carbon analyzer (Toray Engineering Co., Ltd., TOC-650). The amount of PAA adsorbed on Al₂O₃ particles was determined by subtracting the amount of PAA measured in the supernatant from the added amount.

2.4. Sedimentation test under gravity

Polyacryl tubes with an inner diameter of 20 mm were used for the sedimentation test. The initial slurry heights were 150 mm, and the depth of the clarification zones was measured after being allowed to settle for 4 weeks while kept at a constant temperature of 23 °C. The sediment packing volume fraction was calculated from the sediment height and the initial particle concentration.

3. Results and discussion

3.1. Effects of sintering additive on slurry properties without PAA

Fig. 1 shows a comparison of the zeta potential of the AES12 and AES11E slurries as a function of pH. Here, the solid content concentration was 20 vol.%. AES11E slurry was more cohesive than AES12 slurry, because the absolute value of the zeta potential of AES11E slurry was smaller than that of AES12. Fig. 2 shows a comparison of the rheological properties of the AES12 and AES11E slurries. Compared with AES12 slurry, AES11E slurry had high viscosity and exhibited pseudoplastic behavior. When metal ions are present in aqueous solution, the electric double layer of colloidal particles on the surface is compressed. Consequently, the colloidal particles aggregate more readily. This phenomenon is known as coagulation. In order to consider the effects of metal ions, AES12 and AES11E slurry was prepared with the addition of 25 mM ethylenediaminetetraacetic acid disodium salt dehydrate (EDTA-2Na). EDTA-2Na is a polyamino carboxylic acid. It is widely used to dissolve scale. Its usefulness arises from its role as a chelating agent, that is, its

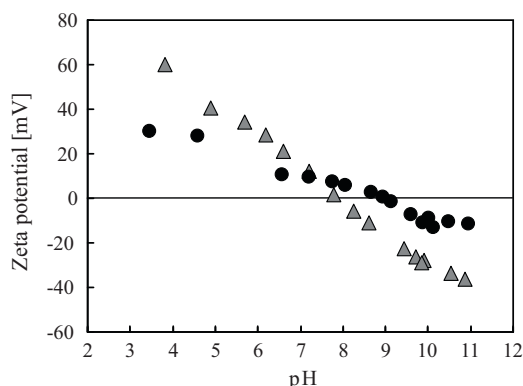


Fig. 1. Zeta potential of 20 vol.% Al₂O₃ slurries. ▲ : AES12; ● : AES11E.

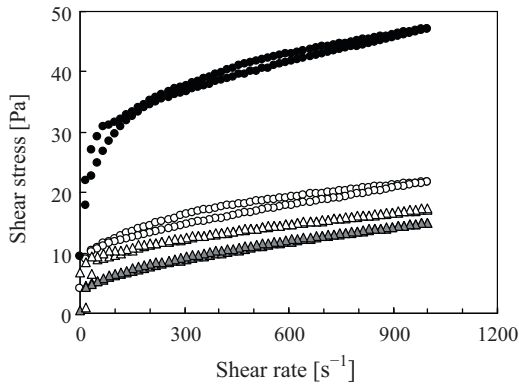


Fig. 2. Flow curves of 20 vol.% Al₂O₃ slurries. ▲ : AES12; ● : AES11E; △ : AES12 with 25 mM EDTA-2Na; ○ : AES11E with 25 mM EDTA-2Na.

ability to chelate metal ions such as Ca²⁺ and Fe³⁺. After being bound by EDTA-2Na, metal ions remain in solution but exhibit diminished activity.

Fig. 2 shows the collected results. The viscous behavior of AES12 slurry did not change even if EDTA-2Na was added. On the other hand, the viscosity of AES11E slurry became similar to that of AES12. This showed that metal ions were present in the AES11E slurry, because the decrease in viscosity as a result of EDTA-2Na addition inactivated the metal ions. The critical coagulation concentration, which is the minimum concentration at which metal ion coagulates to form colloid particles, is dependent on the valence of the metal ion.^{11,12} Therefore, this coagulation by the metal ion can be attributed to the effects of dissolved Mg²⁺ from the Mg additive.

These results show that the Mg²⁺ ions dissolved from sintering aids significantly affect the viscosity of AES11E slurry.

3.2. Relationship between apparent viscosity and packing fraction of sediment

Fig. 3 shows the apparent viscosity of the 40 vol.% Al₂O₃ slurry as a function of the quantity of dispersant added. In this figure, the horizontal axis shows quantity of dispersant addition per unit mass of Al₂O₃. We found that the viscosity of the slurries reached a minimum at 0.7 mg g⁻¹-Al₂O₃ in AES12 and 1.5 mg g⁻¹-Al₂O₃ in AES11E. We expected the slurries to be

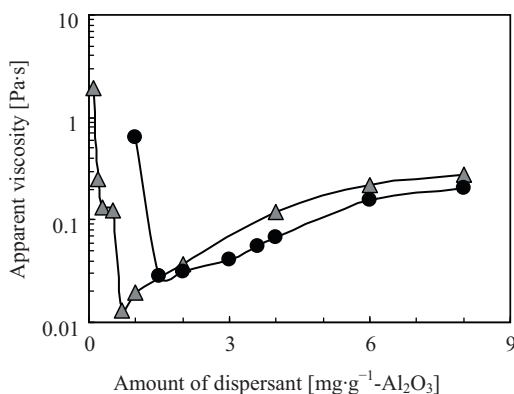


Fig. 3. Apparent viscosity of prepared slurries. ▲ : AES12; ● : AES11E.

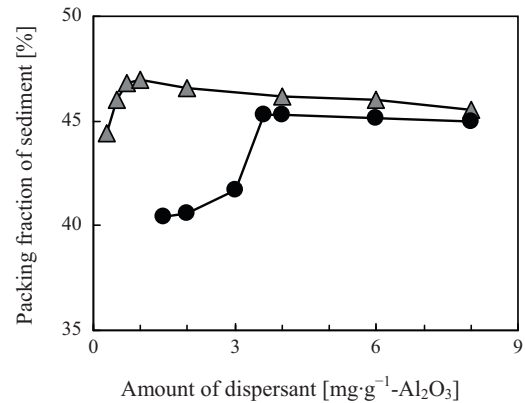


Fig. 4. Packing fraction of sediment after 4 weeks at rest at 23 °C. ▲ : AES12; ● : AES11E.

most dispersed at these quantities of dispersant addition. The packing fractions of sediment are shown in Fig. 4, and photographs of samples in polyacryl tubes are shown in Fig. 5. The sedimentation test under gravity is an evaluation of packing properties, and the packing fraction of sediment was calculated from the height of sediment after 4 weeks left at rest. The gravity sedimentation examination is known as a simple technique for evaluating slurry dispersibility,³ which indicates that the packing of particles is good when the packing fraction of sedimentation is high. A high packing fraction indicates that the interparticle interactions are weak, and that particles disperse well in slurries.

In an actual manufacturing process, such as spray drying, the material is granulated by drying of slurry. It is known that the characteristics of the resulting granules are strongly affected by the dispersion of the slurry.^{13,14}

Evaluating the change in dispersibility with the change in distance between particles from not only the viscosity of the slurry but also the gravity sedimentation test is extremely important for ensuring that the distance between particles decreases with drying.¹⁵

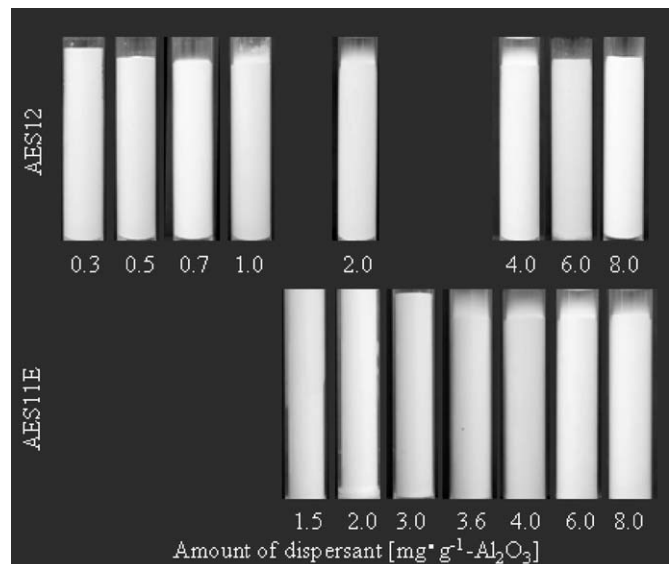


Fig. 5. Photographs of polyacryl tubes after 4 weeks at rest at 23 °C.

In the AES12 slurry, dispersibility correlated with apparent viscosity. The packing fraction of the sedimentation was highest when the amount of added dispersant was $0.7 \text{ mg g}^{-1}\text{-Al}_2\text{O}_3$ at the minimum apparent viscosity. However, the packing fraction of the AES11E slurry with dissolved Mg^{2+} ions did not correspond to apparent viscosity. The packing fraction of the sedimentation was low when the amount of added dispersant was $1.5 \text{ mg g}^{-1}\text{-Al}_2\text{O}_3$ at the minimum apparent viscosity, which indicates that the particles in the slurries were in an aggregated state. On the other hand, a high packing fraction of sediment signifies that the particles in the slurry are well-dispersed, which was observed when the amount of added dispersant was greater than $3.6 \text{ mg g}^{-1}\text{-Al}_2\text{O}_3$.

The disagreement between the apparent viscosity and the packing fraction of sediment has been reported by Tsubaki et al.^{3,16} and Takahashi et al.¹⁷, but the underlying mechanism has not been clarified. Furthermore, Mori et al.^{18,19} have reported similar result for alumina slurry, in which dispersion was controlled by adjusting pH. They considered the disagreement between apparent viscosity and packing fraction by analyzing the interaction potential curve for the particles of the slurry, which was calculated using the DLVO theory. Therefore, we considered the adsorption state of the dispersant by focusing on the differences in the repulsion between the particles.

3.3. Adsorption behavior of dispersant on Al_2O_3 powder

The adsorption isotherms for PAA on Al_2O_3 powder suspended in each slurry are shown in Fig. 6. The horizontal axis shows quantity of dispersant addition per unit mass of Al_2O_3 and the vertical axis shows the adsorption amount of dispersant per unit mass of Al_2O_3 . A line representing complete (100%) adsorption of PAA is also shown. The shape of the isotherm differed between the AES12 and AES11E slurries. For AES12, the isotherm conformed to a Langmuir adsorption isotherm; the well-dispersed slurries were observed from 0.7 to $4.0 \text{ mg g}^{-1}\text{-Al}_2\text{O}_3$, and adsorption was saturated at $2.0 \text{ mg g}^{-1}\text{-Al}_2\text{O}_3$. On the other hand, for AES11E with Mg^{2+} ions dissolved in the slurry, the isotherm was quite unique in that the adsorbed amount had a local maximum, and after reaching the maximum value, the adsorbed amount gradually decreased with increasing amounts of additive. Additionally, the amount of added PAA was higher in comparison with AES12.²⁰ Many researchers have attributed this specific adsorption isotherm to the effects of multivalent ions on the adsorption state. The multivalent ion interactions can promote association of the carboxylate group of PAA via multivalent ion bridging^{7,8,11} and charge neutralization of the carboxylate group.^{6,9}

For AES11E, the packing fraction of sedimentation was highest when the amount of added dispersant was $3.6 \text{ mg g}^{-1}\text{-Al}_2\text{O}_3$ at the maximum adsorption isotherm, not $1.5 \text{ mg g}^{-1}\text{-Al}_2\text{O}_3$ at the minimum apparent viscosity.

3.4. Changes in apparent viscosity with time

Tsubaki et al.¹⁶ and Mori et al.¹⁸ explained the above-mentioned phenomenon that slurry dispersibility, as measured

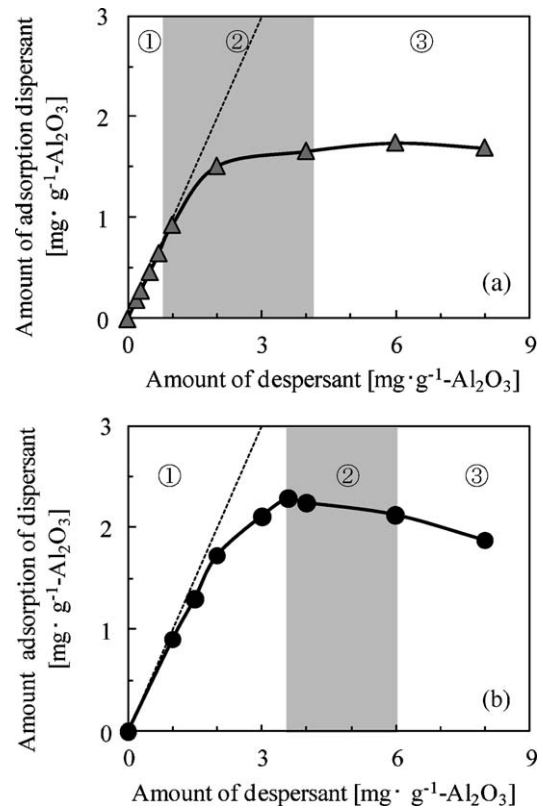


Fig. 6. Effect of added amount of dispersant on dispersant adsorption for (a) AES12 and (b) AES11E. ①: network cohesion area (insufficient dispersant); ②: well-dispersed; ③: likely coagulation (excess dispersant).

in the sedimentation test under gravity, does not correspond to the viscosity immediately after preparation as follows. They hypothesized that the potential barrier of the slurries is lower than that of well-dispersed slurries, owing to the decrease in distance between particles with the progression of sedimentation thickening, which increases the aggregation of the particles. It takes time to make sedimentary layers. In contrast, the viscosity of the slurry immediately after preparation is a short-term evaluation. Therefore, it would appear that it is difficult to evaluate the slurry properties by the apparent viscosity alone.

The slurry is under high shear stress during ball milling. Consequently, the particles in the slurries immediately after preparation maintain a distance corresponding to the density for solidity, and the frequency of collisions between particles is lower. Thus, we observed the changes in the apparent viscosity with time to examine the distance between particles and the influence of the frequency of collisions between particles. The apparent viscosity of the slurries left at rest at 23°C and subsequently agitated for 30 s in an ultrasound bath was measured. Fig. 7 shows the changes in the apparent viscosity with time for AES11E and AES12 slurries having the same apparent viscosity immediately after preparation. The amount of dispersant added to each was $1.0 \text{ mg g}^{-1}\text{-Al}_2\text{O}_3$ in AES12 and $2.0 \text{ mg g}^{-1}\text{-Al}_2\text{O}_3$ in AES11E. AES12 had a packing fraction that corresponded to the apparent viscosity immediately after preparation, and thus the viscosity had changed very little over the 7 days. Mean-

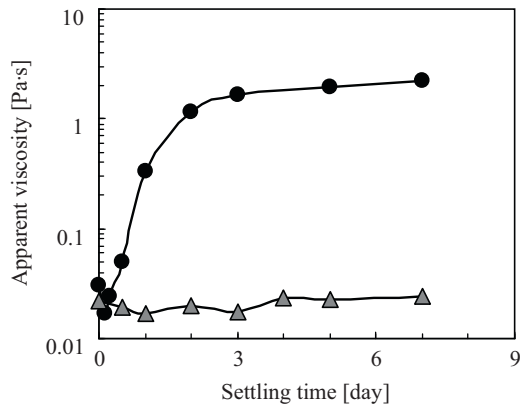


Fig. 7. Viscosity dependence on settling time of 40 vol.% Al_2O_3 slurries. \blacktriangle : AES12, $1.0 \text{ mg g}^{-1}\text{-Al}_2\text{O}_3$; \bullet : AES11E, $2.0 \text{ mg g}^{-1}\text{-Al}_2\text{O}_3$.

while, the viscosity of AES11E which dissolved Mg^{2+} ions had drastically after being left to settle for 3 days.

Fig. 8 shows the respective apparent viscosity of the slurries after 7 days left at rest compared to those immediately after preparation. The apparent viscosity did not change after 7 days at rest in the case of AES12. Similar to the slurry immediately after preparation, the viscosity of the slurry was a minimum when the amount of added dispersant was $0.7 \text{ mg g}^{-1}\text{-Al}_2\text{O}_3$, which produced the maximum packing fraction. On the other hand, the apparent viscosity changed considerably and agreed with the

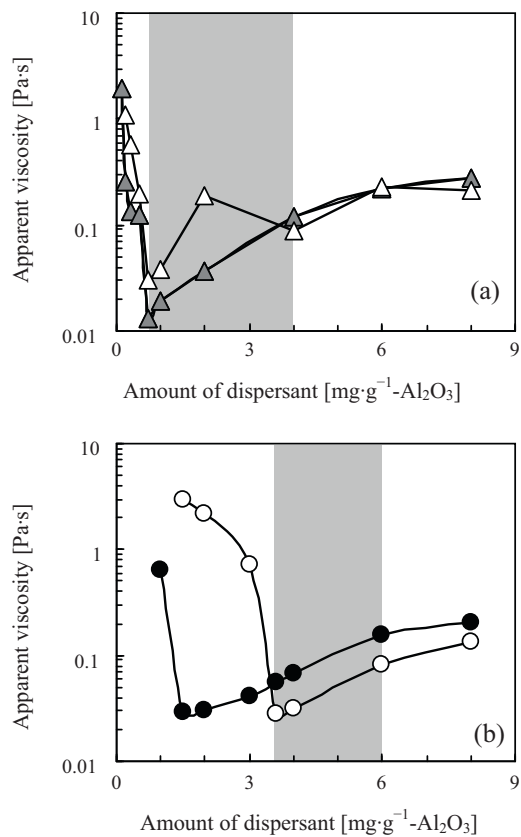


Fig. 8. Comparison of apparent viscosities after 7 days left at rest for (a) AES12 and (b) AES11E. \blacktriangle , \bullet : just after preparation; \triangle , \circ : after 7 days left at rest. Gray area indicates domain where particles are well-dispersed.

packing fraction on AES11E. For an amount of added dispersant of $1.5 \text{ mg g}^{-1}\text{-Al}_2\text{O}_3$, for which the apparent viscosity immediately after preparation was a minimum but the packing fraction was low, the viscosity increased 68-fold. When the amount of added dispersant was $3.6 \text{ mg g}^{-1}\text{-Al}_2\text{O}_3$, which corresponded to the maximum packing fraction and the peak of the adsorption isotherm, the apparent viscosity was a minimum. For both AES12 and AES11E, the slurries showed the maximum packing fraction at the amount of added dispersant that produced the minimum apparent viscosities of the slurries when the distance between the particles decreased by sedimentation.

It was difficult to confirm the effect of Mg^{2+} ions by the evaluation of the viscosity with addition of EDTA. PAA is a compound containing carboxylates, which form strong complexes with metal ions, the same as EDTA-2Na. It would appear that EDTA-2Na offers little benefit, owing to the fact that the Mg^{2+} ions had already been masked by dispersant.

As seen from the above results, for Al_2O_3 slurry including the polyacrylic acid (PAA) dispersant, it was confirmed that dissolved multivalent ions in the slurry significantly influenced the adsorption behavior of PAA and the packing fraction of slurries. In addition, the packing properties could not be predicted from the apparent viscosity that was measured immediately after preparation. We attribute these findings to changes of the dispersion state with time, owing to the increased frequency of collisions due to the decrease in the distance between particles. In the manufacturing process, the distance between the particles decreases with drying of the slurry. Therefore, evaluating not only the viscosity of the slurry immediately after preparation but also the changes of the dispersion states with time is essential for optimizing the slurry conditions.

In addition, Pugh et al.²¹ have made an interesting investigation that the influence of Mg^{2+} ions on electrokinetic and stability behavior of $\alpha\text{-Al}_2\text{O}_3$ powders has been determined at a series of concentrations and a range of pH value. Mg^{2+} ions in the solutions exist in hydrated form depending on the condition of aqueous solutions. The distribution of the various Mg^{2+} species in solution depends primarily on pH and concentration of Mg^{2+} ions. They reported that the adsorbed amount of Mg^{2+} ions on surface of powder had increased under the high pH values or high Mg^{2+} ions concentration conditions. These results suggest that attention needs to be paid to the change of pH and/or Mg^{2+} ions concentration by the preparation condition.

4. Conclusions

We investigated the influence of a sintering aid on Al_2O_3 slurry containing polyacrylic acid (PAA) dispersant. The viscosity behavior of the slurries, the adsorption behavior of PAA on Al_2O_3 particles, and sedimentation under gravity were evaluated.

- (1) In AES11E slurry, which contained a Mg additive with 0.1 mass% in oxide equivalent for suppressing abnormal grain growth of Al_2O_3 , Mg^{2+} ions dissolved in the slurry, and the isotherm was unique in that the adsorbed amount had a local maximum.

- (2) When dispersant was added, the apparent viscosity of slurry immediately after preparation was minimal, and did not correspond to the maximum packing fraction. Instead, the maximum packing fraction corresponded to the amount of added dispersant that produced the local maximum of the adsorption isotherm.
- (3) It would appear that the discordance between the apparent viscosity immediately after preparation and the dispersion evaluation result obtained by the weight sedimentation examination could be attributed to the effects of the change of the aggregate state of Al_2O_3 particles in a suspension with time, which was induced by the increased frequency of collisions between particles, which was caused by the decrease in the distance between the particles. However, the apparent viscosity of the slurries, which decreased of the distance between the particles after 7 days left at rest, was minimal when the amount of added dispersant corresponded to the maximum packing fraction. We intend to provide more information concerning these results in a future paper.

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