

# Effects of trace amounts of additives on the aging of the slurry properties

## 微量添加物がスラリー特性経時変化に及ぼす影響

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### 1. Introduction

#### In wet process

The quality of the product depends on the particle dispersion state of slurries.

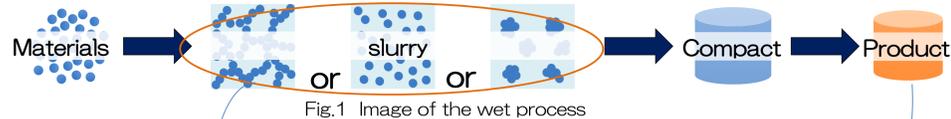


Fig.1 Image of the wet process

ex. Battery material, Paint, Ceramics...

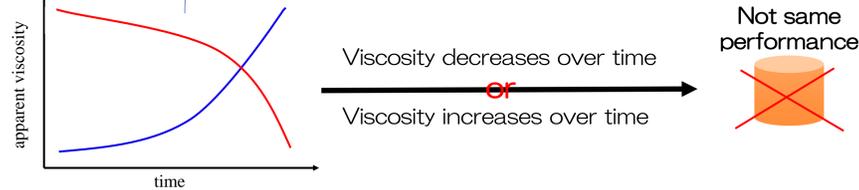


Fig.2 Effect of aging of slurry properties

In previous studies, it has been reported that the slurry properties change over time. However the mechanism of time change of slurry properties has not been clarified yet.

#### Purpose

In this study, the purpose is to clarify what type of slurry changes its property over time as the first step to predict/suppress slurry characteristics change over time.

### 2. Experiment

#### materials

##### Particles

Two types of Alumina - $\text{Al}_2\text{O}_3$ -  
 (AES11E, Sumitomo Chemical Co., LTD.  $d_{50} = 0.48 \mu\text{m}$ ,  
 / AES12, Sumitomo Chemical Co., LTD  $d_{50} = 0.48 \mu\text{m}$ )

| mass%  | $\text{Al}_2\text{O}_3$ | $\text{SiO}_2$ | MgO | $\text{Fe}_2\text{O}_3$ | $\text{Na}_2\text{O}$ | L.O.I | $\text{H}_2\text{O}$ |
|--------|-------------------------|----------------|-----|-------------------------|-----------------------|-------|----------------------|
| 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                 |

##### Media

Deionized water

##### Additive

Magnesium chloride - $\text{MgCl}_2$ -  
 (Wako Pure Chemical Industries, Ltd.)

##### Polymer

PCA -Polycarboxylic acid ammonium-  
 (Celuna D-305, CHUKYO YUSHI CO., LTD.)

### Slurry preparation and evaluation

#### Slurry preparation

Materials weighing and mixing

Ball milling (55 rpm, 1 hour)

Vacuum deforming (10 min)

Stirring (300 rpm, 5 days)

**AES11E**  
 Particle conc. : 20 vol%  
 PCA additive dosage : 0, 1.6, 2.0 mg/g  $\text{Al}_2\text{O}_3$

**AES12**  
 Particle conc. : 20 vol%  
 PCA additive dosage : 1.6, 2.0 mg/g  $\text{Al}_2\text{O}_3$   
 Additive conc : 0, 10 mM  
 (Two kinds of solvents, Deionized water and aqueous solution containing Mg ions were used.)

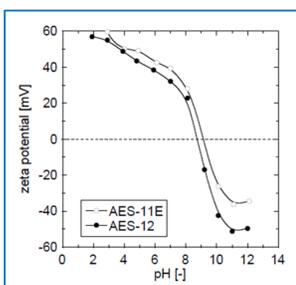


Fig.3 Zeta potential of particle

#### Physical property evaluation

##### Flow properties evaluation

Device : Coaxial cylinder viscometer (Anton Paar, Rheolab QC)  
 Shear rate : 0 ~ 1000  $\text{s}^{-1}$  Preset temperature : 25 $^\circ\text{C}$

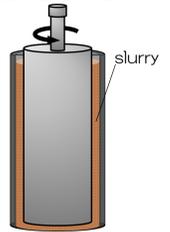


Fig.4 Illustration of viscometer

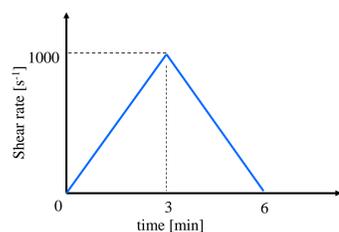


Fig.5 Measurement condition

##### Sedimentation test

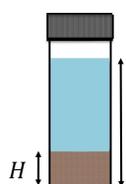


Fig.6 Illustration of centrifuged slurry

Packing fraction  $\Phi$  [-]

$$\Phi = \frac{h}{H} \phi$$

Particle conc.  $\phi$  [-]

##### Polymer adsorbed amount, Measurement of Ion elution



Fig.7 Illustration of centrifuged slurry

**TOC**  
 (SHIMAZU SCIENCE Co., LTD, TOC-V)  
 Combustion temperature : 680  $^\circ\text{C}$

**ICP-OES**  
 (Hitachi High-Tech Science Co., LTD, SPS7800)  
 Measuring element: Magnesium

### 3. Results and discussion

#### Flow properties evaluation

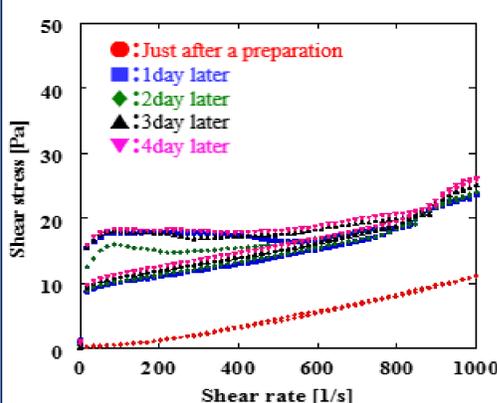


Fig.8 Flow curves of AES11E (PCA 2.0 mg/g  $\text{Al}_2\text{O}_3$ ) slurry

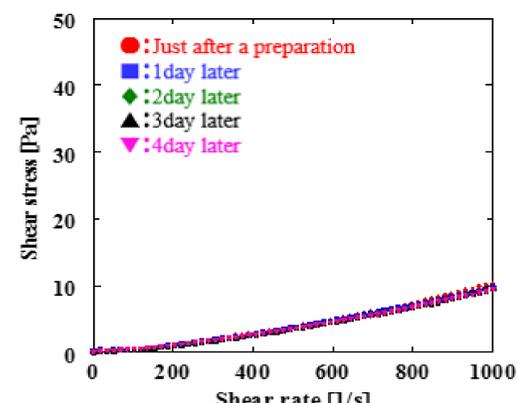


Fig.9 Flow curves of AES12 (PCA 2.0 mg/g  $\text{Al}_2\text{O}_3$ ) slurry

- The apparent viscosity of AES11E slurry, in which elution of  $\text{Mg}^{2+}$  was confirmed, increased after one day passed after preparation.
- No increase in apparent viscosity like AES11E was observed in AES12 slurry without elution of  $\text{Mg}^{2+}$ .

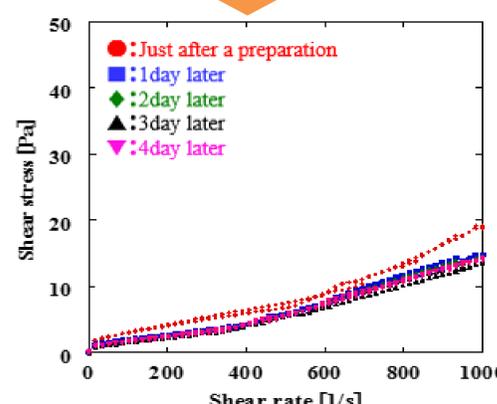


Fig.10 Flow curves of AES12 (PCA 2.0 mg/g  $\text{Al}_2\text{O}_3$ ) +  $\text{Mg}^{2+}$  slurry

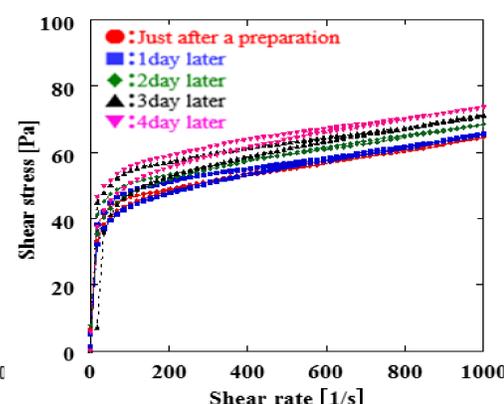


Fig.11 Flow curves of AES11E (PCA 0 mg/g  $\text{Al}_2\text{O}_3$ ) slurry

#### Sediment test

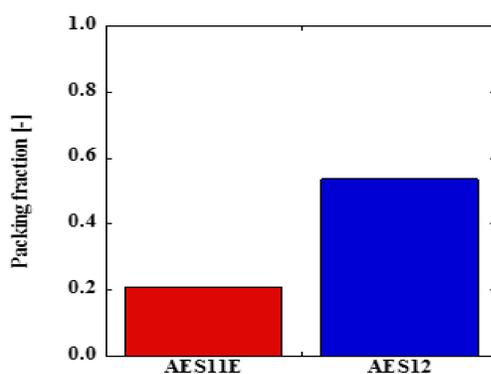


Fig.12 Packing fraction of sediment (PCA 1.6 mg/g  $\text{Al}_2\text{O}_3$ )



Fig.13 Images of final packing sedimentation

#### Polymer adsorbed amount

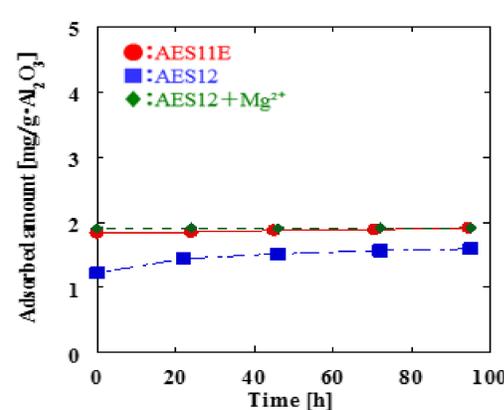


Fig.14 Adsorbed amount of polymer

#### Measurement of Magnesium ion elution

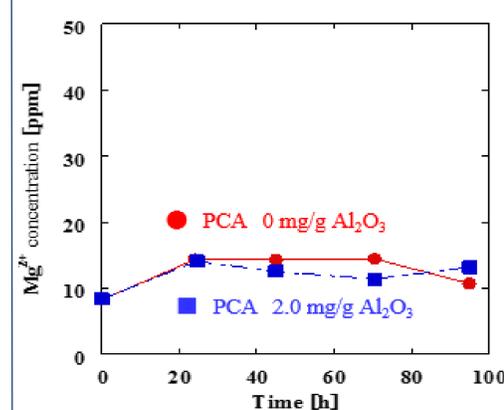


Fig.15 Magnesium ion concentration

### 4. Conclusion

It was found that the adsorbed amount of polymer and Magnesium ion concentration did not change over time. These results indicated that ion elution should not be the main factor to change slurry properties.

#### Access

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