

## Characteristics on Stable Suspensions of Iron Oxide Powder

E. H. Lee\*, S. H. Kim, K. M. Kim, J. W. Na, B. S. Choi, and W. Y. Maeng  
Nuclear Materials Research Division, Korea Atomic Energy Research Institute,  
1045 Daedeok Street, Yuseong, Daejeon 305-353, Republic of Korea  
\*ehlee@kaeri.re.kr

### 1. Introduction

Corrosion products in the secondary-side systems of pressurized-water reactors (PWRs) can be deposited on steam generator (SG) tubes. In order to inhibit the deposition of corrosion products on the surfaces of SG tubes, dispersants, which are generally negatively charged polymers, are used to solve this problem. One of the most well-known dispersants for metal oxides is a poly (acrylic acid) (PAA) [1,2]. For PWR plants, systems are particularly sensitive to water conditions due to the risk of corrosion. As a result, it is important to confirm the polymeric solutions and their suspensions for preventing deposition of the corrosion products onto the PWR systems.

In this study, we investigate the effects of solution pH, and molecular weights and concentration of PAA on the stability of magnetite suspensions.

### 2. Methods and Results

#### 2.1 Materials and Test Procedure

Magnetite nanoparticles, PAA (SIGMA-ALDRICH, USA) were chosen for this study. For convenience, the PAA with a molecular weight of 1,800 is denoted in this paper as PAA18, while the PAA with a molecular weight of 250,000 is represented by PAA25. For sedimentation tests, the suspensions were prepared, as follows. The pH of 100 ml deionized water ( $>17 \text{ M}\Omega\cdot\text{cm}$ ) was adjusted by adding ETA solution. Next, either 1 ml of 100 ppm PAA18 or PAA25 was added to the above solutions, followed by magnetic stirring. After one-day equilibrium, magnetite particles were added to the above solutions and then the solutions were sonicated for 15 min. Transmittance of the suspensions was determined by using a dispersion stability analyzer [3]. For a visual observation, behaviors of the suspensions were observed every day. Transmission electron micrographs (TEM) were measured to identify the morphology of dispersed particles.

#### 2.2 Dispersion Behaviors of Suspensions

The relative concentrations of COOH and COO<sup>-</sup> groups in aqueous solutions with PAA18 as a function of pH were measured. As the COOH groups are ionized, many negatively charged groups along the polymer backbone repel each other, which leads to a conformational change in solution and has an influence

on efficiency of the PAA [4]. In order to minimize the variation in pH values of the testing solutions, 0.005 wt% magnetite particles and 1 ppm PAA were adopted to prepare the suspensions. When the magnetite nanoparticles were mixed with PAA solutions, the solutions became turbid by sonication. For the suspensions with PAA18 and PAA25, lots of magnetite particles aggregated and then settled down in a day. This is because there was not enough PAA to adsorb onto the particles, which promotes a flocculation.

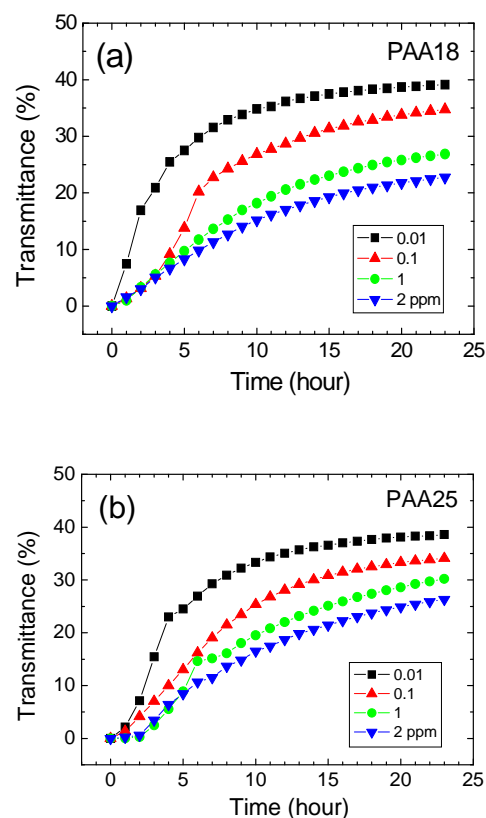


Fig. 1. Plots of % transmittance measured with suspensions (0.005 wt% magnetite, pH 9.0, conc. 0.01, 0.1, 1, 2ppm) containing (a) PAA18 (b) PAA25 as a function of time.

The effects of the concentrations are shown in Fig. 1. The stability of the suspensions increased as the concentrations of PAA increased at pH 9.0, which indicates that more COOH groups were ionized, and that more adsorption occurred. The particles in the suspensions that had 0.01 M PAA 18 and PAA25 agglomerated and then settled down quickly. The

amount of PAA was too low to adsorb onto the particles (0.005 wt%), which led to a bridging flocculation and then deposition.

The dispersion stability decreased as the pH increased in the following order: in pH 7.7, pH 9.0, pH 9.2 solutions. By comparing the data, the suspensions with PAA18 are more stable than those with PAA25. During the one-day equilibrium, many ionized  $\text{COO}^-$  groups bonded with the  $\text{NH}_3^+$  groups of ETA. As pH of the solutions increases, the concentration of  $\text{NH}_3^+$  groups in ETA also increases.

Figure 2 shows a TEM image of the particles in the suspension prepared with PAA25 at pH 9.0 in 210 days. By visual observation, all of the particles were aggregated and deposited. However, a few dispersed particles were observed by TEM. The size of the particles was between 50 and 100 nm, which is smaller than the particles observed in the suspension prepared with PAA18. The particles in the above suspensions formed a bridging flocculation between 3 and 4 particles. From this study, it is suggested that PAA18 is useful in suspending the particles for a long time, whereas PAA25 can be applicable to systems where suspensions are removed continuously for a short period of time.

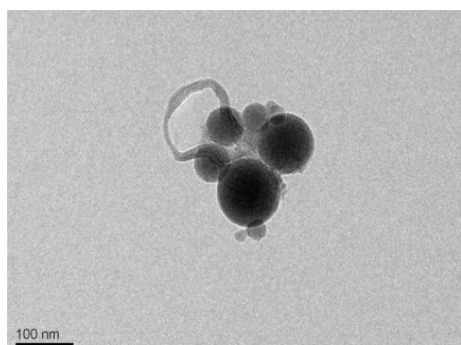


Fig. 2. TEM images of magnetite particles obtained for suspensions with PAA25 after 210 days.

### 3. Conclusions

Aqueous suspensions with PAA18 were more stable than those with PAA25 at room temperature. The stability of the suspensions increased as the pH and concentration of PAA18 and PAA25 increased. The particles dispersed in suspensions were sustained for more than 300 days.

### ACKNOWLEDGMENTS

This research was funded by the Ministry and Knowledge Economy of the Republic of Korea.

### REFERENCES

- [1] A. Sehgal, Y. Lalatonne, J. F. Berret, and M. Morvan, Precipitation-Redispersion of Cerium Oxide Nanoparticles with Poly 9acrylic acid); Toward Stable Dispersions, *Langmuir*, Vol. 21, No. 20, pp. 9359-9364, 2005.
- [2] J. A. Lewis, Colloid Processing of Ceramics, *J. Am. Ceram. Soc.*, Vol. 83, No. 10, pp. 2341-2359, 2000.
- [3] E. H. Lee, M. K. Lee, and C. K. Rhee, Preparation of Stable Dispersions of Ni Nanoparticles by Using a Polymeric Dispersant in water, *Mat. Sci. Eng.*, Vol. A449-451, pp. 765-768, 2007.
- [4] A. A. Zaman, R. Tsuchiya, and B. M. Moudgil, Adsorption of a Low-Molecular-Weight Polyacrylic Acid on Silica, Alumina, and Kaolin, *J. Colloid Interf. Sci.*, Vol. 256, No. 1, pp. 73-78, 2002.
- [5] J. Cesarano III and I.A. Aksay, and A. Bleier, Stability of Aqueous  $\alpha$ - $\text{Al}_2\text{O}_3$  Suspensions with Poly(methacrylic acid) Polyelectrolyte, *J. Am. Ceram. Soc.*, Vol. 71, No. 4, pp. 250-255, 1988.