

Preparation and Characterization of Stable Dispersions of Ni Nanoparticles

Eun Hee Lee, Min Ku Lee and Chang Kyu Rhee

Nuclear Nano Materials Development Lab, Korea Atomic Research Institute, P.O. Box 105,
Yuseong-gu, Daejeon, South Korea 305-600
ehlee@kaeri.re.kr, leeminku@kaeri.re.kr, ckrhee@kaeri.re.kr

Abstract

The effects of several experimental parameters on the formation of stable Ni nanoparticles dispersion were investigated. The suspensions of Ni nanoparticles were produced in organic solvents using Hypermer KD-2 as a dispersant. The transmission profiles, particle size distribution, zeta potential, and visual inspection results were used to discuss the stability of the dispersion. The optimal conditions for the formation of stable dispersion are evaluated.

Keywords : dispersion, suspension, Ni nanoparticles, zeta potential

1. Introduction

When finely dispersed in solutions, Ni nanoparticles have important applications in catalysts, electrophoretic deposition, and electrohydro-dynamics [1-5]. In particular, the Ni-base alloy (Alloys 600, 690, and 800) is the principal material used for the tubing of steam generators in nuclear power plants (NPPs) [6]. These Ni-base alloy nanoparticles can be used to repair cracks that occur during the operation of an NPP. However, the preparation of a stable suspension of Ni nanoparticles is difficult because of the high density and large size of the particles. Therefore, in the present work, we prepared suspensions of Ni nanoparticles in various organic solvents with a dispersant, and then investigated their dispersion stability.

2. Experimental and Results

To obtain Ni nanoparticles, we used levitation-gas condensation (LGC) [7]. We found that the Ni nanoparticle sizes range in size from 10 nm to 40 nm and we observed some NiO due to passivation on the particle surface in an air environment. Ni particles had a specific surface area of 30.36 m²/g. Next, we used Hypermer KD-2 (Uniquema, UK) as a dispersant, without additional purification. For the solvents, we used ethanol, 2-propanol, ethyl acetate, toluene, and xylene. We prepared the suspensions with 0.01 wt% of Ni nanoparticles and Hypermer KD-2 (0.6 wt% to 4 wt%) in organic solvents. To characterize the dispersion stability, we used Turbiscan LAb (Formulation, France) and 90 Plus particle size analyzer (Brookhaven Instruments, USA). We prepared the suspensions in various polar and non-polar solvents with 0.01 wt% of Ni nanoparticles and 1 wt% of Hypermer KD-2. To quantify the dispersion stability, we

used Turbiscan LAb to measure transmission profiles in clear to turbid dispersions [8]. We immediately analyzed the prepared suspensions without further dilution. Coalescence and flocculation phenomena related to the increase in particle size, and sedimentation related to the particle migration were observed [9,10]. Figure 1 shows the calculated mean value of the kinetics for each ΔT profile as function of time for the suspensions with 0.01 wt% of Ni nanoparticles and 1 wt% of the dispersant in various organic solvents. The suspensions prepared in xylene and ethyl acetate rapidly increased in the mean ΔT values. As a result, sedimentation occurred within 1 day and the agglomerated particles were large. However, for each suspension prepared in toluene and 2-propanol, the sedimentation occurred within 2 days and 15 days even though the mean ΔT values were low and the agglomerated particles were small. The suspension prepared in ethanol has a low mean ΔT variation, and visual inspection has confirmed the dispersion stability for more than a year. These results appeared that the polarity of solvents influenced the dispersion stability. Figure 2 shows the concentration effect of Hypermer KD-2 for the suspensions prepared in ethanol with 0.01 wt% of Ni nanoparticles. For suspension without the dispersant, the mean ΔT values increased rapidly, which indicates that the particle size increased continuously. This suspension, as evaluated by visual inspection, showed dispersion stability for 10 days. The suspensions with the dispersant (0.6 wt% - 2 wt%) now have been stable for more than a year. Figure 3 shows the dispersant concentration dependence of the particle size in suspensions with 0.01 wt% of Ni nanoparticles. Without the dispersant, the particle size is about 300 nm. As the concentration of the dispersant increased, the particle size decreased in size about 200 nm. This decrease in particle size indicated that Ni nanoparticles

were well coated with Hypermer KD-2. As a result, electrostatic repulsion between particles is fully provided to prevent the agglomeration. The concentration effect of Hypermer KD-2 on the zeta potential showed that the zeta potential values varied from a positive charge to a negative charge as the dispersant was added and were about -50mV (0.6 wt% - 2wt% dispersant). If we compare particle size data in Fig. 3, it is well corroborated with the zeta potential results. For the suspension with 4 wt% of the dispersant, the decrease in zeta potential was about 8mV, while the dispersion stability was unaffected. From these results, we were able to find the optimal conditions for suspensions with 0.01 wt% of Ni nanoparticles and varying concentration of Hypermer KD-2. The suspensions prepared in ethanol with Hypermer KD-2 are stabilized by the steric effect because of the high molecular weight and the chemical structure of the dispersant

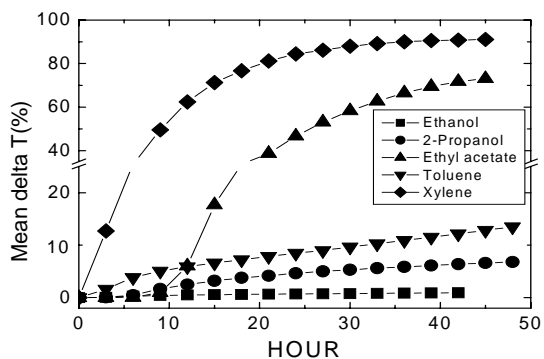


Fig. 1. Effect of the solvents on dispersion stability.

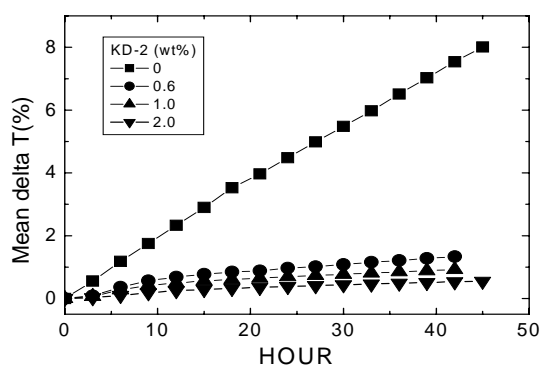


Fig. 2. Effect of Hypermer KD-2 concentration on dispersion stability of the suspensions.

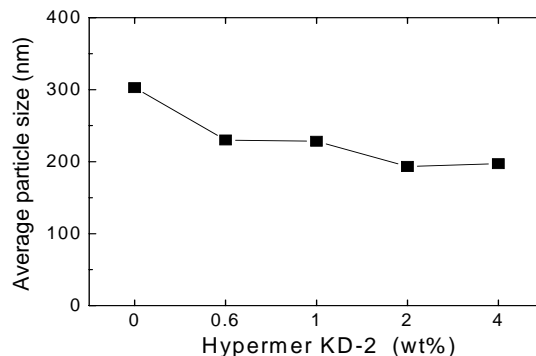


Fig. 3. Effect of Hypermer KD-2 concentration on particle size distribution of Ni nanoparticles.

3. Summary

Through characterization of the dispersion stability of Ni nanoparticles in several organic solvents, we found that ethanol was an optimal solvent to prepare a stable suspension. For suspensions of Ni nanoparticles prepared in ethanol, there appears to be good correlation between transmission profiles, particle size distribution, zeta potential, and visual inspection results.

4. References

1. L. N. Lewis, Chem. Rev. **93**, 2693 (1993).
2. B. C. Gates, Chem. Rev. **95**, 511 (1995).
3. P. Sarkar, X. Haung and P. Nicholson, J. Am. Soc. **76**, 1055 (1993).
4. M. Trau, D. A. Saville and L. A. Aksay, Langmuir **13**, 6375 (1997).
5. W. E. Winder and I. E. Reimanis, Surface and Coating Technology **157**, 267 (2002).
6. T. Shibata and S. Fujimoto, Corrosion **46**, 793 (1990).
7. A. Y. Yermakov, M. A. Uimin, A. A. Mysik and T. Goto, Mater. Sci. Forum, **386-388**, 455 (2002).
8. Formulation, France, user guide (2005).
9. E. H. Lee, M. K. Lee and C. K. Rhee, Mater. Sci. Forum, **510-511**, 206 (2006).
10. B. P. Singh, R. Menchanvez, C. Takai, M. Fugi and M. Takahashi, Colloid and Interface Science **291**, 181 (2005).

Acknowledgements

This research was supported by a grant from the ETEP R&D program funded by the Ministry of Commerce, Industry and Energy (MOCIE), Republic of Korea.