

# **Dispersion of Alloy 625 Nanoparticles in Ethanol**

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## Abstract

The influence of several experimental parameters on the formation of stable Alloy 625 nanoparticles dispersion in ethanol was investigated. Several analyzing methods were applied, like trsnsmission profiles measured by Turbiscan, transmission electron microscopy, X-ray diffraction, gas chromatography, and particle size analyzer. The correlation among the increase of particle sizes, caused by nanoparticle coalescence and collision, concentration of dispersant and time was presented and discussed. The optimum conditions for the formation of stable dispersion are evaluated.

Keywords : dispersion, suspension, Alloy 625 nanoparticles, particle size

## 1. Introduction

Many authors have recently studied various syntheses and applications of metallic nanoparticles because the nanostructure of metal differs significantly from the nanostructure of bulk phases [1,2]. When finely dispersed in solutions, Ni and Ni-base alloy nanoparticles have important applications in catalysts, electrophoretic deposition, and electrohydro-dynamics [3,4]. 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) [5]. These Ni-base alloy nanoparticles can be used to repair cracks that occur during the operation of an NPP. In the present work, we used Alloy 625 nanoparticles because of their high chromic content. First, we prepared suspensions of the Alloy 625 nanoparticles in ethanol with a dispersant, and then we investigated the dispersion stability. To characterize the dispersion stability, we used visual inspection, transmission profiles, and zeta potential measurements.

# 2. Experimental and Results

To obtain Alloy 625 nanoparticles, we used levitation-gas condensation (LGC) [6]. Uhm, et al. demonstrated a schematic illustration of equipment for preparing nanoparticles via LGC [7]. We then used transmission electron microscopy, X-ray diffraction, and gas chromatography for the characterization. A transmission electron micrograph shows that the Alloy 625 nanoparticles range in size from15 nm to 40 nm. From X-ray diffraction, the particles appeared to be nickel chromium iron austenitic steel. A gas chromatograph shows some oxide in Alloy 625 nanoparticles that formed as a result of the passivation on the particle surface in an air environment. Next, we used Hypermer KD-2 (Uniquema, UK) as a dispersant

without additional purification. The Hypermer KD-2 has a relatively high molecular weight and one or more anchoring groups. The suspensions of the Alloy 625 nanoparticles were prepared as follows: first, we fully dissolved the Hypermer KD-2 (0.4 wt% to 2 wt%) in 500 ml of ethanol under magnetic stirring; we then added 0.01 wt% of Alloy 625 nanoparticles, and ultrasonicated the suspensions for 3 hours. To characterize the dispersion stability, we used Turbiscan LAb (Formulaction, France) and we took measurements every 3 hours for 2 days. In addition, we measured the zeta potential with a 90 Plus particle size analyzer (Brookhaven Instruments, USA). We prepared the suspensions with 0.01 wt% of Alloy 625 nanoparticles and Hypermer KD-2 (0.4 wt% to 2 wt%) in ethanol. To quantify the dispersion stability, we used transmission profiles ( $\triangle$ T) measured by Turbiscan [8]. Using these  $\triangle T$  profiles, we calculated mean value of kinetics. Coalescence and flocculation phenomena related to the increase in particle size were observed [9,10]. Figure 1 shows the concentration effect of Hypermer KD-2 for the suspensions of 0.01 wt% of Alloy 625 nanoparticles, as well as the calculated mean value of the kinetics for each  $\triangle T$  profile as a function of time. Even without the dispersant, the suspension was dispersed well due to its positive charge. The visual inspection confirmed that the suspension maintained stability for more than 60 days. For the suspension with 0.4 wt% of the dispersant, there was an agglomeration of Alloy 625 nanoparticles, which consequently showed sedimentation occurring within 10 days. The suspension with 0.6 wt% of the dispersant has a low mean  $\triangle T$  variation. In this case, the sedimentation also occurred within 60 days. For the suspensions with 1 wt% and 2 wt% of the dispersant, the suspensions were observed to remain stable for more than 150 days. This shows that there is no correlation between the dispersion stability and the transmission profiles measured by Turbiscan LAb. Figure 2 shows the zeta potential of 0.01 wt% of Alloy 625 nanoparticles as a function of the Hypermer KD-2

concentration. The zeta potential values vary from a positive charge to a negative charge when the dispersant was added. Without the dispersant, a hydroxyl group of ethanol formed a hydrogen bond with oxygen on the nanosized Alloy 625 surface, and as a result, a positive zeta potential was obtained. For suspensions with 0.4 wt%, 0.6 wt%, and 1 wt% of the dispersant, we did not observe any variation in the zeta potential. However, the suspensions with 0.4 wt% and 0.6 wt% of the dispersant were unstable, regardless of the high negative zeta potential. The amount of dispersant was apparently inadequate to produce a stabilization barrier on the particle surface. Particle-particle interactions consequently occurred, leading to an agglomeration of the particles. In addition, the magnitude of the zeta potential slightly decreased for 2 wt% of the dispersant. As the concentration of Alloy 625 nanoparticles increased from 0.01 wt% to 0.02 wt%, the increase in zeta potential was about - 10mV. Based on the foregoing results, we were able to identify the optimal conditions for stable suspensions of Alloy 625 nanoparticles with Hypermer KD-2. The concentrations of the dispersant and Alloy 625 nanopartilces affect the dispersion stability. Moreover, the suspensions are stabilized by the steric effect because of the high molecular weight and the chemical structure of the dispersant.



Fig. 1. Effect of the dispersant concentration on the dispersion stability of the suspensions.



Fig. 2. Effect of the dispersant concentration on the zeta potential in ethanol suspensions.

### 3. Summary

We found that the suspensions of 0.01 wt% of Alloy 625 nanoparticles in ethanol with 1 wt% and 2 wt% of Hypermer KD-2 showed the dispersion stability for more than 150 days. However, the zeta potential was about -40mV for suspensions with the dispersant. For dispersion stability, there appears to be no correlation between transmission profiles, zeta potential, and the dispersant concentration.

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### 5. References

- 1. L. N. Lewis, Chem. Rev. 93, 2693 (1993).
- 2. B. C. Gates, Chem. Rev. 95, 511 (1995).
- P. Sarkar, X. Haung and P. Nicholson, J. Am. Soc. 76, 1055 (1993).
- M. Trau, D.A. Saville and I.A. Aksay, Langmuir 13, 6375 (1997).
- 5. T. Shibata and S. Fujimoto, Corrosion 46, 793 (1990).
- A. Y. Yermakov, M. A. Uimin, A. A. Mysik and T. Goto, Mater. Sci. Forum, 386-388, 455 (2002).
- Y. R. Uhm, W. W. Kim and C. K. Rhee, Phys. Stat. Sol. 210, 1934 (2004).
- 8. Formulaction, France, user guide (2005).
- I. Sondi, D. V. Goia and E. Matijevi, J. Colloid Interface Sci. 260, 75 (2003).
- 10.E. H. Lee, M. K. Lee and C. K. Rhee, Mater. Sci. Forum, **510-511**, 206 (2006).