N O T E S

Studies on Coprecipitation of Metal Ions with Aluminum Hydroxide. II.[1] Nickel(II)

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The method of coprecipitation with a carrier has been often used for separation and concentration of minute amounts of metals. The coprecipitation percentage of metals is dependent on many factors.[2-4] The effects of these factors must be thoroughly investigated for obtaining sufficiently high percentage of coprecipitation. In this study, nickel was chosen as the metal to be coprecipitated with aluminum hydroxide. The precipitate of aluminum hydroxide was formed in the solution containing nickel salt, but in some case, it was formed before nickel salt solution was added to it.

Experimental

Standard nickel salt solution (1×10^{-2} M) was prepared from nickel chloride and kept slightly acidic with hydrochloric acid. All dilute nickel solutions were prepared before use from this solution. Aluminum salt solution (5×10^{-2} M) was prepared from aluminum chloride.

To the mixture of the solutions of nickel chloride, aluminum salt and ammonium chloride in a 100 *ml* beaker, ammonia water or hydrochloric acid was added with a pipette so as to obtain desired pH value. The final volume of the solution was 100 *ml*. The solution was heated on a water bath for 10 min and kept in a thermostat at 25.0±0.1°C for more than 12 hr. Then, the pH value of the solution was measured, and the precipitate was filtered through Toyo-Roshi filter paper No. 5A. The precipitate on the filter paper was dissolved with 10 *ml* of 6 N hydrochloric acid, washed with water, and the whole solution was evaporated on a water bath almost to dryness. The residue was dissolved with water and transferred into a 50 *ml* volumetric flask. Then, 25 *ml* of 2 M ammonia-ammonium chloride and 5 *ml* of 0.1% gelatin were added to it and diluted to the mark with water. The amounts of nickel in the solution were measured polarographically after deaeration with purified hydrogen. The half-wave potential was −1.1 V *vs.* SCE.

Results and Discussion

Effect of Aging of Precipitate. The relation between the percentage of nickel coprecipitated with aluminum hydroxide and aging time is shown

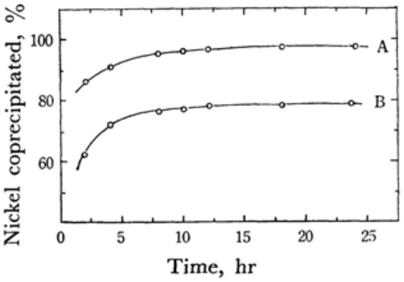

Fig. 1. Effect of aging.
4×10^{-4} M $NiCl_2$, 5×10^{-3} M $AlCl_3$, pH ≒ 8.4,
Temp. 25.0±0.1°C
Curve A: 2×10^{-2} M NH_4Cl
Curve B: 2×10^{-1} M NH_4Cl

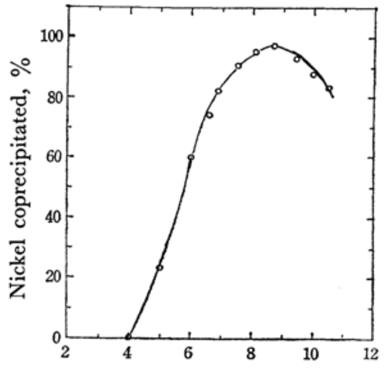

Fig. 2. Effect of pH.
4×10^{-4} M $NiCl_2$, 5×10^{-3} M $AlCl_3$, 2×10^{-2} M NH_4Cl

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[1] Part I of this series: T. Yonekubo, *Nippon Kagaku Zasshi (J. Chem. Soc. Japan, Pure Chem. Sect.)*, **84**, 502 (1963).

[2] I. M. Kolthoff and B. Moskovitz, *J. Phys. Chem.*, **41**, 629 (1937).

[3] M. H. Kurbatov, G. B. Wood and J. D. Kurbatov, *J. Phys. Chem. & Colloid Chem.*, **55**, 1170 (1951).

[4] M. Ishibashi, T. Fujinaga, T. Kuwamoto and H. Sawamoto, *Nippon Kagaku Zasshi (J. Chem. Soc. Japan, Pure Chem. Sect.)*, **85**, 763 (1964).

in Fig. 1. The coprecipitation percentage increased in earlier stage, but after about 12 hr, it showed a tendency to reach a constant value.

Effect of pH of the Solution. The pH value of the solution had much influence on the coprecipitation of nickel as shown in Fig. 2. The coprecipitation percentage was maximum at pH 8—9, reaching almost 100%. The percentage decreased rapidly outside this range and became zero at pH 4. At such pH, the carrier itself is soluble as well as nickel hydroxide.

Effect of Concentration of Aluminum Salt. Keeping the other variables constant, coprecipitation percentage of nickel ions was observed at various concentrations of aluminum chloride. When the concentration of aluminum salt was small, the coprecipitation precentage was approximately 100%, and it decreased slightly in more concentrated solutions as shown in Fig. 3.

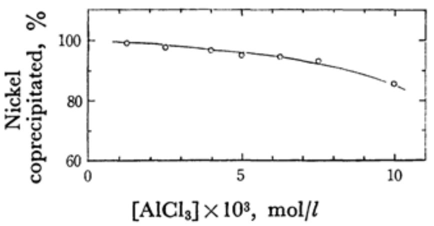

Fig. 3. Effect of aluminum salt concentration.
4×10^{-4} M NiCl$_2$, 2×10^{-2} M NH$_4$Cl, pH 8.4

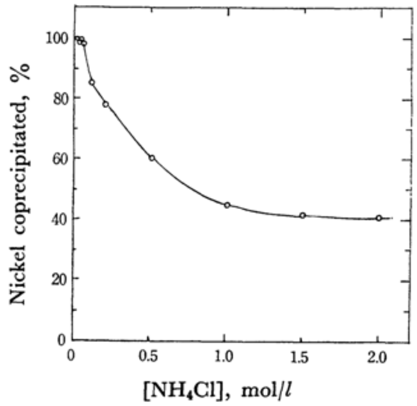

Fig. 4. Effect of concentration of ammonium chloride.
4×10^{-4} M NiCl$_2$, 5×10^{-3} M AlCl$_3$, pH 8.5

Effect of Concentration of Ammonium Chloride. The relation between the coprecipitation percentage of nickel and the concentration of ammonium chloride is shown in Fig. 4; the coprecipitation percentage was nearly 100% in the range of low concentration—less than 0.02 M—of ammonium chloride, and it decreased rapidly as the concentration of the salt increased, but it reached to a constant value in the solutions more concentrated than 1 M.

Efficiency of Pre-formed Carrier. In the above experiments, precipitate of aluminum hydroxide was formed directly in the solution containing nickel salt, and now, another experiment was tried to examine the efficiency of pre-formed aluminum hydroxide that was formed as follows: 25 ml of 1 N ammonia water and 10 ml of 2 N hydrochloric acid were added to 10 ml of 0.01 M aluminum chloride in a 50 ml tube, centrifuged at a rate of 2000 rpm for about 3 min. The obtained precipitate of aluminum hydroxide was washed three times with water and poured into a 100 ml beaker, then 10 ml of 4×10^{-3} M nickel chloride, 10 ml of 2 M ammonium chloride and 0.1 N (1 N) ammonia water or 0.1 N hydrochloric acid were added to the solution to obtain desired pH value.

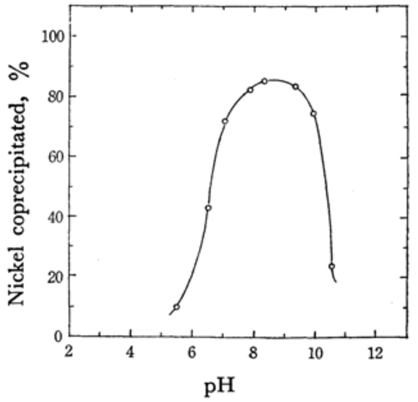

Fig. 5. Efficiency of pre-formed carrier.
4×10^{-4} M NiCl$_2$, 2×10^{-2} M NH$_4$Cl

The solution was kept in a thermostat for more than 12 hr, and the coprecipitation percentage of nickel was measured in the same way as above. As shown in Fig. 5, the coprecipitation percentage showed maximum at pH 7.5—9.5, reaching about 85%, and it decreased rapidly outside this range. The smaller percentage compared with the value in Fig. 2 is attributed to smaller adsorptive surface of this carrier.

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