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Synthesis of cobalt nanoparticles via surfactant-assisted hydrothermal reduction process in near critical and supercritical water

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Introduction

Cobalt nanoparticles were synthesized with or without surfactants through hydrothermal reduction process at 340°C and 380°C in batch type reactor. Decomposition of formic acid was used as reduction agent and various organic modifiers were used for protection and functionality. Reaction kinetics, crystal structure and morphology change were studied here. The result show good crystalline cobalt nano particles were synthesized and morphology of cobalt nano particles also can be controlled at a certain experimental conditions.

Experiment

Pressure-resistant tube reactors (Hastelloy) whose inner volume was 5 ml were used for hydrothermal synthesis with in-situ surface modification. Preparing pre-cursors and loading chemicals to reactors were done in the argon gas based glove box. The reactor was loaded cobalt (II) acetate aqueous solution depends on conditions. In order to reduce the cobalt (II) acetate, formic acid was used. Formic acid is well decomposed to hydrogen and CO_2 in the presence of water. For the modification of surface of cobalt, various modifiers were added. The reactors were capped tightly and came out of the Glove box. Then the reactors were put in an electric furnace whose temperature was maintained at 350°C or 400°C (inner temperature was 340°C or 380°C each). The reaction was performed from 5 to 60 min and terminated by quenching the reactors in a water bath (24°C). After quenched, the particles were corrected by hexane (or methanol in the case of without modifiers). The obtained products were once centrifuged to remove hexane and then purified by three times of a combination of decantation and centrifugation using methanol. Finally, the products were collect by cyclohexane or methanol again and dried in the deciccator which was installed in the glove box.

Results and discussion

Cobalt nano particles were synthesized at different reaction time and different reaction temperature. Obtained particles weights were measured with electric balance. Very small particles which were easily stick to the reactor wall were also corrected by Nitric acid solution (1:3 volume ratio) then analyzed with Atomic Absorption Spectroscopy. Sum of two results shows yield of produced cobalt NPs. Obtained particles were analyzed by XRD and the represented peaks showed hcp or fcc cobalt. Figure 1 shows the XRD pattern of one of obtained Cobalt NPs. Figure 2 shows TEM image of modified cobalt NPs.

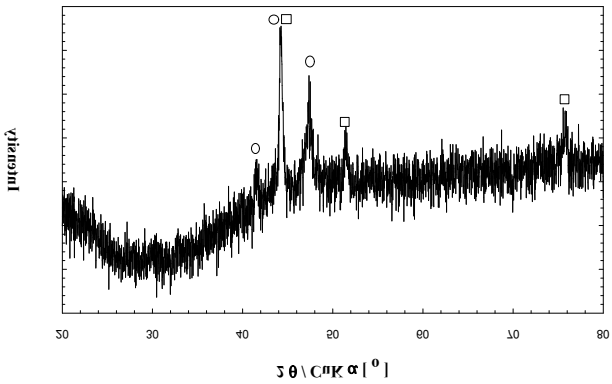

Fig 1. XRD pattern of obtained Co NPs: (□) fcc cobalt; (○) hcp cobalt.

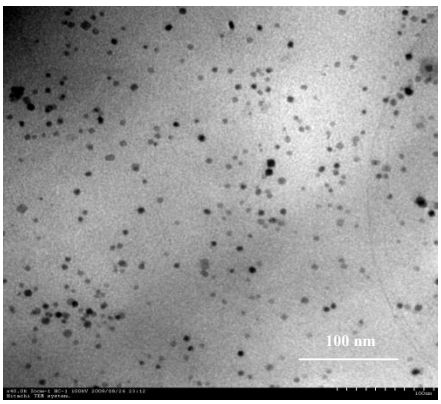

Fig 2. TEM image of modified cobalt NPs with oleylamine synthesized at 380°C.

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