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Electrochemical synthesis and structure characterization of nickel sulfide nanoparticles

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ABSTRACT

Nickel sulfide (NiS) nanoparticles were synthesized by a simple electrochemical route. In this study, synthesis conditions of nickel sulfide nanoparticles were optimized by Taguchi robust design. The current application of the Taguchi method was successful in optimizing the experimental parameters of the electrochemical synthesis procedure of nickel sulfide nanoparticles. The effects of several main procedure factors (i.e., sulfide ion concentration, applied voltage, stirring rate and reactor temperature) on the particle size of synthesized nickel sulfide particles were investigated. The significance of these factors on the diameter of nickel sulfide particles was evaluated by the analysis of variance (ANOVA) and the optimum conditions for the preparation of nickel sulfide nanoparticles by the proposed electrochemical method was proposed. The prepared NiS nanoparticles at optimum conditions were characterized by various techniques i.e., SEM, TEM, XRD, EDX, FT-IR, UV–vis and PL.

1. Introduction

Investigation on the facile methods for preparation of inorganic compounds with nano-sized dimensions and special morphology has been obtained a wonderful attraction and interest in different fields of science especially in material science and nanotechnology. Among wide range of inorganic compounds, metal sulfides present valuable electronic properties and applied in different products and technologies [1,2]. Nickel sulfide with some special properties such as paramagnetic–antiferromagnetic transition and metal insulator have been used widely as a catalyst for hydrodenitrogenation and hydrodesulfurization reactions, transformation toughener in window glasses, IR detectors, rechargeable lithium batteries and solar storages [3–8]. Until today, there have been published only limited reports on the preparation of nickel sulfide. These reports proposed some techniques including chemical vapor deposition, solid state reaction [9], and precipitation from aqueous and organic media [10–12] for the preparation of nickel sulfide powder. Therefore, any effort put to introducing new methods or optimizing experimental conditions of previous techniques for the synthesis uniformly NiS nanoparticles is interested. On the other hand, developing any procedure for the generation of nanoparticles requires an optimization step. Thus, in this investigation Taguchi robust design as an orthogonal array design (OAD) was used for this aim. The methodology, theory, and also application of OAD in optimization of the nanoparticle synthesis procedures have been illustrated in detail previously [13–20]. This study was aimed to investigate the role of several experimental variables in the size of nickel sulfide particles and identify the best conditions of synthesis procedure in order to preparation of NiS nanoparticles. Therefore, effect of some experimental variables i.e., sulfide ion concentration, applied voltage, stirring rate and temperature on the particle size of

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nickel sulfide was investigated by the experimental design procedure. Up to date, several reports deal with preparation of uniform metal sulfides by different procedures have been published [1–3,6,10]. To the best our knowledge, this is the first report on the optimization of conditions for synthesis of nickel sulfide nanoparticles via the electrodeposition procedure.

2. Experimental

2.1. Materials and procedure

Reagent-grade sodium sulfide was used as received from Merck Company (Germany). Nickel electrode ($2 \times 3\ cm^2$ in size) with 99.99% purity as anode and a steel electrode ($1 \times 3\ cm^2$ in size) as cathode were inserted into the sulfide solution. Before electrodeposition, the electrodes were mechanically polished several times by a wire brush and then rinsed with the distilled water [20]. The electrolyte solution was prepared by adding the definite amounts of sodium sulfide into the distilled water to achieving the adequate concentrations. The electrodes inserted into the electrolyte solution were connected to a programmable power supply in order to adjust desired voltages. The beaker containing electrolyte solution was placed on a magnetic hot plate, while the temperature of the solution was adjusted via surrounding water. Concentration, voltage, stirring rate and temperature at each experiment were adjusted according to the designed trial conditions (Table 1). Electrodeposition was started by applying a direct current to the electrodes while the time was taken. At the end of process, the electrodes were removed from the electrolyte solution. Collecting of the products was carried out by centrifuging the solution. The precipitate was washed with distilled water thrice. Then, the resulted precipitate on the filter was washed with ethanol and dried at 90 °C for about 2 h. Experimental parameters of the electrodeposition process for synthesis of nickel sulfide nanoparticles were optimized via Taguchi robust design. The studied variables (S^{-2} concentration, applied voltage, stirring rate and temperature of reactor) and their levels are shown in Table 1.

2.2. Characterization of synthesized nanoparticles

The morphology of electrosynthesized nickel sulfide samples was investigated by scanning electron microscope (SEM). A Hitachi S-4160 series instrument was used to record SEM images, while dried particles were loaded on the instrument by a gold film. The particles were coated onto the gold films using a sputter coater model SCD005 made by BAL-TEC (Switzerland). Transmission electron microscope (TEM) images were obtained on a Ziess-EM10C scanning electron microscope. The sample preparation was carried out via coating on formvar carbon coated grid Cu Mesh 300 prior to the measurement. X-ray powder diffraction (XRD) analysis was carried out by a Rigaku D/max 2500V diffractometer equipped with a graphite monochromator and a Cu target. The product was also analyzed by energy-dispersive analysis by X-rays (EDX). Fluorescence properties of the produced NiS were measured on a Beckman (LS-45) spectrofluorometer using a 150W xenon lamp as an excitation source. The synthesized NiS nanoparticles dispersed in distilled water were analyzed by UV–vis spectrophotometer made by PerkinElemer (Lambda 35) in wavelength range of 200–700 nm. The IR spectrum of NiS nanoparticles was obtained on a FT-IR spectrophotometer (Bruck Equinox 55) using the KBr pellet technique.

3. Results and discussion

3.1. Nanoparticles synthesis procedure and optimization strategy

By the use of fractional factorial experiments, i.e. Taguchi robust design, the required number of experiment trials for parameters optimization could be reduced efficiently [21–24]. Furthermore, the other advantage of fractional factorial in comparison with the sequential design is the possibility of extracting more information from the resulted experimental data. In Taguchi robust designs, depending on the number of studied parameters and their levels an orthogonal array is used to perform a series of experiments whose results are analyzed via the well-known mathematical processes. In this method, the average effect of the studied factors at each preselected level could be independently calculated. Thus, arranging the experiments orthogonal permits us to separate the effects of different parameters at their various levels [25–27].

Tuning the size of particles formed in electrodeposition processes is a complicated step and comprehension of the relations between the process parameters and size of product is time-consuming. However, experimental identification of the effects of parameters on the size of product particles by their statistical optimization could be a shortcut.

Table 1
The OA_9 (3^4) matrix for parameter optimization in synthesis of nanoparticles via electrochemical reaction and mean diameter of produced nickel sulfide.

Experiment number	S^{2-} Concentration (M)	Voltage (V)	RPM	Temperature (°C)	Average diameter of NiS particles (nm)
1	0.01	5	200	20	22
2	0.01	10	400	40	25
3	0.01	15	600	60	27
4	0.05	5	400	60	21
5	0.05	10	600	20	20
6	0.05	15	200	40	23
7	0.1	5	600	40	17
8	0. 1	10	200	60	22
9	0.1	15	400	20	21