

Morphological Properties of Nickel Selenide Synthesis Through Hydrothermal Routes with The Use of Different Time

Nur Amalini Zulkifli^{1*}, Siti Nadiah Zulkifli¹, Lee Han Kee¹, Zainal Abidin Talib¹,
Tan Sin Tee¹,

¹*Department of Physics, Faculty of Science, Universiti Putra Malaysia, 43400 UPM
Serdang, Selangor, Malaysia*

** Corresponding author: amalinizulkifli@gmail.com*

Abstract: This study was undertaken to investigate the effects of reaction time of nickel selenide (NiSe) synthesized. In this work, nickel selenide has been synthesized via hydrothermal method from a well-mixed solution containing nickel chloride 6-hydrate and selenium tetrachloride as a precursors, hydrazine hydrate as a reducing agent, and distilled water as a reaction medium. The samples were prepared by using different reaction of time, which are 12, 24 and 36 hours at 200 °C. As a results, the Scanning Electron Microscopy (SEM) images showed that morphology of the samples consists of aggregation of nanoparticles with almost uniform shape with different particle sizes.

Keywords: Nickel silinide, Hydrothermal, Nanoparticles, Nucleation and growth

The investigation of synthesis nickel selenide (NiSe) nanoparticles has attracted remarkable interest. This is because, more than in current years, many studies attempts were made reserved for research on semiconductor nanocrystallites because of its quantum size and potential application impacts. In the area of material science, nickel selenide semiconductors exhibit attractive electronics and magnetics properties and have discovered a few applications, which has attracted a lot of research attention over the last one decade. It is well recognized that the shape of nanoparticles plays a crucial role in determining their properties. However, there is challenges in order to fabricate of size and shape-controlled nanoparticles and their self-assembly into materials. One of the problems in the preparation of nanoparticles is the control of growth by varying the reaction parameters. Experimental factors which includes concentration, temperature, pH, capping agent as well as the reaction time are critical parameters that effect the size and the morphology of the nanoparticles.

There are a lot of techniques were used to synthesis of nickel selenide. The most reported one is by solvothermal technique. But, this approach remains a challenge to gain a series of nickel selenides (NiSe₂, Ni_{1+x}Se and Ni₃Se₂) and systematically control their compositions, phase structures and morphologies in aqueous solution. Lately, the hydrothermal approach has been also reported to provide metal chalcogenide nanostructural materials [1].

In this work, we demonstrated nickel selenide synthesized through hydrothermal route. Hydrothermal method was selected in this work because the method is simple, reaction condition can be controlled easily and also non-toxic precursor and solvent

were used in this work. Experimental factor such as reaction of time, play significant role in the formation of the nickel selenide. The effect of reaction time on the morphology and optical properties of NiSe prepared by hydrothermal method, were characterized by Scanning Electron Microscopy (SEM).

In this study, nickel selenide were synthesized from an aqueous solution containing of nickel chloride 6-hydrate, selenium tetrachloride, hydrazine hydrate and distilled water by the hydrothermal process. nickel chloride 6-hydrate and selenium tetrachloride with a molar ratio of 1:1 were dissolved in of distilled water at room temperature under magnetic stirring for 15 minutes. Then, hydrazine hydrate were added drop wise to the above solution. After five minutes, the reactants were put in a teflon-lined autoclave of 50.0 ml capacity. The autoclave was maintaining at 200 °C for 12, 24 and 36 hours and cooled down to room temperature naturally. The black precipitate formed was separated by centrifugation at 3000 rpm for two minutes. In order to remove other impurities, the precipitate was washed with distilled water for two times and an ethanol and dried at 60°C for six hours. The powder formed was grind with pastel and mortar sets to reduce substances to very fine powder and better homogeneity after heat treatment process. After that, the samples were transferred into the sample bottle. Finally, sample formed were characterized by scanning electron microscopy (SEM).

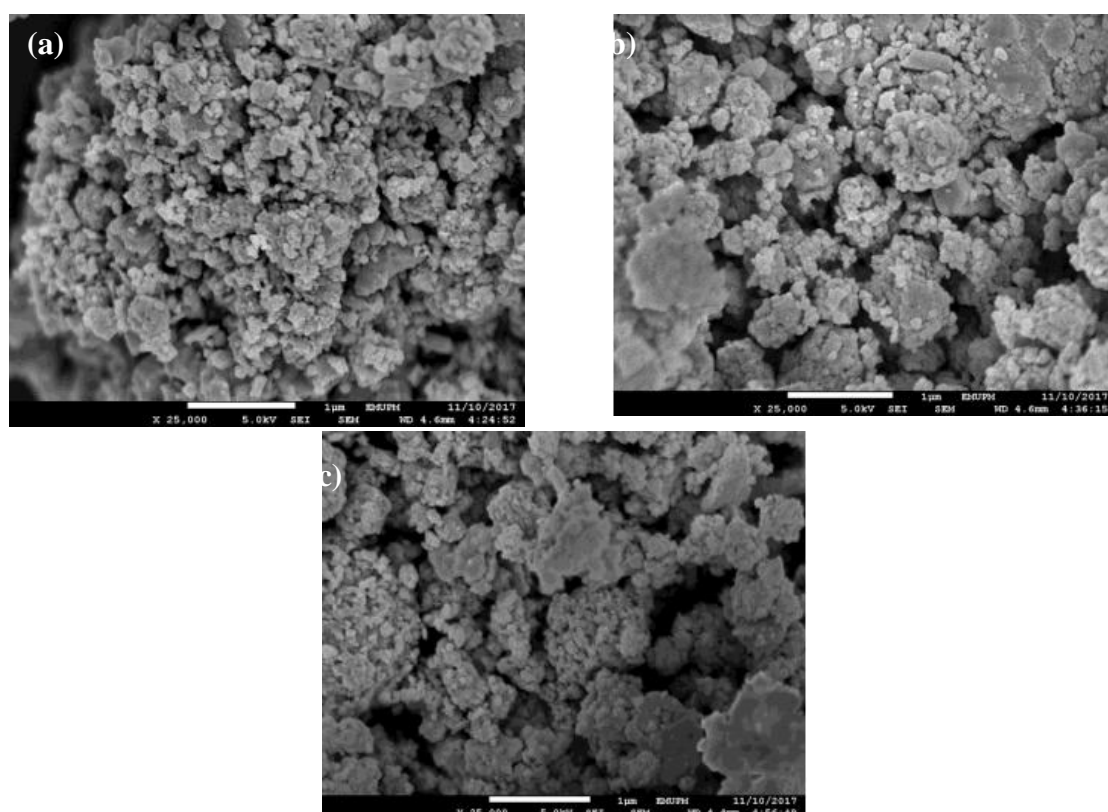


Figure 1: SEM image for a)12 hours, b) 24 hours and c) 36 hours reaction of time of nickel selenide synthesized.

The morphologies of the synthesized nickel selenide were examined by using scanning electron microscopy (SEM). The SEM micrographs of the particle

synthesized at different time are shown in figure above. The effect of reaction time on the morphology of nickel selenide nanostructure was investigated. The SEM image of the particles synthesized at 200°C for 12 hours, based on Figure 2, shows agglomerates of small nanoparticles, with a size lower than 60 nm. When increasing the reaction time to 24 hours, the size of the nanoparticles increased and their aggregation of the nanoparticles to form the flower-like shaped structures. However, on prolonged the time to 36 hours, the size of the particles decreased and flower-like shaped structures obtained.

Sobhani et al. [2], proposed a growth mechanism of these structures, wherein the individual nanoparticle becomes unstable and aggregates into three-dimensional flowers when no surfactant is used and protection disappears. Libert et al. Suggested that there were two dynamic processes and a theoretical model for the formation of nanoparticles in order to control synthesis of CdS nanoparticles. According to the model, firstly, the process is nucleation, and the nuclei grow to form the nanoparticles. The sequential second process is aggregation of the nanoparticles to form the flower-shaped structures. The NiSe flower-like structures could be formed in a similar way. For the formation of the NiSe nanoparticles, the aggregated and flower-like structured formed because of the nucleation takes place immediately after mixing the precursors. The nucleation rate for the samples could increase rapidly when there is no surfactant to control the size of the nuclei.

Besides, the formation of the flower-like NiSe should be related to the layered anisotropic lattice structure during the hydrothermal synthesis. During the synthesis process, a free Se atom or Se^{2-} ion tends to bond with the atoms on the growing crystal surface via covalent bonding. Since a van der Waals bonding is not strong enough to hold the atom on the atomic surface, a single Se atom or Se^{2-} ion from the solution attaching itself to a Se layer crystal surface will probably jump back into the solution. Therefore, a NiSe crystal will grow faster in the a- and b-axis directions than in the c direction.

Sobhani et al., [3] mentioned that the reason why the flower-like NiSe were obtained because of the hydrazine is relatively slower in the reduction rate while Salavati-Niasari and Sobhani [1] stated that because of the $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ salts are used which caused the nucleation occur slowly. These reasons allowed ample time for the NiSe particles to agglomerate and grow into more regular shapes.

Nickel selenide (NiSe) were successfully prepared by a hydrothermal route. In this work, we conclude the hydrothermal method synthesis of NiSe and the effect of reaction time on the morphological and optical properties of the nickel selenide synthesized. Based on SEM morphology, the flower-like formed because no surfactant were used in this experiment, to control the size of nuclei, nickel salt and hydrazine used caused nucleation and reduction occur slowly respectively. Lastly, the

formation of the flower-like NiSe should be related to the layered anisotropic lattice structure during the hydrothermal synthesis.

References

- [1]. Salavati-Niasari, M., & Sobhani, A. (2013). Effect of nickel salt precursors on morphology, size, optical property and type of products (NiSe or Se) in hydrothermal method. *Optical Materials*, 35, 904–909.
- [2]. Sobhani, A., Davar, F., & Salavati-Niasari, M. (2011). Synthesis and characterization of hexagonal nano-sized nickel selenide by simple hydrothermal method assisted by CTAB. *Applied Surface Science*, 257, 7982–7987.
- [3]. Sobhani, A., Salavati-niasari, M., & Davar, F. (2012). Shape control of nickel selenides synthesized by a simple hydrothermal reduction process. *Polyhedron*, 31, 210–216.