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Original Research Paper

Determination of optimum condition to produce nanocrystalline pyrite by solvothermal synthesis method

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ABSTRACT

In this research, FeS_2 was produced by solvothermal synthesis method. For this purpose, various solvents including ethanol, 1-propanol, 2-propanol, 1-butanol and ethylenediamine were used. Results of XRD analysis showed that ethanol and 1-butanol were fairly proper to produce FeS_2. In addition, the effects of raw materials, temperature and molar ratio of materials, on process performance were investigated. The produced iron disulfide was analyzed by XRD and SEM analyses. Results indicated that usage of the couple of $FeSO_4$ and $Na_2S_2O_3$ with molar ratio 1:4 in ethanol at temperature around 200 °C were the most appropriate condition for producing pyrite. Also, result of SEM analysis showed products with nanostructures.

1. Introduction

Iron disulfide (FeS_2) has been used as a semiconductor material in solar cells because of its high optional absorption coefficient ($\alpha \approx 5 \times 10^5\ cm^{-1}$ when $\lambda < 750$), much higher than that of silicon and suitable energy band gap ($Eg \approx 0.95$ eV) which is comparable to that of silicon [1,2]. Also iron disulfide is nontoxic, ample and cheap.

FeS_2 has been prepared so far by different methods such as thermal sulfuration [3–7], mechanochemical milling [8–10], but the conversion efficiency of iron disulfide solar cells has been low because FeS_2 can crystallize not only into a cubic pyrite structure but also into an orthorhombic marcasite structure with low band gap ($Eg \approx 0.34$ eV) [11]. These methods also could not produce pyrite in a single stage process and they required high temperature and additional sulfur to convert other phases such as FeS or FeS_{2-x} into pyrite [11]. Thus, there is a necessity to design a low temperature method such as solvothermal synthesis [12–14].

Xuefeng et al. [12] have prepared one dimensional nano rod FeS_2 in cubic structure via a solvothermal method for the first time. Kar and Chaudhur [13,14] reported FeS_2 nano wires, nano ribbons and nano tubes by solvothermal synthesis method.

In the previous studies, a stainless-steel cylindrical chamber was used to produce pyrite [12–14] and none of them have used the stainless-steel stirred reactor. In other words, previous works were in laboratory scale and applied reaction systems were not appropriate to scale up pyrite production process.

In this research, it is reported the synthesis of nano structure FeS_2 in a stirred reactor. This issue is one of the paper's innovations. Using impeller increased the mass transfer, thus reaction time was reduced from 12 to 5 h. Also, quality of the product was increased and the particle size was decreased. It is noticeable that method used in this research is capable to scale up.

In addition, in this study, the effects of solvent, raw materials, temperature and molar ratio of raw materials are investigated.

2. Experimental

All reagents were analytical grade and were used without further purification. Experiments were carried out in 2000 ml stainless-steel stirred reactor equipped with a temperature controller, a barometer and an axial impeller with four blades. All experiments were performed at stirring speed 1000 rpm. In a typical process, an appropriate amount of iron source and sulfur source were mixed with 600 ml of solvent and the mixture was taken into the reactor. Then the reactor was maintained at desired temperature. After 5 h the reactor was cooled down to the room temperature (24 °C). The resulted black precipitates were filtered off and washed sequentially with distilled water and CS_2 to remove impurities (sulfur). The final products were dried at 60 °C for 4 h to get black powder.

The obtained samples were characterized by SEM to observe structure and XRD to determine composition. SEM images were taken by a VEGA\\TESCAN scanning electron microscope. The XRD analysis was carried out by a Philips X-ray diffractometer PW 1800 with Cu $K\alpha$ radiation ($\lambda = 1.542$ Å) (Fig. 1).

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