# Continuous Production of ws, Nanoparticles by Co-Precipitation Method

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#### **Abstract**

This manuscript demonstrates the design, modification and initial investigation of a co-precipitation method for manufacturing of inorganic fullerene WS<sub>2</sub> nanoparticles. Different preparation methods starting with various precursors have been investigated. Furthermore, the influence of temperature, reaction time, and reaction gases etc, on the synthesis of inorganic fullerene WS<sub>2</sub> nanomaterials was investigated, and these parameters were optimised based on combined characterisations using XRD, SEM and TEM.

**Keywords:** WS<sub>2</sub> Nanoparticles, XRD, SEM, TEM, I-V Characteristics

## Introduction

This photocatalysis is widely regarded as one of the most promising environmental remediation technique due to the clean energy utilization method [1,2]. Generally, some accepted that high-efficient photo catalysts with wide forbidden gap, such as WS<sub>2</sub> an ultraviolet irradiation [3]. As to practical application, photocatalysis strategy will be a huge boost once a photo catalyst can favourably absorb the abundant solar energy in visible region [4-6]. It is widely accepted that the solar cell energy can improve the separation probability of light-induced charge because the contact interfacial region of photocatalysis will provide an internal electric field to restrain the recombination probability, thus resulting in an efficient photocatalytic performance. In general, the designed photocatalysis will adopt at least one narrow band semiconductor to harvest more visual-light energy and then to generate more photos induced charges [7].

Layered transition metal dichalcogenides are widely regarded as a kind of promising loading material because of their analogous hexagonal structure [8]. For example, monolayered and few-layer  $WS_2$  have recently paid the attention of the scientific community in photocatalysis research, which describes the lack of interlayer coupling and the absence of inversion symmetry resulting in the symmetry and the photoelectric property that differ markedly from those of the bulk [9]. For instance, the hierarchical WS2 composites exhibited an efficient performance for photocatalytic oxidation of visual light photo catalyst has been reported.

These WS2nanomaterials, in addition to their significant mechanical, biocompatible and electronic properties, are excellent solid lubricants [10-12]. Accordingly, the incorporation of these nanomaterials into a proper matrix in composites will lead to new products with

hugely improved physical and mechanical properties. Another extraordinary property of WS<sub>2</sub> nanostructures is their superb shock absorbing performance [13-15], which suggests an important field of application in lightweight and high performance protective composites [16]. Such applications will obviously demand large amounts of WS<sub>2</sub> supply, however the synthesis was only obtained in gram level at the early stage, which was far too less for any practical work. Hence realising a great industrial level success in nanomaterials.

# **Materials and Methods**

The WS<sub>2</sub> samples were synthesized using a facile co-precipitation method. Typically, 2 mmol of Bi(NO<sub>3</sub>)<sub>3</sub>.5H<sub>2</sub>O was added to 10 ml of ethylene glycol solution containing dissolved Na<sub>2</sub>MoO<sub>4</sub>.2H<sub>2</sub>O with molar ratio of 2:1 under magnetic stirring. An appropriate amount of exfoliated WS<sub>2</sub> nanoslices was dispersed into 20 ml ethanol and ultrasonicated at room temperature for 45 min. Then, it was slowly added into the above solution, followed by stirring for 10 min to form a homogeneous phase. The resulting solution was transferred into a 50-ml. Subsequently, the autoclave was cooled to room temperature gradually. Finally the precipitate was centrifuged and was washed with ethanol and deionized water several times and dried in a vacuum oven at 80°C for 6 hr.

# **Structural Characteristics**

In order to confirm the composition and crystal structure of the as prepared samples, an XRD study was carried out. As shown in Fig. 1, it can be found that the pure WS $_2$ , peaks located at for 0.1 molar concentration  $23.94^{\circ},28.86^{\circ},33.78^{\circ}$  and 0.2 molar concentration  $16.37^{\circ},28.56^{\circ},31.82^{\circ}$  and 0.3 molar concentration  $27.54^{\circ}.31.82^{\circ},44.96^{\circ}$  have been observed, which matched well with the (106), (109), (008), (114), (208) crystal planes of WS $_2$  (JCPDS card no. 87-2417).

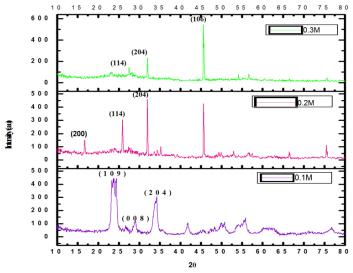
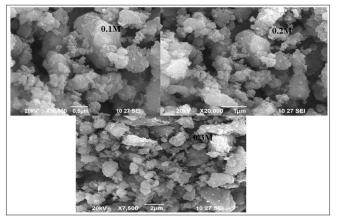


Figure 1: XRD Pattern of Tungsten sulphide 0.1, 0.2, 0.3 molar concentration

The XRD pattern only displays the characteristics diffraction peaks of hexagonal phases  $WS_2$  lattice. Furthermore, compared with the standard data for  $WS_2$  (84-1398), the existence of few layer  $WS_2$  did not change the diffraction peak positions of  $WS_2$  in the composite sample, indicating that tungsten sulphide Nano flakes grown on few-layer and then rather Nano slices incorporated into the tungsten sulphide lattice. There is no trace of any impurity phase under the present solution, which suggests the high purity of the as prepared samples [17].

## **Scanning Electron Microscopy**

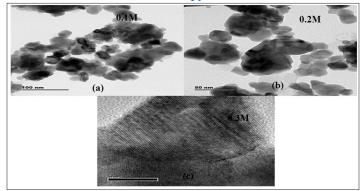


**Figure 2**: (a-c) SEM Images of Tungsten sulphide 0.1, 0.2, 0.3 molar concentration

The morphologies of the as synthesized samples were investigated using SEM. For comparison, SEM images of the bulk raw WS $_2$  without sonicated treatment and exfoliated nanoslices are shown in Fig.2. The former displays a distinct layer laminated morphology with about 2µm in thickness, while the latter exhibits with like more morphology with thickness varying from dozens of nanometers to 1-2µm. The results demonstrate that the layered commercial WS $_2$  have been stripped to few layer nanoslices. It can be clearly seen that the surfaces of WS $_2$  nanoslices were uniformly covered by numerous two dimensional nanoplates and that formed a hexagonal

structure [18-20].

## **Transmission Electron Microscopy**



**Figure 3**: (a-c) TEM Images of Tungsten sulphide 0.1, 0.2, 0.3 molar concentration.

TEM and HRTEM were utilised to further characterise all the samples. Products from WS<sub>2</sub> particles dominantly around 100nm in size are presented, with no coating observed. In fig.3 (a-c) there appears to be a layer of nanosheet at the bottom part of the image surrounding the particles, possibly being a WS<sub>2</sub> or nanosheet, as both possess similar morphologies at this magnification. The TEM images of sample WS<sub>2</sub> nanoparticles exhibit a clean surface and presents an agglomeration containing both dark and bright spheres. Even at this low magnification, it is apparent that the bright parts of from spray pyrolysis a mixture of styrene and acetone, at high temperature is not particles surrounding the outer edge of these agglomerates are rather than WS<sub>2</sub>. The presence of particles surprising [21]. However, the critical temperature varies with different processing conditions.

As confirmed by the high resolution TEM image in fig.3. This layer has been observed to appear thicker on particles collected from different areas of the reaction tube and is considered due to the longer time exposed to the hot zone of the furnace. The coating layers are observed particularly lighter, compared to the darker through the layer is thinner in the top arrowed area than that in the bottom area [22,25].

## **Conclusions**

X-ray diffraction analysis indicated the formation of WS2 hexagonal with type of nanocrystal structures respectively. The transparent conducting oxide electrodes, and separated electrons could transfer from the conduction band of the tungsten disulphide compound semiconductors to respectively. Formation of the high quality in active layers with inorganic nanoparticles would improve the efficiencies of the solar cells.

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