

# A New Insight in the Physical and Photoelectrochemical Properties of Molybdenum Disulfide Alpha-Hematite Nanocomposite Films

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## Abstract

The alpha (a)-hematite  $(Fe_2O_3)$  as photoanode has been used for photoelectrochemical applications due to low bandgap, low cost, high chemical stability, nontoxicity, and abundance in nature. The doping with various transition metals, formation of nanostructured and nanocomposite of *a*-Fe<sub>2</sub>O<sub>3</sub> have been attempted to enrich the carrier mobility, surface kinetics and carrier diffusion properties. The manuscript is an attempt to improve the photoelectrochemical properties of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> by formation of nanocomposite with dichalcogenide (molybdenum disulfide (MoS<sub>2</sub>) nanomaterials. The nanocomposite of MoS<sub>2</sub>- $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> have been synthesized by varying the amount of MoS<sub>2</sub> in sol-gel synthesis process. The nanocomposite MoS<sub>2</sub>-a-Fe<sub>2</sub>O<sub>3</sub> materials were characterized using UV-visible, FTIR, SEM, X-ray diffraction, Raman and particle analyzer. The photoelectrochemical properties were investigated using cyclic voltammetry and chronoamperometry studies. The optical and structural properties of  $MoS_2$ -*a*-Fe<sub>2</sub>O<sub>3</sub> nanocomposite have been found to be dependent on MoS<sub>2</sub> doping. The band gap has shifted whereas; the structure is more prominent as flower-like morphology, which is a result of doping of MoS<sub>2</sub>. The photocurrent is more pronounced with and without light exposition to  $MoS_2$ -a-Fe<sub>2</sub>O<sub>3</sub> based electrode in photoelectrochemical cell. We have understood the photoelectrochemical water splitting using nanocomposite a-Fe<sub>2</sub>O<sub>3</sub>-MoS<sub>2</sub> through schematic representation based on experimental results. The enhanced photoelectrochemical properties of nanocomposite a-Fe<sub>2</sub>O<sub>3</sub>-MoS<sub>2</sub> films have been observed as compared to pristine a-Fe<sub>2</sub>O<sub>3</sub> and transition metal doped  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanostructured films.

#### **Keywords**

Water Splitting, Photoelectrochemical, Hematite (*a*-Fe<sub>2</sub>O<sub>3</sub>), MoS<sub>2</sub>, Nanocomposite

## **1. Introduction**

Photoelectrochemical cell (PEC) produces hydrogen through splitting the water using renewable sources (*i.e.*, the sun) [1] [2] [3]. Photoelectrochemical (PEC) cells have been used to convert solar energy to hydrogen gas by splitting water into hydrogen and oxygen, hence offering clean and renewable energy [4]. Moreover, photoelectrochemical (PEC) has attracted attention since Honda and Fujishima utilized the first application of titanium dioxide (TiO<sub>2</sub>) in 1972 [5]. Nevertheless, the large bandgap of  $TiO_2$  (3.1 - 3.3 eV) impedes the absorption of visible light, and limits the solar-to-hydrogen efficiency to 2.2% [5]. So, it is necessary to use material that has small bandgap and easy to harvest energy from sunlight (visible light 53%) [6]. Iron oxide, bismuth vanadate, tungsten oxide, and tantalum nitride are the examples of low band gap semiconducting materials [5]. The  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> is one of the most attractive photo-anode materials with efficiency of 16% to convert solar-to-hydrogen [5] [7]-[14]. The *a*-Fe<sub>2</sub>O<sub>3</sub> has been applied for photoelectrochemical applications due to low bandgap (2.1 - 2.2 eV), low cost, high chemical stability, nontoxicity, and abundance in nature [5]. However, a-Fe<sub>2</sub>O<sub>3</sub> also has several drawbacks such as shorter hole diffusion length, low conductivity, shorter life time of photoexcitation and deprived reaction kinetics of oxygen evolution in photoelectrochemical applications [15]. The doping with several metallic ions such as zinc [16], titanium [17] [18], molybdenum [19], aluminum [20], platinum [21], silicon [22] [23] [24], graphene [25] [26], and cadmium sulfide [27] have shown improved PEC performance. The zinc and aluminum doped  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> have shown enhanced photoelectrochemical properties compared to  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanostructures [28] [29] [30].

Recently, two-dimensional (2D) dichalcogenide material "molybdenum disulfide (MoS<sub>2</sub>)" with bandgap of 1.8 eV has been used as n-and p-types structures for photoelectrochemical studies [5]. The MoS<sub>2</sub> shows stimulating photocatalytic activity due to its bonding, chemical composition, doping, and nanoparticles growth on various film matrices, and has been used for hydrogen production in nanocluster structures [2] [31] [32] [33] [34]. Besides, MoS<sub>2</sub> has shown different applications in photocatalyst, phototransistors and sensors applications [5]. It is understood that MoS<sub>2</sub> could help to play an important role as the charge transfer with slow recombination of electron-hole pairs created due to photoenergy with the charge transfer rate between surface and electrons [35].

Under this work,  $MoS_2$  particles were used to promote electron transport properties of the *a*-Fe<sub>2</sub>O<sub>3</sub> nanomaterial by doping and homogenous structure due to  $MoS_2$ -*a*-Fe<sub>2</sub>O<sub>3</sub> nanomaterials. The doping of  $MoS_2$  particles varied by 0.1%, 0.2%, 0.5%, 1%, 2% and 5% in a-Fe<sub>2</sub>O<sub>3</sub>. The MoS<sub>2</sub>-a-Fe<sub>2</sub>O<sub>3</sub> nanomaterials were characterized using X-ray diffraction, SEM, FTIR, Raman spectroscopy, particle analyzer, and UV-vis techniques. The cyclic voltammetry (CV) and impedance measurements were utilized to understand the electrochemical electrode/electrolyte interface and photoelectrochemical properties of MoS<sub>2</sub>-a-Fe<sub>2</sub>O<sub>3</sub> based nanostructures for water splitting applications.

#### 2. Experimental Details

#### 2.1. Materials

The materials iron chloride (FeCl<sub>3</sub>), aluminum chloride (AlCl<sub>3</sub>), sodium hydroxide (NaOH), MoS<sub>2</sub>, and ammonium hydroxide (NH<sub>4</sub>OH) were purchased from Sigma-Aldrich. The fluorine tin oxide (FTO) coated glass with resistance of ~10  $\Omega$  was also procured from Sigma-Aldrich. The centrifuged containers were purchased to clean the synthesized nanomaterials from the solution.

#### 2.2. Experimental Procedure

a-Fe<sub>2</sub>O<sub>3</sub> and MoS<sub>2</sub>-a-Fe<sub>2</sub>O<sub>3</sub> were synthesized by a sol-gel technique as shown in Equation (1).

$$\operatorname{FeCl}_{3} \cdot 6\operatorname{H}_{2}O + \operatorname{MoS}_{2} \xrightarrow{} \operatorname{NaOH}(\operatorname{heat} 110^{\circ}C)/\operatorname{sonination}/\operatorname{drv}/\operatorname{heat}} \operatorname{MoS}_{2} \operatorname{-Fe}_{2}O_{3}$$
(1)

**Table 1** shows the amount of chemicals used for the synthesis of  $MoS_2$ -*a*-Fe<sub>2</sub>O<sub>3</sub>. Different concentrations of FeCl<sub>3</sub> with  $MoS_2$  were prepared in 500 ml round bottom flasks. NaOH solution was added to the resulting solution and stirred with a magnet for an hour. A condenser was connected to the round bottom flask, which allowed chemical reaction to proceed at 90°C - 100°C. The reaction was terminated after 24 hours, and the solution was cooled at room temperature. The synthesized material was separated using a centrifuge and continuous cleaning with water and initially left drying in room temperature. The synthesized materials containing different ratios of *a*-Fe<sub>2</sub>O<sub>3</sub> to MoS<sub>2</sub> in MoS<sub>2</sub>-*a*-Fe<sub>2</sub>O<sub>3</sub> were obtained. Figure 1 shows the photographs of the MoS<sub>2</sub>-*a*-Fe<sub>2</sub>O<sub>3</sub> materials synthesized using various percentage of MoS<sub>2</sub> to *a*-Fe<sub>2</sub>O<sub>3</sub>. The immediate doping of 0.1% MoS<sub>2</sub> changes the color of *a*-Fe<sub>2</sub>O<sub>3</sub>, whereas the dark red color can be visualized with the increase of MoS<sub>2</sub> percentage in *a*-Fe<sub>2</sub>O<sub>3</sub>. The MoS<sub>2</sub>-*a*-Fe<sub>2</sub>O<sub>3</sub> at various ratio were dried at various temperatures (100°C, 200°C, 300°C, 400°C,

<b>Table 1.</b> The amount of chemical used for synthesis of $MoS_2$ -composite <i>a</i> -hem	iatite.
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Chemicals	0.1% MoS <sub>2</sub> w.r. to FeCl <sub>3</sub>	0.2% MoS <sub>2</sub> w.r. to FeCl <sub>3</sub>	0.5% MoS <sub>2</sub> w.r. to FeCl <sub>3</sub>	1% MoS <sub>2</sub> w.r. to FeCl <sub>3</sub>	2% MoS <sub>2</sub> w.r. to FeCl <sub>3</sub>	$5\% \text{ MoS}_2$ w.r. to FeCl <sub>3</sub>
FeCl <sub>3</sub>	6.8 g	6.8 g	6.8 g	6.8 g	6.8 g	6.8 g
$MoS_2$	0.013 g	0.026 g	0.065 g	0.1296 g	0.2592 g	0.648 g
NaOH	4.8 g	4.8 g	4.8 g	4.8 g	4.8 g	4.8 g
C <sub>19</sub> H <sub>42</sub> BrN	0.5 g	0.5 g	0.5 g	0.5 g	0.5 g	0.5 g

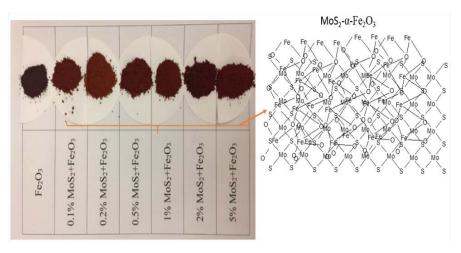


Figure 1. The synthesized *a*-hematite (*a*-Fe<sub>2</sub>O<sub>3</sub>) and MoS<sub>2</sub>-*a*-Fe<sub>2</sub>O<sub>3</sub> composite materials.

and 500°C). In each case, the temperature was maintained in furnace for one hour. The materials were collected by cooling at room temperature and stored in a tight bottle for characterization as well preparation of electrodes for electrochemical and photochemical studies.

## 2.3. The Film Formation of Substrate

The MoS<sub>2</sub>-a-Fe<sub>2</sub>O<sub>3</sub> was prepared at different concentrations by mixing with acetic acid to obtain the homogenous solution to cast film on various substrates. 500 mg of MoS<sub>2</sub>-*a*-Fe<sub>2</sub>O<sub>3</sub> (0.1%, 0.2%, 0.5%, 1%, 2%, and 5%) was grinded and then mixed into 10 ml acetic acid in a container, and left for 10 hours. Later, homogenous colloidal solution containing  $MoS_2$ - $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> with acetic acid were used to make films on quartz, silicon, and fluorine tin oxide (FTO) coated glass plates. The films were cured at different temperatures (100°C, 200°C, 300°C, 400°C, and 500°C) for an hour. It has been observed that the nanomaterials treated at 100°C to 200°C could still have the water molecules. However, the temperature at around 300°C allowed to have a solid material. The nanomaterials were further treated to 400°C and 500°C. The XRD, SEM, cyclic voltammetry, and UV-vis characterizations were performed in room temperature in cooled samples which were heated till 500°C of  $MoS_2$ -a-Fe<sub>2</sub>O<sub>3</sub> films. We had observed passivation, change in structure and morphology in the samples treated at 300°C, 400°C and 500°C. However, the results are presented for the samples treated at 500°C due to their enhance photocurrent.

## 3. Results and Discussions

#### 3.1. UV-Vis Studies

**Figure 2** shows the UV-vis spectra of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>, MoS<sub>2</sub> and  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>-MoS<sub>2</sub>-prepared at a different ratio of MoS<sub>2</sub> to  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>. An UV-Vis Spectrometer Jasco V-530 was used to measure the absorption spectra on various samples deposited on glass plates. Figure 2(a) shows the UV-vis absorption at around 550 nm for the

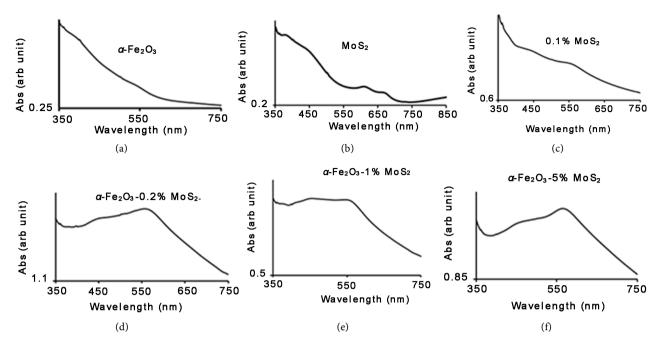


Figure 2. UV-vis absorption spectra of  $MoS_2$  with  $\alpha$ -hematite nanocomposite.

pristine *a*-Fe<sub>2</sub>O<sub>3</sub> similar to shown in literature. Figure 2(b) shows the characteristics absorption bands 388, 453, 618 and 679 nm for the MoS<sub>2</sub> nanomaterial film on glass plates. Figures 2(c)-(f) shows the UV-vis absorption spectra for MoS<sub>2</sub> doped in different percentage (0.1%, 0.2%, 1% and 5%) with *a*-Fe<sub>2</sub>O<sub>3</sub> nanomaterial. Figure 2(c) shows the absorption bands at 282, 454, 463 nm. Figure 2(d) shows the absorption bands at 446 and 565 nm. The distinct peaks can be seen at 382, 461 and 570 nm. Figure 2(e) absorbs the UV-vis band at 382, 456 and 559 nm whereas Figure 2(f) shows the absorption band at 382, 459 and 572 nm. There is a blue shift as an increase of MoS<sub>2</sub> in *a*-Fe<sub>2</sub>O<sub>3</sub> [36]. However, the band observed for 0.1% MoS<sub>2</sub> doping is shifted at 572 nm in 5% MoS2 doping in *a*-Fe<sub>2</sub>O<sub>3</sub> nanomaterial. Such results are consistent with the result shown of transition composite metal ions [37]. The UV-vis spectra of the composite hematite have been estimated to be 2.17 eV for the band at 572 nm.

#### 3.2. XRD Studies

The crystalline structure of  $MoS_2$ -*a*-Fe<sub>2</sub>O<sub>3</sub>was investigated by using Powder X-ray diffraction (XRD), model PANalytical X'Pert Pro MRD system, with Cu K *a* radiation (wavelength = 1.5442 Å) operated at 40 kV and 40 mA. **Figure 3** shows X-ray diffraction curves for different percentage of MoS<sub>2</sub> (0.1%, 0.2%, 0.5%, 1%, 2%, and 5%) to *a*-Fe<sub>2</sub>O<sub>3</sub>; *a*-Fe<sub>2</sub>O has polycrystalline structure as revealed from XRD pattern. The diffraction common peaks in MoS<sub>2</sub>-*a*-Fe<sub>2</sub>O<sub>3</sub> nanocomposite at different percentage of MoS<sub>2</sub> displays bands at 31.2°, 33.2°, 37.5°, 40.9°, 49.5°, 54.1°, 62.2°, and 64.2° which can be indexed to (012), (104), (110), (113), (024), (116), (214), and (300) for crystal planes of hexagonal iron oxide [8]. It is clear from strong and sharp diffraction peaks that Fe<sub>2</sub>O<sub>3</sub> is well crystallized

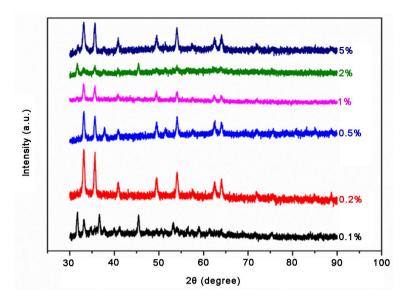


Figure 3. X-ray diffraction pattern of  $MoS_2$  with *a*-hematite nanocomposite.

in the synthesis process for all percentage of  $MoS_2$  in  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> [38]. The peak at 54.1° is due to the presence of  $MoS_2$  in  $MoS_2$ - $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>-structure.

#### 3.3. FTIR Studies

Perkin Elmer spectrum one was utilized to study FTIR spectroscopy of various samples of  $MoS_2$ -*a*-Fe<sub>2</sub>O<sub>3</sub>-nanocomposite. The  $MoS_2$ -*a*-Fe<sub>2</sub>O<sub>3</sub>-nanocomposite was mixed with KBr, the pellets were made using the hydraulic press, and the samples were measured using the transmission mode from 400 to 4000 cm<sup>-1</sup>. FTIR spectra of  $MoS_2$ -*a*-Fe<sub>2</sub>O<sub>3</sub> shows the change of percentage of  $MoS_2$  doping with *a*-Fe<sub>2</sub>O<sub>3</sub> with Curve 1% to 5%, Curve 2% to 0.2%, Curve 3% to 2%, Curve 4 to 1%, Curve 5% to 0.5%, and Curve 6% to 0.1% of  $MoS_2$  in  $MoS_2$ -*a*-Fe<sub>2</sub>O<sub>3</sub> in shown in **Figure 4**. The infrared bands of each  $MoS_2$  doping to *a*-Fe<sub>2</sub>O<sub>3</sub> are shown in **Table 2**.

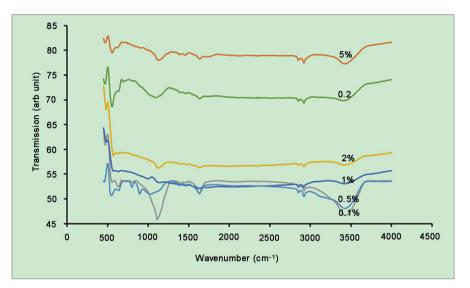
The hydroxyl (OH) groups in a-Fe<sub>2</sub>O<sub>3</sub> is related to infrared band at 3414 cm<sup>-1</sup>. The band at 1642 cm<sup>-1</sup> is due to v (OH) stretching. The band at 562 cm<sup>-1</sup> is due to Fe-O vibration mode in Fe<sub>2</sub>O<sub>3</sub>. The band at 620 - 654 and 474 - 512 are related to the lattice defects in Fe<sub>2</sub>O<sub>3</sub> [39] [40]. The infrared band at 474 - 512 cm<sup>-1</sup> is due to stretching vibration depicting the presence of MoS<sub>2</sub> in the MoS<sub>2</sub>-a-Fe<sub>2</sub>O<sub>3</sub> structure. The doping of 0.1% to 5% of MoS<sub>2</sub> shifts the infrared band from 512 cm<sup>-1</sup> to 474 cm<sup>-1</sup>. The band at 474 cm<sup>-1</sup> is the band observed for exfoliated MoS<sub>2</sub> nanosheets revealing that maximum doping in MoS<sub>2</sub>-a-Fe<sub>2</sub>O<sub>3</sub> structure [41].

#### 3.4. SEM Studies

The scanning electron microscopy (SEM) of various  $MoS_2$ -*a*-Fe<sub>2</sub>O<sub>3</sub> samples were measured using FE-SEM, S-800, Hitachi. Figure 5 shows SEM images of  $MoS_2$ -*a*-Fe<sub>2</sub>O<sub>3</sub> nanomaterials which consisted of different percentages from 0.1 to 5%  $MoS_2$  to Fe<sub>2</sub>O<sub>3</sub> in  $MoS_2$ -*a*-Fe<sub>2</sub>O<sub>3</sub>. SEM images reveals that the morphology

4. MoS <sub>2</sub>	Wavenumber (cm <sup>-1</sup> )
5%	474, 562, 620, 1136, 1193, 1472, 1642, 2858, 2924, 3436
2%	484, 562, 620, 1136, 1193, 1472, 1642, 2858, 2924, 3436
1%	474, 570, 640, 1006, 1134, 1388, 1470, 1670, 2854, 2924, 3436
0.5%	458, 554, 644, 802, 898, 1042, 1386, 1468, 1634, 2856, 2922, 3438,
0.1%	512, 522, 654, 802, 1114, 1396, 1434, 1666, 2836, 2952, 3448

**Table 2.** The infrared bands of each  $MoS_2$  doping to *a*-Fe<sub>2</sub>O<sub>3</sub>.

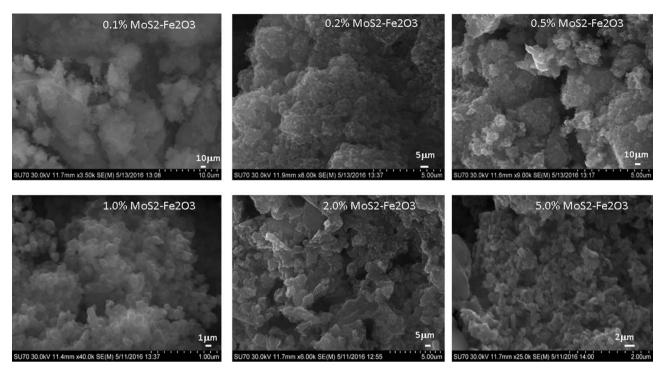


**Figure 4.** FTIR spectra of  $MoS_2$  with *a*-hematite nanocomposite. Each curve  $MoS_2$  doping with  $Fe_2O_3$  is given as: Curve  $1 = 5\% MoS_2$ , Curve  $2 = 0.2\% MoS_2$ -Fe<sub>2</sub>O<sub>3</sub>, and Curve  $3 = 2\% MoS_2$ -Fe<sub>2</sub>O<sub>3</sub>, Curve  $4 = 1\% MoS_2$ -Fe<sub>2</sub>O<sub>3</sub>, Curve  $5 = 0.5\% MoS_2$ -Fe<sub>2</sub>O<sub>3</sub> and Curve  $6 = 0.1\% MoS_2$ -Fe<sub>2</sub>O<sub>3</sub>.

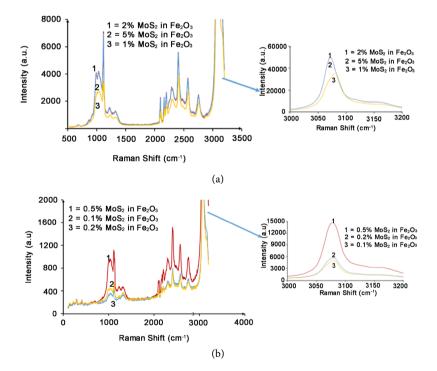
 $MoS_2-a-Fe_2O_3$  resembles blooming flower-like nanoparticles. The blooming flower-like morphology is a result of doping  $MoS_2$  with a-Fe<sub>2</sub>O<sub>3</sub> [42]. The images reveal that the size of the particle changes with the increase of  $MoS_2$  doping from 0.1% to 5% in  $MoS_2-a$ -Fe<sub>2</sub>O<sub>3</sub> nanomaterial. Besides, it is difficult to differentiate simple a-Fe<sub>2</sub>O<sub>3</sub> nanoparticles from  $MoS_2$  nanosheets; this shows a strong interface formation between Fe<sub>2</sub>O<sub>3</sub> and  $MoS_2in MoS_2-a$ -Fe<sub>2</sub>O<sub>3</sub> nanomaterial [43].

#### 3.5. Raman Spectroscopy

The Raman spectrum is measured which is also a rapid and nondestructive surface characterization technique to probe the vibrational properties of bonding of  $MoS_2$  to  $Fe_2O_3$  in  $MoS_2$ -*a*- $Fe_2O_3$  nanomaterial. **Figure 6** shows the Raman spectra of  $MoS_2$ -*a*- $Fe_2O_3$  film excited by 532 nm laser [44]. The Raman shift at 532 cm<sup>-1</sup> resonates with the electronic transition in ring structures for aromatic clustering processes in sp<sup>2</sup>-dominated particles. The shift associated at 374 and 417 cm<sup>-1</sup> are due to in-plane vibrational ( $E_{2g1}$ ) and the out-of-plane vibrational ( $A_{1g}$ ) modes. The enhanced  $MoS_2$  is indicative of energy difference between Raman shifts due to  $MoS_2$  content in  $MoS_2$ -*a*- $Fe_2O_3$  nanomaterial.



**Figure 5.** Scanning electron micrographs (SEM) of  $MoS_2$  with *a*-hematite nanocomposite. The percentage of  $MoS_2$  with *a*-hematite nanocomposite is shown in **Figure 5**.



**Figure 6.** Raman spectra of  $MoS_2$ - $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> film sample and ITO substrate as various percentage of  $MoS_2$  as shown in figures.

## **3.6. Particle Analysis**

The Zetasizer Nano particle analyzer range model was used to measure the average particle size of various  $MoS_2$ - $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> samples. Initially, the  $MoS_2$ - $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>

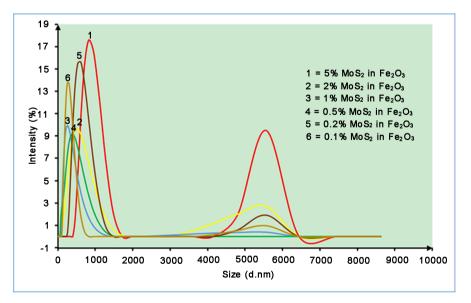
nanomaterial was dispersed in water and ultra-sonicated to have aggregated free colloidal sample. **Figure 7** shows the particle size of  $MoS_2$ -*a*-Fe<sub>2</sub>O<sub>3</sub> as a function of  $MoS_2$  doping in *a*-Fe<sub>2</sub>O<sub>3</sub>. The average particle size in liquid sample ranges from 459 nm (0.1%) to 825 nm for (5%) do pant of  $MoS_2$  respectively. Although these particles are small, there are few particles which are larger than 5 microns. These larger particles that can be detected through SEM measurement are a result of aggregation. The average size of particles is important for the fabrication of the electrodes from the particles. This information of nanomaterial dispersion of  $MoS_2$ -*a*-Fe<sub>2</sub>O<sub>3</sub> can be exploited for the electrode fabrication or other applications.

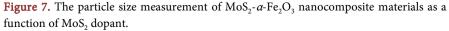
### **3.7. Electrochemical Studies**

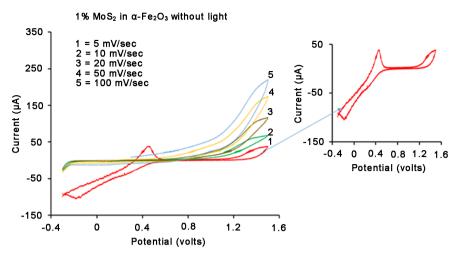
#### 3.7.1. Cyclic Voltammetry

The electrochemical measurements on various  $MoS_2$ -*a*-Fe<sub>2</sub>O<sub>3</sub> electrodes were measured from electrochemical workstation (Volta lab). The electrochemical set-up was adopted similar to our earlier studies on hybrid films [45] [46]. **Figure 8** shows the cyclic voltammetry (CV) of 1%  $MoS_2$ -*a*-Fe<sub>2</sub>O<sub>3</sub> in 1M NaOH as working electrode, platinum (Pt) as counter and Ag/AgCl as reference electrode in three electrodes based electrochemical cell. The continuous increase of CV current was observed with an increase in function of scan rate. The presence of  $MoS_2$  ions induces the electrochemical properties and 1.3 V can be seen as oxidation potential of water that is less than the Aluminum-doped from our previous studies [30].

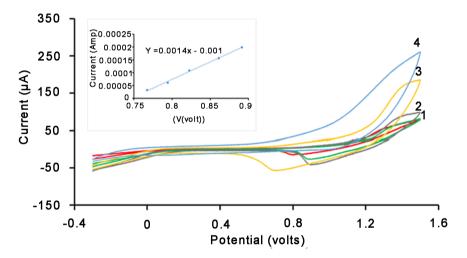
The CV is shown in **Figure 9** with application of light simulated for solar radiation. However, with the scan rate of 100 mV/sec, there was a maximum photocurrent absorbed for  $MoS_2$ -*a*-Fe<sub>2</sub>O<sub>3</sub> film. The diffusion coefficient was







**Figure 8.** The cyclic voltammetry of 1% MoS<sub>2</sub> with Fe<sub>2</sub>O<sub>3</sub> nanocomposite without light in 1 M NaOH in three electrodes where platinum as reference and Ag/AgCl as reference electrode.



**Figure 9.** The cyclic voltammetry of 1% MoS<sub>2</sub> with Fe<sub>2</sub>O<sub>3</sub> nanocomposite with light in 1 M NaOH in three electrodes where platinum as reference and Ag/AgCl as reference electrode.

calculated by using peak current for a reversible cyclic voltammetry is given by the Randles-Sevcik equation (Equation (2)).

$$I_{P} = (2.69 \times 10^{5}) n^{3/2} A C D^{1/2} v^{1/2}$$
<sup>(2)</sup>

where:

*n* = number of electrons

 $A = \text{electrode area} (\text{cm}^2)$ 

- $C = \text{concentration (mole/cm}^3)$
- D = diffusion coefficient (cm<sup>2</sup>/s)
- v = potential scan rate (V/s)

$$I_p$$
 = current.

The diffusion coefficient has been estimated to be  $0.24 \times 10^{-16}$  cm<sup>2</sup>/s.

#### 3.7.2. Chronoamperometry Study

We made an attempt to deposit  $MoS_2-a-Fe_2O_3$  film on ITO coated glass substrates uniformly using the homogenous paste obtained using acetic acid. The thickness of  $MoS_2-a-Fe_2O_3$  was around 30 µm. Figure 10(a) & Figure 10(b) shows the chronoamperometry study of two electrodes cell consisting of  $MoS_2-a-Fe_2O_3$ film as working and steel as counter in various concentrations (0.01 0.1, 1 M) of NaOH based electrolyte. The potential from -1000 mV to 1500 mV was applied, and the chronoamperometry photocurrent was studied. Figure 10(a) & Figure 10(b) shows the chronoamperometry photocurrent plot with t<sup>-1/2</sup> for oxidation and reduction processes for  $MoS_2-a-Fe_2O_3$  film. The rise of photocurrent showed a linear relationship with t<sup>-1/2</sup> due to excitation of light. The current transient was different from the excitation of light. The diffusion-controlled photocurrent is calculated using Cottrell equation in Equation (3) [47] [48] [49].

$$i = \left[ nFAD^{\frac{1}{2}}C \right] / \left[ \pi t^{\frac{1}{2}} \right]$$
(3)

where:

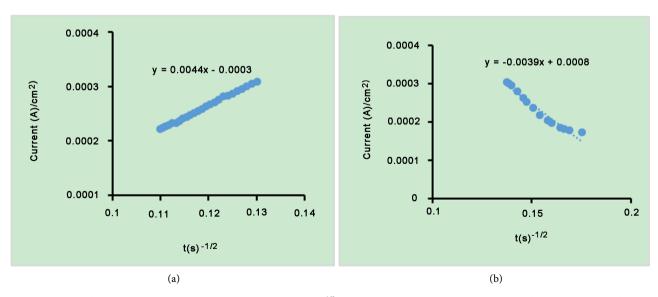
n = the electron participating in the reaction

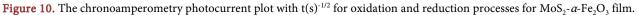
- F = the faraday constant
- A = the area of the electrode
- *i* = the transient current
- D = the diffusion coefficient
- C = the concentration of the electrolyte

The D has been estimated to be  $1.057 \times 10^{-14} \text{ cm}^2/\text{sec.}$ 

#### 3.7.3. Impedance Study

Figure 11(a) and Figure 11(b) show the Nyquist plot in 1 M NaOH without and with light irradiation in  $MoS_2$ - $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> film in a photoelectrochemical





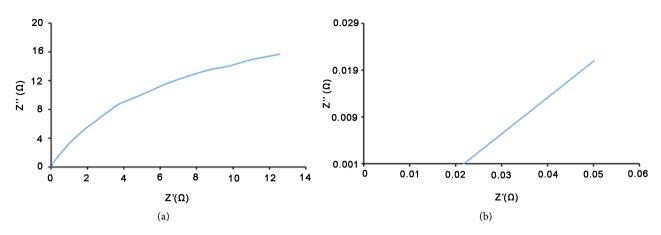


Figure 11. Nyquist plot of MoS<sub>2</sub>-*a*-Fe<sub>2</sub>O<sub>3</sub> film in 1 M HCl in photoelectrochemical cell without (a) and with (b) light irradiation.

set-up. The change in the impedance value has been observed for real and imaginary without light irradiation as shown in Figure 11(a) and Figure 11(b). The photocurrent is able to make process more conducting in presence of light.

#### 3.7.4. Half Sweep Potential

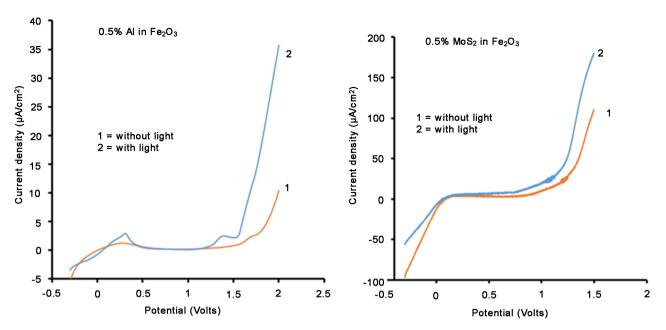
**Figure 12** shows the half sweep potential with and without light for both aluminum doped-*a*-Fe<sub>2</sub>O<sub>3</sub>and MoS<sub>2</sub>-*a*-Fe<sub>2</sub>O<sub>3</sub>. Our previous study on aluminum doping has shown the photocurrent to be 35  $\mu$ A whereas for the same type of electrode for MoS<sub>2</sub>-*a*-Fe<sub>2</sub>O<sub>3</sub> showed the current to be 150  $\mu$ A. Schottky type current-voltage is experienced for both aluminum doped as well as MoS<sub>2</sub>-*a*-Fe<sub>2</sub>O<sub>3</sub> based electrode in photoelectrochemical cell.

#### 3.8. Schematic of MoS<sub>2</sub>-α-Fe<sub>2</sub>O<sub>3</sub> Reaction Process

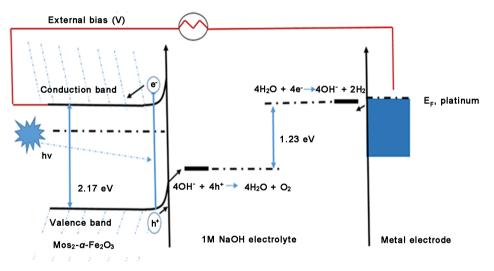
A schematic was drawn to understand the effect of  $MoS_2$  with a-Fe<sub>2</sub>O<sub>3</sub>. The schematic of hydrogen production using  $MoS_2$ -composite a-Fe<sub>2</sub>O<sub>3</sub> photocatalyst in 1 M NaOH is shown in **Figure 13**. The bandgap of  $MoS_2$  varies from 1.2 - 1.9 eV, whereas the band gap of a-Fe<sub>2</sub>O<sub>3</sub> is 2.1 eV. It was estimated the bandgap of  $MoS_2$ -composite a-Fe<sub>2</sub>O<sub>3</sub> in range of 1.94 to 2.40 eV based on UV-vis measurements, which is well in the region of visible light.  $MoS_2$  doping also increased the conductivity of the samples. The schematic in **Figure 12** shows the photogenerated electrons from conduction band of  $MoS_2$  is transferred to conduction band (CB) of a-Fe<sub>2</sub>O<sub>3</sub> whereas holes from  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> e are transferred to valence band (VB) of  $MoS_2$ . This enhances the photocatalytic activity of  $MoS_2$  composite with a-Fe<sub>2</sub>O<sub>3</sub> in  $MoS_2$ -a-Fe<sub>2</sub>O<sub>3</sub> nanomaterial-based electrode.

#### 4. Conclusions

The synthesized  $MoS_2-a$ - $Fe_2O_3$  observed the shift in the band gap to 2.17 eV with  $MoS_2$  doping. There is a marked change in the band due to  $MoS_2$  doping in a- $Fe_2O_3$ . The increase of  $MoS_2$  dominated the structure as marked from SEM measurements. The photocurrent can be clearly distinguishable with and without light irradiation through various electrochemical studies on  $MoS_2-a$ - $Fe_2O_3$ .



**Figure 12.** Half sweep potential with and without light for Aluminum doped- $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> and MoS<sub>2</sub>- $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> film with and without light exposure.



**Figure 13.** The schematic of hydrogen production using  $MoS_2$ -composite *a*-Fe<sub>2</sub>O<sub>3</sub> photocatalyst in 1 M NaOH.

nanomaterial. The enhanced photocurrent is observed with  $MoS_2$  doping in  $MoS_2$ -*a*-Fe<sub>2</sub>O<sub>3</sub> nanomaterial. The  $MoS_2$ -*a*-Fe<sub>2</sub>O<sub>3</sub> nanomaterial thin film has the potential to produce hydrogen using a PEC water splitting process that could have renewable energy applications. Our future work is based on the use of  $MoS_2$ -*a*-Fe<sub>2</sub>O<sub>3</sub> as n-type in p-n photoelectrochemical studies for efficient water splitting applications.

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