# Synthesis of Tungsten sulphide(WS<sub>2</sub>)-rGO nanofibers for efficient water treatment

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The nanofibers of Tungsten sulphide and rGO (WS<sub>2</sub>-rGO) are prepared by electrospinning process. The structural and morphological features of nanofibers are examined by SEM. Material analysis is done by FTIR. The nanofibers are used to prepare membranes for water filtration in various aspects and levels. First of all Tungsten sulphide (WS<sub>2</sub>) powder 3g is dissolved in 100 ml of Dimethylformamide (DMF) for a complete and quick-dissolving solution is placed on a hot magnetic stirrer and solution is heated at 100 °C. Next, the solution is stirred for an hour. The final solution is then filled in a syringe fitted on an infusion pump which is set up by electrospinning apparatus. The Nanofibers were synthesized successfully which can serve as membranes for water treatment.

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# 1. Introduction

The definition of Nanotechnology is the research and technology expansion at the length gauge of about 1- 200 nm range, to deliver a central comprehensive empathetic of wonders and materials at the nano-scale and to generate and use assemblies, devices, and systems that have innovative properties and occupations because of their small and/or intermediate size [1]. Electrospinning is a process that makes nanofibers by applying an electrical field to the polymer solution. This method is the simplest of the techniques and contains three main parts: the first part is a syringe with a steel needle that will hold the polymer solutions, the second part is a high voltage power source which creates an electrical field between the polymer solution and collector. and, the third part is an aluminum plate as a collector for nanofibers. FTIR technique is used to find an infrared spectrum of absorption and emission in gases, liquids and solids [2]. FTIR spectrometer gathers information at a high spectral resolution as compared to a very wide spectrum [3]. This gives a very high advantage of FTIR spectrum over a dispersive spectrometer. The surface morphology of nanofibers was observed by using a nova nanometer scanning electron microscope. After the preparation of nanofiber of rGO-WS2 composite, its application as filtration membrane for tap water was analyzed. For this purpose separated membrane was put in the funnel. The tap water from Momin pura industrial area was collected. The tap water source was 400 ft from ground level. Sample temperature and pH were checked on the spot. Then the sample was sealed with tape to make it air-tight and to reduce microbial activity. After that, it was shifted to the laboratory for filtration purpose [4]

Filtration was done under very controlled conditions at standard atmospheric pressure and temperature. After putting the membrane on the funnel, water grabbed from the industrial area poured on the membrane settled on the funnel with a dropper. About 20 ml water was filtered with help of a dropper as the membrane was fragile this was completed slowly. The sample was analyzed by the authorized method and the result of the samples shows some difference in values of treated and untreated samples this membrane has shown significant resistance against bacteria

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found in tap water. Furthermore, it is also seen that water flux can be maintained by increasing some more adhesives to the main solution.

# 2. Experimental work

#### 2.1. Materials and chemicals

Tungsten disulfide (WS<sub>2</sub>), PVA (polyvinyl alcohol) 90% mol wt 30,000 to 70,000, Polyvinylidene fluoride (PVDF) Mw = 180,000, (NMP) N-Methyl-2-pyrrolidone, Ethanol. Carbon, graphite powder, sulphuric acid, hydrofluoric acid (HF), Potassium Permanganate(KMnO4), Phosphoric acid (H<sub>3</sub>PO<sub>4</sub>), Hydrogen Peroxide (H<sub>2</sub>O<sub>2</sub>), Hydrochloric acid (HCl), deionized water.

#### 2.2. Synthesis of graphene oxide

Graphene oxide was prepared by using the improved Hummer's (Tour's) method. Graphite powder was first purified by using hydrofluoric acid as it contains some impurities between its planes [5-6]. Took 10 grams of graphite powder and 200mL of HF having ratio1:10. Pour HF on graphite powder with vigorous stirring for one hour to fully remove the impurities. First of all, purified graphite powder (6g, 1 wt. equiv.) and KMnO<sub>4</sub> (36g, 6 wt. equiv.) were added to the glass beaker and mixed. 9:1 mixture of H<sub>2</sub>SO<sub>4</sub> and H<sub>3</sub>PO<sub>4</sub> (720:80 mL) was added with vigorous stirring continuously for 12 hours. After continuous stirring color changes to dark brown. Then pour this mixture on 1400mL Deionized water ice and the color changes to brownish-purple. Further 5mL of H<sub>2</sub>O<sub>2</sub> is added which reduces the manganese heptoxide and stops the further oxidization and color changes to milky yellow. Following this process, a mixture of DI waterethanol and HCl each 300mL was added to remove the K<sup>+</sup>, Mn<sup>+2</sup> and SO<sub>4</sub><sup>-2</sup> ions and further wash with DI water until pH 7 was attained. The obtained mixture was shifted to petri dishes and dried at 40<sup>o</sup>C in the oven [7].

The obtained graphite oxide from the previous step was then dispersed in DI water and DMF separately having a ratio (1mg: 1mL) in an ultrasonicated bath for two hours to separate the graphite oxide layers [8]. This separation was done by increasing the d-spacing between the layers of graphite oxide as there are weak forces present between these layers. Further, the obtained dispersion was centrifuged at 6000 rpm for a half-hour to remove the unreacted graphite powder. The surfactant was removed which gave the dark brown exfoliated GO.

#### 2.3. Synthesis of WS<sub>2</sub>-rGO composite

 $WS_2$ -rGO composite was prepared by hydrothermal method.3g of  $WS_2$  was dissolved in 100mL of DMF and 40mL of rGO dispersion in rGO was added to it and stirred for one hour to intercalate the tungsten ions between the layers of rGO. Then the solution was set aside for 24 hours to check the stability of the solution. After 24 hours it was stable and the solute was not settled down which shows the stability [9]. The obtained material was washed with ethanol and deionized water multiple times and dried at 80<sup>o</sup>C overnight. This intermediate product was WS<sub>2</sub>-rGO composite. Then the obtained powder was sintered at 200<sup>o</sup> C for six hours termed as WS<sub>2</sub>-rGO composite.

#### 2.4. Synthesis of rGO-WS<sub>2</sub> nanofibers

 $WS_2$ - rGO composite nanofibers were prepared by electrospinning technique. Then few drops of 0.5 molar PVA solution were added to the solution as a binder to set the viscosity and then stirred for six hours to get a homogenous solution for electrospinning. Electrospinning was done having 15cm distance between needle tip and collector. The potential difference was maintained at 24 kV by controlling flow rate 1mL/h. Sample collected on aluminum foil was sintered at 250<sup>o</sup>C for seven hours which is designated as WS<sub>2</sub>-rGO composite nanofiber [10].

#### 2.5. Collection of sintering nanofibers

Nanofibers are deposited on an aluminum foil are collected in the form of a little cloudy cloth. Which is then stored in the glass petri dish for further process. First of all nanofibers were sintered at  $250^{9}$ C [11]. Sintering is important because nanofibers contain a lot of polymers and DMF contents which are required to be removed from our fibers and the material that has been used must be thermally decomposed to get the pure metal whose nanofibers are required.

# 3. Results and discussion

#### 3.1. FTIR spectroscopy result of WS<sub>2</sub>-rGO nanofibers

FTIR spectrometer gathers information at a high spectral resolution as compared to a very wide spectrum. This gives a very high advantage of FTIR spectrum over a dispersive spectrometer. FTIR spectroscopy was performed having a range of 4000cm-1 to 650cm-1. Figure 1 shows the FTIR spectra of rGO and WS<sub>2</sub> having two major peaks at 1628 cm<sup>-1</sup> and 634 cm<sup>-1</sup> respectively.[12]

FTIR spectroscopy is a very little instinctive approach to abstract similar data. As opposed to shining a monochromatic light ray (as this technique uses a monochromatic wavelength) for example, this strategy sparkles a ray containing various frequencies of visible light without any delay and measures the sum of those rays which are absorbed by the sample as shown in Figure 1. Then the ray is adjusted to have an alternate blend of frequencies, which gives subsequent information [13]. This procedure is quickly rehashed many times over a brief span range. A short time later, a PC collects this information and process it back to gives information about the spectrum at each wavelength [14].



Fig. 1. FTIR results of WS<sub>2</sub> and rGO.

# 3.2. Scanning Electron Microscopy

The surface morphology of nanofibers was observed by using a nova nanometer scanning electron microscope. Nanofibers which were prepared and collected on aluminum foil were sintered at  $250^{\circ}$ C for seven hours. Sintering improved its surface morphology and sharpened its diameter up to few nanometers [15]. No surface irregularity is seen. A high voltage and fine collection of nanofibers improved their surface texture as shown in Fig. 2.

Polyvinyl alcohol which is used as polymer played a very important role in the fine fabrication of delegate nanofibers. Although that polymer is later removed from principle metal by thermal decomposition in a furnace at  $350^{\circ}$ C [16].



Fig. 2. SEM images of  $WS_2$ -rGO nano fibers at (a)  $5\mu m$  (b)  $10\mu m$  (c)  $2\mu m$ .

# 4. Application

# 4.1. Preparation of membrane

Nanofiber generated after then used to prepare filter water purification. For the preparation of membrane after synthesis, it is removed from aluminum foil then it is taken for drying purpose. Considering the fragile behavior of membranes all the process was done in a very controlled environment.

# 4.2 Separation of membrane

Firstly nanofiber sheet is removed from the substrate which is aluminum foil after that is rinsed with DI water then kept in 0.6 M NaCl solution for 12h for preservation purpose [17]. The membrane samples in the filtered cells were pressurized with DI water at a pressure of 10 bar for about 2 h to make sure that the membrane is stable or not before further test. The tested membrane was washed under a vessel with DI water for about 5 min to remove the fouling impurity molecules which were placed on the surface of the membrane and the water flux was another time monitored under the same pressure.

### 4.3. Sample preparation

Two samples were prepared before and after the treatment of water and dispatched to the water testing laboratory for characterization of different parameters which will be discussed in the next chapter [18]. These parameters will show the difference of results showing the ability of membranes to treat water at various levels. Fig. 3 shows the samples carried out for testing.



Fig. 3. (a)Untreated and (b) treated water samples.

#### 4.4. Laboratory results

The samples were analyzed by the authorized method and the result of samples shows some difference in values of treated and untreated samples. So the membrane has shown significant resistance against bacteria found in tap water, as also reported in [19]. Furthermore, it is also seen that water flux can be maintained by increasing some more adhesives to the main solution [20-22]. Table 1 and Table 2 show the results of treated and untreated samples.

Sr. No	Parameter	Unit	Result	PEQS
1	Turbidity	NTU	0	<5
2	Total hardness	mg/l	110	<500
	CaCO <sub>3</sub>			
3	Nitrogen	mg/l	0.7	≤50
	(Nitrate)			
4	Nitrogen	mg/l	BDL	≤3
	(Nitrite)			
5	Boron	mg/l	BDL	0.3
6	Chloride	mg/l	35	<250
7	Fluoride	mg/l	0.2	<1.5
8	Zinc	mg/l	0.4	5
9	Arsenic	mg/l	BDL	≤0.05
10	Manganese	mg/l	0.02	≤0.5
11	Total Coliform	Number/100	130	0
		ml		
	E Coli	Number/100	100	0
		ml		

Table 1. Tap water analysis results (before treatment).

\*BDL = Below detection level

The bacterial growth of water samples before and after treatment is shown in the bar chart 1.



Chart 1. Bacterial growth of water samples before and after treatment.

Sr.	Parameter	Unit	Result	PEQS
No				
1	Turbidity	NTU	0	<5
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5	Boron	mg/l	BDL	0.3
6	Chloride	mg/l	35	<250
7	Fluoride	mg/l	0.2	<1.5
8	Zinc	mg/l	0.4	5
9	Arsenic	mg/l	BDL	≤0.05
10	Manganese	mg/l	0.02	≤0.5
11	Total Coliform	Number/100	0	0
		ml		
	E Coli	Number/100	0	0
		ml		

Table 2. Tap water analysis results (After treatment).

\*BDL = Below detection level

# 4. Conclusions

This article gives a view of the use of electrospun tungsten sulfide nanofibers and their related composites as modern materials terminals for high productivity and capacity, which doesn't just report the advancement of the structure and manufacture of different one-dimensional nanostructures. However furthermore outlines their noteworthy applications in nano filtration membranes.

This paper presents preparation of nanofibers of Tungsten sulphide-rGO by electrospinning process and then a membrane has been prepared from these nanofibers. The preparation of nanofibers and membranes played an important role in increasing the efficiency of nano filetration. The experiment provides an important platform to use different metals for the synthesis of more efficient membranes for water treatment and filtration. The composite nanofiber of tungsten sulphide has high porosity and good flux rate. The Tungsten Disulphide is highly recommended for the treatment of heavy metals found in industrial effluents. These nanofiber membranes are efficient and are cheap to prepare for water and wastewater treatment.

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