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# Nanofibers synthesised from Jawar stem (Agro-Waste) for removal of Cr (VI) from aqueous solution

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## ABSTRACT

Hexavalent chromium (VI) is a toxic form of chromium metal generally used in many industrial applications for its anticorrosive properties. Cr (VI) has been deemed to be a class-A human carcinogen. Among all the techniques available, adsorption is one of the most suitable techniques. The efficiency of the process depends on the adsorption medium. Although there are several adsorbents available, there is still huge scope to develop a number of adsorption media for the benefit of the common people. In this study, nanomaterial synthesised from Jawar stem (Agro-Waste) has been used as an adsorbent for removal of Cr (VI) from aqueous solution. The Jawar stem nanofibers have been synthesised using chemical vapour deposition technique (CVD). The characterisation of adsorbent was carried out using various analytical techniques like XRD, BET, SEM, TGA and FTIR. The adsorption capacity of Jawar stem nanofibers was found to be  $76.10 \text{ m}^2 \text{ gm}^{-1}$ . The removal of hexavalent chromium was studied as a function of contact time, dosage of adsorbent and initial concentration of Chromium (VI). The applicability of Langmuir and Freundlich isotherms was tested. Freundlich isotherm was found to be most suitable for all adsorbent dosages.

## ARTICLE HISTORY

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## KEYWORDS

Jawar stem nanofibers;  
Agro-Waste; hexavalent chromium; synthesis of carbon nanofibers

## 1. Introduction

Water, the most vital resource for all kinds of life on this planet, is adversely affected by various human activities. Water pollution by heavy metals is now a unique challenge to the environmentalists in recent years. The incredible use of heavy metals over the past few decades has inevitably resulted in an increased influx of metallic substances in aquatic environment. Chromium (Cr) in particular has received a great deal of attention due to its perilous nature. Chromium is a natural metal, commonly found in wastewaters, which originates from electroplating, textile, paint, tannery and pigment industries [1]. Chromium generally exists in two forms of wastewater: trivalent Cr (III) and hexavalent Cr (VI). Cr (VI) is more toxic than Cr (III). Cr (VI) is highly toxic, carcinogenic, and mutagenic to most of the living organisms when its concentration level is higher than 0.05 ppm and extremely mobile than Cr (III). Therefore, there is a great importance to remove Cr (VI)

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from wastewaters to prevent the deleterious impact of the Cr (VI) on the human health [2] and [3]. Several methods, such as membrane filtration [4], photocatalytic degradation [5], electrochemical methods [6], chemical precipitation [7], adsorption [8] and ion exchange [9], have been used for removal of Cr (VI) from industrially polluted wastewaters. However, most of these techniques have several limitations and drawbacks, they require high energy or massive use of reducing agents, and they are not widely used. In particular, adsorption is considered to be simple, economical and remains one of the most attractive approaches for treating Cr (VI).

The efficiency of adsorption process depends on the adsorption media; although there are several adsorbents available, there is still huge scope for adsorption media for the benefit of common people [10]. [11] used polypyrrole polyaniline (PPy-PANI) nanofiber for removal of Cr (VI). The study revealed that PPy-PANI nanofibers have a maximum adsorption capacity of 227 mg/g of adsorbent for removal of Cr (VI). [12] have used porous magnetite nanoparticles prepared from titanium residue for removal of Cr (VI) from wastewater. The study revealed that porous magnetite nanoparticles have promising applications for removal of Cr (VI) from wastewater with rapid adsorption kinetics and good adsorption capacity. [13] used sodium dodecyl sulphate coated  $\text{Fe}_3\text{O}_4$  nanoparticles (SDS -  $\text{Fe}_3\text{O}_4$ ) for removal of Cu (II), Ni (II) and Zn (II) from water and wastewater samples. The study showed maximum absorption capacity for Cu (II), Ni (II) and Zn (II) ions were 24.3, 41.2 and 59.2  $\text{mg}\cdot\text{g}^{-1}$ , respectively. [14] used Cysteine-modified polymer nanofibers for removal of Cr (III) from tannery wastewater samples. The study revealed a maximum removal of 1.75 gm of Cr (III)/gram of polymeric matter. [15] used polypyrrole of sepiolite nanofiber for removal of Cr (VI) from aqueous solution. The study revealed that sepiolite fibres have a maximum absorption capacity of 302 mg for removal of Cr (VI). [16] used magnetic multi-walled carbon nanotubes for removal of Cr (VI). The study shows that the efficiency of Cr (VI) increases with increase in adsorption dosage but the equilibrium adsorption capacity decreases significantly.

Many studies have been conducted for removal of Cr (VI) from water and wastewater. [17] used carbon nanotubes, [18] used oil palm kernel-based powdered activated carbon nanofibers, [19] use silver-yttrium oxide nanocomposites (SYON's) and [20] used graphene oxide composites.

In this work, nanofibers synthesised from Jawar stem (Agro-Waste) have been used for removal of Cr (VI) from aqueous solution. The study was conducted by varying dosage of adsorbent, contact time and initial concentration of Cr (VI). The objectives addressed in the present study include the following:

- Synthesise and characterise Jawar stem (Agro-Waste) nanofibers.
- Performance assessment of Jawar stem nanofibers for removal of Cr (VI) with reference to influencing parameters
- Determination of adsorbent dosage for varied concentration of Cr (VI).
- Determination of applicable isotherm model and constants.

  
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## 2. Materials and methods

### 2.1. Synthesis of carbon nanofibers by using CVD technique

Chemical Vapour Deposition (CVD) technique has been recognised as a versatile and robust method to synthesise carbon nanofibers (CNFs) on different substrates. Considering the variety of applications in which Carbon Nano Materials (CNMs) can be exploited due to their outstanding mechanical, optical, electrical and electrochemical properties, remarkable attention has been paid to CVD as a facile and reliable synthesis method for this material. Basically, CVD uses a controlled atmosphere containing a carbonaceous species at a suitable temperature to decompose the carbon precursor on a catalytic surface and subsequently, by supersaturating the catalyst particle, makes the extra carbon to precipitate in a certain crystalline form, namely graphite.

#### 2.1.1. Experimental procedure for synthesis of CNFs

**Materials** – In this study, Jawar stem, which is an agro-based waste material, has been used for synthesis of carbon nanofibers (CNFs).

**Carrier Gas** – Argon has been used as a carrier gas.

**CVD-Air atmospheric pressure (Pyrolysis set-up)** – Jawar stems were washed, dried and then cut into to small pieces. These small pieces are burnt in a Chemical Vapour Deposition (CVD) machine at a temperature of 800–850°C to carry out the pyrolysis process. Pyrolysis was carried out for 1–2 h, and thereafter, the furnace was cooled to room temperature, during this process, carbon nanofibers (CNFs) were deposited in the quartz boat. Figure 1 shows the components of CVD machine.

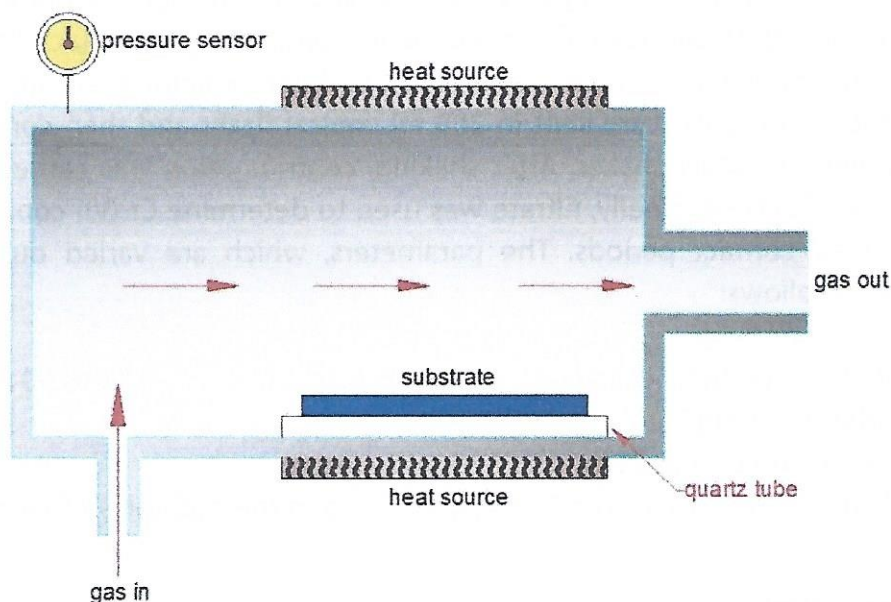


Figure 1. CVD reactor used for pyrolysis of Jawar stem.

  
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## 2.2. Preparation of synthetic chromium solution

All chemicals of AR grade were used in the study. Hexavalent chromium [Cr (VI)] reacts with diphenylcarbazide ( $C_{13}H_{14}N_4O$ ) under acidic condition to form a red-violet soluble complex. Traces of chromium have been estimated by spectrophotometry using this reagent. Lower valent Cr was oxidised to Cr (VI) by  $KMnO_4$ . Iron, copper and nickel, which may also be present in water, will interfere with this method and were removed from the sample by peroxidation treatment.

### 2.2.1. Reagents used

*Sulphuric acid (1:1) solution:* Add with cooling 50 ml conc.  $H_2SO_4$  to 50 ml water.

*Diphenylcarbazide reagent:* Dissolve 0.2 reagents in 100 ml of 95% ethyl alcohol. Add to this solution and mix well.

*Potassium permanganate solution (0.002 M):* Dissolve 0.0316 g solid in 100 mL water

*Sodium Azide:* 0.5% solution in water.

*Standard  $K_2Cr_2O_7$  solution:* Dissolve 0.3535 g  $K_2Cr_2O_7$  (AR) in water and make up the solution to 100 ml. Pipette 10 ml of this and dilute to 100 ml. Pipette 5 ml of the second solution and make it up to 250 ml. This diluted solution contains 1 ppm chromium (VI) [21].

## 2.3. Batch adsorption experiments

Laboratory studies on the removal of Cr (VI) from aqueous solution were conducted using nanofibers synthesised from Jawar stem (Agro-Waste). The adsorption experiments were designed to study the effect of various influencing parameters, such as contact time, dosage of adsorbent and initial chromium concentration. In these experimentations, the chromium solution and adsorbents were kept in 250 ml conical flasks and then continuous shaking was carried on rotary shaker. After shaking, centrifugation was carried at 12,000 rpm to separate adsorbent. Finally, filtrate was used to determine Cr (VI) concentration at predetermined contact periods. The parameters, which are varied during experimentations, are as follows:

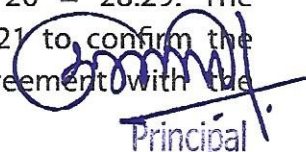
- (1) The concentration of synthetic chromium solution varied from 10 mg/lit to 50 mg/lit in the multiples of 10 mg/L.
- (2) The contact period varied from 20 min to 180 min.
- (3) The dosage of adsorbent varied from 20 mg to 100 mg in the multiples of 20 mg.

## 3. Results and discussions

### 3.1. Characterisation of carbon nanofibers synthesised from Jawar stem

#### 3.1.1. X-Ray diffraction (XRD) analysis

The XRD pattern of carbon nanofibers synthesised from Jawar stem (Agro-Waste) is shown in Figure 2. Carbon nanofibers showed peaks at  $2\theta$  value of 24.19, 28.29, 30.04, 31.29 and 40.49. The maximum peak was observed at  $2\theta = 28.29$ . The obtained XRD-pattern was coordinated with JCPDS file no. 75-1621 to confirm the presence of carbon nanofibers. The observed values are in agreement with the



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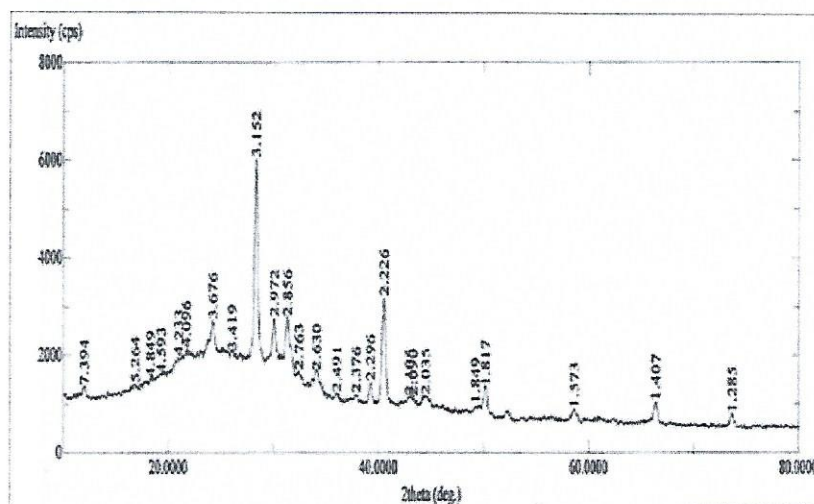


Figure 2. XRD pattern of carbon nanofibers synthesised from Jawar stem.

standard JCPDS file number 75–1621. The size of nanofiber was calculated using Debye-Scherrer formula and was found to be 43 nm. The X-Ray wavelength ( $\lambda$ ) 1.49 Å was taken for diffraction analysis.

### 3.1.2. Surface area analysis of Jawar stem CNFs


BET surface area analysis was used to find the surface area of nanofibers synthesised from Jawar stem. The surface area was found to be 76.10 m<sup>2</sup>/g; this large surface area confirms the availability of more active sites for adsorption. The surface area of other nanomaterials reported in the literature are manganese oxide 14.6 m<sup>2</sup>/g [22], 10 m<sup>2</sup>/g polyacrylonitrile (PAN)/ferrous chloride (Fe Cl<sub>2</sub>) composite [23] and 15.3 m<sup>2</sup>/g for montmorillonite-supported magnetite nanoparticles [24]. The obtained surface area is relatively higher than that of nanomaterials used in the literature. This increased surface area is one of the reasons for the increase in removal efficiency of metal ions.

### 3.1.3. Scanning electron microscopy (SEM)

A typical scanning electron microscopy (SEM) image of carbon nanofibers (CNFs) synthesised from Jawar stem is shown in Figure 3. The average diameter of carbon nanofibers (CNFs) has been found to be less than 100 nm.

### 3.1.4. Thermogravimetric analysis (TGA)

Thermal stability of carbon nanofibers synthesised from Jawar stem was assessed by TGA analysis by varying the temperature from 20°C to 900°C. Figure 4 shows three different slopes of weight reduction. The first-stage reduction was observed for an increase in temperature from 25°C to 220°C. The weight loss of 14.46% was observed in the first stage, and it is attributed to moisture removal. In the second-stage loss registered at 220°C to 650°C with marginal weight reduction of 0.82%. This indicates its strong stability for variation in temperature from 220°C to 650°C. The third stage registered at 650°C to 900°C where loss of weight is 35.23%. This major loss is attributed to degradation of the material.

  
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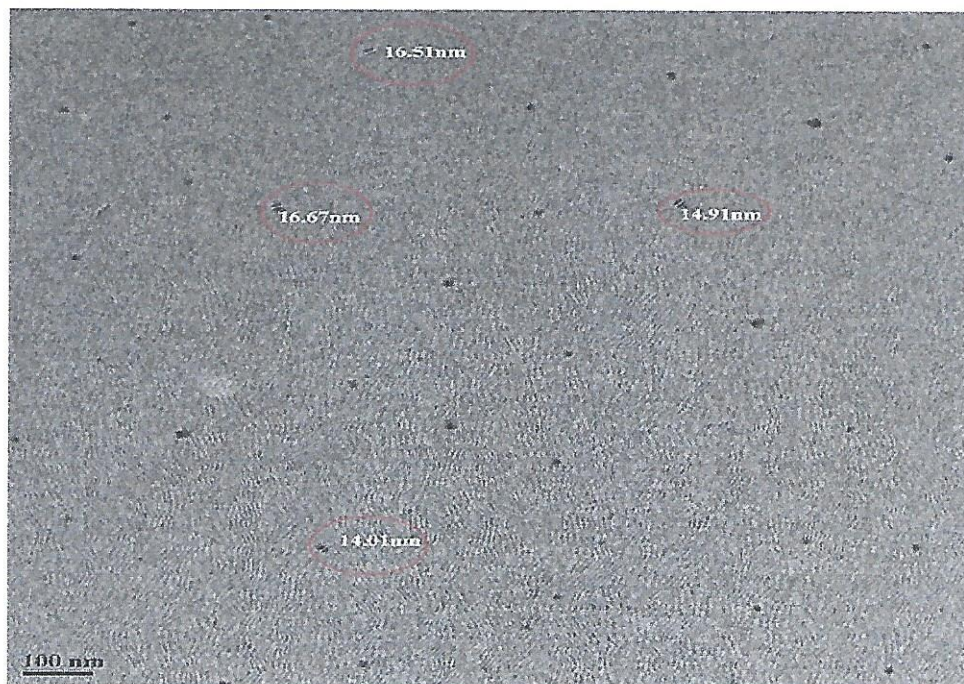


Figure 3. Scanning electron microscopy (SEM) image of carbon nanofiber.

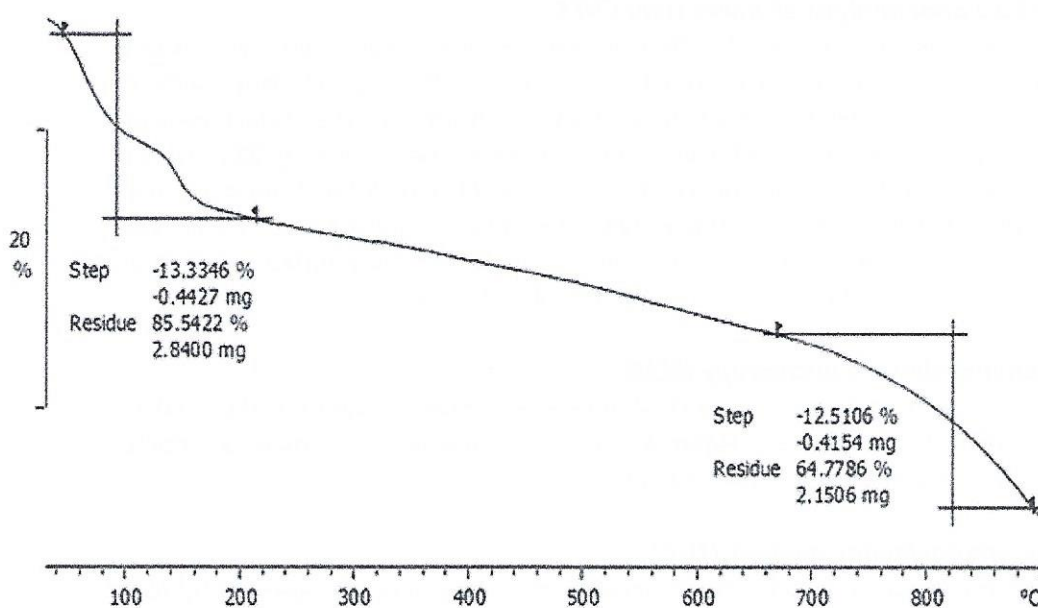


Figure 4. TGA analysis showing thermal stability of CNFs synthesised from Jawar stem.

### 3.1.5. FTIR analysis of Jawar stem

Comparative FTIR spectra before and after batch adsorption study for the nanofibers synthesised from Jawar stem are shown in Figures 5 and 6. In Figure 5 characteristics peaks may be observed at wave numbers  $699\text{ cm}^{-1}$  and  $832\text{ cm}^{-1}$  corresponds to  $\text{C}\equiv\text{C}$  stretching  $1011\text{ cm}^{-1}$  and  $1098\text{ cm}^{-1}$  corresponds to alkoxy C-C stretching  $1376\text{ cm}^{-1}$  belongs to  $(\text{O}=\text{C}-\text{OH})$  carboxyl stretching  $1535\text{ cm}^{-1}$  and  $1631\text{ cm}^{-1}$  corresponds to N-H hexamine stretching  $2916\text{ cm}^{-1}$  corresponds methyl C-H stretching and  $3656\text{ cm}^{-1}$  corresponds to O-H free hydroxyl stretching.

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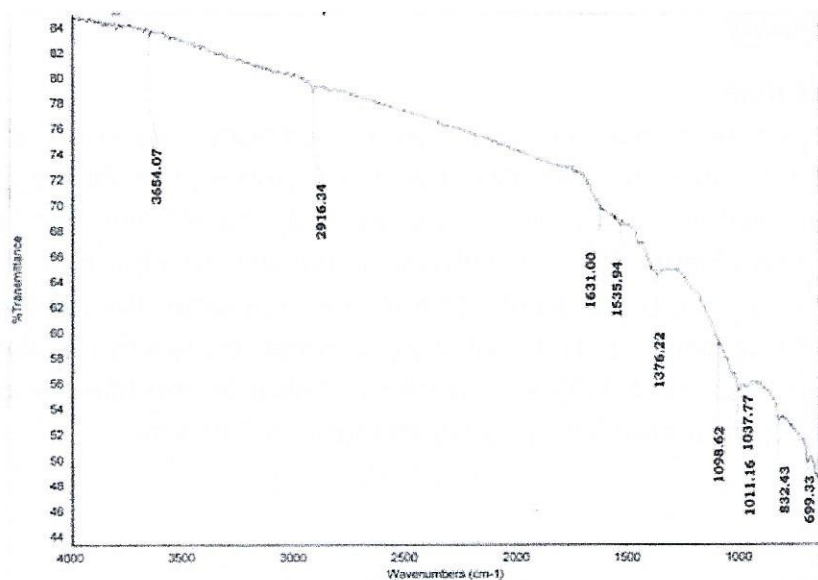


Figure 5. FTIR spectra of nanofibers synthesised from Jawar stem before adsorption.

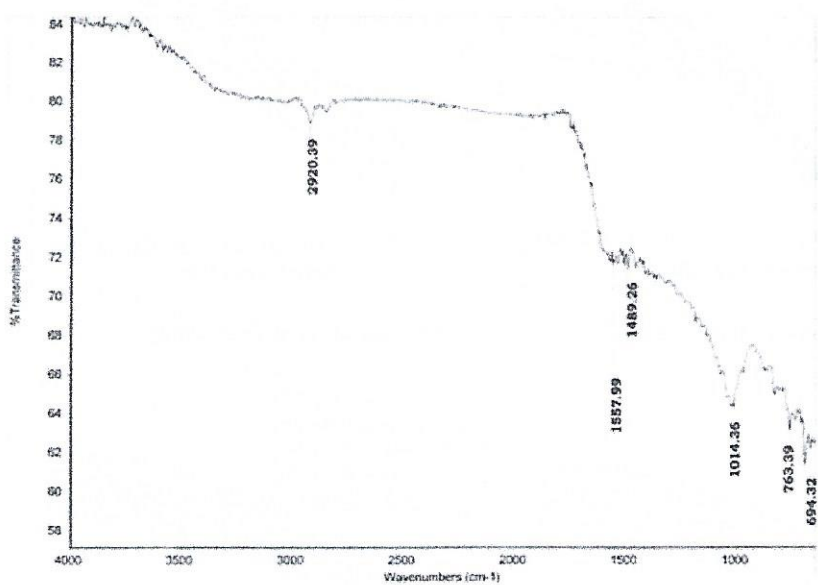


Figure 6. FTIR spectra of nanofibers synthesised from Jawar stem after adsorption.

Similar findings for the peak position were also made by other researchers (Talreja et al., 2014). Characteristic peaks after adsorption may be observed at wave numbers  $694\text{ cm}^{-1}$  and  $763\text{ cm}^{-1}$  attributes to alkoxy C-C stretching 2920 representing hydrochloric functional group. From Figures 5 and 6, it can be observed that almost all peaks are reduced, and peak attributes to O-H bonding have disappeared indicating the adsorption of metal on the surface of the nanofibers.

  
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### 3.2. Results of batch study

#### 3.2.1. Effect of contact time

The adsorption of chromium by carbon nanofibers synthesised from Jawar stems at varied contact time (20 min to 180 min in the multiples of 20 min) is presented in Figure 7. It can be observed that the adsorption of chromium increases with contact time and reaches equilibrium in 180 min. From Figure 7, it is also observed that the rate of uptake of metal ions was very rapid up to a contact period of 140 min, and thereafter, the removal rate increases marginally; this is because as removal percentage approaches towards its saturation, the rate of uptake of chromium ions decreases. This indicates that the adsorption rate starts reaching its saturation level at a contact time of 140 min.

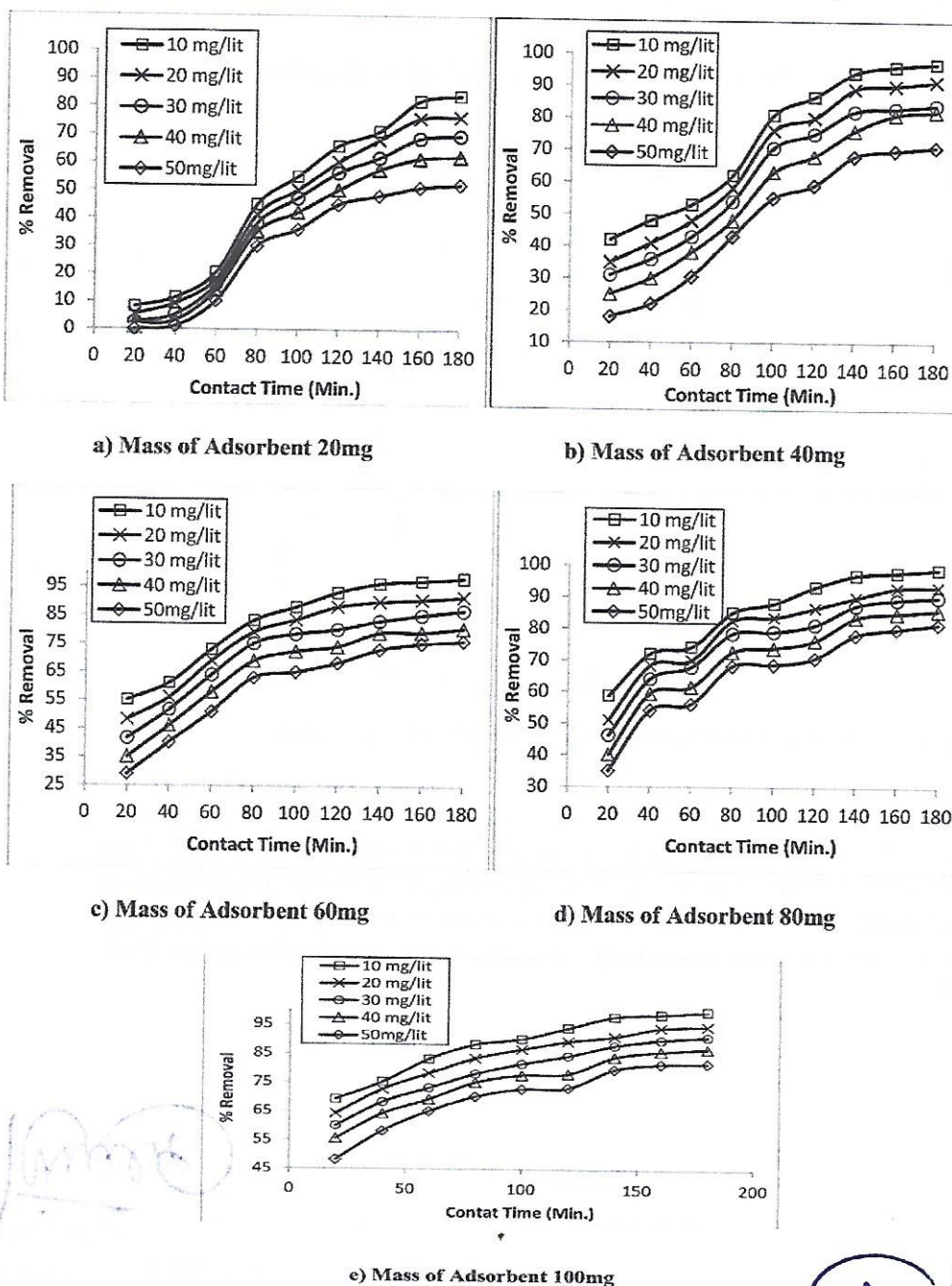


Figure 7. Effect of contact time on removal efficiency of Chromium (VI).

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### 3.2.2. Effect of initial concentration of chromium on removal rate

In this study, the concentration of Cr (VI) was varied from 10 mg/L to 50 mg/L in the multiples of 10 mg/L. The effect of the initial concentration on the removal efficiency is presented in Figure 8. From Figure 8, it can be observed that as the initial concentration of chromium increases, the removal efficiency decreases because there were no adsorption sites on the adsorption surface of the adsorbent material. The maximum Cr (VI) removal was observed at an initial concentration of 10 mg/lit for all adsorbent dosages (20–100 mg).

### 3.2.3. Effect of dosage of CNF adsorbent

In this study, the dose of adsorbent varied from 20 mg to 100 mg in the multiples of 20 mg. Figure 9 shows the removal efficiency of CNF for varying mass of adsorbents and initial chromium ion concentration. From Figure 9, it can be observed that the removal

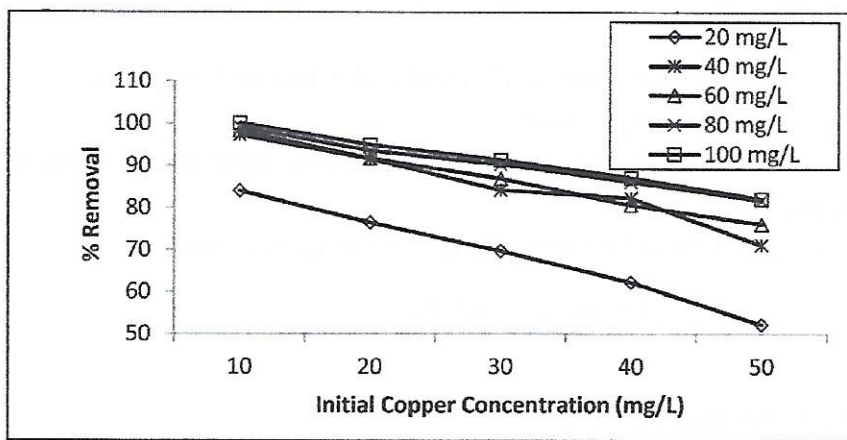


Figure 8. Effect of initial conc. of Chromium (VI) on its removal (%).

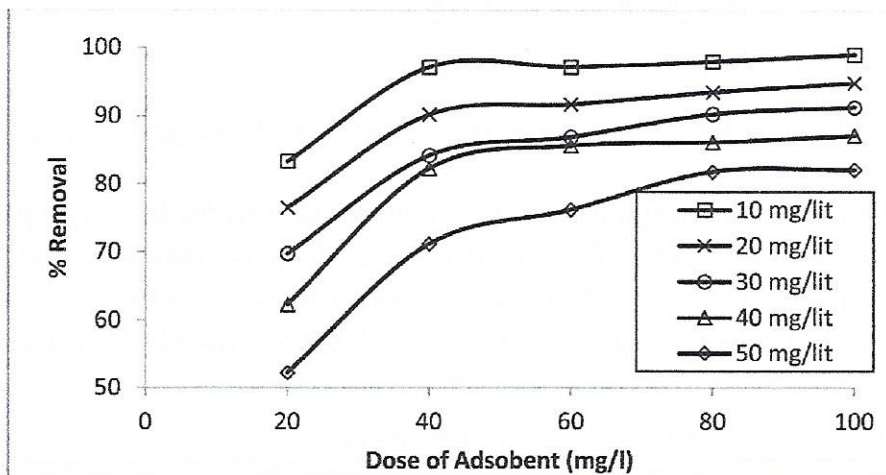


Figure 9. Removal of Chromium (VI) for varied adsorbent dosage.

  
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efficiency increases as the dose of adsorbent increases. When the dose of adsorbent was varied from 20 mg to 40 mg, the removal rate increased rapidly, but after dosage of 40 mg, there was a marginal increase in the percentage removal efficiency.

### 3.2.4. Adsorption isotherms

The adsorption isotherms are generally used to simulate the adsorption process. Further, these isotherms provide an idea of adsorption capacity and intensity of the adsorbent. In this study, Langmuir and Freundlich isotherms are tried to describe the adsorption data. The linearised forms of these isotherms are represented by the following equations 1 and 2, respectively.

The linearised Freundlich adsorption isotherm is

$$\ln(q_e) = \ln K_f + 1/n(\ln C_e) \quad (1)$$

Where,

$q_e$  = the amount of Cr (VI) adsorbed per unit weight of adsorbent (mg/gm);

$C_e$  = Equilibrium concentration (mg/L) and

$K_f$  and  $1/n$  are the Freundlich constants, and they represent adsorption capacity and adsorption intensity, respectively.

In this experimentation,  $q_e$  is calculated by using following equation

$$q_e = V(C_0 - C_e)/m$$

Where,

$C_e$  = initial copper concentration (mg/L)

$m$  = Mass of adsorbent (gm)

$V$  = Volume of sample taken = 1 lit.

The linear form of Langmuir adsorption is given by

$$1/q_e = (1/Q_0) + (1/b.Q_0.C_e) \quad (2)$$

where  $Q_0$  and  $b$  are the Langmuir constants related to capacity and energy of adsorption, respectively.

The equilibrium data and corresponding isotherm computations for both Langmuir and Freundlich are presented in Table 1. The reason for providing two isotherm plots for lower and higher dosages is due to the fact that the lower dosage results in a wider range of chromium levels and vice versa in Cr (VI) removed water. Typical Langmuir and

**Table 1.** Equilibrium data and corresponding isotherm computations.

Mass of Adsorbent (mg)	Initial chromium Conc. ( $C_0$ ) (mg/L)	Equilibrium Chromium Conc. ( $C_e$ ) (mg/L)	$q_e = (1(C_0 - C_e)/m)$ (mg/g)	1/C	1/q	ln ( $C_e$ )	ln ( $C_0$ )
20	10	1.2	0.44	0.83	2.27	0.18	-0.82
	20	5.86	1.03	0.17	0.83	1.78	1.89
	30	13.93	1.80	0.07	0.55	0.26	0.59
	40	24.41	2.27	0.04	0.44	3.19	0.82
	50	36.47	2.67	0.03	0.37	3.56	0.98
100	10	0.15	0.5	6.67	2.03	-1.9	-0.71
	20	*1.035	1.44	0.97	0.69	0.03	0.37
	30	3.78	2.31	0.26	0.43	1.33	0.83
	40	7.5	3.12	0.30	0.32	2.01	1.13
	50	12.91	3.85	0.08	0.26	2.55	1.34

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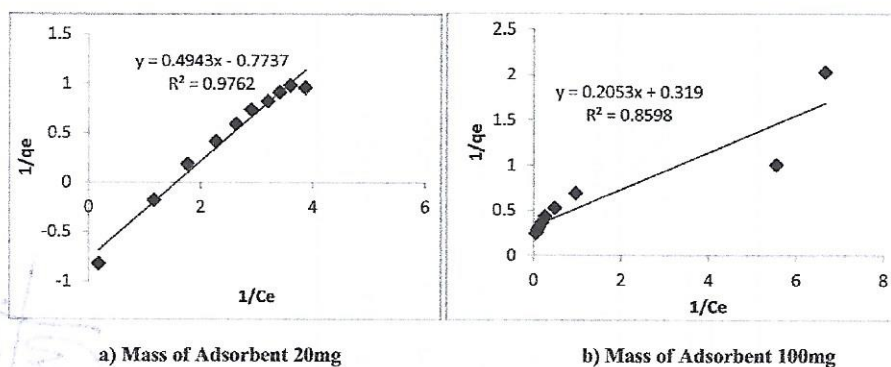


Figure 10. Typical Langmuir isotherms for removal of Chromium (VI).

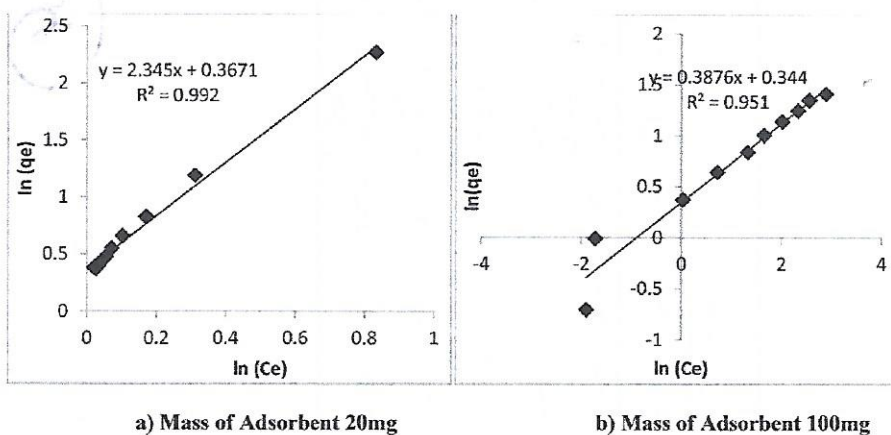


Figure 11. Typical Freundlich isotherms for removal of Chromium (VI).

Freundlich isotherms are presented Figures 10 and 11, respectively. The Langmuir and Freundlich equations and their constants for all adsorbent dosages are presented in Table 2. The applicability of isotherm for various adsorbent dosages has been determined based on the coefficient of correlation ( $C_c$ ) expressed as  $R^2$ . It can be observed from Table 2 that for all adsorbent dosages, Freundlich isotherm performs better than Langmuir isotherm.

#### 4. Conclusions

Laboratory studies on the removal of Cr (VI) from aqueous solution were conducted using nanofibers synthesised from Jawar stem (Agro-Waste). The adsorption experiments were designed to study the effect of various influencing parameters, such as contact time, dosage of adsorbent and initial chromium concentration. Based on the overall studies of chromium adsorption, few important observations have been made. The synthesised material is in powdered form. The hybrid material is in the nano-order range, which is evident from the particle size analysis. The material is found to be partially amorphous in nature as marked in the XRD analysis. The structure of the

  
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Table 2. Isotherm equations and corresponding constants for removal of Chromium (VI).

Sr. No.	Adsorbent	Parameter Mass of Adsorbent (gm)	Langmuir Equation $1/q_e = (1/b \cdot Q_0) \cdot (1/C_e) + (1/Q_0)$	Langmuir Constants		Freundlich Equation $\ln q_e = (1/n) \cdot (\ln C_e) - \ln K_f$	Freundlich Constants			
				B(L/mg)	$Q_0$ (mg/g)		$C_s$ ( $R^2$ )	$K_f$	1/n	$C_s$ ( $R^2$ )
1.	Nanofiber (Synthesised from Jawar stem)	20	$1/q_e = 0.494 \cdot (1/C_e) - 0.773$	-1.57	-1.294	0.976	1.44	2.345	0.992	Freundlich
		40	$1/q_e = 0.664 \cdot (1/C_e) + 0.290$	0.44	3.4482	0.901	0.987	0.428	0.933	Freundlich
		60	$1/q_e = 0.419 \cdot (1/C_e) + 0.329$	0.79	3.0395	0.865	1.1	0.405	0.945	Freundlich
		80	$1/q_e = 0.237 \cdot (1/C_e) + 0.332$	1.4	3.012	0.8	1.31	0.398	0.941	Freundlich
		100	$1/q_e = 0.205 \cdot (1/C_e) + 0.319$	1.55	3.1347	0.859	1.41	0.387	0.951	Freundlich



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material is confirmed by the FTIR data, which show the presence of metal-oxygen bonding. From all the above observations, it may be concluded that the material is suitable to act as adsorbent for the removal of chromium (VI) from water. The conclusions of batch study are as follows:

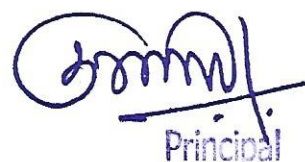
- As contact time increases, removal efficiency increases and maximum removal efficiency is observed at 140 min.
- As the initial concentration of chromium increases, the removal efficiency decreases.
- As the dosage of adsorbent increases, the removal efficiency increases. The maximum removal efficiency is observed at a dosage of 80 mg.
- For batch study, Freundlich isotherm is most suitable.

### Disclosure statement

No potential conflict of interest was reported by the author(s).

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