



## REMOVAL OF CHROMIUM (VI) IONS FROM AQUEOUS SOLUTION BY USING *LAPLAP PURPUREUS* (DOLICHOS BEAN) PLANT STEMS AS BIOSORBENT

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### ABSTRACT

The main intention of the present work is to explore the removal effectiveness of Cr<sup>+6</sup> ions from their aqueous solutions by adsorption method using *Laplap purpureus* stem powder (LPSP) as a biosorbent. The most important purpose of research is to analyze the batch mode of adsorption performance such as influences of pH, the dosage of biosorbent, reaction time, concentration of Cr<sup>+6</sup> ions solution, and temperature. The data obtained from the experiment results were studied by means of both Langmuir and Freundlich adsorption isotherm models. The pseudo-first-order and pseudo-second-order models have been analyzed to examine the kinetic models of adsorption. The adsorption isotherm models and kinetic study of analysis were divulged that an adsorption method was most favored for the Langmuir isotherm model. This investigation exhibits that the pseudo-second-order model plays an important responsibility in the adsorption mechanism to remove Cr<sup>+6</sup> ions. The biosorbent (LPSP) has better adsorption ability and also the removal of competence for Cr<sup>+6</sup> ions from its aqueous solution.

**Keywords:** Adsorption, Biosorbent, *Laplap purpureus* stem powder (LPSP), Kinetics, Langmuir, Freundlich.

### 1. INTRODUCTION

Environmental pollution arises due to rapid increasing of industries, which leads to deposition of excessive heavy metal on the earth surface [1]. Heavy metal stress is a most important concern against to all living things and major sources of heavy metal pollution derive from mining, smelting and other chemical industries [2, 3]. Especially, Chromium has been used enormously in various industries like textiles, metal finishing work, electroplating technique, leather tanning, production of stainless steel, preparation of chromate, dyes making for plastics, wood and manufacturing of paint, pigments and chemicals [4]. Chromium is one of the heavy metals in the top 16 metals that have ruinous effects on human beings [5]. Chromium metal exists in the form of its trivalent (Cr<sup>+3</sup>) or hexavalent (Cr<sup>+6</sup>) in the environment. The species of Chromium metal act as highly toxic in the biological system like carcinogen, teratogen and mutagen [6, 7]. Especially, the oxidation state of Chromium<sup>+6</sup> has more poisonous character than Chromium<sup>+3</sup> to human health [8]. The hexavalent Chromium is almost quite

soluble in water over the entire range of pH, hence it easily contaminates the environment [9]. It causes liver damage, edemas, pulmonary congestion, skin allergies and cancer on human beings by prolonged exposure of Chromium metal species, but the recommended limits of chromium in potable water is 0.05 mg/L [10]. Even though, Chromium (III) metal is needed 50-200 mg per day to the human body for the utilization of fat, protein and sugar [11]. Therefore, the removal of harmful heavy metal chromium from industrial waste is a significant one in the world to guard our surroundings. An Ordinary technique like chemical precipitation, chemical reduction, ion exchange, evaporation, membrane process and adsorption are applied for the elimination of hazardous heavy metals from industrial effluents. Particularly, the adsorption process proposes preferable uses such as low operational cost, minimizing the chemical apply or biological sludge, high rate of removal performance to heavy metals from metal aqueous solutions, retrieval of biosorbents, the possibility of metal regeneration and eco friendly [12]. The mechanism of

surface sorption is mainly dependent upon physio-chemical interactions in between metals and the surface of biosorbent and they are active appreciably more than assumed in equilibrium models [13]. The physico-chemical phenomenon during the adsorption process is rapid and reversible [14]. As a final point, the adsorption method is proficient to removal of heavy metal from its metal aqueous solutions by preparation of low-cost and eco-friendly bio-sorbent. In recent years most of the researchers had studied a variety of bio-adsorbents. In the present work, the adsorption of  $\text{Cr}^{+6}$  ions from its aqueous solution with the biosorbent prepared from *Laplap purpureus* plant stems powder (LPSP) was studied. The adsorption kinetic models and the equilibrium data were obtained by adsorption experiments and it was used to analyze the biosorbent sample. Consequently, the percentage of adsorption and quantities of adsorption to  $\text{Cr}^{+6}$  was calculated by using LPSP from the aqueous solution of chromium. The basic kinetic models like pseudo-first-order and pseudo-second-order models have been studied to exhibit the adsorption efficiency of  $\text{Cr}^{+6}$  ions in the adsorption process by using LPSP as a biosorbent.

## 2. MATERIAL AND METHODS

### 2.1. Adsorbent

The *Laplap purpureus* (Dolichos Bean) stems were obtained in the home garden after the harvest of bean fruits at Sevvaypatti village in Pudukkottai district, Tamil Nadu, India. The stems were cut into tiny pieces and systematically washed with tap water to remove unwanted dirt impurities and subsequently washed with distilled water and then dried in direct sunlight only for five days. After that, the stem pieces were ground as very fine powder using a domestic grinder and then stem powder was screened by manual experimental (Jayant Test Sieves) sieves in the size of  $< 90 \mu\text{m}$ . These separated *Laplap purpureus* stem powdered (LPSP) particles were stockpiled in a good-quality, conditioned air-tight plastic bottle for further use in biosorption studies [15]. There was no other chemical treatment for making adsorbent.

### 2.2. Batch adsorption studies

The effects of various parameters were studied for the removal of  $\text{Cr}^{+6}$  ions by *Laplap purpureus* stem Powder (LPSP). The AR grade of chemicals were used for experimental analysis at a high range of purity. The stock solution of the adsorbate consists of 1000 mg/L of  $\text{Cr}^{+6}$  metal ions solution prepared by dissolving the measured

quantity of  $\text{K}_2\text{Cr}_2\text{O}_7$  in double-distilled water. The investigational stock solution was diluted for the required initial concentration. In each adsorption experiment, 50 ml of  $\text{Cr}^{+6}$  ion solution with a well-known concentration was an addition to 200 mg of *Laplap purpureus* stem powder (LPSP) in a 250 ml of stopper flask, the adsorption experimental procedure was conducted at  $35^\circ\text{C}$ , then the combination was stirred for 50 minutes in a mechanical shaker at pH 3. The pH values were altered with 1N HCl and in 1N NaOH solution. The samples were withdrawn from the mechanical shaker at the proper time period and then adsorbent was separated by filtration with the help of No.40 Whatman filter paper. The concentration of  $\text{Cr}^{+6}$  metal ion solutions was investigated before and after treatment of the experiment by a UV-visible Spectrophotometer (Systronics PC-based double beam Spectrophotometer 2202).

## 3. RESULTS AND DISCUSSION

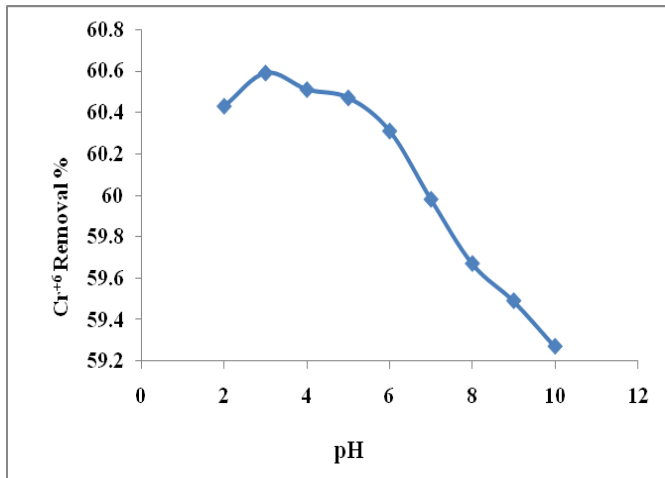
### 3.1. Effects of pH on Chromium (VI) ion adsorption

The effect of pH was studied by varying the pH of the solution (2, 3, 4, 5, 6, 7, 8, 9 and 10), initial concentration of  $\text{Cr}^{+6}$  metal ion solution 150 (ppm), taction time 50 minutes, temperature  $35^\circ\text{C}$ , adsorbent dose 200 mg, 50 ml of  $\text{Cr}^{+6}$  solution and agitation speed 360 rpm. The results shown in fig.1 indicated that at lower pH 3 maximum (60.59%) removal occurred. Hence, at higher pH 10 percentage of  $\text{Cr}^{+6}$  slightly decreased to 59.27%. This may be owing to the surface of biosorbent covered with various functional groups containing hydroxyl and alkyl groups with the increasing pH. The availability of functional group concentration increases which competes with  $\text{Cr}^{+6}$ . Therefore, the percentage removal of  $\text{Cr}^{+6}$  slightly decreases at higher pH [16, 17].

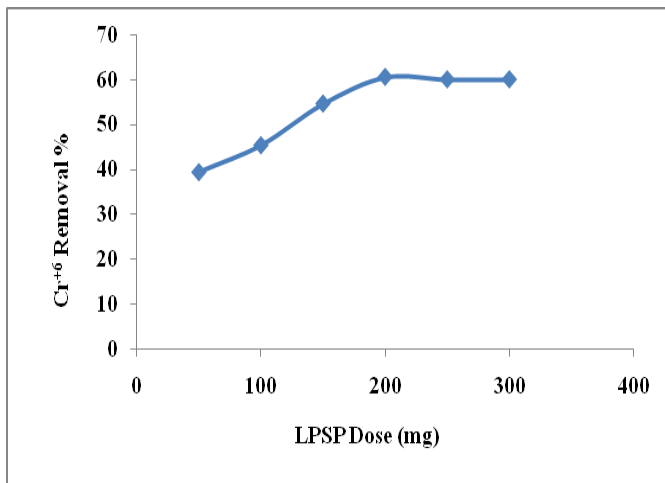
### 3.2. Effects of LPSP dose on Chromium (VI) ions adsorption

The adsorption of  $\text{Cr}^{+6}$  onto LPSP was studied by varying the adsorbent dosage (50, 100, 150, 200, 250 and 300 mg), initial concentration of  $\text{Cr}^{+6}$  metal ion solution 150 (ppm), temperature  $35^\circ\text{C}$ , taction time 50 minutes, 50 ml of  $\text{Cr}^{+6}$  metal solution, agitation speed 360 rpm and pH 3. Figure-2 exhibits the retention of  $\text{Cr}^{+6}$  ions against the dosage of biosorbent as shown in the graph, the adsorbent dosage increases while the adsorption of  $\text{Cr}^{+6}$  ions increases and then a certain value is reached. After a certain dose, the adsorption remained constant significantly and the stability of  $\text{Cr}^{+6}$  on the biosorbent

was attained at an adsorbent dose of 200 mg. At equilibrium, the removal percentage of Cr<sup>+6</sup> became constant due to the saturation of the available adsorbent site on the biosorbent [18].



**Fig. 1: Effects of pH on Chromium (VI) ion adsorption**

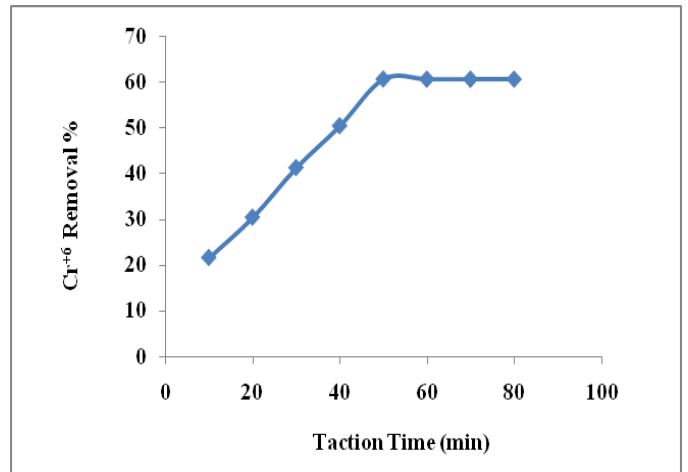


**Fig. 2: Effects of LPSP dose on Chromium<sup>+6</sup> ions adsorption**

**3.3. Effects of taction time on Chromium (VI) ions adsorption**

The experimental study of taction time is one of the main factors to fix time for analyzes the various parameters. In this parameter, taction time varied like 10, 20, 30, 40, 50, 60, 70 and 80 minutes, Temperature 35°C, pH 3, adsorbent dose 200 mg, initial concentration of Cr<sup>+6</sup> ion solution 150 (ppm), 50 ml of Cr<sup>+6</sup> solution and agitation speed 360 rpm were kept constant. Figure-3 represented that the biosorbption efficiency increased with increases in taction time from 10 to 80 min. Maximum removal

percentage for Cr<sup>+6</sup> ions was achieved up to 50 min after that did not change until 80 min. It may be due to that the large number of surface area available on biosorbent in initial stage, there after it slowed down later, because of the exhaustion of leftover surface area and repulsive forces between the bulk phase and the solute molecules [19].



**Fig. 3: Effects of taction time on Chromium (VI) ions adsorption**

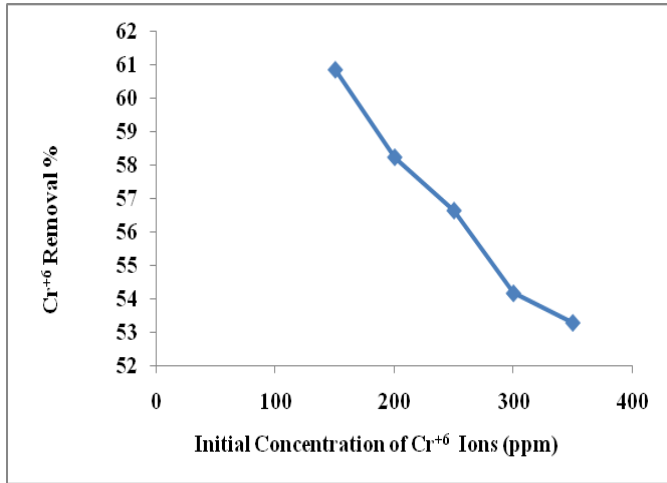
**3.4. Effects of various Concentrations on Chromium (VI) ions adsorption**

Concentration of the metal ion is an important factor in the adsorption process. In this parameter, the initial concentration varied likes as 150, 200, 250, 300 and 350 ppm and other parameters such as temperature 35°C, pH 3, agitation speed 360 rpm, time 50 minutes and 200 gm of adsorbent dose were kept constant. The removal efficiency of the initial Cr<sup>+6</sup> ions concentration was obtained from the experimental data which are represented in Fig.4. The maximum removal was possible in 150 ppm which clearly exhibit that the removal efficiency decreases with increasing initial concentration of Cr<sup>+6</sup> ion solution. It may be due to immediate saturation of limited obtainable biosorbent sites at higher concentration and the other hand at a lower concentration of Cr<sup>+6</sup> the removal efficiency achieved maximum due to the ratio of available surface sites of biosorbent to the number of moles of Cr<sup>+6</sup> ions [20].

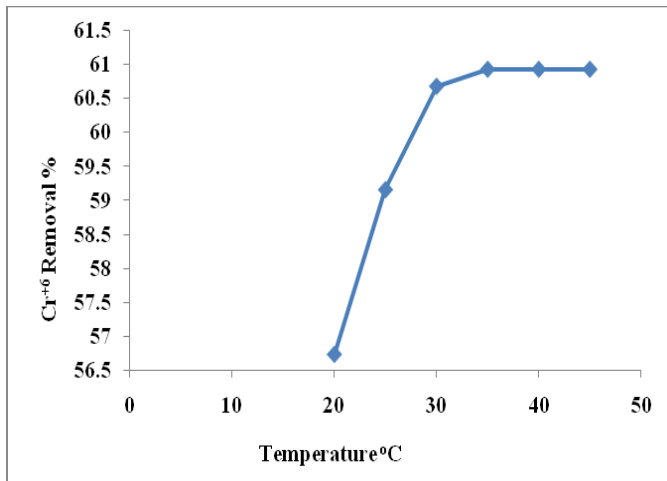
**3.5. Effects of temperature on Chromium (VI) ions adsorption**

Temperature is a great impact factor in the adsorption process. In this experiment, temperatures changed from

20 to 45°C, initial concentration of Cr<sup>+6</sup> ion 150 (ppm), under pH 3, agitation speed 360 rpm, taction time 50 minutes and 200 gm of adsorbent dose. In several adsorption experiments, the percentage of removal efficiency increases as the temperature increases. The changes in removal efficiency of LPSP adsorption for Cr<sup>+6</sup> as a function of temperature is shows in Fig.5.



**Fig. 4: Effects of various Concentrations on Chromium (VI) ions adsorption**



**Fig. 5: Effects of Temperature on Chromium (VI) ions adsorption**

Just as in the previous reports, the Cr<sup>+6</sup> metal ions removal efficiency is slowly improved with the increased temperature of adsorption. However, the changes of temperature had small effect on LPSP adsorption Cr<sup>+6</sup>. The percentage removal efficiency was increased only by 4.19% while temperatures changed from 20 to 45°C. Cr<sup>+6</sup> mobility increases at higher temperatures which leads to penetration of the harmful substances into the

internal voids of LPSP particles. Thus, the removal efficiency was progressed with increasing the adsorption temperature. However, by further increase in temperature, the presence of inside vacant site pores was almost packed and thereafter the removal efficiency remains stable. The optimization process of the temperature effect was informed to us that continuance increases of the temperature do not bring any important changes in the process of adsorption [21].

**3.6. Adsorption Isotherm Models**

The adsorption isotherm specifies that how adsorbate molecules partitioned between adsorbent and liquid phase at equilibrium as a role of adsorbate concentration. In this learning, equilibrium studies were carried out to understand the character of the adsorbent of Cr<sup>+6</sup> onto LPSP at equilibrium conditions by considering Langmuir and Freundlich adsorption isotherm models.

**3.6.1. Langmuir isotherm model**

The Langmuir isotherm model studied about saturated monolayer formation for solute on the surface of the adsorbent in adsorption process [22]. The linear form of Langmuir isotherm model used in the term

$$C_e/q_e = 1/q_m K_L + C_e/q_m$$

C<sub>e</sub> = Equilibrium constant for metal ions (mg / L), q<sub>e</sub> = Amount of metal ions adsorbed at equilibrium (mg/g), q<sub>m</sub> = Constant related to maximum adsorption capacity (mg/g), K<sub>L</sub> = Langmuir constant related to energy of adsorption.

The equation for the linear plot of C<sub>e</sub> / q<sub>e</sub> against C<sub>e</sub> should be a straight line. It shows that adsorption obeys the Langmuir isotherm model. The constant q<sub>m</sub> can be derived from slope and intercept of the plot and the values are shown in table 1 and fig.6. It demonstrates that the Langmuir isotherm model fitted well for the chosen biosorbent and adsorbate system with respect to r<sup>2</sup>= 0.972. Furthermore, q<sub>m</sub> of the LPSP is 91.24 (mg/g) was compared with the previous studies and q<sub>m</sub> values were represented in table 2.

The important characteristics of a Langmuir isotherm can be explained in terms of a dimensionless separation factor, R<sub>L</sub> [23], which is defined by the following equation:

$$R_L = 1 / 1 + (q_{max} \times K_L) C_o$$

Where C<sub>o</sub> is the highest initial concentration and the value of separation factor, R<sub>L</sub> represents the type of isotherm and nature of the adsorption process. Recognizing the R<sub>L</sub> values as the adsorption can be unfavorable (R<sub>L</sub>> 1); linear (R<sub>L</sub>=1); favorable (0< R<sub>L</sub><1)

or irreversible ( $R_L=0$ ) [24]. Hence, as the  $R_L$  value nearer to zero, the adsorption will be done better. In this experimental study, the  $R_L$  value 0.1976 (Table 1) favourable adsorption.

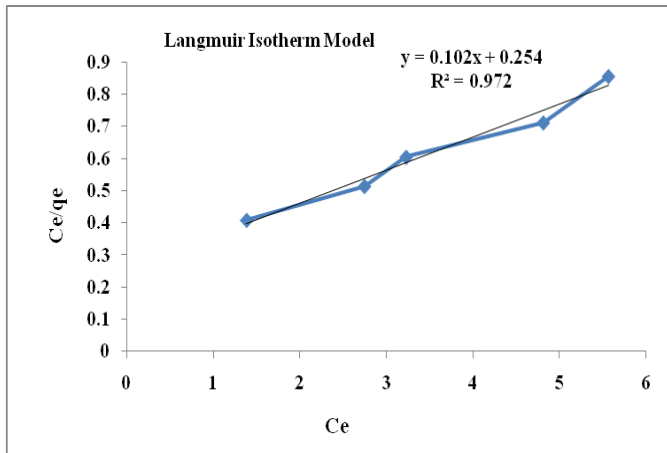


Fig. 6: Langmuir adsorption Isotherm Model

3.6.2. Freundlich Isotherm

The metal ions distribution between the liquid and solid phases can be illustrated by Freundlich isotherm model [25]. It best describes the adsorption onto heterogeneous surface. The common equation for the Freundlich isotherm model is represented as

$$\log q_e = \log k_f + 1/n \log C_e$$

Where,  $q_e$  = Amount of  $Cr^{+6}$  ions adsorbed on per unit weight of biosorbent (mg/g),  $K_f$  = Freundlich constant, which is correlated to calculate of adsorption capacity (mg/g).  $1/n$  = Sorption intensity (mg/L) and  $C_e$  = Equilibrium concentration (mg/L). Linear plots of  $\log q_e$  against  $\log C_e$ . The values of  $K_f$  and  $1/n$  can be derived from the intercept and slope respectively and their values

are exhibited in table 1. When  $1/n$  lies in between 1 to 10 values, the linearity of Freundlich plot suggests the favorable adsorption for  $Cr^{+6}$  on the biosorbent surface. These parameters have been determined from a plot  $\log q_e$  vs  $\log C_e$  (Fig.7). Thus the values of  $K_f$  and  $n$  were found as 4.1836 and 1.532 respectively at  $35^\circ C$  with  $r^2$  of 0.965.

The Langmuir adsorption isotherm exhibited that it has fitted well due to high  $r^2$  ( $r^2=0.972$ ) value compared with Freundlich adsorption isotherm model. The results stated that the nature of adsorption was a monolayer, which means a formation of  $Cr^{+6}$  molecules in single layer on the LPSP adsorbent surface [26]. It was suggesting that the equilibrium adsorption of  $Cr^{+6}$  onto LPSP might be best demonstrated with the Langmuir isotherm, because of the correlation between experimental and calculated values along with regression factors are in good agreement with Langmuir isotherm, it was exhibited that  $Cr^{+6}$  was most favorably adsorbed by LPSP.

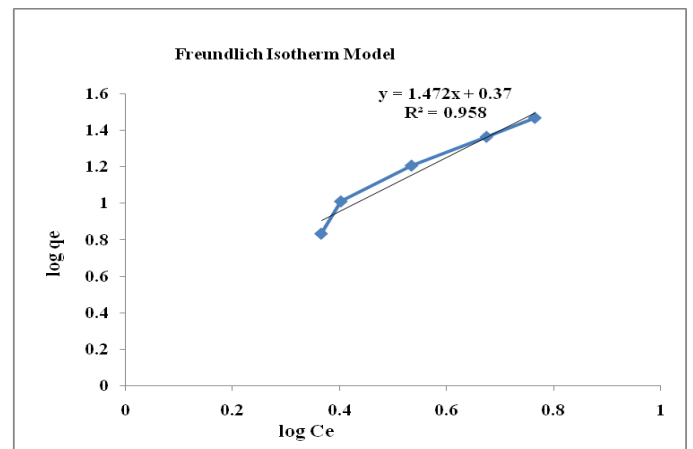


Fig. 7: Freundlich adsorption Isotherm Model

Table 1: Langmuir and Freundlich model parameters

$C_i$	Langmuir Model				Freundlich Model		
	Temp $^\circ C$	$q_m$ (mg/g)	$R_L$ (L/mg)	$r^2$	$K_f$ ( $mg^{1-n} g^{-1} L^n$ )	$n$ ( $mg^{1-n} g^{-1} L^n$ )	$r^2$
150	35 $^\circ C$	91.24	0.1976	0.972	4.1836	1.532	0.965

Table 2: Comparative study of adsorbent capacity in Langmuir constant  $q_{max}$  (mg/g) of various biosorbent

Adsorbent	Adsorbent capacity (mg/g)	[Reference]
Saw dust	20.70	[27]
<i>S. quadricuada</i>	12.00	[28]
<i>Ficus auriculate</i> leaves powder	13.33	[29]
Banana peel	10.42	[30]
<i>Moringa stenopetala</i> seed powder	09.70	[31]
<i>Trametes versicolor polyporus</i>	45.10	[32]
<i>Sargassum dentifolium</i>	41.20	[33]
<i>Rhizopus</i> sp.	09.95	[34]
Gooseberry seed	19.23	[35]
<i>Laplap purpureus</i> stem powder	91.24	This study

### 3.7. Kinetic Study

The Kinetics of adsorption studies have been carried out to illustrate adsorption mechanism and diffusion. The generated data had tested by Pseudo first order and Pseudo second order kinetics equation in order to determine the rate of the chromium ion adsorption on the LPSP, which controls the equilibrium time.

#### 3.7.1. The Pseudo first-order model

The possibility of adsorption data obtained from Lagergren pseudo first-order rate of equation can be described by the following equation:

$$dq_t/dt = k_1(q_e - q_t)$$

Where,  $k_1$  is the rate constant for first order adsorption ( $g.mg^{-1}.mn^{-1}$ ),  $q_e$  is  $Cr^{+6}$  adsorbed at equilibrium per unit mass of the sorbent ( $mg/g$ ),  $q_t$  is  $Cr^{+6}$  adsorbed ( $mg/g$ ). The combined form of above equation becomes  $\log(q_e - q_t) = \log(q_e) - (k_1/2.303)t$

Fig. 8 represents a plot  $\log(q_e - q_t)$  versus (t) represents a straight line of slope ( $k_1/2.303$ ) and an intercept of  $\log(q_e)$  [36].

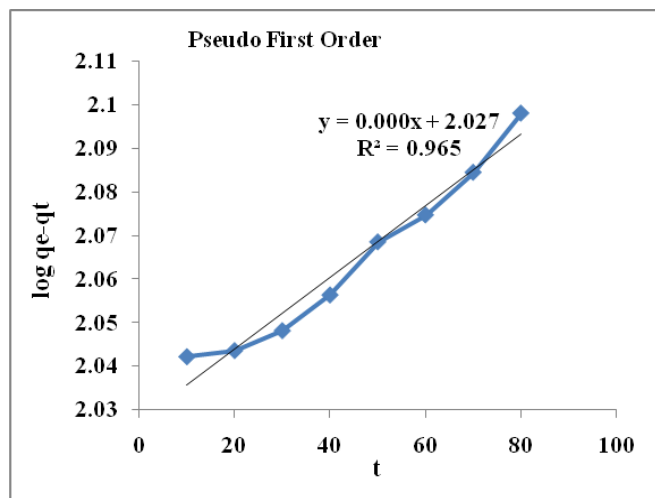


Fig. 8: Pseudo First Order Kinetic Model

#### 3.7.2. The Pseudo second-order model

This adsorption kinetic model equation developed as pseudo second-order rate of equation representation is based on the sorption capacity of solid phase is commonly expressed as:

$$dq_t/dt = k_2(q_e - q_t)^2$$

Where,  $k_2$  is the rate constant for pseudo second order adsorption ( $g.mg^{-1}.min^{-1}$ ), For the same boundary condition the integrated form of above equation becomes

$$t/q_t = 1/k_2q_e^2 + 1/q_e(t)$$

where, the  $k_2$  can be calculated by the slope and intercept of the plots of  $(t/q_t)$  versus time (t) (fig. 9). The pseudo second-order adsorption capacity ( $q_e$ ) values and correlation coefficient ( $r^2$ ) values are represented in table 3. The reasonable degree of conformity between the calculated data values and experimental data values have been found in the pseudo-first-order model compared with pseudo-second-order model. The correlation coefficient ( $r^2$ ) for the adsorption of  $Cr^{+6}$  was found to be very high ( $r^2=0.986$ ) in pseudo-second-order model with  $q_e$  46.56. This value represents that, the adsorption capacities ( $q_e$ ) is nearer to the calculated adsorption capacity, hence the pseudo-second-order model obeys the sorption of  $Cr^{+6}$  ions on biosorbent (LPSP).

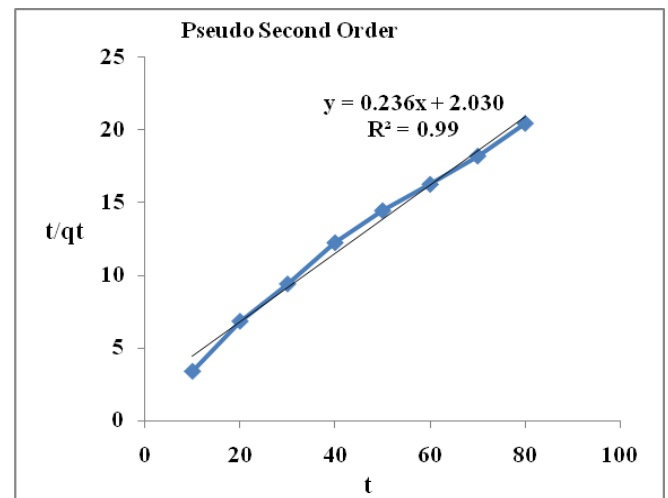


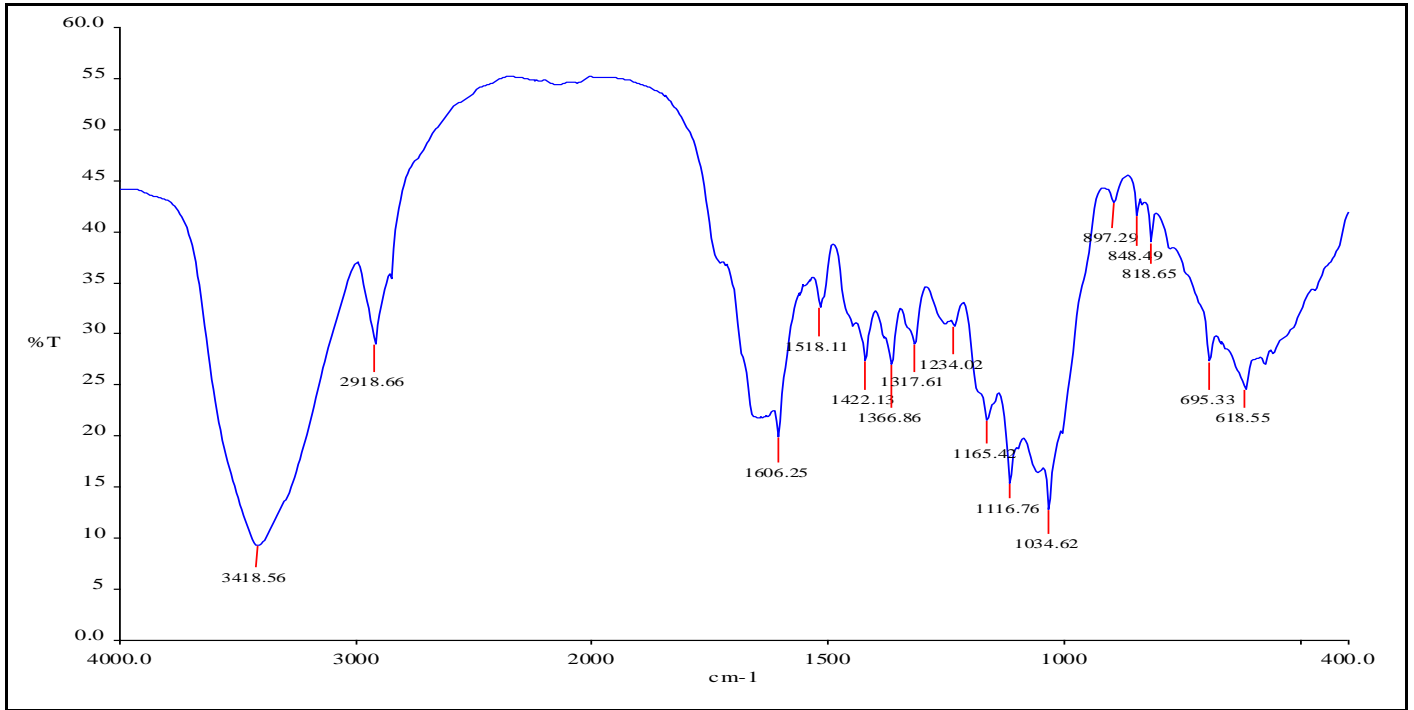
Fig. 9: Pseudo Second Order Kinetic Model

### 3.8. FT-IR spectrum

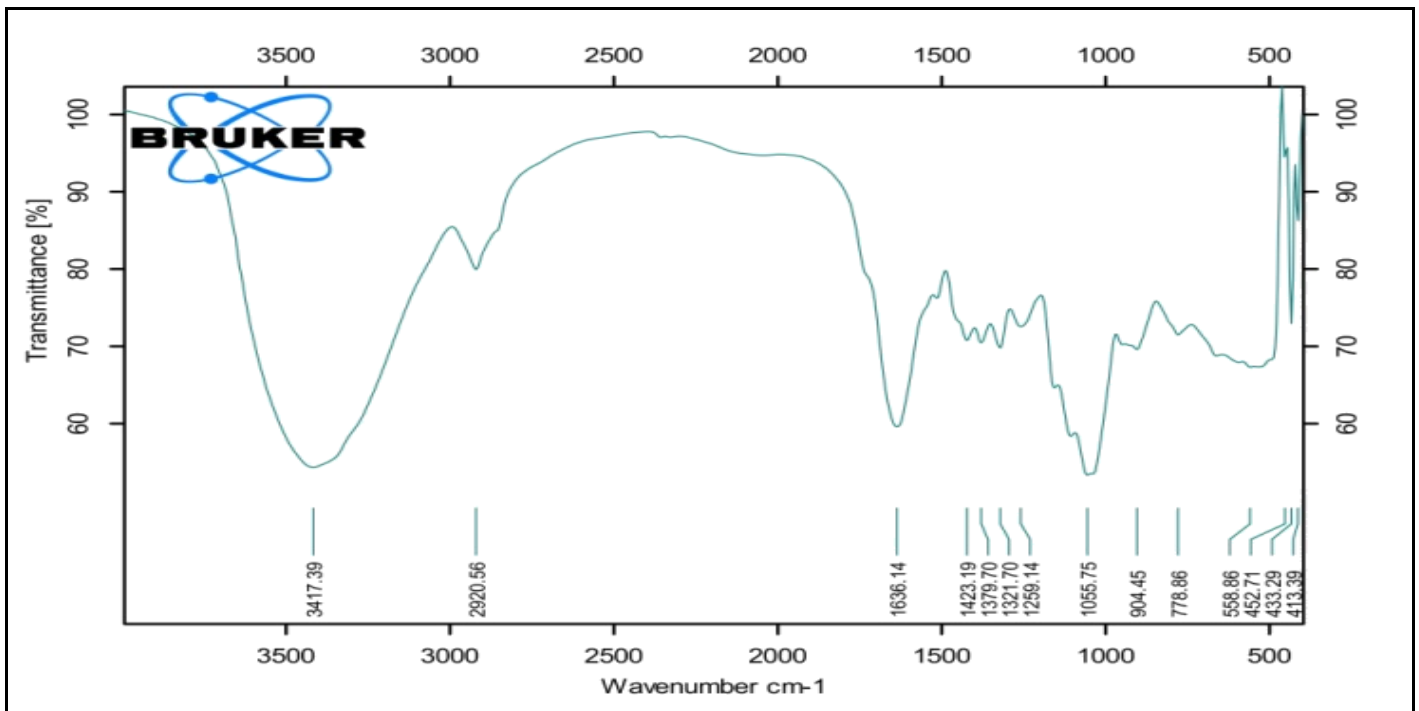
The FTIR spectrum analysis provides information about the changes in functional groups on biosorbent (LPSP), the spectra of LPSP before and after the  $Cr^{+6}$  metal ions adsorption are represented in the fig. 10 and 11. The FTIR spectrum of biosorbent displays a number of adsorption peaks range between of  $400-4000\text{ cm}^{-1}$ , which clearly shows only a complex of chemical nature for this biosorbent. The band at  $3418\text{ cm}^{-1}$  representing -OH groups, the band at  $2918\text{ cm}^{-1}$  corresponding to C-H stretching, the band around at  $1606\text{ cm}^{-1}$  could be assigned to the C=O stretching, the band at  $1518\text{ cm}^{-1}$  and at  $1422\text{ cm}^{-1}$  indicating as  $CH_2$  bonding vibration, the band at  $1034\text{ cm}^{-1}$  indicating C-O stretching. Amongst these adsorption peaks predominantly bonded -OH groups, C=O stretching and carboxyl groups were involved in  $Cr^{+6}$  ions biosorption [37, 38].

**Table 3: Pseudo-first-order and Pseudo-second-order kinetic parameters**

C <sub>i</sub>	Temp <sup>o</sup> C	Pseudo first order			Pseudo second order		
		q <sub>e</sub>		r <sup>2</sup>	q <sub>e</sub>		r <sup>2</sup>
		Theo.	Exp.		Theo.	Exp.	
150	35 °C	43.10	47.14	0.965	45.19	46.56	0.990



**Fig. 10: FTIR spectra of *Laplap purpureus* stems powder before adsorption**



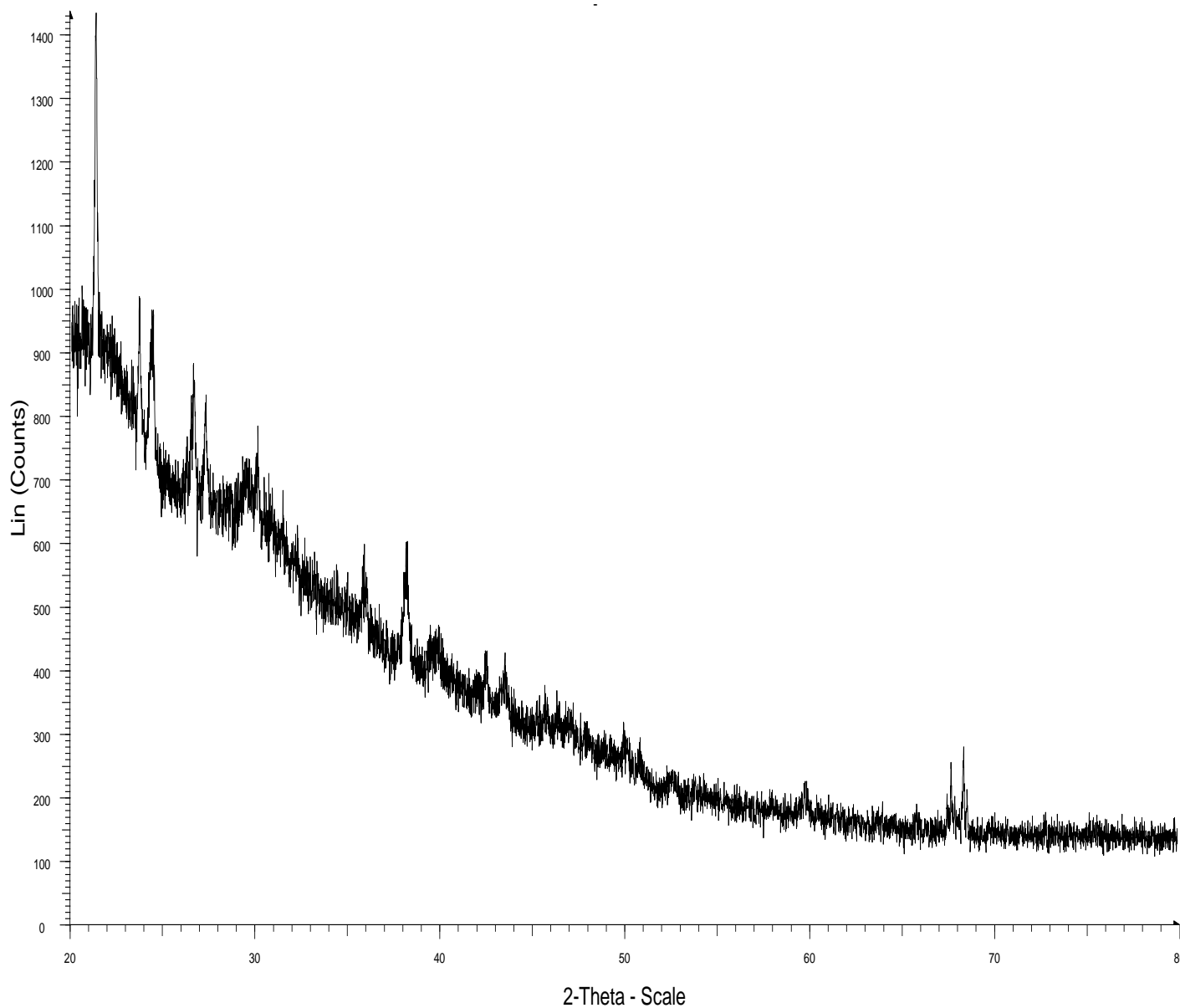
**Fig. 11: FTIR spectra of *Laplap purpureus* stems powder after adsorption**

### 3.9. X-Ray Diffraction of LPSP

The X-ray diffraction (XRD) technique is a powerful technique to analyze the crystalline amorphous nature of the adsorbent material. The LPSP before and after adsorption were recorded in Fig.12 (sample 1) and 13 (sample 2) respectively. The intense main peak (Fig.12) shows that the presence of highly organized crystalline nature of raw LPSP. After the adsorption of  $\text{Cr}^{+6}$  metal ions (Fig.13), the intensity of the main peak is slightly diminished and broadens. This means that the physical adsorption takes place on the upper layer of LPSP crystalline structure after adsorption of  $\text{Cr}^{+6}$  metal ions.

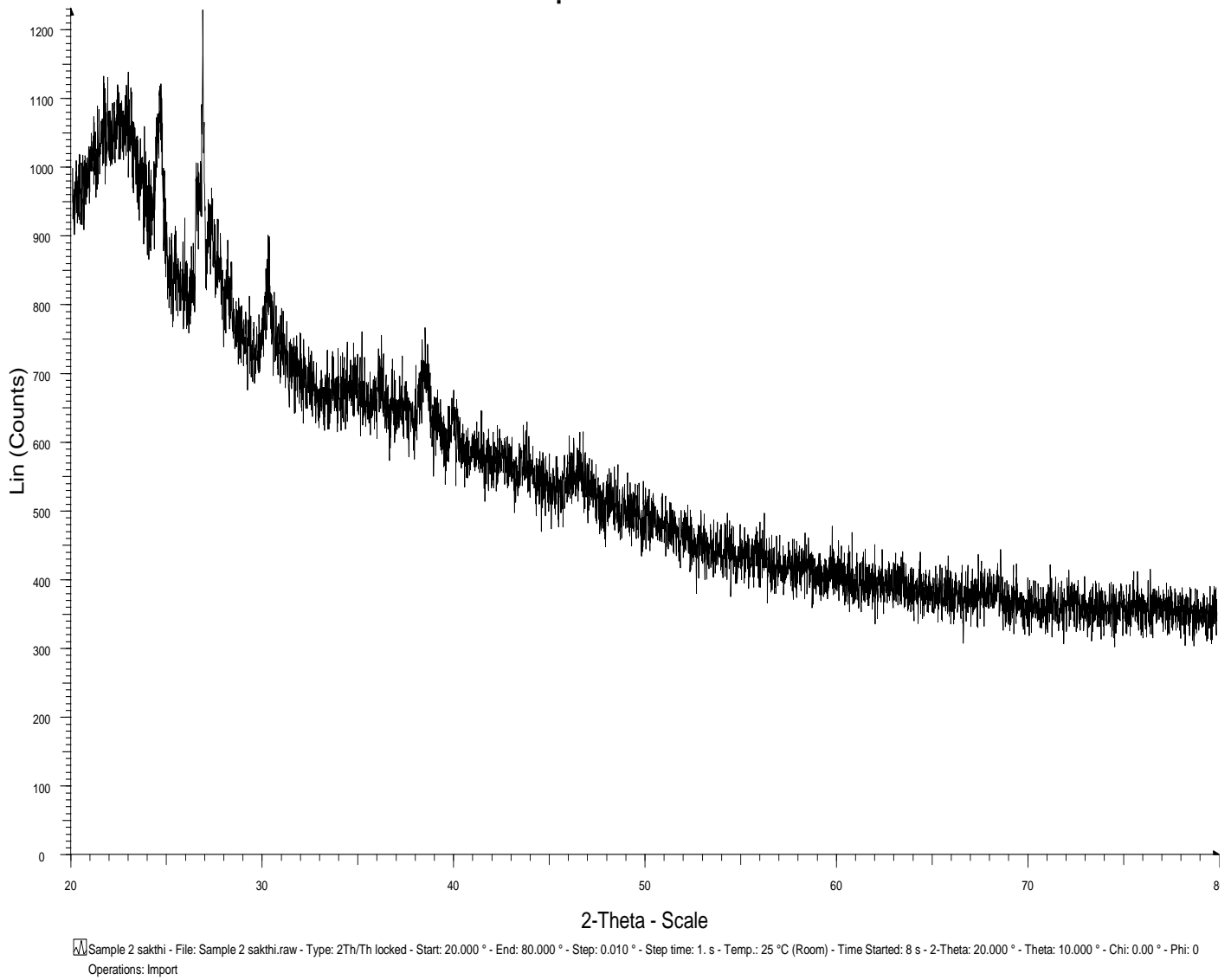
### 3.10. SEM Analysis for $\text{Cr}^{+6}$ Ions on Adsorption

Scanning Electron Microscope (SEM) studies provides the information about morphological property of the biosorbent (LPSP). Fig.14 is SEM image of unloaded LPSP material which represents the irregular and rough surface. The Fig.15 is SEM image of  $\text{Cr}^{+6}$  metal ions loaded on biosorbent (LPSP) surface. After the  $\text{Cr}^{+6}$  ions sorption, a significant change is viewed in the surface of biosorbent (LPSP). This property should be mentioned as a reason for sorption of metal ions. The biosorbent appears to have small pores like new cavities due to effective adsorption takes place.

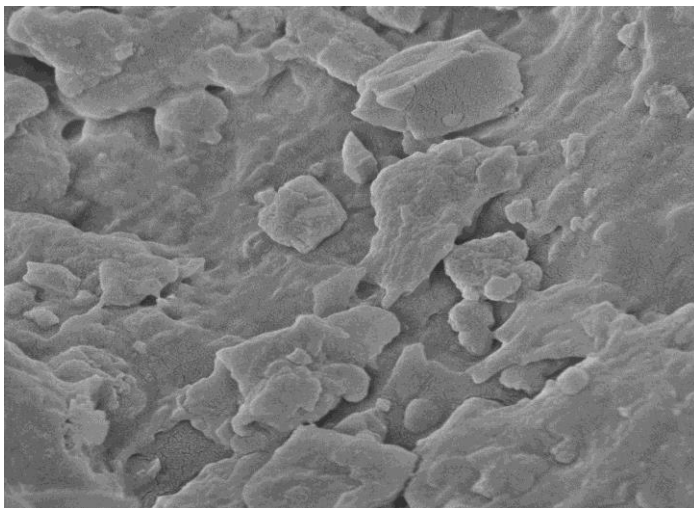


**Fig. 12: XRD Pattern of *Laplap purpureus* stems powder before adsorption**

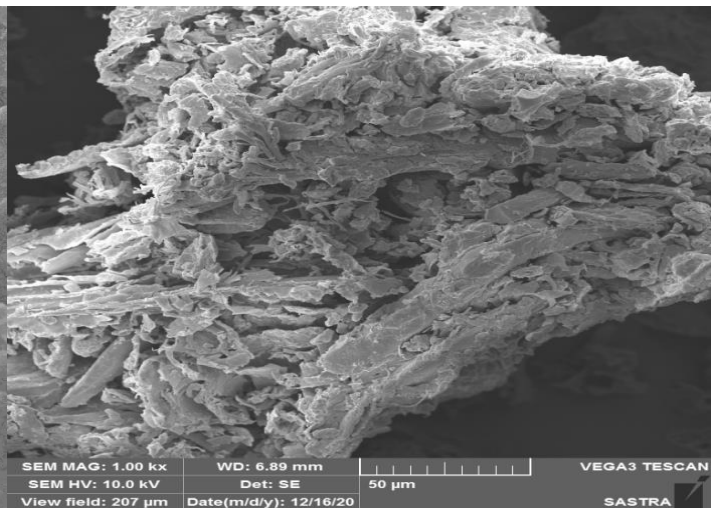




**Fig. 13: XRD pattern of *Laplap purpureus* stems powder after adsorption**



**Fig. 14: SEM image before adsorption**



**Fig. 15: SEM image after adsorption**

#### 4. CONCLUSION

The results demonstrated that *Laplap purpureus* stem powder (LPSP) is an efficient biosorbent for the removal of Cr<sup>+6</sup> ions from its aqueous solution. The present study investigated that biosorbent is an essential alternate for the adsorption of Cr<sup>+6</sup> metal ions. The percentage of removal efficiency was dependent upon pH, biosorbent dose, reaction time, initial Cr<sup>+6</sup> ions concentration and temperature of the system. It was concluded that the optimum biosorption phenomenon occurred at pH 3, the optimal dose was 200 mg, the suitable reaction time was noted to be 50 minutes, reasonably acceptable Cr<sup>+6</sup> ion concentration was 150 ppm and the best adsorption temperature was 35°C. The adsorption equilibrium better fitted with Langmuir isotherm model compared with Freundlich isotherm model. The adsorption kinetic process was found to be better fitted with pseudo-second-order model than pseudo-first-order. The SEM, FTIR and XRD spectral analysis clearly exhibited the biosorption of Cr<sup>+6</sup> ions on biosorbent LPSP. Therefore, LPSP may be used as a cheap biosorbent for removal of Cr<sup>+6</sup> metal ions.

#### 5. ACKNOWLEDGEMENT

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