



Exploring the Potential of Biochar Activated with Phosphoric Acid towards Hexavalent Chromium Removal

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Authors' contributions

This work was carried out in collaboration among all authors. Author RK designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Authors EP and VD managed the analyses of the study. Author DJSS managed the literature searches. All authors read and approved the final manuscript.

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ABSTRACT

The biochar derived from the dreadful weed water hyacinth (*Eichhornia crassipes*) was activated with phosphoric acid was utilized for adsorption of hexavalent chromium (Cr (VI)) from the simulated solution. The characterization (Zeta potential, specific surface area, Scanning Electron Microscope analysis) of biochar was done to study the surface properties of the biochar. The modified biochar ($379 \text{ m}^2\text{g}^{-1}$) recorded 49.80% greater surface area in comparison to the pristine biochar ($253 \text{ m}^2\text{g}^{-1}$). Zeta potential of modified biochar is 4.2 mV and pristine biochar is -13.9 mV. Positively charged modified surface enhance the sorption of negatively charged Cr (VI) ions. Study on effect of pH and concentration revealed that maximum sorption occurred at pH 2 and the increasing trend of concentration resulted in the decreased sorption respectively. The isotherm study was well supported by Langmuir isotherm (R^2 value=0.976) defining the monolayer adsorption. Maximum adsorption capacity was recorded for phosphoric acid modified biochar (50.0 mg g^{-1}) than the pristine biochar (35.7 mg g^{-1}). Pseudo second order kinetics (R^2 value=0.998) sounds good in explaining the rate of reaction. The findings reflect the utility of acid modified *Eichhornia crassipes* for heavy metal removal from aqueous medium.

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1. INTRODUCTION

Chromium is a pernicious heavy metal that taints the water resources strenuously. Chromium (VI) is the toxic form which is 500 times malignant in comparison to Cr (III) [1]. Overexposure of Cr (VI) uptake leads to development of tumor cells in liver and stomach whereas Cr (III) in considerable amount is essential for certain metabolism [2,3]. Cr (VI) in the industrial effluents is almost 5-200 mgL⁻¹ which is several times higher than the admissible concentration of 0.05-0.10 mgL⁻¹ [4]. The crucial role is played by the tanning industries because even a single unit could contaminate the surrounding 8 kilometer (km) of groundwater sources [5]. Basic chromium sulfate is used by them which results in 60% of chromium in liquid and solid waste expelled out. Thus a tannery would expel the solid waste containing about 3 kg of chromium and wastewater containing 5 kg of chromium. It is very intense to avert these toxic ions from entering the food chain and protect the precious water resources [6,7].

Different treatment methods such as precipitation, ion exchange, adsorption and membrane filtration pave way to get rid of this heavy metal. Amidst them adsorption is an enticing biological technique which evict the lacuna of the other methods including difficulties in operation, cost and efficiency. Although activated carbon is found to be widely used for adsorption, the need for cost efficiency necessitated the production of alternatives [8]. Hence in order to make the adsorption process more efficient it is better to use the organic waste materials rich in surface area and porosity [9]. For this process, one of the best preferences is biochar prepared from water hyacinth (*Eichhornia crassipes*). It is marked as one of the 100 most dreadful exotic weeds in the world. They hibernate for a longer while and sprout during summer. This aquatic weed is known for its notorious effects affecting nearly 50 countries [10]. They interfere in power generation, irrigation and avert the sunlight penetration leading to eutrophication. Moreover, they are made up of lignin, cellulose, hemicelluloses and prosperous in hydroxyl groups that assist in adsorption [1,11]. Therefore it will play the dual role of treating the pollutant as well as eradicating the toxic weed [12].

Biochar produced by the thermal decomposition possess had inferior properties to some extent.

Hence to overcome this issue, the modified biochar is prepared using acids, bases, metal oxides, magnetic material and nano composites. Acid modification with phosphoric acid was concentrated in this study. Phosphoric acid modification enhanced the porous structure and also enjoys the benefit of low cost, low pollution and low temperature [13]. The present study aims at reducing the burden of chromium toxicity in an eco friendly manner. The main objective is to study the significance of removal efficiency of Cr (VI) ions by phosphoric acid modified water hyacinth biochar and also the adsorption study is supported by the isotherm and kinetics study.

2. MATERIALS AND METHODS

2.1 Adsorbent Preparation

Eichhornia crassipes was collected from freshwater resource and cleansed with water to get rid of the debris and other aquatic weeds. Then it was allowed to dry and milled to obtain the powdered biomass. Pristine water hyacinth biochar (P-WBC) was prepared by pyrolysis of the biomass at 450°C for 3 hours. In case of phosphoric acid modified biochar (H₃PO₄ – WBC), the powdered biomass was sieved and plunged in 85% H₃PO₄ for 10 hours. Later it was kept in furnace for 1 hour at 450°C. The material was cooled and washed to bring the pH neutral. Finally it was dried in hot air oven for 8 hours at 60°C and sifted [9].

2.2 Characterization of Adsorbent

The pH of the biochar was determined by immersing the electrode of pH meter in a mixture of biochar: water (1:5) given by Jackson,1973 [14]. Zeta potential establishes the surface charge. It was carried out by adding 0.5 mg of the biochar to 20 ml of Millipore water. The solution was subjected to shaking at 180 rpm for 2 hours. Then it was sonicated for equal distribution of colloids and filtered through Whatman Filter Paper Grade No.42. The filtered solution was analyzed by particle size analyzer [15]. Surface texture and properties of the biochar was recorded with Scanning Electron Microscope (M/s. FEI- Quanta 250, Czech Republic). The elemental composition was examined using Energy Dispersive X-ray Analysis (EDXA). Point of zero charge (pH_{ZPC}) was assessed by solid addition method. 0.1 M KNO₃ solution was taken and the pH was set

from 2 to 10. Each pH solution was added with 1.0 g of biochar and agitated for 48 hours. The equilibrated solution was centrifuged and pH change was observed. The point of intersection between initial and difference in pH gave the pH_{ZPC} [16].

2.3 Adsorption Experiment

Adsorption study was carried out using simulated Cr (VI) solution through batch studies. Simulated solution was prepared by adding 2.83 g of Potassium dichromate solution in 1 liter of millipore water. Further concentration (100-1000 mgL^{-1}) for the experiment were diluted from the stock solution. The batch study results in studying the parameters such as effect of pH (1-8), dosage (1,1.5,2 and 2.5 g/100 ml), size (0.2,0.5,1.0 and 2.0 mm), concentration (100, 200, 300, 400, 500, 600, 700, 800, 900,1000 $mg L^{-1}$) and time (18,24,36,42,48 hrs). The fixed parameters are pH-2, dosage-2 g/100 ml, size-1.0 mm, concentration-200 $mg L^{-1}$ and time-36 hrs. It was done in Erlenmeyer conical flask (250 ml) by taking 100 ml of the solution. Biochar was added to it and shaken at 250 rpm. Then it was subjected to centrifugation at 8000 rpm for 10 minutes and finally pH and the Cr (VI) ions were examined. Residual Cr (VI) concentration in the solution after adsorption was analyzed spectrophotometrically by Diphenyl carbazide method. Spectrophotometer follows the principle of Beer's law which reads the purple violet colour at 540 nm [1]. The percent removal of Cr (VI) ions was assessed by the following equation (1).

$$\text{Adsorption(\%)} = \frac{(C_i - C_e)}{C_i} \times 100 \quad (1)$$

Where C_i ($mg L^{-1}$) is the initial Cr (VI) concentration in the simulated solution and C_e ($mg L^{-1}$) is the concentration when equilibrium is attained.

2.4 Isotherm Study

Langmuir and Freundlich isotherm models expound the formation of monolayer and multilayered structure respectively [17]. Langmuir isotherm [18] is represented by the following equation (2).

$$q_e = \frac{q_{\max} dC_e}{1 + dC_e} \quad (2)$$

Where C_e (mg/L) and q_e (mg/g) are concentration and amount of solute adsorbed, q_{\max} (mg/g) is the maximum monolayer adsorption and d is the

constant. Intercept between C_e/q_e and C_e gives the value of q_{\max} and d . d is obtained from the dimensionless constant (K_R) given by equation (3).

$$K_R = \frac{1}{1 + dC_o} \quad (3)$$

Freundlich isotherm [19] is expressed by the equation (4)

$$q_e = K_F C_e^{1/n} \quad (4)$$

Where K_F (mg/g) and n are constants derived from the graph plotted between q_e and $\log C_e$. K_F denotes the relative adsorption capacity whereas n denotes the intensity.

Temkin [20] isotherm is given by the corresponding equation (5)

$$q_e = \frac{RT}{B} \ln A + \frac{RT}{b} \ln C_e \quad (5)$$

Where R (8.314 J/molK) and A (\log^{-1}) are found to be universal gas constant and Temkin constant respectively. T ($25^\circ C$) represents the temperature at which sorption is carried out. B (J/mol) is the heat.

2.5 Kinetics Study

The linearized forms of kinetics first order [21] and kinetics second order reactions [22] are shown in the equation below (6 & 7).

$$\log(q_e - q_t) = \log q_e - \frac{K_1}{2.303} t \quad (6)$$

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e} t \quad (7)$$

Where K_1 (min^{-1}) and K_2 ($g \text{ mg}^{-1} \text{ min}^{-1}$) are first order and second order rate constants. q_e (mg/g) amount of biochar adsorbed at equilibrium. q_t (mg/g) is the adsorption performed at time period t (min). K_1 is computed from the slope of $\log(q_e - q_t)$ Vs t and K_2 from the intercept of t/q_t and t .

The Elovich model [23] presented in 1939 used for heterogeneous adsorbing surfaces in chemisorption is expressed in the equation (8)

$$q_t = \left(\frac{1}{b}\right) \ln(ab) + \left(\frac{1}{b}\right) \ln t \quad (8)$$

Where a and b were obtained from the graph plotted between $q(t)$ and $\ln(t)$, $1/b$ (mg/g) is the

available sites for sorption and a (mg/g min) is the rate of initial sorption.

In order to discuss about the diffusion mechanism, intra particle diffusion model [24] was investigated by the following equation (9):

$$q_t = K_{id}t^{1/2} + C \quad (9)$$

Where C obtained from intercept between q_t and $t^{1/2}$ is the constant representing boundary layer thickness and K_{id} ($\text{mg g}^{-1} \text{min}^{-1}$) is the intra particle diffusion rate constant.

3. RESULTS AND DISCUSSION

3.1 Characterization of Adsorbent

Physical characteristics of the P-WBC and H_3PO_4 -WBC were tabulated (Table 1). Point of zero charge (pH_{ZPC}) is the condition when net charge on the surface of the biochar is zero. It contains more of positive charges compared to the negative charges. The negatively charged surface is created on the adsorbent due to the OH^- ions sorption at $\text{pH}_{ZPC} < \text{pH}$ while the positively charged surface is created on the biochar due to H^+ ions sorption at $\text{pH}_{ZPC} > \text{pH}$. Hence the Cr (VI) ions sorption is favored greatly by the condition of $\text{pH}_{ZPC} > \text{pH}$ [25].

Table 1. Characteristics of biochar

Parameters	P-WBC	H_3PO_4 -WBC
pH	9.70	2.52
pH_{ZPC}	7.60	5.80
Zeta potential(mV)	-13.9	4.2
BET surface area(m^2g^{-1})	253	379

The zeta potential represents the charge of the adsorbent surface. Zeta potential of P-WBC was recorded as -13.9 mV which indicates that surface dominated by negative charge. In case of H_3PO_4 -WBC, it was 4.2 mV filled with positive charges. Hence the adsorption of Cr (VI) anions was higher in H_3PO_4 -WBC comparison to the P-WBC that attracts towards their positive charged surface. Specific surface area of P-WBC and H_3PO_4 -WBC was $253 \text{ m}^2\text{g}^{-1}$ and $379 \text{ m}^2\text{g}^{-1}$ respectively. Specific surface area strongly influences the adsorption. Hence H_3PO_4 -WBC shows higher sorption as it is 49.80% higher surface area in comparison to the P-WBC.

Morphological micrographs and its EDXA examination of P-WBC is shown in Fig. 1. SEM

image before (Fig. 1a) and after (Fig. 1c) adsorption shows clear striated structure and compact aggregated structure respectively. This fact is due to the agglomeration of Cr (VI) ions on the surface after adsorption [26]. EDXA image before and after sorption of P-WBC is given in the Fig. 1b and Fig. 1d respectively. EDXA image before adsorption of Cr (VI) ions represents 2.19% of Ca and 0.49% Na which reduced after sorption into 1.99% Ca and 0% Na. The reduction in Ca peak is due to the occurrence of prominent Cr peaks [27]. Similarly the composition of Mg, Cl and K reduced after adsorption replaced by the Cr (VI) peaks.

3.2 Adsorption Experiment

3.2.1 Effect of pH

Variation in pH showed greater difference in the adsorption efficiency. It was studied with varying pH from 1 to 8 by adding 1 N (Normality) HCl or 1 N NaOH. The result showed that the higher Cr (VI) removal occurred at pH 2 and the efficiency decreases as the pH increases from 2 (96.7 %) to 8 (67.2%) for H_3PO_4 -WBC treatment whereas in P-WBC it decreased from 90% to 59.8% (Fig. 2). This is because at lower pH the surface of the biochar gets dominated by the H^+ ions creating the favourable positive charge environment for adsorption of negatively charged Cr (VI) ions. In contrast, the higher pH brings about the surface enriched with OH^- ions creating the negative charge surface which would result in the electrostatic repulsion between the surface and the Cr (VI) ions [28]. In addition, reduction of Cr (VI) ions to Cr (III) ions is favored by the pH below 2. The Cr (III) ions are further to be detoxicated where it occurrence would bring about minor health problems [29]. Hence pH 2 was found to be the best for removal of Cr (VI) ions.

The type and nature of adsorbent plays an important role in differing the pH. Hence the changes in the functional groups present on the modified biochar surface creates pH variation [30]. This in turn improves the removal efficiency of the H_3PO_4 -WBC as the number of protons is higher than the P-WBC. This is also in evidence from the relation between pH and pH_{ZPC} . As aforesaid, the anionic adsorption is favoured by the condition where $\text{pH}_{ZPC} > \text{pH}$ [28]. Hence the adsorption was higher for H_3PO_4 -WBC ($\text{pH}_{ZPC} > \text{pH}$) when compared to P-WBC ($\text{pH}_{ZPC} < \text{pH}$).

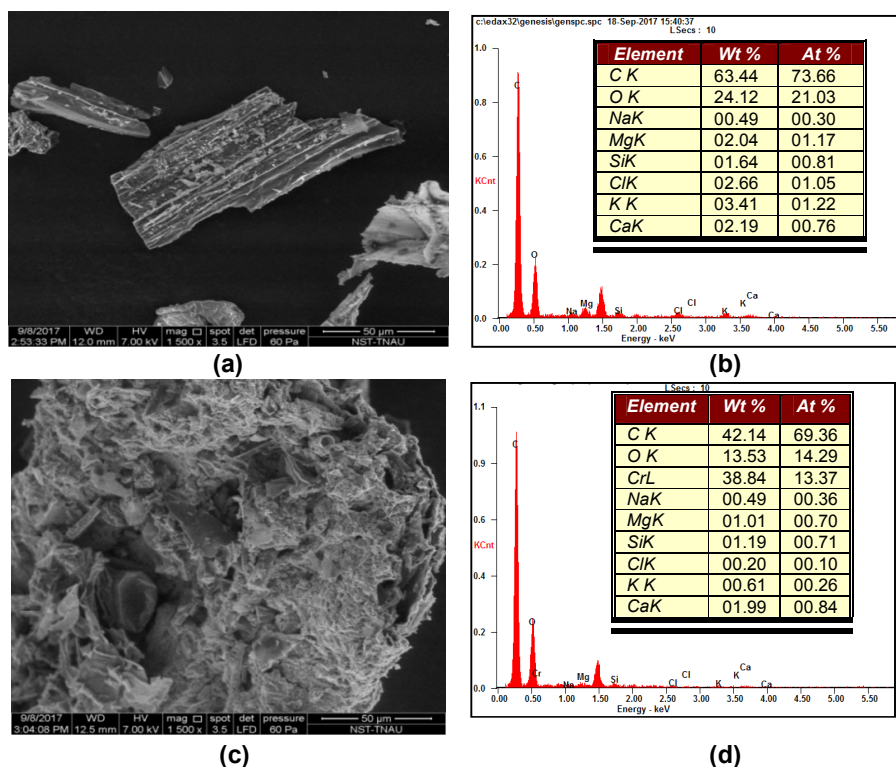


Fig. 1. Scanning electron micrographs of P-WBC a) before Cr (VI) loaded, b) after Cr (VI) loaded, EDXA image of P-WBC c) before Cr (VI) loaded and d) after Cr (VI) loaded

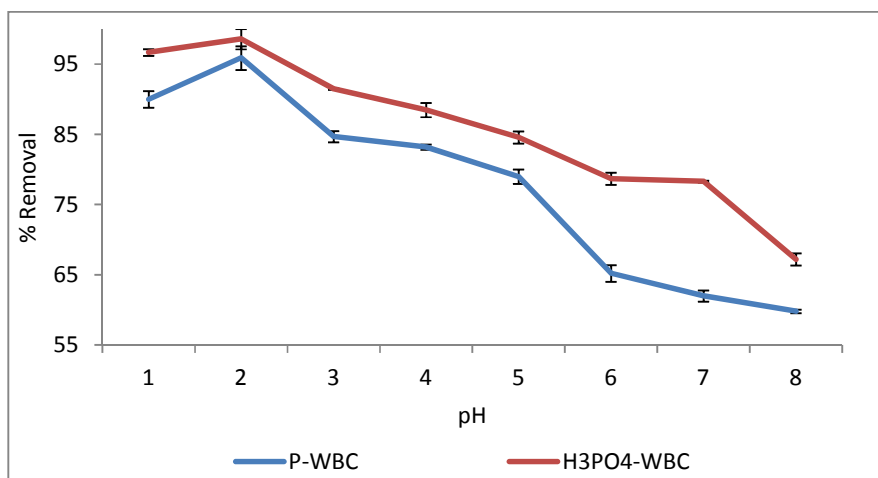


Fig. 2. Effect of pH on adsorption of Cr (VI) ions

3.2.2 Effect of dosage

The potential of biochar to remove the Cr (VI) ions were found to be maximum at 2.0 g/100 ml. It is 97.6% by H₃PO₄-WBC and 91.9% through P-WBC. Increase in dosage above 2.0 g/100 ml resulted in the lesser efficiency (Fig. 3). The

increase in dosage increases the surface area facilitating the more number of active sites for sorption. After reaching equilibrium the efficiency decreases. But the adsorption per unit weight decrease in increasing the dosage due to lack of solute for all exchangeable sites on the surface. Hence the removal percentage is higher and

sorption per unit weight is lower for higher dosage [31,32].

3.2.3 Effect of size

The effect of adsorbent size on the adsorption capacity was studied by fixing other factors with varying size of biosorbent. Adsorption efficiency is maximum at 0.5 mm size and then decreased on further increase in size (Fig. 4). Increase in biochar size decreased the surface area which in turn reduces the binding sites [25]. Hence the sorption is maximum at smaller size.

3.2.4 Effect of concentration

The initial concentration of the simulated solution affects the adsorption potential in a greater manner. The adsorption efficiency decreased from 99% (100 mg L^{-1}) to 88.8% (1000 mg L^{-1}) in H_3PO_4 -WBC whereas 86.6% (100 mg L^{-1}) to 63.6% (1000 mg L^{-1}) in P-WBC (Fig. 5). Decrease in adsorption efficiency from lower to higher concentration is due to the saturation of the restricted sites to the fixed dosage. Hence the adsorbent material exhaust after the equilibrium leading to the decreased sorption efficiency [17, 33].

3.2.5 Isotherm study

Isotherm is a necessary phenomenon to be examined that sounds good in explicating the separation of solute from the solution and attachment to an adsorbent surface. The isotherm was studied by fitting into the Langmuir, Freundlich and Temkin isotherm equations. The non-linear curves of Langmuir isotherm were plotted in Fig. 6. The isotherm parameters obtained from the plotted graphs are illustrated in the Table 2. Maximum adsorption capacity of P-

WBC and H_3PO_4 -WBC are 35.7 (mg/g) and 50.0 (mg/g) respectively. Hence the modified biochar tends to show efficacy in the adsorption potential of Cr (VI) ions. The higher regression coefficient (R^2) values confirm the validity of monolayer adsorption and the activation energy is distributed equally over each molecule. Thus the adsorption is highly facilitated by Langmuir isotherm portraying the homogeneous structure of the biochar followed by Temkin isotherm. Furthermore the favorability of adsorption is also represented by the Langmuir dimensionless constant (K_R). When K_R value is equal to 1 the sorption is linear while K_R value is between 0 and 1 favorable adsorption takes place. It becomes unfavorable when K_R value is greater than 1 [34]. From the Table 3 it was clear that all the dimensionless constant value is between 0 and 1 which reflects good adsorption.

The Temkin isotherm shows relatively higher R^2 value due to linearity which may be the proof of intrinsic heterogeneity of surface [35]. In case of Freundlich isotherm, the R^2 value is lesser but the n value stands in the prescribed limit of $1 < n < 10$ which portrays that sorption is also favored by the heterogeneous sites [36].

3.2.6 Effect of time

Adsorption of Cr (VI) ions was found to increased with increase in time until it attained the exhaustion stage. Time depends on the nature of adsorbent and the presence of exchangeable sites on its surface. The removal of Cr (VI) ions was higher at 36 hrs. The percent removal was 91.2% by P-WBC and 99.5% by H_3PO_4 -WBC (Fig. 7). Initially the metals ions are adsorbed at a faster rate due to abundance of sites resulting in monolayer formation. Later the sites are filled and the sorption decreases [28].

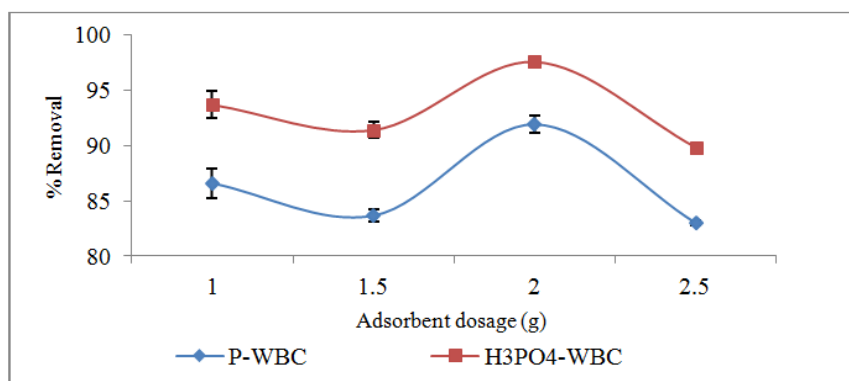


Fig. 3. Effect of dosage on adsorption of Cr (VI) ions

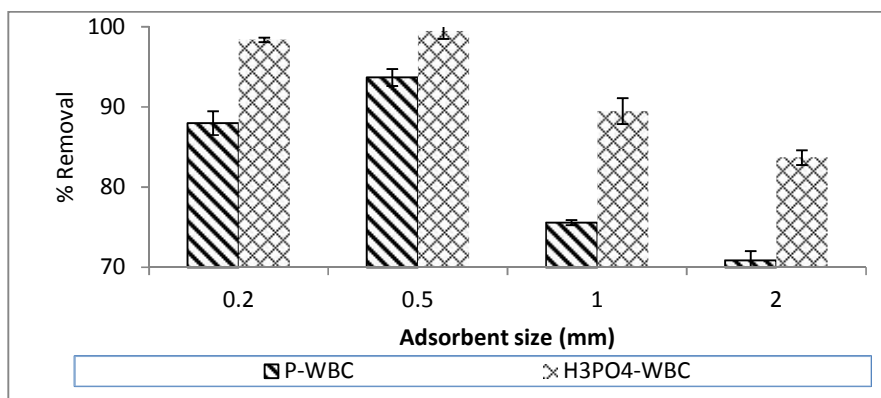


Fig. 4. Effect of adsorbent size on adsorption of Cr (VI) ions

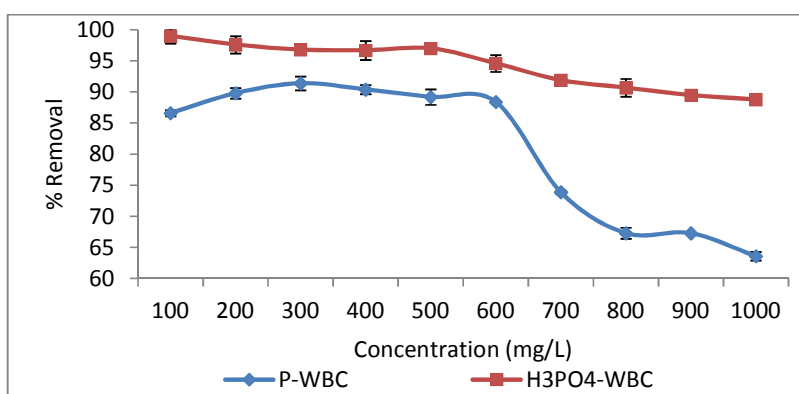


Fig. 5. Effect of concentration on adsorption of Cr (VI) ions

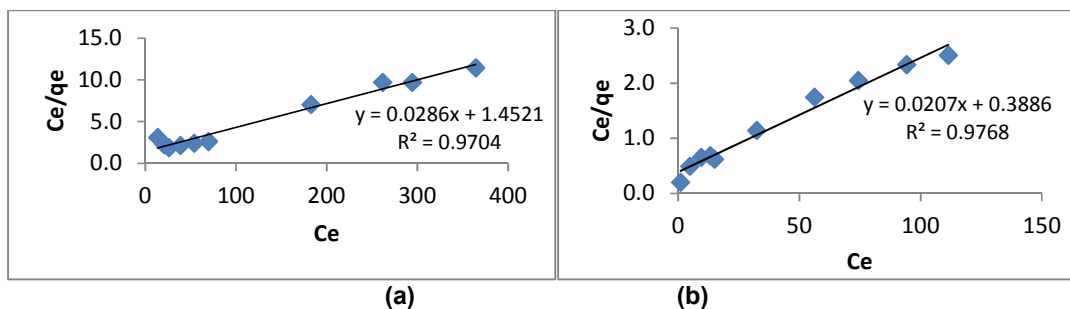


Fig. 6. Langmuir isotherm plots of a) P-WBC, b) H₃PO₄-WBC

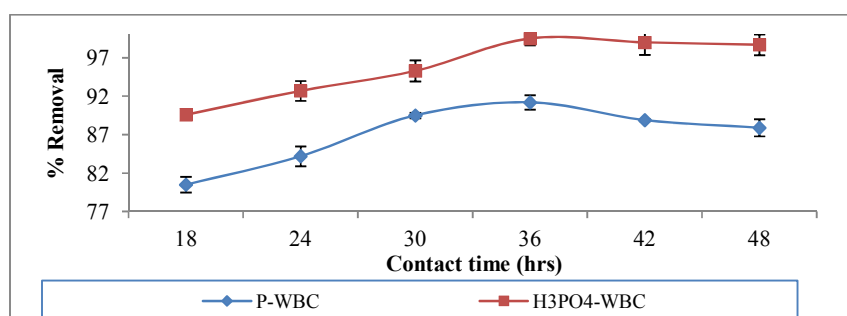


Fig. 7. Effect of contact time on adsorption of Cr (VI) ions

Table 2. Isotherm parameters for Cr (VI) ions adsorption

Constants	P-WBC	H ₃ PO ₄ -WBC
Langmuir isotherm		
q _{max}	35.7	50.0
d	0.019	0.051
R ²	0.970	0.976
Freundlich isotherm		
n	2.18	2.17
K _F	2.52	5.32
R ²	0.744	0.972
Temkin isotherm		
B	7.24	8.56
A	0.24	0.95
R ²	0.881	0.940

Table 3. Langmuir dimensionless constant (K_L) of pristine and modified biochar

C ₀ (mg/L)	P-WBC	H ₃ PO ₄ -WBC
100	0.344	0.163
200	0.208	0.089
300	0.149	0.061
400	0.116	0.046
500	0.095	0.037
600	0.080	0.031
700	0.069	0.027
800	0.061	0.003
900	0.055	0.021
1000	0.050	0.019

3.2.7 Kinetics study

Kinetics study determines the rate of chemical processes that aids in assessing the performance of adsorption. It will aid in working of the batch process in a better manner and to design and model the process efficiently by regulating the rate and equilibrium time [34]. Pseudo first order and pseudo second order kinetics were studied and their respective datas were explained in Table 4. Pseudo R² value for

pseudo first order is 0.257 and 0.635 for P-WBC and H₃PO₄-WBC respectively. R² value for pseudo second order is 0.993 for P-WBC and 0.998 for H₃PO₄-WBC (Fig. 8). As R² value supports the validity of the kinetics, adsorption is governed by the pseudo second order kinetics. Moreover the pseudo second order shows closeness of calculated value towards the experimental value, it is strongly recommended [25]. The q_e values of H₃PO₄-WBC is higher than the q_e values of P-WBC. This might be due to the increased functional groups such as -COOH and -OH resulting in greater interaction between Cr (VI) and H₃PO₄-WBC [37].

Table 4. Kinetics constants of the biochar

Constants	P-WBC	H ₃ PO ₄ -WBC
Pseudo first order		
(q _e) _{exp}	9.12	9.95
(q _e) _{cal}	1.487	6.958
K ₁	0.035	0.093
R ²	0.257	0.635
Pseudo second order		
(q _e) _{exp}	9.12	9.95
(q _e) _{cal}	9.398	10.660
K ₂	0.045	0.027
R ²	0.993	0.998
Elovich		
a	6.586 x 10 ²	3.209 x 10 ²
b	1.157	0.956
R ²	0.635	0.903
Intra particle diffusion		
C _i	7.006	7.455
K _{id}	0.299	0.374
R ²	0.573	0.873

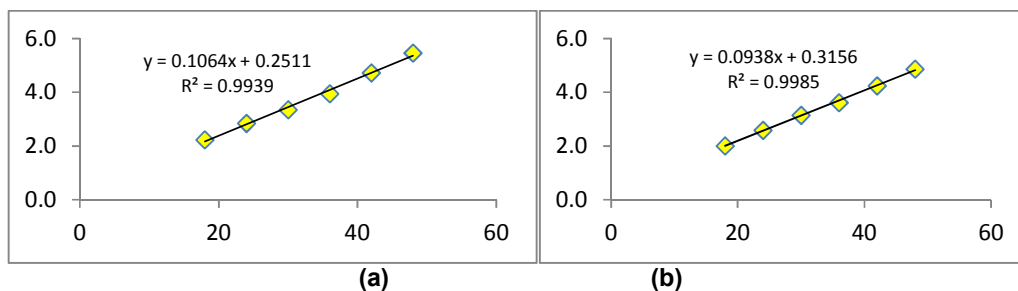


Fig. 8. Pseudo second order kinetics plots of a) P-WBC, b) H₃PO₄-WBC

Elovich rate of reaction was also studied to in order to support the adsorption phenomena. But the R^2 value is lower in comparison to the pseudo second order kinetics. The diffusion mechanism was investigated by employing the obtained data in intra-particle diffusion model. Though the R^2 value is not much higher the intra particle diffusion model is suggested as the experimental q_e values are quite near to the calculated q_e values. Multiple linear plots were obtained from the plots of diffusion model. In general, the intra particle diffusion model was assumed by three phases. Faster adsorption is enhanced by the outer surface at the initial stage succeeded by the intra particle diffusion stage. At last the diffusion step is lowered by the slower adsorption stage where equilibrium is attained at the condition where the adsorbate molecules resided in the solution are lesser [34].

3.3 Summary

Generally acid modification reveals a greater number of H/C, O/C and N/C groups increasing the "O" content in comparison to the other modifications. It leads to increased hydrophilicity. The adsorption capacity is also enhanced by the micropores and specific surface area. Moreover, the addition of more functional groups such as hydroxyl, carboxylic, ketonic and other oxygen containing moieties improved the morphology of the H_3PO_4 -WBC [38,39]. Increased micropores generation by addition of H_3PO_4 may be the result of prevention of contraction of pores due to phosphate and polyphosphate bridges and destruction of aliphatic, aromatic and lignocellulosic materials [40]. Hence activation of water hyacinth biochar with phosphoric acid showed maximum chromium adsorption than pristine biochar.

4. CONCLUSION

The study arrives the conclusion that the adsorption mechanism was improved by modification of biochar with phosphoric acid as the increased H^+ ions allure the hexavalent Cr ions strongly. The data was well fitted with Langmuir isotherm explaining the chemisorption on a homogeneous substrate. Kinetics of adsorption is highly influenced by the pseudo second order kinetics proceeded by the chemical reactions in the initial stage followed by pore diffusion and ultimately by the equilibrium stage. The vast availability and its severe toxicity will make the water hyacinth to be utilized in a useful manner as an adsorbent. Hence it was chosen to

remediate the heavy metal from the polluted water.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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