

A comparative study on the efficiency of KOH and H₃PO₄ impregnated jackfruit leaf based carbon as adsorbent for removal of Cr (VI) from its aqueous solution

Merlin Thomas *,Sagar P. Patel, Ankit V. Patel, Jigar V. Patel

Department of Industrial Chemistry, Institute of Science & Technology for Advanced Studies & Research (ISTAR), Vallabh Vidyanagar, Gujarat - 388120, India.

Abstract— This research work investigates the application of KOH and H₃PO₄ impregnated jackfruit leaf powder to remove Cr (VI) from wastewater. The removal characteristics of Cr (VI) from wastewater was investigated under various operating variables like contact time, adsorbent dosage, initial metal concentration, solution pH, temperature and stirring speed. The results showed that the H₃PO₄ impregnated jackfruit leaf powder is a better adsorbent for Cr (VI) from waste water than KOH impregnated jackfruit leaf powder. The experimental results were found to fit well with Langmuir and Freundlich isotherms. The maximum adsorption capacity of the KOH impregnated jackfruit leaf powder was found to be 62.7 mg/g and that of H₃PO₄ impregnated jackfruit leaf powder was found to be 68.37 mg/g.

Keywords — Cr (VI), adsorption, Jackfruit leaf powder, adsorption isotherm

I. INTRODUCTION

Drinking water scarcity and the demand for non-contaminated water for daily consumption have been cumulatively increased due to rapid industrialization. Water of high quality is essential to human life and water of acceptable quality is essential for agriculture, industrial, domestic and commercial activities. The release of toxic heavy metals like Cr(VI) and Ni into water has been a major challenge to environmental engineers today due to the pollution of water. Removal of these from water is necessary because of the frequent appearance of these metals in waste streams. Priority should be given to regulate the concentration of these pollutants at the discharge level. Different sources of Cr(VI) pollution include effluents from mining, electroplating, dye & plastic industries, leather tanning, metallurgical, electroplating, battery and chemical manufacturing industries. Cr(VI) refers to chemical compounds that contain the element chromium in the +6 oxidation state.

Currently methods like Chemical precipitation, coagulation, ion exchange, solvent extraction, and membrane filtration are used for the removal of these contaminants from industrial effluents. But these methods produce large amounts of sludge and waste that need to be disposed off and can consequently cause a lot of problems to the environment due to the presence of high content of contaminants.

Adsorption which uses a strong and selective interaction of adsorbent with adsorbate has gained attention as a purification, separation and recovery process on an industrial scale since the last few years. Adsorption using activated carbon has been gaining momentum. Various natural adsorbents have been tried successfully for the removal of pollutants from water. Adsorption by natural adsorbents, especially waste materials as adsorbent not only solves the problem of waste disposal and also converts a potential waste to a valuable product. Any cheap material with a high carbon content, low inorganics can be used as a raw material for the production of activated carbon [1]. Low cost adsorbents made from agricultural wastes like coconut shells, almond shells, rice husk and date pits [2-8] are found to be effective in the removal of pollutants from water.

Both physical activation and chemical activation are widely used for the production of Activated Carbon [9]. Carbonisation of raw materials at high temperatures (between 800 and 1100 °C) in the presence of gases like carbon dioxide or steam is done in physical activation [10-15]. These agents burn off the pore blocking structures created during the carbonization phase and so, they develop a porous, three-dimensional graphite lattice structure. In chemical activation, pyrolyzed at moderate temperatures in the absence of air is done after the mixing of a chemical agent with the precursor [16-23]. Carbonization and activation are carried out simultaneously in chemical activation. The disorganized carbon is removed by the activation by exposing the crystallites to the action of activating agent which leads to the development of porous structure. Though it is the porous structure

of the activated carbon which determines its adsorption capacity, it is also strongly influenced by a relatively small amount of chemically bonded heteroatoms (mainly oxygen and hydrogen). The various properties which are related to the high adsorption capacities of activated carbon are surface area, pore volume and pore size distribution. These unique characteristics are functions of the type of raw materials employed for preparation of Activated Carbon and the method of activation.

The main objective of this study is to investigate the feasibility of using KOH and H₃PO₄ impregnated jackfruit leaf powder based carbon to remove Cr (VI) from wastewater. The choice of this material is based on its low cost, considering its abundance in in the tropical regions. Jackfruit seeds are rich in protein. The fruit is also rich in vitamin B₆, potassium, calcium, and iron. Carbonized Jackfruit leaf and jack fruit peel are already established as efficient adsorbents for basic dyes, Cd (II) and chlorophenols [24-29]. KOH and H₃PO₄ impregnated jackfruit leaf powder is already established as an effective adsorbent for removal of Nickel from waste water [30-31].

2. MATERIALS AND METHODS

Jackfruit leaves were collected locally and all the chemicals used were of LR grade.

2.1 Impregnation and Carbonization

Jackfruit leaves were collected, sun dried, crushed and ground in a ball mill. The ground sample was sieved to obtain the particles of uniform size of 0.3 – 0.8mm. The precursor obtained was washed to remove surface bounded impurities and mud and dried at 100°C for 12 h. This was impregnated with H₃PO₄ and KOH in varying impregnation ratios, holding time and carbonisation temperatures and the yield was determined. Impregnation ratio was varied from 10% to 50% for H₃PO₄ and 0.5 to 2.5 for KOH. Holding time of 30 min to 90 min and carbonisation temperature of 400°C to 700°C were tried and the yield of the resultant carbon determined. Impregnation ratios varying from 0.5 to 2.5 and holding time of 30 min to 90 min and carbonisation temperature of 400 °C to 700 °C were tried and the yield of the resultant carbon determined.

2.2 Porosity Determination

Porosity was determined using Mercury porosimeter (Model: Pascal-140, Pascal-440, Make: Thermo Quest). The technique involves the intrusion of mercury at high pressure into a material through the use of a porosimeter. The test is performed assuming cylindrical pore geometry. Porous materials are prone to absorb water and hence the experiment is done after removal of moisture.

Optimum values of carbonisation temperature, holding time and impregnation ratio were determined using the results of yield and porosity.

2.3. Proximate Analysis

Moisture Content, Volatile Matter, Ash Content and Fixed Carbon of the the prepared sample was determined using process described in IS 1350, 1984(Methods of test for coal and coke)

2.4 Surface Area determination

The particle size distribution and surface area of the prepared sample was determined using Particle size analyser (Model: Helos (H1004) & Suceil Make : Sympatec, German). Surface area is important to be measured in adsorption studies as it affects the adsorption capacity of adsorbents. For particles of small size, surface area will be high.

2.5 Study of Surface Morphology

The surface morphology was studied using Scanning Electron Microscope (Model: Nova Nano SEM 450 Make: FET Ltd) respectively.

SEM study is used to study the morphology and surface characteristics of adsorbent material. The pores present in activated carbon can be divided into three basic classes' viz., macropores transitional pores and micropores [32]. The effective radius of transitional pores usually falls within 40-200 Å and their surface area lies between 20-70 m²/g. Micropores, which have radius less than 20 Å usually amounts to at least 95 % of total surface area. Micropores are of greatest significance for adsorption due to their large surface area.

2.6 Adsorption Studies

Jack fruit leaf based KOH and H₃PO₄ impregnated carbon were used as adsorbents to carry out batch adsorption tests for removal of Cr (VI) from water. Stock solution of 100 mg/L of Cr (VI) was prepared by dissolving 282.9 gm of potassium dichromate in 1 L distilled water. The stock solution was then diluted as per requirement. The tests were carried out by varying adsorbent dosage, contact time, pH, temperature, initial Cr concentration and stirring rate. After the adsorption, the sample was filtered and the filtrate was checked for the Cr(VI) concentration using UV-Visible spectrophotometer(Model : Lambda19,Make : Perkin Elmer, U.S.A.) at 540 nM by complexing Cr (VI) with 1,5 –diphenylcarbazine. The effects of all the factors were studied as per Table 1.

Table 1: factors studied for their effect on adsorption efficiency

Adsorbent dosage(gm/100 ml)	0.2 -1
initial concentration of Cr(ppm)	1-10
pH	2-9
Contact time(min)	30-120
Temperature (°C)	30-55
rpm	200-500

2.7 The adsorption isotherm

The adsorption isotherm for Cr(VI) was studied using langumir and Freundlich isotherms using the optimum conditions.

Langumir isotherm is defined as $\left(\frac{C_e}{q_e}\right) = \frac{1}{b q_{max}} + \frac{C_e}{q_{max}}$ and the Freundlich isotherm is defined as $\log(q_e) = \log(k) + \frac{1}{n} \log(C_e)$ where q_{max} is the maximum adsorption capacity (mg/g), and b is the adsorption equilibrium constant (l/mg). C_e is the equilibrium concentration of metal ions, q_e is the amount of metal ions adsorbed per unit weight of adsorbent (mg/g). K and n represent the adsorption capacity and measure of heterogeneity respectively. The type of isotherm can be favourable if $1/n < 1$ and unfavourable if $1/n > 1$

3 RESULTS

3.1 selection of KOH & H₃PO₄ impregnation ratio, holding time and carbonisation temperature

The results of effect of KOH and H₃PO₄ impregnation ratio on the yield is shown in table 1, effect of holding time and carbonisation temperature on yield & porosity given in table 2 to 4 for H₃PO₄ and KOH impregnated samples. The transformation from lignocellulosic materials into carbon involved releasing of O and H atoms as H₂O, CO, CO₂, CH₄ and distillation of tar. The drop in yield after a certain range of impregnation ratio may be due to enhancement of carbon burn off by extra amount of extra amount of H₃PO₄ and KOH. The % yield decreased with increase in temperature due to carbon burn off and tar volatilization at higher temperatures.

Table 2: Effect of Impreg. Ratio on Yield

H ₃ PO ₄	% yield	KOH	% yield
10%	56%	0.5	75.5
20%	62%	1	78.6
30%	71.5%	1.5	80.3
40%	71%	2	83.3
50%	70.5%	2.5	81.1

Table 3 :Effect of Holding Time and Carbonisation Temp.on Yield

min	Carbonisation temperature in °C					
	400		500		600	
	H ₃ PO ₄ (%)	KOH (%)	H ₃ PO ₄ (%)	KOH (%)	H ₃ PO ₄ (%)	KOH (%)
30	56	55.2	46.5	52.4	38.2	36.2
60	52.3	53.4	43.5	51	36.1	35.6
90	48.4	52.6	40.5	49.6	34.3	35

The effect of carbonization temperature on the porosity of samples is given in tables 4.

Table 4: Effect of Carbonisation Temperature on porosity of H₃PO₄ impregnated adsorbent

Carbonisation temperature in °C	Holding time in min	Porosity (%)	
		H ₃ PO ₄	KOH
400	30	50.7	22.8
500	30	44.3	25.49
600	30	40.06	26.3

From the study of table 2 to 4, the impregnation ratio, holding time and carbonisation temperature were fixed as shown in table 5.

Table 5: Selected values of impreg. Ratio, holding time and carbonisation temperature

	KOH	H ₃ PO ₄
Impregnation ratio	2	30 %
Holding time(min)	30	30
Carbonisation temperature(°C)	600	400

3.2 Proximate Analysis

Low amount of moisture, ash and volatile matter, indicate that the particle density is relatively small and that the biomaterial should be an excellent raw material for adsorbent. Ash content can also affect activated carbon as it reduces the overall activity of activated carbon. It also reduces the efficiency of reactivation, the lower the ash value therefore the better the activated carbon for use as adsorbent. The results of proximate analysis for both KOH and H₃PO₄ impregnated samples are given in table 6. The H₃PO₄ impregnated sample is found to have a fixed carbon value of 73.3 % as against 69.4 % for KOH impregnated sample.

Table 6: Proximate Analysis of the H₃PO₄ and KOH impregnated adsorbent

	H ₃ PO ₄	KOH
Moisture content	7.9 %	8 %
Volatile matter	18.1 %	19 %
Ash content	0.70%	3.6 %
Fixed carbon	73.3%	69.4 %

3.3 Surface area determination

The surface area of adsorbent was determined using particle size analyser. High surface area implies that the nature of the prepared activated carbon consists mainly of mesopores which are very favourable for adsorption of large molecules. The H₃PO₄ impregnated sample was found to have a total specific surface area of 66.77 m²/g as against 44.2 m²/g for KOH impregnated sample. The H₃PO₄ impregnated sample was found to have a total porosity of 50.74 % as against 36 % for KOH impregnated sample (table 7).

Table 7 : Surface area of H₃PO₄ and KOH impregnated sample

	H ₃ PO ₄	KOH
Total cumulative volume (cc/g)	.5897	.4227
Total specific surface area (m ² /g)	66.777	44.258
Average pore diameter (Micron)	.0845	0.72
Total porosity (%)	50.74	36.35
Bulk density (g/cm ³)	.8605	.62335
Apparent density (g/cm ³)	1.747	.84637
Sample volume correction	.9914	.9878

3.4 Morphology Study

Scanning electron micrographs of the surface morphology of the sample of the activated carbon impregnated with H₃PO₄ and KOH are given in Fig. 1 a & b . The SEM images show the adsorbents to be an effective. SEM image shows the presence of some wide pores which resulted due to chemical activation with the activating agents. This activation depends on the cellulose content of the adsorbent. Under the electrolytic action of the activating agent, the cellulose swells during which the arrangement of molecules in the direction of longitudinal axis remains unchanged as the lateral bonds are broken down with the result that inter and intra micelle voids increase.

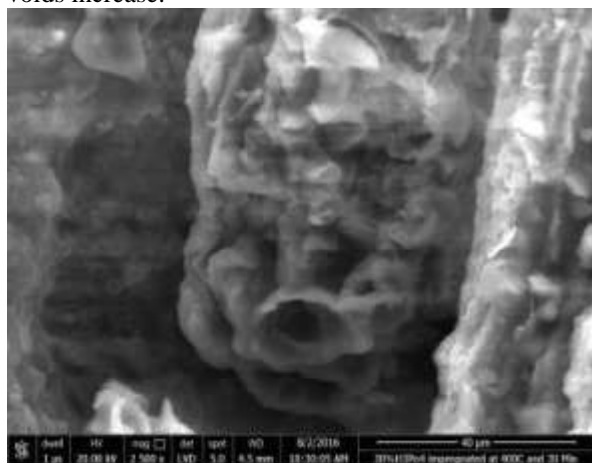


Fig. 1a :SEM of H₃PO₄ impregnated adsorbent

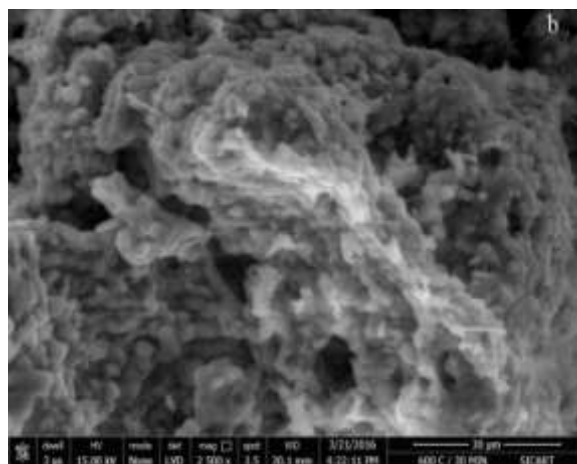


Fig 1b. SEM of KOH impregnated adsorbent

3.5 Adsorption Studies

3.5.1 Effect of adsorbent dosage on % adsorption

The adsorbent dosage was varied from 0.2 to 1 gm per 100 mL of solution, keeping all other parameters constant. The highest removal of Cr(VI) was observed as 99 % for H₃PO₄ impregnated sample and 97.4 % for KOH impregnated sample at 1m of adsorbent dosage (Fig 2) after which there was no substantial increase in Cr removal. As the dose of adsorbent increases, the number of adsorbent sites increases. This results in attachment of more ions to their surfaces.

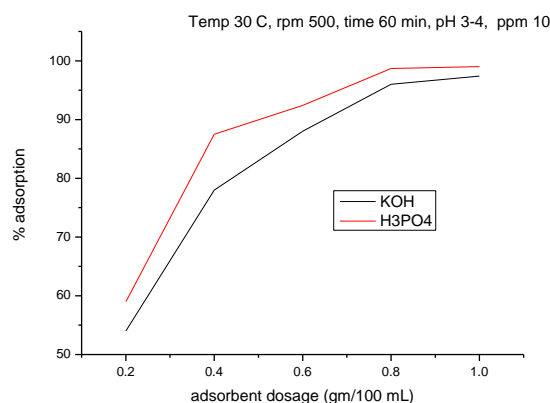


Fig 2: effect of adsorbent dosage- KOH & H₃PO₄ impregnation-

3.5.2 Effect of initial Cr (VI) concentration

Adsorption studies were conducted within the initial Cr concentration of 1 ppm to 10 ppm. At low Cr concentrations, sufficient adsorption sites are available for adsorption of Cr ions. At higher concentrations, relatively less available sites caused reduction in % adsorption (Fig 3). Increasing the initial Cr concentration decreased percentage removal of Cr from the solution due to the saturation of adsorbent with Chromium ions.

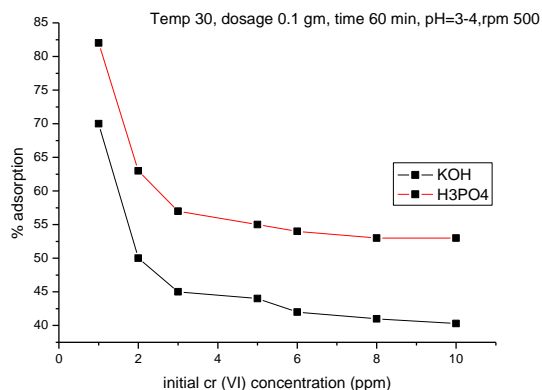


Fig 3: effect of initial Cr concentration- KOH & H₃PO₄ impregnation

3.5.3 Effect of contact time on % adsorption

Cr(VI) adsorption was studied by varying the contact time between the adsorbate and adsorbent in the range 30-120 min while keeping all other parameters constant. The data showed that the sorption of Chromium ions on jackfruit leaf powder based carbon was relatively better for H₃PO₄ impregnated adsorbent where the equilibrium was reached at 120 minutes in both the cases. A maximum adsorption of 99 % was observed in the case of H₃PO₄ impregnated sample while 97.6 % adsorption was observed in the case of KOH impregnated sample min (Figure 4). The adsorption process is fast at the beginning of the reaction due to the adsorption of Cr on the surface sites of adsorbent.

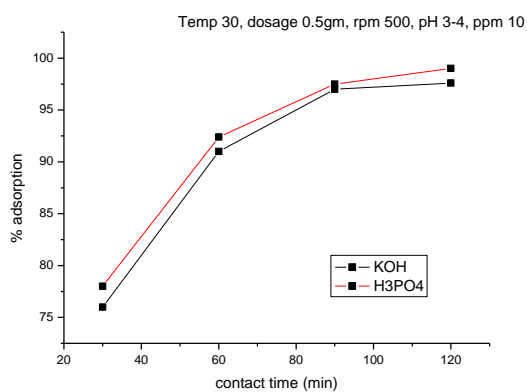


Fig 4: effect of contact time-KOH & H₃PO₄ impregnation

3.5.4 Effect of temperature on % adsorption

Adsorption studies were conducted between 30°C to 55°C. The adsorption efficiency was found to sharply increase from 30°C to 40°C and progressing slowly thereafter (Fig 5). There was no significant increase after 50°C. At higher temperatures, the rate of diffusion to the pores of adsorbent increases since

diffusion is an endothermic process. Thus the % adsorption increases at higher diffusion rate of Cr. Moreover, at low temperatures, the kinetic energy of Chromium species is low and hence contact between adsorbate and adsorbent is insufficient. Throughout the operation, H₃PO₄ impregnated adsorbent was found to perform better than that of KOH impregnated sample.

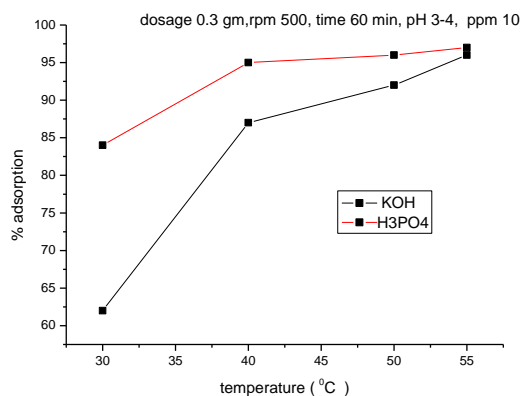


Fig 5: effect of temperature -KOH & H₃PO₄ impregnation

3.5.5 Effect of solution pH on % adsorption

Solution pH is an important variable which controls the adsorption of the heavy metal from wastewater. The effect of pH on the removal of Cr ions by H₃PO₄ and KOH impregnated jackfruit leaf based carbon powder was investigated. The adsorption of Cr was studied in the pH range 2–9, keeping all other parameters constant. The results (Fig 6) show that the adsorption of Cr decreased from 99 % to 89 % for H₃PO₄ impregnated adsorbent and from 97 % to 88 % for KOH impregnated adsorbent.

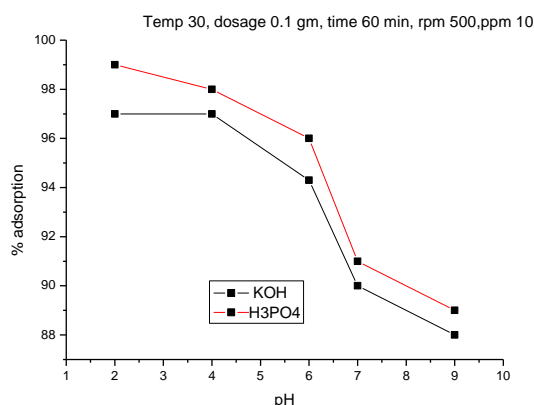


Fig 6: effect of pH -KOH & H₃PO₄ impregnation

3.5.6 Effect of rpm on % adsorption

The adsorption efficiency was observed to be low at small speeds whereas the maximum efficiency was observed at 500 rpm. The adsorption efficiency increases with the change in rpm from 200 to 500 rpm (fig 7). Due to agitation, proper contact was

developed between metal ion in solution and the binding sites, which promoted effective transfer of adsorbate ions onto the adsorbent sites. But the effect of rpm on adsorption efficiency is less as compared to other parameters for both H₃PO₄ and KOH impregnated samples. Variation in adsorption efficiency from 92-99 % was observed for H₃PO₄ impregnated adsorbent and 90-94 % was observed for KOH impregnated adsorbent.

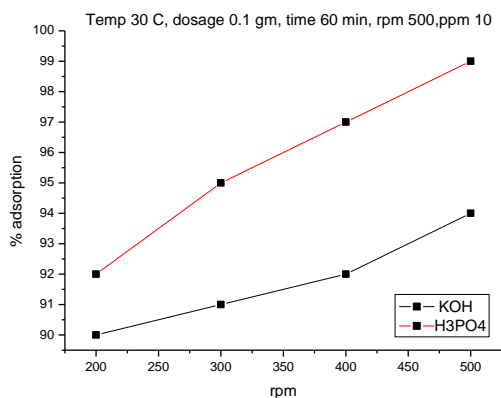


Fig 7: effect of rpm -KOH & H₃PO₄ impregnation

3.11 Adsorption isotherm

Isotherm parameters are given in table 8. Using Langmuir isotherm, the maximum adsorption capacity of the adsorbent was found to be 68.37 mg/g in the case of H₃PO₄ impregnated jackfruit leaf powder and 62.7 mg/g in the case of KOH impregnated adsorbent. n values between 1 and 10 for the Freundlich isotherm indicate effective adsorption[33]. n values of 2.28 and 2.4 were obtained for KOH impregnated and H₃PO₄ impregnated jackfruit leaf powder respectively. The experimental data in both the isotherms were found to fit well with R squared values above 98 %.

Table 8 Isotherm parameters

Langmuir Isotherm			
KOH		H ₃ PO ₄	
qmax	62.7	qmax	68.37
b	0.01	B	0.03
R squared	98 %	R squared	98.29 %
Freundlich Isotherm			
KOH		H ₃ PO ₄	
n	2.28	n	2.4
k	1.82	k	1.81
R squared	99.26 %	R squared	99.8 %

Conclusion

The results obtained in present study indicate the H₃PO₄ impregnated jackfruit leaf powder based carbon is a better adsorbent than KOH activated jackfruit leaf powder based carbon for removal of Cr (VI) from waste water. The yield of activated carbon impregnated by potassium hydroxide is lower than those activated with phosphoric acid at high temperature and so, the carbon content is less than fixed carbon. The presence of metallic potassium will intercalate to the carbon matrix, yielding lower yield of activated carbon, less than the carbon content of the raw material.

The amount of Cr(VI) adsorbed in both the cases was found to be heavily dependent on adsorbent dose, initial Cr concentration, temperature contact time and solution pH. The effect of rpm on adsorption efficiency was negligible. The sorption of Cr(VI) on the adsorbent was relatively fast with H₃PO₄ than KOH impregnated carbon. The maximum Cr(VI) adsorption capacity of H₃PO₄ impregnated jackfruit leaf based carbon was found as 68.37 mg/g whereas it was found to be 62.7 for KOH impregnated adsorbent

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