# A Comparative Study on the Efficiency of Koh Impregnated and H₃PO₄ Impregnated Jackfruit Leaf Based Carbon as Adsorbent for Removal of Ni from Its Aqueous Solution

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**Abstract:** This research work investigates the efficiency of $H_3PO_4$ and KOH impregnated jackfruit leaf powder based carbon as adsorbent to remove Ni from wastewater. The removal characteristics of Nickel using both the adsorbents were investigated under various operating variables like contact time, adsorbent dosage, initial metal concentration, solution pH, temperature and stirring speed. The results showed that the $H_3PO_4$ impregnated jackfruit leaf powder based carbon is a better adsorbent than the KOH impregnated adsorbent. The experimental results were found to fit well with langmuirand freundlich isotherms.The maximum adsorption capacity of the$H_3PO_4$impregnated adsorbentwas found to be 62.98 mg/g and that of the KOH impregnated adsorbent to be 54.66 mg/g.

**Key words:** Ni, adsorption, Jackfruit leaf powder, adsorption isotherm.

## I.     INTRODUCTION

Water is the greatest gift of nature. Increase in pollution of this natural resource has become a matter of grave concern recently due to increased release of toxic chemicals in water. Rapid industrialization and non-implementation of regulations have jointly aggravated the problem. Water of highquality is essential to human life and water of acceptable quality is essential for agriculture, industrial, domestic and commercial activities. So the development of new purification and extraction methods for removal of pollutants from water has become essential. Some of these techniques have been successful, but at great cost or with detrimental effects to environment. The release of toxic materials like Ni into water has been a major challenge to environmental engineers today due to the pollution of water. Priority should be given to regulate the concentration of these pollutants at the discharge level. Ni is a heavy metal used in a large no. of industries like electroplating, Ni-Cd batteries manufacturing, forging, mining [31] coal and oil-burning power plants, etc.[32]. The discharge of heavy metals in water has adverse effects on the health of humans, animals, plants and other organisms [27].The maximum contamination limit of Ni in drinking water is 0.1 mg/L, as set by United States Environmental Protection Agency.

Methods like Chemical precipitation, coagulation, ion exchange, solvent extraction, and membrane filtration were widely used for the removal of 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. This has led to the development of new adsorption techniques.

Over the last few decades, adsorption using activated carbon has gained importance as a purification, separation and recovery process on industrial scale. Adsorption by various 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. Cheap material with high carbon content and low inorganics can be used as a raw material for the production of activated carbon [6]. Low cost adsorbents made from agricultural wastes like coconut shells, almond shells, rice husk and date pits [5,8,13] are found to be effective in the removal of pollutants from water.

Production of AC is achieved typically through two routes, physical activation and chemical activation [14]. In physical activation, the raw material is carbonized at high temperatures (between 800 and 1100°C) in the presence of gases like carbon dioxide or steam [4,24,32,33]. 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, a chemical agent is mixed with the precursor and then pyrolyzedat moderate temperatures in the absence of air [10,20,21,26,28,29,34]. Carbonization and activation are carried out simultaneously in chemical activation. During activation,the disorganized carbon is removed by exposing the crystallites to the action of activating agent which leads to the development of porous structure. Along with the porous nature, the efficiency of activated carbon is also strongly influenced by a relatively small amount of chemically bonded heteroatoms (mainly oxygen and hydrogen) [7]. Properties such as surface area, pore volume

and pore size distribution influence the adsorption capacities of activated carbons. These unique characteristics are functions of the type of raw materials employed for preparation of AC and the method of activation. Impregnated activated carbons are carbonaceous adsorbents which have chemicals finely distributed on their internal surface. The impregnation optimizes the existing properties of the activated carbon giving a synergism between the chemicals and the carbon [15]. KOH and $H_3PO_4$ are widely used as an impregnating agents [23,25,30]. Porosity, pore size distribution, surface area and degree of surface reactivity are the important factors of the adsorbents which determine their efficiency [22].

The main objective of this study is to investigate and compare the efficiency of KOH and $H_3PO_4$ impregnated jackfruit leaf powder based carbon to remove Nickel from wastewater. The choice of this material is based on its low cost, considering its abundance in in the tropical regions. Conversion of jackfruit peel into activated carbon would increase its economic value, help reduce the cost of waste disposal, and provide a potentially inexpensive raw material for commercial activated carbon [25]. Jackfruit seeds are rich in protein. The fruit is also rich in vitamin $B_6$, potassium, calcium, and iron. Carbonised Jackfruit leaf and jack fruit peel are already established as efficient adsorbents for basic dyes , Cd (II) , Ni [9] and chlorophenols [2,14,16,17,18,19,30]. However, no work is reported about the use of KOH and $H_3PO_4$ impregnated carbonized jackfruit leaf as adsorbent for removal of Ni from aqueous solution.

## II.    MATERIALS AND METHODS

**Raw materials and chemicals**

Jackfruit leaves were collected from Anand Agricultural University, Anand (Gujarat, India). Potassium hydroxide, Nickle sulphate ($NiSO_4.6H_2O$) were purchased from Sigma (St. Louis, U.S.A). Ammonia, Hydrochloric acid, sodium hydroxide, phosphoric acid, Potassium Iodide, Iodine, Sodium Citrate and DMG (Dimethyl Glyoxime) were purchased from Hi-media (Mumbai, India). All the chemicals used in the present study were of analytical grade and of the highest purity available.

**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 100ºC for 12 h. This was impregnated with $H_3PO_4$ for 12h in varying impregnation ratios, holding time and carbonisation temperatures and the yield was determined. Impregnation ratio was varied from 10% to 50% for $H_3PO_4$ and 0.5 to 2.5 for KOH. Holding time of 30 min to 90 min and carbonisation temperature of 400ºC to 700ºC were tried and the yield of the resultant carbon determined.

**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. The most important variable to be controlled for getting proper porosity of activated carbon is the ratio of the chemical agent to the precursor [12]. Optimum values of carbonisation temperature, holding time and impregnation ratio were determined using the results of yield and porosity.

**Proximate Analysis**

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

**Surface Area determination**

The particle size distribution and surface area of the prepared sample was determined using Particle size analyser (Model: Helos (H1004) &Sucell 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.

**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 [7]. Pores of effective radius greater than 500- 1000 Å are classified as macropores. In activated carbon, the effective radius of macropores frequently lies in the range of

5000-20000 Å having surface area ranging between 0.5 and 2.0 $m^2/g$. The effective radius of transitional pores usually falls within 40-200 Å and their surface area lies between 20-70 $m^2/g$. 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 [1].

**Adsorption Studies**

Jack fruit leaf based KOH and $H_3PO_4$ impregnated carbon were used as adsorbents to carry out batch adsorption tests for removal of  Nickel  from water. Stock solution of 100 mg/L of  Ni was prepared by dissolving 447.8 mg of  Nickel Sulphate ( $NiSO_4.6H_2O$) in 1L 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 Ni concentration and stirring rate. After the adsorption, the sample was filtered and the filtrate was checked for the Ni concentration using UV-Visible spectrophotometer ( Model : Lambda19,Make : Perkin Elmer, U.S.A.) at 540 nm by complexing Ni with DMG (Dimethyl Glyoxime). The effects of all the factors were studied as per Table 1.

**The adsorption isotherm**

The adsorption isotherm for Ni was studied using Langmuir and Freundlich isotherms using the optimum conditions.

Langmuir 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 qe=log K + (1/n) log Ce where $q_{max}$ is the maximum adsorption capacity (mg/g), and b is the adsorption equilibrium constant (l/mg). Ce is the equilibrium concentration of metal ions, qe 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.

## III.    RESULTS AND DISCUSSION

**Selection of KOH and H₃PO₄ impregnation ratio, holding time and carbonisation temperature.**

The results of effect of $H_3PO_4$  and KOH impregnation ratio on the yield is shown in table 2, effect of holding time and carbonisation temperature on yield & porosity given in table 3 a&b and 4 a &b  respectively. The transformation from lignocellulose materials into carbon involved releasing of O and H atoms as $H_2O$, CO, $CO_2$, $CH_4$ 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 $H_3PO_4$ andKOH. The % yield decreased with increase in temperature due to carbon burn off and tar volatilization at higher temperatures.

From the study of table 2, 3a & b and 4 a & b, the impregnation ratio, holding time, carbonisation temperature were fixed as shown in table 5.

**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.

**Surface area determination**

The total specific surface area of $H_3PO_4$ and KOH adsorbents were determined as 66.777$m^2/g$and14.258 $m^2/g$ respectively (Table7a & b) 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.

**Morphology Study**

Scanning electron micrographs of the surface morphology of the sample of the activated carbon impregnated with $H_3PO_4$ and KOH are given in Fig. 1 a & b. The SEM image shows the adsorbent to be an effective one. SEM image shows the presence of some wide pores which resulted due to chemical activation with $H_3PO_4$ and KOH. 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.

**Effect of adsorbent dosage on % adsorption**

The adsorbent dosage was varied according to data in table 1, keeping all other parameters constant. The highest removal of Ni was observed at 0.07gm (98 %) for $H_3PO_4$ impregnated sample and  0.15gm (96 %)

for KOH impregnated sample (Fig 3) after which there was no substantial increase in Ni removal. As the dose of adsorbent increases, the number of adsorbent sites increases. This results in attachment of more ions to their surfaces [1].

**Effect of contact time on % adsorption**

Ni 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 Nickel ions on jackfruit leaf powder based carbon was relatively fast for $H_3PO_4$ impregnated adsorbent where the equilibrium was reached after 80 min (Figure 4). The adsorption process is fast at the beginning of the reaction due to the adsorption of Ni on the surface sites of adsorbent.

**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 Ni ions by $H_3PO_4$ and KOH impregnatedjackfruit leaf based carbon powder was investigated. The adsorption of Ni was studied in the pH range 2–11, keeping all other parameters constant. The results (Fig 5) show that the adsorption of Ni decreased from 98 % to 88 % for $H_3PO_4$ impregnated adsorbent and from 95.3 % to 86 % for KOH impregnated adsorbent.

**Effect of initial Ni concentration) on % adsorption**

Adsorption studies were conducted within the initial Nickel concentration of 2 ppm to 11 ppm. At low Ni concentrations, sufficient adsorption sites are available for adsorption of Ni ions. At higher concentrations, relatively less available sites caused reduction in % adsorption (Fig 6). Increasing the initial Ni concentration decreased percentage removal of Ni from the solution due to the saturation of adsorbent with Ni.

**Effect of temperature on % adsorption**

Adsorption studies were conducted between 28ºC to 50ºC. The adsorption efficiency was found to sharply increase from 28ºC to 35ºC and progressing slowly thereafter (Fig 7). There was no significant increase after 45ºC. 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 Nickel. Moreover, at low temperatures, the kinetic energy of Nickel species is low and hence contact between adsorbate and adsorbent is insufficient.

**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 550 rpm (fig 8). 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 minimum as compared to other parameters.

**Adsorption isotherm**

Isotherm parameters are given in table 8. The maximum adsorption capacity of the adsorbent was found to be 66.98 mg/g for $H_3PO_4$ and 54.66 mg/g for the KOH impregnated sample using Langmuir isotherm, n values between 1 and 10 for the Freundlich isotherm indicate effective adsorption [27]. The experimental data fits well into both the isotherms with R squared values of 97.2 and 99.7 respectively for $H_3PO_4$and KOHforLangmuir isotherm and R squared values of 98.76 and 99.28 respectively for $H_3PO_4$and KOHFreundlich isotherms.

## IV. CONCLUSIONS

The results obtained in present study indicate the $H_3PO_4$ impregnated jackfruit leaf powder based carbon is a better adsorbent than KOH activated jackfruit leaf powder based carbon though both can be successfully used for the removal of Nickel from aqueous solutions. 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 [14], yielding lower yield of activated carbon, less than the carbon content of the raw material.

The amount of Nickel adsorbed in both the cases was found to be heavily dependent on solution pH, adsorbent dose and initial Ni ppm, temperature and contact time. The effect of rpm on adsorption efficiency was negligible. The sorption of Nickel on the adsorbent was relatively fast with $H_3PO_4$ than KOH impregnated carbon.

The optimum conditions of above parameters were tested with Freundlich and Langmuir isotherm models. Based on R squares values obtained from different isotherm models, it was found that both Freundlich and Langmuir isotherms gave good data fitting for both $H_3PO_4$ than KOH impregnated jackfruit leaf based carbon with R squared values above 97. The maximum adsorption capacity of the $H_3PO_4$ impregnated jackfruit leaf powder was found to be 62.98 mg/g and that with KOH impregnated jackfruit leaf powder was found to be 54.66 mg/g.
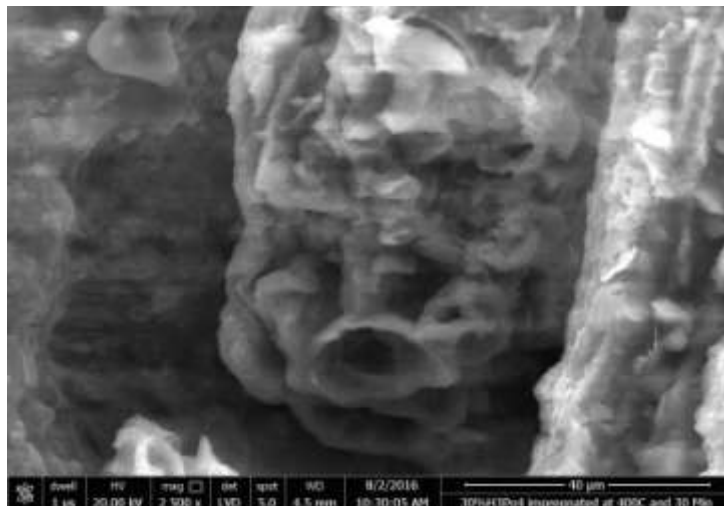


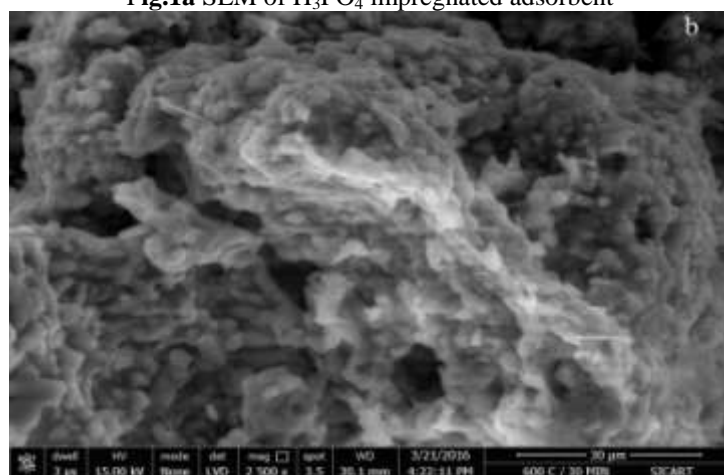F**ig.1a** SEM of $H_3PO_4$ impregnated adsorbent



**Fig 1b.** SEM of KOH impregnated adsorbent
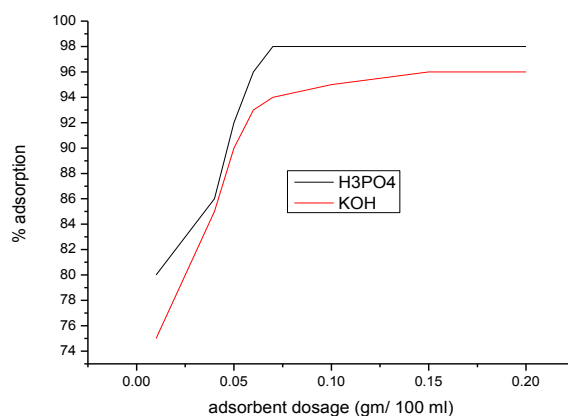


**Fig. 3** effect of adsorbent dosage on % adsorption
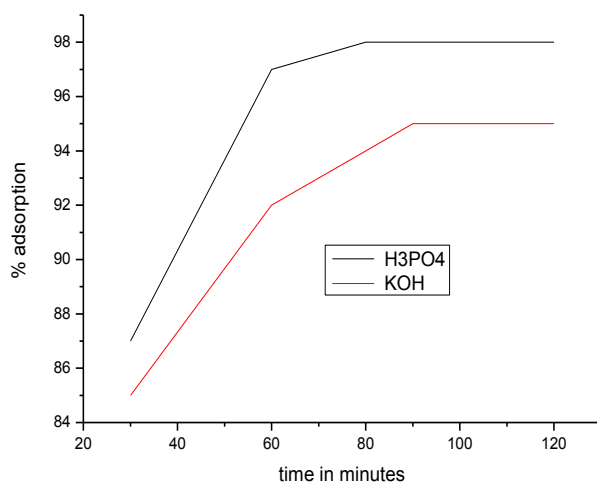
**Fig 4.** Effect of contact time on % adsorption
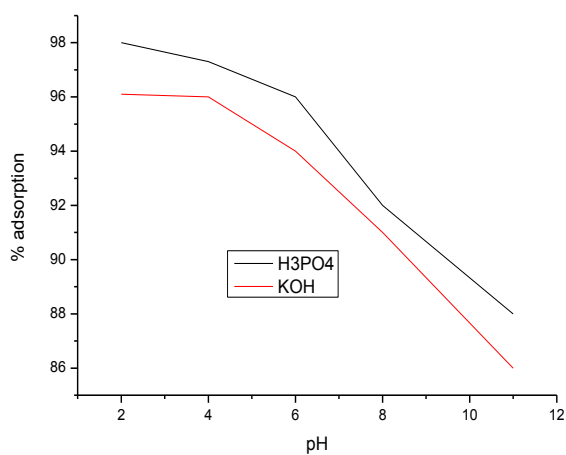
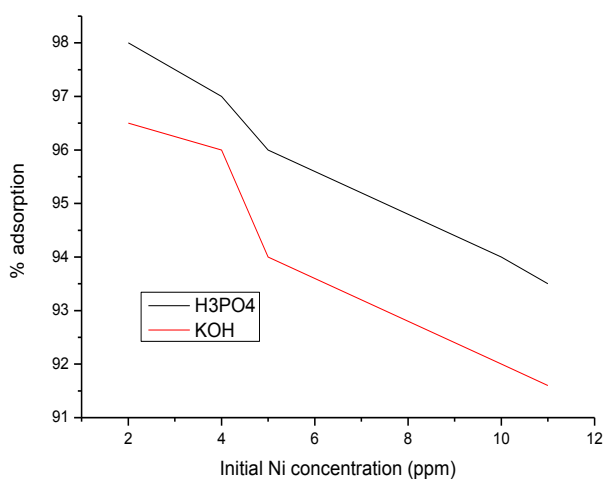

**Fig 5.** Effect of pH on % adsorption



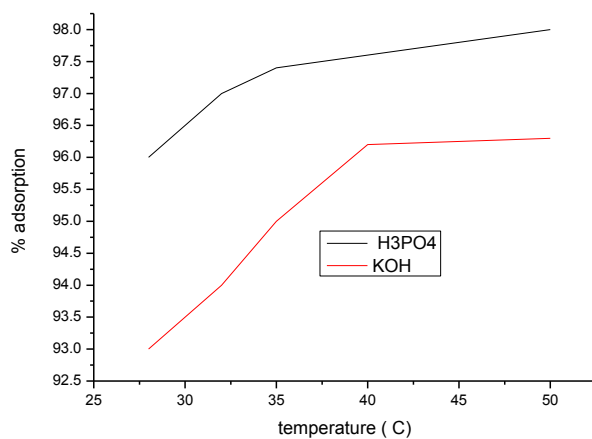**Fig. 6** Effect of initial Ni concentration on % adsorption

**Fig. 7**Effect of
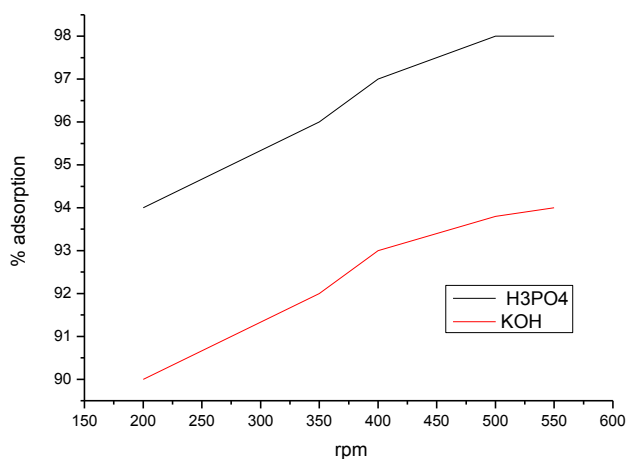


**Fig. 8**Effect of rpm on % adsorption temperature on % adsorption

**Table 1**

| Variable | Value |
|---|---|
| Adsorbent dosage | 0.01 -0.2gm/100 ml |
| Initial concentration of Cr(VI) | 2-11 ppm |
| pH | 2-11 |
| Contact time | 30-120 min |
| Temperature | 28-50$^{\circ}$C |
| Rpm | 200-550 |

**Table 2** Effect of Impregnation Ratio of H₃PO₄ and KOH on Yield

| Sr. No. | H₃PO₄ impregnation ratio | % Yield | KOH impregnation ratio | % Yield |
|---|---|---|---|---|
| 1 | 10% | 56% | 0.5 | 75.5 |
| 2 | 20% | 62% | 1 | 78.6 |
| 3 | 30% | 71.5% | 1.5 | 80.3 |
| 4 | 40% | 71% | 2 | 83.3 |
| 5 | 50% | 70.5% | 2.5 | 81.1 |

**Table 3a** Effect of Holding Time and Carbonisation Temperature of 30% H₃PO₄ on Yield

| Holding time in min | Carbonisation temperature in $^{\circ}$C | | |
|---|---|---|---|
| | 400 | 500 | 600 |
| 30 | 56 % | 46.5% | 38.2% |
| 60 | 52.3% | 43.5% | 36.1% |
| 90 | 48.4% | 40.5% | 34.3% |

**Table 3b** Effect of Holding Time and Carbonisation Temperature of KOH on Yield

| Holding time in min | Carbonisation temperature in °C | | | |
|---|---|---|---|---|
| | 400 | 500 | 600 | 700 |
| 30 | 55.2 | 52.4 | 51.1 | 36.2 |
| 60 | 53.4 | 51 | 49.2 | 35.6 |
| 90 | 52.6 | 49.6 | 47.6 | 35 |

**Table 4a** Effect of Carbonisation Temperature on porosity of H$_3$PO$_4$ impregnated adsorbent

| Carbonisation temperature in °C | Holding time in min | Porosity (%) |
|---|---|---|
| 400 | 30 | 50.7 |
| 500 | 30 | 44.3 |
| 600 | 30 | 40.06 |

**Table 4b** Effect of Carbonisation Temperature on porosity of KOH impregnated adsorbent

| Carbonisation temperature in °C | Holding time in min | Porosity (%) |
|---|---|---|
| 400 | 30 | 22.8 |
| 500 | 30 | 25.49 |
| 600 | 30 | 26.3 |

**Table 5** Selected values of impregnation ratio, holding time and carbonisation temperature

| | KOH | H$_3$PO$_4$ |
|---|---|---|
| Impregnation ratio | 2 | 30 % |
| Holding time | 30 | 30 |
| Carbonisation temperature | 600 | 400 |

**Table 6** Proximate Analysis of the 30 % H$_3$PO$_4$ and KOH impregnated Sample

| | 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 % |

**Table7a :** 30% H$_3$PO$_4$ Impregnated sample

| | |
|---|---|
| Total cumulative volume (cc/g) | .5897 |
| Total specific surface area (m$^2$/g) | 66.777 |
| Average pore diameter (Micron) | .084502 |
| Total porosity (%) | 50.7453 |
| Bulk density (g/cm$^3$) | .8605 |
| Apparent density (g/cm$^3$) | 1.74704 |
| Sample volume correction | .9914436 |

**Table 7b:** KOH Impregnated sample

| | |
|---|---|
| Total cumulative volume (cc/g) | .4227 |
| Total specific surface area (m$^2$/g) | 14.258 |
| Average pore diameter (Micron) | 0.720006 |
| Total porosity (%) | 26.3506 |
| Bulk density (g/cm$^3$) | .62335 |
| Apparent density (g/cm$^3$) | .84637 |
| Sample volume correction | .9878572 |

**Table 8** Isotherm parameters

| KOH Impregnation | | | H$_3$PO$_4$ Impregnation | |
|---|---|---|---|---|
| Ce | Qe | | Ce | Qe |
| 10 | 11.11 | | 11 | 15.876 |
| 20 | 18.18 | | 19 | 23.333 |
| 40 | 26.66 | | 41 | 30.666 |
| 60 | 33.33 | | 59 | 39.12 |
| 80 | 36.36 | | 81 | 45.43 |
| **Langmuir Isotherm** | | | | |
| R squared value | 99.77 % | | Rsquared value | 97.2 % |
| qmax | 54.66 | | Qmax | 62.98 |
| b | 0.025 | | B | 0.028 |

| Freundlich Isotherm | | | |
|---|---|---|---|
| n | 1.72 | N | 1.97 |
| k | 3.01 | K | 1.99 |
| R squared value | 99.28 % | Rsquared value | 98.76 % |

## Acknowledgements

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