Removal of Heavy Metals from Aqueous Solution Using Ion Exchange Resin MBHPE-TKP

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Abstract— The aim of this study is to synthesis of TKP (MBHPE-TKP) resin for the removal of heavy metals from aqueous solution. Ion exchange resins are polymers that are capable of exchanging particular ions within the polymer with ions in a solution that is passed through them. This ability is also seen in various natural systems such as soils and living cells. The synthetic resins are used primarily for purifying water, but also for various other applications including separating out some elements. Factorial design of experiments is employed to study the effect of above factors pH, time and sorbent used. The new synthesized resins i.e. MBHPE–TKP is hydrophilic and biodegradable, so after effluent treatment used resins can be disposed off without facing any environmental problem .This study focuses on synthesis of new cation exchange resin (MBHPE – TKP) and developing method for treatment of highly contaminated industrial effluents.

Key words: TKP (MBHPE-TKP) resin, hydrophilic, biodegradable, water purification, adsorption.

I. INTRODUCTION

A. *Heavy metals in waste water:*

Heavy metals are often problematic environmental pollutants, with well-known toxic effects on living systems. Nevertheless, because of certain useful physical and chemical properties, some heavy metals, including silver, cobalt, cadmium, copper, nickel, Pb^{2+}, Zn^{2+} etc. are intentionally added to certain consumer and industrial products such as batteries, electroplating. Cobalt was once widely used in pharmaceutical products and agricultural chemicals while cesium has taken more importance after nuclear reactor construction has been expanded. Large amount of any of them may cause acute or chronic toxicity.

Since copper is a widely used material, there are many actual or potential sources of copper pollution .Copper may be found as contaminant in food, especially shellfish, liver, mushrooms, nuts, and chocolate. Briefly, any processing or container using copper material may contaminate the product such as food, water or drink. Copper is essential to human life and health but, like all heavy metals, is potentially toxic [2]

The presence of heavy metals in both surface and underground water has led to increase environmental concern by both scientists and engineers. One of such heavy metals is Cd (II) which is non-essential for human consumption. Cadmium, Cd (II) has an extremely long biological half-life (greater than 20 years) and is listed as one of the 126 priority contaminants known as 'carcinogen' by the International Agency for Research on Cancer. Cadmium is released through natural processes such as volcanic activity. Its release meets water bodies directly through the effluent of industries causing a mark increase in its concentration [14]

B. *Heavy Metals Pollution:*

The toxicity of heavy metals lays in the fact, that almost all of them have the ability to bind with cysteine, and cause protein denaturation. Some of them, for example copper, have additional affinity for histidine. From the cellular point of view, heavy metals are toxic and unneeded in the concentration present in polluted waste water. Almost all living cells, despite their origins, possess cellular mechanisms that help them to keep the heavy metal ions from damaging their system. Mainly, specially designed proteins called metalloproteinase are involved in the removal of heavy metals from the cell. Metalloproteinase are sulfur rich proteins, containing many cysteine residues which irreversibly binds metals to the protein. Usually the metal-bonded protein is useless for the organism, but in many organisms there is no possibility for the cell to remove such proteins out of it. The accumulation of toxic metal is unavoidable, and after reaching certain level of concentration, the cell dies. The following section lists out the most popular polluting heavy metals. Copper – the most widespread anthropogenic pollutant. The industry of copper, includes wires and brasses production. In normal, healthy organisms traces of copper are necessary for enzymes in oxidative-reduction reactions. The higher levels of copper enhance the reactions of reactive oxygen species, which damage the cytoplasm by oxidating the proteins. Copper also has high affinity for methionine, cysteine and histidine, and thus blocks the active sites of proteins, containing these three amino acids [2]. Mercury – the main source of mercury is mercury production itself, gold production, coal combustion, caustic soda and cement production, pig iron, steel and non-ferrous metals smelting and others. In the nineteenth century mercury was widely used as an agent for softening materials, especially for hats (nota bene Mad Hatter character from Alice in the Wonderland). The toxicity of mercury lays in the fact that this metal mainly exists in form of easily evaporating liquid. The vapors of mercury are easily penetrating the mucus membranes, respiratory system and skin. The mercury is one of the strongest neurotoxins. It devastates the neural system, kidneys and endocrine system. Because of the pathway of intruding the organism, the tongue, teeth and gums are also affected. Long exposition to mercury vapors causes serious brain damage and death. Furthermore, more toxic than mercury itself are metallorganic compounds of mercury, namely dimethylmercury. These compounds are easily fat soluble, and accumulate in bodies of animals, especially fish. Eating fish may lead to acute mercury poisoning and death .Lead – commonly used as an absorbent of high-energy radiation. For years, tetraethyl lead has been used as an anti-knocking agent in gasoline for car engines. Lead bullets have been in use up to nineties of last century. White paint, which is present in many old houses is also a very efficient source of toxic lead. Lead poisoning (also called plumbism) is connected with toxicity of high levels of lead, which interferes with many processes in blood and organs. Lead is poisonous for the heart, kidneys, bones, reproductive and nervous systems. The symptoms of lead poisoning come mainly from the nervous system, i.e. insomnia, delirium, hallucinations and convulsions. Other symptoms are abdominal pain, headache, encephalopathy and kidney failure. Not cured plumbism may lead to death .Cadmium – The most significant source of cadmium in the environment is cadmium mining and nickel-cadmium batteries. Cadmium has toxic influence mainly on respiratory system, bones and kidneys. It can cause fatal renal failure, osteoporosis, osteomalacia, and several lungs diseases. The characteristic symptoms of many patients, who were exposed to cadmium is the loss of the sense of smell.

Nickel – The industrial use of nickel is mainly stainless steel. The other applications are magnets, other than steel alloys and coinage. In organisms, the enzyme urease contains nickel in an active state. This metal is known as cancerogene, especially when inhaled as dust. Many people are allergic to nickel, which appears after the contact with this metal. The symptoms are itchy, red skin and rash over the place of contact. Cobalt – Almost all produced cobalt is consumed for alloys that can be used in turbines, and other equipment that needs high-performance materials. Living organisms need traces of cobalt in the form of cobalamin (vitamin B12).Intoxication with cobalt causes anemia that in many cases is lethal . Bismuth – mainly used in electronics as this metal itself and its alloys are good semiconductors. Bismuth is far less toxic than lead, as does not tend to accumulate, except for gums and kidneys. Thus, it can cause disorder of these organs. There is no evidence of lethal bismuth poisoning. Arsenic – This metalloid is used as wood preserver, green pigment and a part of medicine for syphilis cure (now is banned in many countries). Poisoning with arsenic causes heart disorders, cardiovascular failure and death .Chromium – this element is mainly used in smelting, stainless, and durable steel. The main application in metallurgy is chromium films, which protect other metals against rust. Other applications are yellow dye production, leather tanning (main source of chromium in wastewater) and as a catalyst in chemical reactions. Chromium is a very poisonous and cancerogenic metal. Small amounts of chromium in drinking water can cause cancer.

New Technologies for Removal of Heavy Metals from Wastewater:

- Current Wastewater Treatment Process - Heavy Metals Removal
- Metal Removal from Wastewater Using Peat
- A Novel Method for Heavy Metal Removal Using Fish Scales
- Seashells for Heavy Metals Clean-Up
- Toxic Heavy Metal Ions Removal from Waste Water by Membrane Filtration Of PGA-Based Nanoparticles
- Novel Biofiltration Methods for the Treatment of Heavy Metals from Industrial Wastewater
- Removal of Heavy Metals from Industrial Wastewaters by Adsorption onto Activated Carbon Prepared from an Agricultural Solid Waste
- Physico–Chemical Treatment Techniques for Wastewater Laden with Heavy Metals
- Microbial and Plant Derived Biomass for Removal of Heavy Metals from Wastewater
- Removal of Heavy Metals from Wastewater by Membrane Processes: A Comparative Study
- Low-Cost Adsorbents for Heavy Metals Uptake from Contaminated Water: A Review

II. EXPERIMENTAL WORK

A. Experimental Setup:-

A series of batch experiments were carried out to determine isotherms of selected heavy metals on the adsorbent. Each heavy metal solution of Cu, Cr, Pb was placed in 1000ml beakers at pH 2-8 and a know amount of MBHPE-TKP resin were added each beaker. The mass (g) of MBHPE-TKP resin were 0.2, 0.4 ,0.6 and 0.8. the flasks were shaken at constant rate of rpm to ensure that equilibrium was reach. It was assume that the applied shaken speed allows all the surface area to come into contact with heavy metal iron over the course of the experiment. The study was performing at the constant temperature to be representative of environmentally relevant conditions. To avoid the fluctuations of pH due the exchange of gases during the experiment the bottles were capped and kept closed as depicted.

III. MATERIALS AND METHODS

The cation exchange MBHPE-TKP resin was characterized with regard to its adsorption properties for specific heavy metals.

A. Chemicals:

1) Stock solution preparation

The pH meatier was calibrated using standard buffer solution of known pH. pH adjustment was done by 0.1M NaOH or HCL.

A stack of 1000 ppm of copper was prepared by dissolving 2.68gm of copper chloride dehydrate in 1000ml volumetric flask and then makeup with deionised water. Deionised water was issued throughout the work no other salt were added to adjust ionic strength. A stack of 1000 ppm of lead was prepared by dissolving 1.598gm of pb $(NO_3)_2$ in 1000ml volumetric flask and then makeup with deionised water. [2]

Similarly, a stock solution of Cr(III) (520 mg/L) was prepared by dissolving analytical reagent grade chromium nitrate (Cr(NO3)3·9HO) (Sigma) in distilled water. All working solutions were prepared by adequate dilution of the stock solution with distilled water.[4]

2) Preparation of Sorbent:

The setup used in this work, for synthesis of sorbent TKP (MBHPE-TKP) resin is very simple, easily understandable and economical also as compare to other setup used in different synthesis processes.

Chemicals used for the synthesis of sorbent (MBHPE – TKP) and studying its properties are epichlorohydrin, iso-amyl alcohol , 50% aqueous solution of NaOH containing 4.0 g (0.1 mole) sodium hydroxide , acetone , 83 g (0.5 mole) TKP , dioxane , 70% methanol , acetic acid 0.05 N HCl ,phenolphthalein indicator etc. are purchase from Central Scientific lab. All were of Analar grade and were used as supplied without further purification.[13]

3) Preparation of 1,2-Epoxy propyl-3-methyl butyl ether:

7.8 ml (0.1 Mole) of epichlorohydrin was taken in a 200 ml round bottom flask and 10.9 ml (0.1 mole) of iso-amyl alcohol was added slowly to it with stirring on a magnetic stirrer. The stirring was continued for two hours or till the epichlorohydrin layer disappeared. The product so formed is an open chain viscous compound, miscible with water, to this reaction mixture, 50% aqueous solution of NaOH containing 4.0 g (0.1 mole) sodium hydroxide was added drop wise, using phenolphthalein indicator. Stirring was continued for three hours and then extracted with acetone in order to separate it from solid sodium chloride formed during the reaction. The preparation of 1, 2-Epoxy propyl-3-methyl butyl ether [13]

4) Preparation of 3-(3-Methyl) Butoxy-2-Hydroxy Propyl Ether of TKP (MBHPE-TKP) Resin:

83 g (0.5 mole) TKP was taken in dioxane and 5 ml of 50% aqueous sodium hydroxide was added in order to make it alkaline,1,2-epoxy propyl-3-methyl butyl ether (IV) solution in dioxane was added to the alkaline TKP (Tamarind Kernel Powder), with stirring and the reaction was continued for four hours. The product (V) was filtered and washed with 70% methanol containing some acetic acid and dried in air. A light yellow, free flowing powder obtained.

Different techniques are used to study properties of resin .some techniques are important for coating purposes ,some are for water purification .All the Analytical Techniques Used in literature survey are as follow:[13]

- FTIR(Fourier Transform Infrared Technology)
- TGA(Thermogravimatric Analysis)
- SEM(Scanning Electron Microscope

IV. RESULT AND DISCUSSION

A. Adsorption on MBHPE-TKP resin:-

A series of batch adsorption experiment were conducted to establish the isotherm for Pb, Cd, Cu and Zn adsorption on CRH. This section presents the result of batch adsorption isotherm of different metal with CRH.

B. Chromium Study (Cr):-

1) Effect of pH on Resin MBHPE-TKP:

The effect of pH on the removal of Cr by the resin were studied at room temperature by varying the pH of metal solution-resin suspension from 2.0 to 8.0.The results are shown in Fig.The removal of Cr exhibited that the greater increase in the sorption rate of metal ions on resin were observed in a pH range from 6 to 8. Metals were poorly adsorbed at pH<2. This may be due to increasing Cr adsorption with decreasing pH lead to fewer OH^- ions in low pH conditions that could compete with chromium. On the removal of chromium by the resin MBHPE-TKP. The greater percentage was adsorbed in the pH range 6-8, more than 90% Cr was removed.

2) Effect of contact time on the adsorption of chromium:

Contact time is inevitably a fundamental parameter in all transfer phenomena such as adsorption consequently it is important to study its effect on the capacity of retention of chromium by adsorbent like resin MBHPE-TKP .Fig. indicates the typical form of saturation curves showing that the maximum percentage of adsorption is attained at 120 min For this investigation 0.6g of MBHPE-TKP resin were mixed with 20 mL of 1000 ppm chromium ion at different exposure times (30-120 min). The samples after filtration were analyzed for unadsorbed chromium ion. The results are summarized in Figure. As it is indicated, sorption of metal ion is increased by increasing exposure time.

3) Effect of sorbent dosage on adsorption of Chromium:

In this experiment different weights of adsorbent MBHPE-TKP resin (0.2-0.8g) were treated with 20 mL chromium solutions with constant concentration of 1000 ppm. All the other conditions were the same as used for Figure. The results obtained are shown in Figure. As our results show, with increasing sorbent dosage (MBHPE-TKP), sorption percent increases. According to the results shown in Figure, 0.8 g of MBHPE-TKP resin can 98% clean 20 ml 1000ppm aqueous solution contaminated by chromium ion.

C. Lead Study (Pb):

1) Effect of sorbent dosage on adsorption of Lead

In this experiment different weights of adsorbent MBHPE-TKP resin (0.2-0.8g) were treated with 20 mL lead solutions with constant concentration of 1000 ppm. All the other conditions were the same as used for Figure. The results obtained are shown in Figure. As our results show, with increasing sorbent dosage (MBHPE-TKP), sorption percent increases. According to the results shown in Figure, 0.8 g of MBHPE-TKP resin can 99% clean 20 ml 1000ppm aqueous solution contaminated by lead ion.

2) Effect of contact time on the adsorption of lead:

Contact time is inevitably a fundamental parameter in all transfer phenomena such as adsorption consequently it is important to study its effect on the capacity of retention of lead by adsorbent like resin MBHPE-TKP .Fig.indicates the typical form of saturation curves showing that the maximum percentage of adsorption is attained at 120 min For this investigation 0.6g of MBHPE-TKP resin were mixed with 20 mL of 1000 ppm lead ion at different exposure times(30-120 min). The samples after filtration were analyzed for unadsorbed lead ion. The results are summarized in Figure. As it is indicated, sorption of metal ion is increased by increasing exposure time.

3) Effect of pH on the adsorption of lead:

For this investigation, 0.6 g of sorbent (MBHPE-TKP) was treated with 20 mL of Pb ion (1000 ppm) at different pH values (2-8). The pH of solutions was adjusted using NaOH and HCl solution (0.1M). The results obtained are shown in Fig. As our results show, with increasing the pH of metal solution, sorption of lead ion is increased and in acidic media (pH < 4) the sorption of metal ions is very poor. With increasing the pH of treated solution, the polymer is changed into undoped form, then free amine or imine groups in the polymer will be available for metal chelating, so the sorption of Pb ion is increased considerably.

D. Copper Study (Cu):

1) Effect of contact time on the adsorption of copper:

Contact time is inevitably a fundamental parameter in all transfer phenomena such as adsorption consequently it is important to study its effect on the capacity of retention of copper by adsorbent like resin MBHPE-TKP .Fig.indicates the typical form of saturation curves showing that the maximum percentage of adsorption is attained at 120 min For this investigation 0.6g of MBHPE-TKP resin were mixed with 20 mL of 1000 ppm copper ion at different exposure times (30-120 min). The samples after filtration were analyzed for unadsorbed copper ion. The results are summarized in Figure. As it is indicated, sorption of metal ion is increased by increasing exposure time.

2) Effect of sorbent dosage on adsorption of Copper:

In this experiment different weights of adsorbent MBHPE-TKP resin (0.2-0.8g) were treated with 20 mL copper solutions with constant concentration of 1000 ppm. All the other conditions were the same as used for Figure. The results obtained are shown in Figure. As our results show, with increasing sorbent dosage (MBHPE-TKP), sorption percent increases. According to the results shown in Figure, 0.8 g of MBHPE-TKP resin can 98.5% clean 20 ml 1000ppm aqueous solution contaminated by copper ion.

3) Effect of pH on the adsorption of copper:

For this investigation, 0.6 g of sorbent (MBHPE-TKP) were treated with 20 mL of Cu ion (1000 ppm) at different pH values (2-8). The pH of solutions was adjusted using NaOH and HCl solution (0.1M). The results obtained are shown in Fig. As our results show, with increasing the pH of metal solution, sorption of copper ion is increased and in acidic media (pH < 4) the sorption of metal ions is very poor. With increasing the pH of treated solution, the polymer is changed into undoped form, then free amine or imine groups in the polymer will be available for metal chelating, so the sorption of Cu ion is increased considerably.

E. Freundlich and Langmuir adsorption isotherms:

The adsorption isotherms are very important in describing the adsorption behavior of solutes on the specific adsorbents. In this work, two important isotherm models such as Langmuir and Freundlich were selected and studied. The Langmuir isotherm takes an assumption that the sorption occurs at specific homogeneous sites within the bio sorbent. A general form of the Langmuir equation is

$$qe = \frac{b\ qmaxCe}{1 + b\ C} \quad (1)$$

The linear form of isotherm equation can be written as:

$$\frac{1}{qe} = \left[\frac{1}{b\ qmax}\right]\left[\frac{1}{Ce}\right]\left[\frac{1}{qmax}\right]$$

Where qmax is the maximum metal uptake corresponding to the saturation capacity of the biosorbent and b is the energy of bio sorption. The variables qe and C are, respectively, the amount of metal adsorbed on the bio carbon and the equilibrium metal concentration in solution. The constants qe and b are the characteristics of the Langmuir isotherm and can be determined from (1). Therefore a plot of 1/qe versus 1/C gives a straight line of slope(1=b qmaxe) and intercept (1=q). The data fit the Langmuir isotherms model well for Pb, Cu and Cr ions.

The values of Langmuir parameters for the removal of both Pb, Cu and Cr metals ions are presented in Table . The correlation coefficient (r2) for the bio sorption of Pb, Cu and Cr are 0.989, 0.921and 0.829, respectively. The linearity of the three plots (Fig. , fig and Fig.) Indicates application of the Langmuir equation, supporting monolayer formation on the surface of the bio sorption. The values of b and q 2 indicate that the bio sorption of the metal ions is concentration and pH dependent.

The expression of separation factor (RL) in the dimensionless form of the Langmuir isotherm is RL = 1= (1 + b C0), where C0 is the initial concentration of metal ion and b is the Langmuir constant. The irreversible (RL= 0). It is well observed that, in all selected concentrations (25–125 mg/l) of metal ions, the separation factor (RL) is less than 1.0 indicating the favorable bio sorption conditions.

The Freundlich expression is an empirical equation based on a heterogeneous surface. The general form of Freundlich equation is

$$qe = KfCe^{1/n}$$

And the linearized form of this model is

$$\log q\ qe = \log Kf + 1/n \log Ce$$

Where the intercept log Kf is a measure of biosorption capacity, and the slope 1=n is the intensity of bio sorption. The variables q e and C are the amount of metal ion adsorbed and the equilibrium metal ion concentration in solution. The Freundlich biosorption models are presented in Fig. It is also observed that the Freundlich isotherm model is well fitted for the two metal ions.

Table 1 Comparison of technologies to heavy metal removal from wastewater

Method	Advantage	Disadvantage
Chemical precipitation	• Simple • Inexpensive • Most of metals can be removed	• Large amount of sludge produced • Disposal problems
Chemical coagulation	• Sludge settling • Dewatering	• High cost • Large consumption of chemicals
Ion-exchange	• High regeneration of materials • Metal selective	• High cost • Less number of metal ions removed
Electrochemical methods	• Metal selective • No consumption of chemicals • Pure metals can be achieved	• High capital cost • High running cost • Initial solution pH and current density
Adsorption		
Using activated carbon	• Most of metals can be removed • High efficiency (99%)	• Cost of activated carbon • No regeneration • Performance depends upon adsorbent
Using natural zeolite	• Most of metals can be removed • Relatively less costly materials	• Low efficiency
Membrane process and ultrafiltration	• Less solid waste produced • Less chemical consumption • High efficiency (>95% for single metal)	• High initial and running cost • Low flow rates • Removal (%) decreases with the presence of other metals

Source: (Farooq et al. 2010).

Fig.1: Synthesis of 3-(3-methyl) butoxy-2-hydroxy propyl ether of (mbhpe-tkp) resin

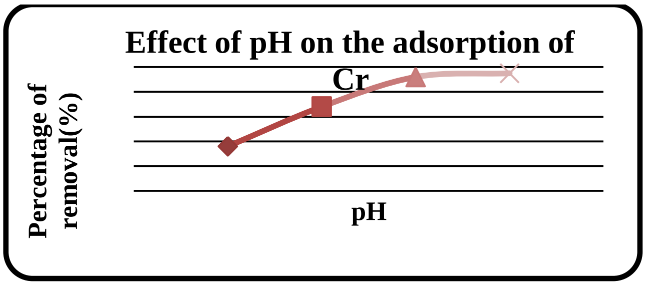

Fig. 2: Effect of pH on the adsorption of Cr

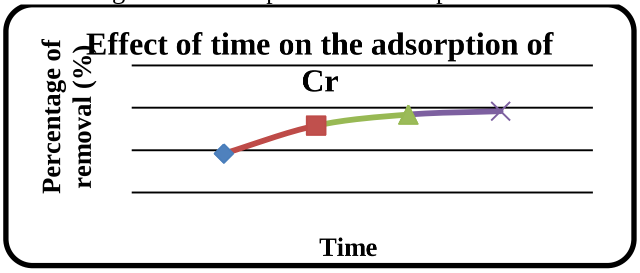

Fig. 3: Effect of time on the adsorption of Cr.

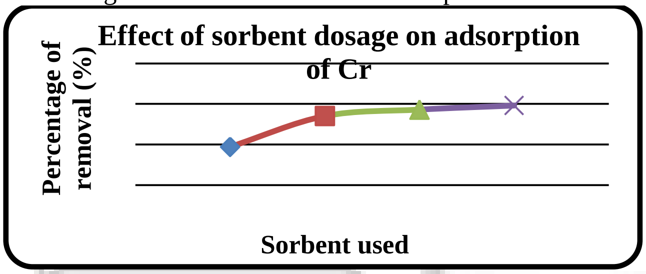

Fig. 4: Effect of sorbent dosage on the adsorption of Cr.

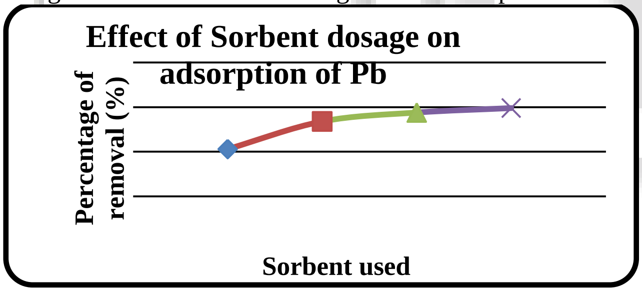

Fig. 5: Effect of sorbent dosage on the adsorption of Pb.

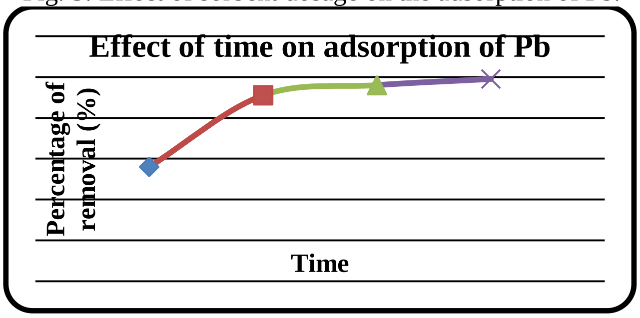

Fig. 6: Effect of time on the adsorption of Pb.

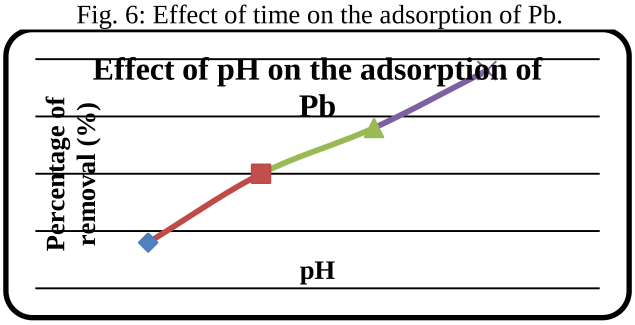

Fig. 7: Effect of pH on the adsorption of Pb.

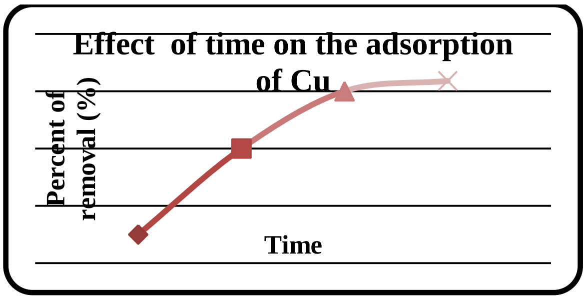

Fig. 8: Effect of time on the adsorption of Cu.

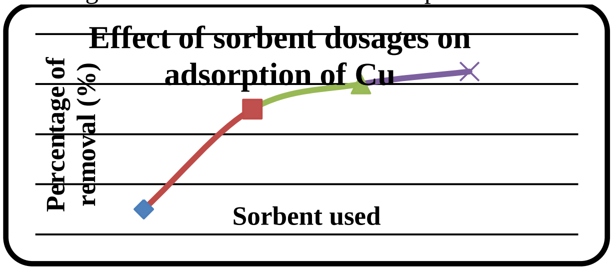

Fig. 9: Effect of sorbent dosages on the adsorption of Cu.

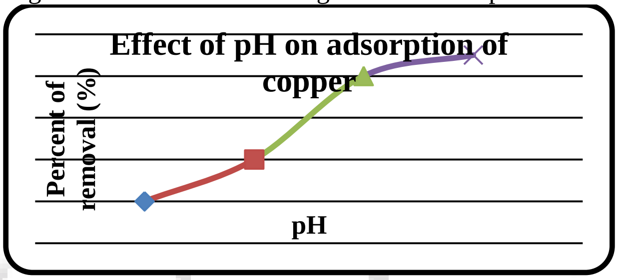

Fig. 10: Effect of pH on the adsorption of Cu.

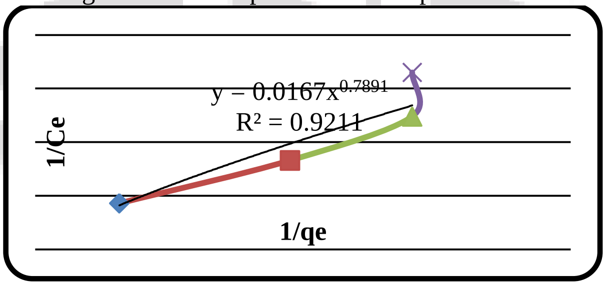

Fig. 11: copper Langmuir adsorption:

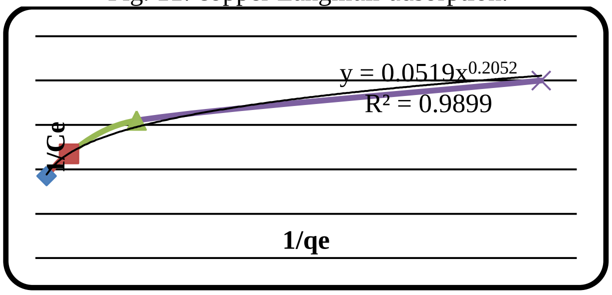

Fig. 12: Lead Langmuir adsorption

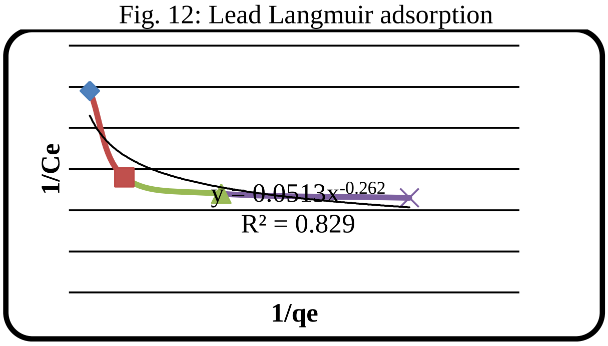

Fig. 13: Chromium Langmuir Adsorption

V. OBSERVATION

In the research study MBHPE-TKP resin is used for the adsorption of heavy metals from aqueous solution the different parameter variation result are shown below:

A. *In case of removing copper (Cu) from aqueous solution:*

- Time requirement: At a time 120 min. when pH of sample was 6 and sorbent used was 0.6gm.the maximum adsorption of copper get is 98%
- PH Requirement: In between the PH range 6-8 maximum adsorption is obtain.
- Sorbent Used: About 98% of copper is adsorbed by using 0.6gm of adsorbent

B. *In case of removing Lead (Pb) from aqueous solution:*

- Time requirement: At a time 120 min. when pH of sample was 6 and sorbent used was 0.6gm.the maximum adsorption of copper get is 99%
- PH Requirement: In between the PH range 6-8 maximum adsorption of lead is obtain.
- Sorbent Used: About 99% of lead is adsorbed by using 0.8gm of adsorbent

C. *In case of removing Chromium (Cr) from aqueous solution:*

- Time requirement: At a time 120 min. when pH of sample was 6 and sorbent used was 0.6gm.the maximum adsorption of Chromium get is 96%
- PH Requirement: In between the PH range 6-8 maximum adsorption of chromium is obtain.
- Sorbent Used: About 98% of chromium is adsorbed by using 0.8gm of adsorbent

VI. CONCLUSION

According to the inferences drawn on the basis of result and discussion from my research work the following conclusions are made.

- From above result it evident that the different heavy metals like Cr ,Cu & Pb are adsorbed from aqueous solution with the percentage 96%, 98% and 99% respectively and hence MBHPE-TKP resin is one of the best adsorbent.
- The MBHPE-TKP resin acts as a flocculent cum metal ion exchanger and can be used as scavenger for toxic and hazardous metal ions present in effluent of mineral & metallurgical industries.
- In this study, the method used to remove these heavy metals is very simple, common and has following advantages:
  - Very Simple Setup,
  - Less time consuming,
  - Can be used both in small scale (Lab level) as well as large scale
- For the process of adsorption, the resin (MBHPE-TKP) used as a adsorbent, is synthesized from natural ingredient Tamarind Kernal Powder (TKP), a hydrophilic polysaccharide obtain from seeds of tamarind. Hence the resin is easily biodegradable and not harmful to environment.

The above experimental work has been conducted in lab only and results in an industrial setup for the same experiment may vary.

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