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An economic removal of Cu^{2+} and Cr^{3+} on the new adsorbents: Pumice and polyacrylonitrile/pumice composite

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Abstract

In this article, the adsorption of Cu^{2+} and Cr^{3+} onto pumice (Pmc) and polyacrylonitrile/pumice (PAn/Pmc) composite has been investigated by using a batch method at room temperature and their adsorption properties were compared. To highlight their application, selected information on pH, initial metal concentration and adsorption capacity of the adsorbents is presented. Depending on the characteristics of the individual adsorbents viz. surface modification, initial concentration of adsorbates, and pH, their adsorption capacities were found to vary. The time required for the removal of metal ions (100–500 mg/g) from aqueous solutions was about 6 h. The experimental data have been analyzed using the Langmuir and Freundlich isotherm models. The results of adsorption were fitted to the Langmuir models and coefficients indicated favorable adsorption of Cu^{2+} and Cr^{3+} on the adsorbents. Adsorption of Cu^{2+} and Cr^{3+} in the solutions was in following order: (PAn/(Pmc) composite > Pmc). The adsorption capacity of the adsorbents Pmc and (PAn/Pmc) were determined from the isotherms equations and were found to be 0.055 and 0.065 mmol/g for Cu^{2+} and 0.031 and 0.268 mmol/g for Cr^{3+}, respectively. More than 80% of studied cations were removed by Pmc and 87% (PAn/Pmc), respectively, from aqueous solutions in single step. Effective removal of metal ions was demonstrated at pH values of 8.0. The mechanism for cations removal by the Pmc and (PAn/Pmc) composite includes complexation and sorption. The process is very efficient especially in case of low concentrations of pollutants in aqueous solutions, where common methods are either economically unfavorable or technically complicated.

Keywords: Pumice; Adsorption; Sorption isotherms; Polyacrylonitrile/pumice composite; Chromium; Copper

1. Introduction

Metallic pollutants are inevitable for industrial growth and some of them are essential nutrients both for plants and human beings. But if these species are present beyond the tolerance limits, these are reported to effect the health adversely [1–3]. Removal of heavy metals is an important problem especially in industrial effluents. Various treatment technologies have been developed for the removal of heavy metals from water. The commonly used technologies for removing metal ions from effluents include chemical precipitation, lime coagulation, ion exchange [4–6], electro dialysis, reverse osmosis membrane filtration [7–9], electro winning [10], solvent extraction and common adsorption [11–13], the latter process being a more useful method for the metal removal than other processes [14,15]. Although heavy metal removal from inorganic effluent can be achieved by conventional treatment such as chemical precipitation or flotation, each treatment has its inherent limitations. Major sources of the heavy metal pollution of the water in particular are the discharges from metal finishing industries, electroplating industries, etc.

In recent years, the search for low-cost adsorbents that have metal-binding capacities has intensified. This has led many workers to search for cheaper alternates such as coal, fly ash, silica gel, wool wastes, agricultural wastes, wood wastes, and clay minerals [16–18]. Due to its large surface area, high adsorption capacity and surface reactivity, adsorption using activated carbon can remove metals from inorganic effluents and the

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treated effluent can meet stringent standards for metal bearing effluents' discharge. Even after its expensive nature, granular activated carbon is the most popular adsorbent and has been used with great success. Complexing agents are also added to activated carbon to further increase its efficiency [19,20]. Materials locally available in large quantities such as natural materials, agricultural wastes or industrial by-products can be utilized as low-cost adsorbents. Some of these materials can be used as adsorbents with little processing. Conversion of these materials into activated carbon, which can be used as an adsorbent for water purification, would improve economic value, helping industries reduce the cost of waste disposal and providing a potential alternative to activated carbon. However, regeneration of contaminated adsorbent is not recommended and should not be preferred to the immediate disposal. The above consideration insists scientists to focus attention on identifying efficient low-cost throwaway adsorbents. On the other hand, natural zeolites are interesting options and are 100–200 times cheaper although their adsorption capacity is 10–20 times lower [21–23]. Zeolite is widely used in water and sewage purification, ammonia and heavy metals removal, ion exchange in radioactive wastewater treatment, removal of oil pollution from water, and adsorption of other components from liquid and gaseous phases [24,25]. The clay minerals are considered very important adsorbents in natural water systems because of their high specific area combined with the structural and pH dependent charge developed on their surfaces. To achieve an economically effective treatment of metal-contaminated wastewater, various low-cost materials have been investigated worldwide [26–29].

To enhance the sorption capacity, the sorbents are modified in various ways, such as treatment by inorganic and organic compounds, acids and bases. While their sorption capacity is usually less than those of synthetic sorbents, these materials could provide an inexpensive alternate for the treatment of wastewaters rich in heavy metals [7,9,16,17,27,30]. Ages ago, granite was exposed to intense heat generated within the earth itself that caused it to expand, turn into molten froth, and boil off its organic and chemical impurities. During volcanic eruptions of the molten mass and in the course of rapid cooling, tiny air cells-trapped in prodigious quantities in the chemically inert granite-formed minute sealed vesicles within the hardened material. The end result of these natural phenomena was a unique, lightweight material now called pumice. It is to clarify that pumice is not any ash, tuff or other agglomerate material.

The use of pumice as an adsorbent to remove metals from wastewater treatment at low cost is a well-established process. Pumice is a light, porous igneous volcanic rock. It has a porous structure and a large surface area and it can be processed easily. The large proportion of free silica sites at the grain surface results in a negatively charged surface. The structure contains open channels that allow water and ions to travel into and out of the crystal structure. It is a valuable scouring, scrubbing, and polishing material both in powdered form and as pumice stone. Pumice has a skeleton structure that allows ions and molecules to reside and move within the overall framework. The structure contains open channels that allow water and ions to travel into and out of the crystal structure. Pumice is used chiefly as an abrasive and is included in many scouring preparations. This rock is so light, that many pumice rocks will float in water. Pumice is actually a kind of glass and not a mixture of minerals [8,9,14,30]. Pumice has been found to be effective for the removal of phosphate ions from water [31].

The aim of this study is to investigate the adsorption of metal ions onto pumice powder and (PAn/Pmc) composite containing polyacrylonitrile and pumice powder, therefore exploring its application as a low-cost adsorbent if its efficacy for adsorbing heavy metals from aqueous solutions and metal bearing water is demonstrated. The adsorption properties of both the adsorbents will be compared with each other. Cu^{2+} and Cr^{3+} ions were the metals selected for the proposed study since they are most common heavy metals in the wastewater of the electroplating industry.

2. Materials and methods

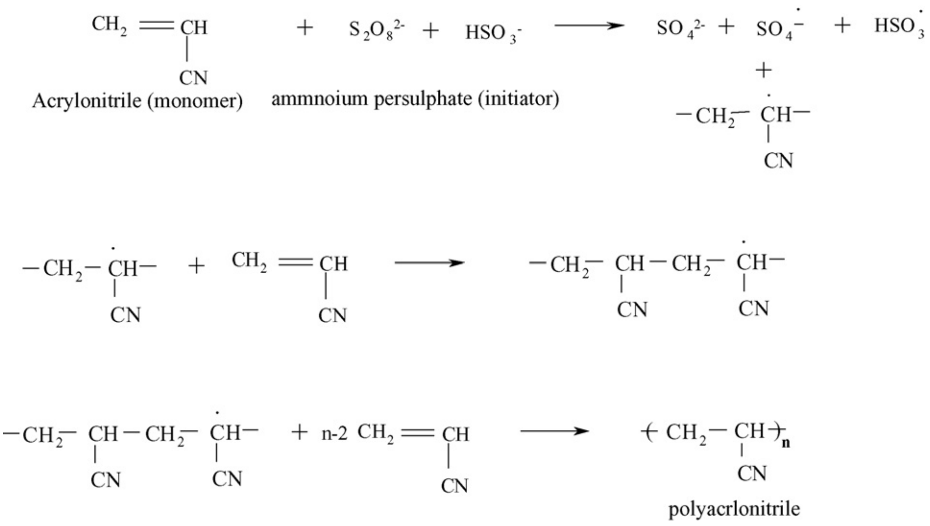
2.1. Chemicals

All solutions were prepared from analytical reagent grade chemicals and Milli-Q filtered deionized water. 1.0×10^{-3} mol/L of metals stock solution were prepared by dissolving of $Cu(NO_3)_2{\cdot}3H_2O$, $Cr(NO_3)_3{\cdot}9H_2O$ (from Merck company, Darmstadt, Germany).

Working solutions imitating wastewater with a concentration of 1.0×10^{-4} to 1.0×10^{-3} mol/L metal ions were prepared by appropriate dilutions of the stock solution immediately prior to their use. The pH was adjusted either with diluted HCl or with NaOH solutions. The electrolyte used to modify the ionic strength in the adsorption experiments was 0.10 mol/L $NaNO_3$.

2.2. Synthesis of polyacrylonitrile–pumice composite samples

Pumice samples were obtained from the Middle Anatolia region (Kayseri-Talas) of Turkey. The material was dried in an oven at 105 °C for 24 h, ground and sieved in the size range of 80–100 mesh and the material was placed in an air-tight container for further use. As natural raw pumice contains various extractable materials, which can affect the adsorption, 0.10 mol/L HCl was used to pre-treat the mineral substrate to remove these residual inorganic salts. 5.0 g pumice was washed with 0.10 mol/L HCl. A solution of 2.0 g linear alkyl benzene sulfonic acid (LABSA), 30.0 mL H_2O and 5.0 mL 2.0 mol/L HCl was prepared in a three neck flask by stirring the solution. Then 5.0 g of the pumice added to the solution and a slurry obtained. The slurry was kept at 80 °C and 10.0 mL aqueous solution of 1.0 g $(NH_4)_2S_2O_8$ and 5.0 mL acrylonitrile (monomer) were slowly added to the slurry [32]. The solution was allowed to polymerization reaction for about 4.0 h. The polymerization was terminated by pouring into the emulsion into methanol. The precipitate was filtered, washed with water, ethanol, 0.1 mol/L HCl solution, and water, respectively. Final product was dried under vacuum at 60 °C for 24 h. The polymerization reaction is given in Scheme 1.



Scheme 1. Schematic representation of acrylonitrile monomer's polymerization.

2.3. Instrumentation

Chemical composition of pumice powder samples was determined by XRF (Panalytical X'PertPro) and given in Table 1. The SEM results of the pumice samples obtained by scanning electron microscopy (SEM) (JEOL 5600-LV Model) were given in Fig. 1 for Pmc, PAn/Pmc, Pmc/Cu^{2+}, (PAn/Pmc)/Cu^{2+}, Pmc/Cr^{3+}, (PAn/Pmc)/Cr^{3+} before and after adsorption of metal ions. FTIR spectrum of each pumice sample was acquired using a Perkin-Elmer BX Model FTIR Spectrometer and given in Fig. 2. The band at around 3600, 1050 and 500 cm^{-1} was assigned to characteristic pumice peaks. The region corresponding to high wave numbers shows a band centered at 2928 and 2250 cm^{-1} can be attributed to (CH_2) and (CN) groups of acrylonitrile. Absorbance values were measured with a Perkin-Elmer AA800 Model AAS. An Orion 900S2 model digital pH-meter and a Gallenkamp Orbital Incubator were used for pH adjustment and shaking, respectively.

2.4. Sorption studies

Sorption isotherm studies were conducted in well-sealed 100 mL polyethylene bottles at room temperature viz. 20 °C. The adsorption of Cu^{2+}, and Cr^{3+} from aqueous solutions onto pumice powder was performed using batch equilibrium technique. For the determination of adsorption isotherms, 30.0 mL of metal solution of known initial concentration was shaken with a certain amount of the adsorbent on a shaker at 200 rpm for 6.0 h. Initial metal concentrations were changed in the range of 1.0×10^{-4} to 1.0×10^{-3} mol/L. Following a 24 h reaction period on a shaker, the samples were taken at 15 min intervals, and the solids separated from solutions by filtration. Then pH was measured and acidified samples were analyzed for equilibrium liquid phase concentration and the residual heavy metal concentrations were determined. The amount of metal ion adsorbed was calculated using the Eq. (1):

$$q_e = (C_0 - C_e)\frac{V}{M} \qquad (1)$$

where C_0 and C_e are the initial and equilibrium concentrations of metal ion (in mmol/L), M the mass of pumice powder (g), and V is the volume of solution (L).

Table 1
Percentage of the main constituents of pumice

Components	SiO_2	Al_2O_3	Na_2O	K_2O	Other
Amount (%)	74.0	15.6	6.1	2.4	1.9

3. Results and discussion

3.1. The effect of pH on the removal of metal ions

The adsorption of metal ions on pumice samples was studied over the pH range 2–8 for a fixed pumice dose of 0.5 g. Comparison of absorbances at different values of pH shows that most cations are hydrolyzed beyond pH 6 and they have low solubility above pH 6. It is well known that the pH of the system is an important variable in the adsorption process. Fig. 3 shows the effect of pH on the K_d of pumice samples and composite. K_d value is equilibrium constant that is resulted from the ratio the equilibrium concentrations of metal ion on the adsorbent and in the solution, respectively. Distribution coefficient (K_d) indicates the capability of pumice to retain a solute and also the extent of its movement in a solution phase [33]. According to Gomes et al. [34], K_d is a useful parameter for comparing the sorptive capacities of different adsorbents for any particular ion, when measured under same experimental conditions. Maximum pH of solution was 8.0, and it is observed that adsorbed cations percentage is intensively decreased at pH (<5.0) relative to pH 8.0.This can be attributed to competition of proton ions with cations in the pumice structure. By increasing the pH from 5.0 to 8.0, the adsorption rises from 20 to 80% for the pumice and 20 to 87% for (PAn/Pmc) composite, respec-

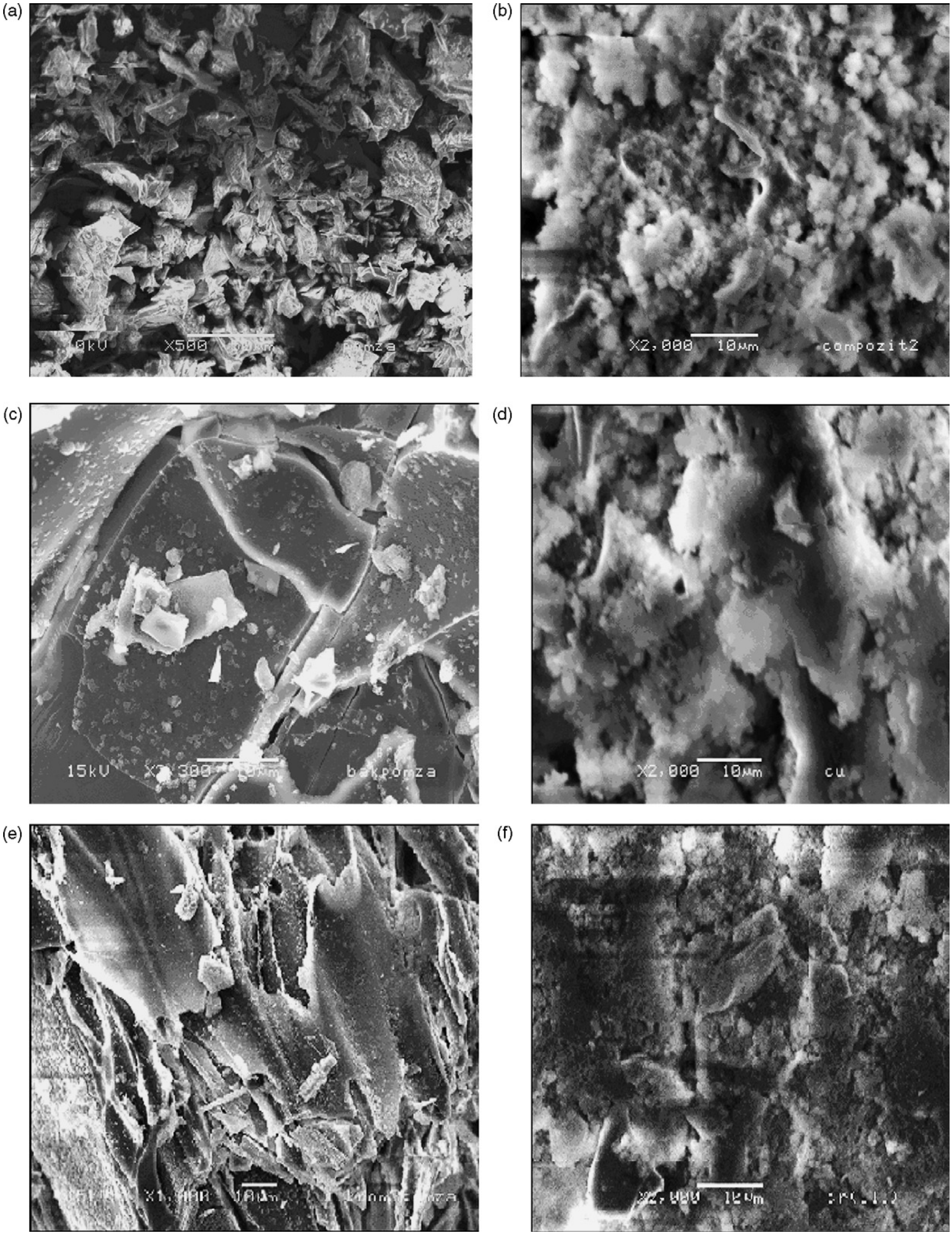


Fig. 1. SEM micrographs of (a) Pmc, (b) PAn/Pmc, (c) Pmc/Cu^{2+}, (d) (PAn/Pmc)/Cu^{2+}, (e) Pmc/Cr^{3+} and (f) (PAn/Pmc)/Cr^{3+} before and after adsorption of metal ion.

tively. All data related to sorption isotherm were given in Table 2 for both cations. It was observed that the percentage of sorption was maximum at pH values between 7.0 and 8.0 for both the adsorbents. As the pH of the solution increases, metal ions will replace hydrogen ions from the surface of the pumice and (PAn/Pmc) composite and therefore the extent of the adsorption will increase.

As can be seen from Fig. 3, adsorption of all heavy-metal ions first increases with increasing pH. Metal adsorption on pumice surfaces has been described in terms of two molecular mech-

Table 2
Freundlich and Langmuir constants of Pmc and (PAn/Pmc) composite

Adsorbent	Species	Freundlich Constants				Langmuir constants			
		k	*n*	R^2	1/*n*	Q_0	*b*	R^2	R_L
Pmc	Cu^{2+}	0.811	1.808	0.951	0.553	0.055	614.300	0.989	0.619
	Cr^{3+}	0.072	5.208	0.915	0.192	0.031	1990.760	0.997	0.334
PAn/Pmc	Cu^{2+}	0.602	1.948	0.940	0.513	0.065	665.450	0.994	0.600
	Cr^{3+}	3.872	0.490	0.800	2.041	0.268	132.200	0.960	0.883

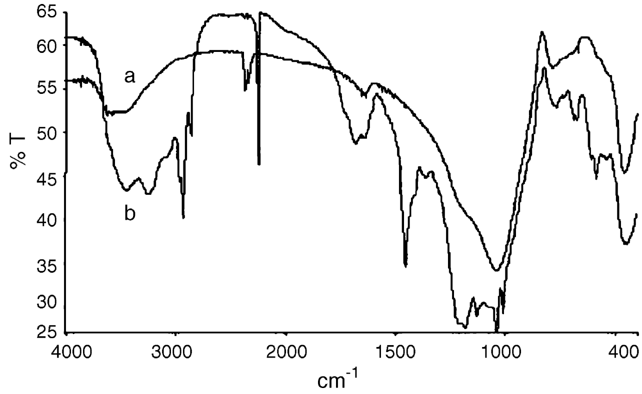


Fig. 2. FTIR spectra of adsorbents (a) Pmc and (b) PAn/Pmc.

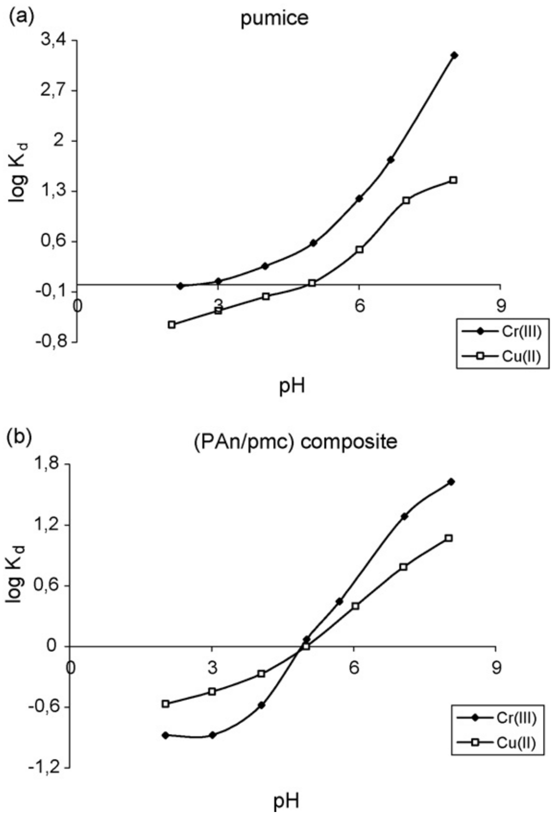


Fig. 3. The distribution coefficient of Cu^{2+} and Cr^{3+} metal ion on the adsorbents as a function of pH (a) Pmc and (b) PAn/Pmc (adsorption conditions: initial concentration of metal ion, 1×10^{-3} M; amount of adsorbent, 50 mg; volume of the solution, 30 mL; adsorption time, 6 h; temperature, 20 °C).

anisms. (i) Inorganic cation exchange in the interlayer and (ii) specific adsorption resulting from surface complexation [35]. Surface complexation adsorption model has been described as a composite of SiOH and AlOH sites.

High adsorption at higher pH values implies that metal ions interact with pumice by chelating. Hydrogen ions affect metal complexation because they have a great affinity for many complexing and ion-exchange sites. At very low pH (<4.0) functional oxidized groups (Al_2O_3, SiO_2 groups, etc.) of the pumice are protonated.

3.2. Effect of time on the removal of metal ions

Batch adsorption experiments were conducted by taking 30 mL of the solution containing 1.0×10^{-3} mol/L of the selected adsorbates. The results are given in Fig. 4. The adsorbent loading was kept at a constant value of 0.5 g and a contact time of 24 h was employed. The majority of metal ions in adsorption equilibrium were achieved in between 6 and 8 h for both cations. This rapid and strong decrease in the metal concentration in aqueous phase is followed by a slow decrease down for the next 15 h. The experimental results indicate that the percentage removal of metal becomes asymptotic to the time axis, nearly representing an equilibrium pattern. For contact times of up to 6 h the amounts of metal adsorbed increase by 5–80%. Adsorption first followed a linear rise in which instantaneous and extremely fast uptake takes place, and then stationary state was observed. In the first parts of the isotherm, the decrease in metal concentration was about 30–60% in the first 6 h and in the second part, a stationary state was observed from 6 to 8 h of contact time with a removal of 87%. Therefore, for practical considerations this time was presumed to represent the equilibrium time for adsorption processes. This phenomenon could be attributed to the instantaneous utilization of the most readily available adsorbing sites on the adsorbent surface. Second part of the isotherm curve may be attributed to a very slow diffusion rate of the metal through the adsorbent micro pores. The initial faster rate of metal transition may be explained by the large, uncovered available surface area of the pumice and its composite. The slower adsorption rate in the second stage is probably due to the diffusion of metal ions into the porous structure of the pumice. The preference of the pumice for metals would appear to be strongly related to the ionic properties of the ions (i.e. ionic radius, electronegativity, or ionic potential). Metal ion having the greatest ionic potential, is most strongly attracted to the adsorbent.

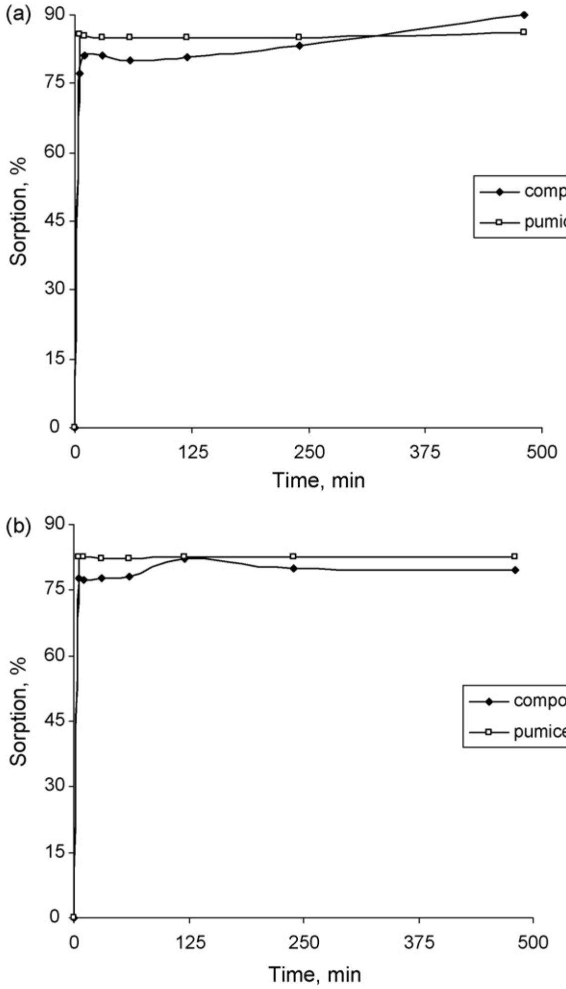


Fig. 4. Effect of contact time on the sorption of (a) Cu^{2+} and (b) Cr^{3+} by adsorbents (adsorption conditions: initial concentration of metal ion, 1×10^{-3} M; adsorption time, 0–24 h; amount of adsorbent, 50 mg; volume of adsorption medium, 30 mL; temperature, 20 °C).

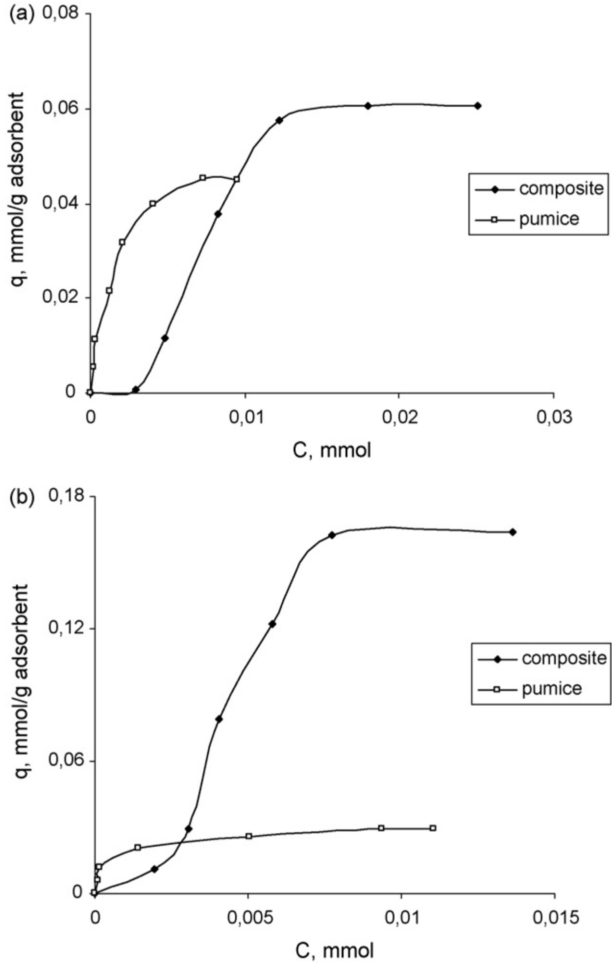


Fig. 5. Sorption isotherms of (a) Cu^{2+} and (b) Cr^{3+} ion on Pmc and PAn/Pmc (adsorption conditions: initial concentration of metal ion, 1×10^{-4} to 1×10^{-3} M; amount of adsorbent, 50 mg; volume of adsorption medium, 30 mL; adsorption time, 6 h; temperature, 20 °C).

3.3. *Sorption isotherm as a function of metal concentration*

Equilibrium isotherm equations are used to describe experimental sorption data in Fig. 5. The equation parameters and the underlying thermodynamic assumptions of these equilibrium models often provide some insight into both the sorption mechanism and the surface properties and affinity of the sorbent. The metal ions removal at the optimum pH values of 7.0 and with initial concentrations of 1.0, 2.0, 4.0, 6.0, and 8×10^{-4} mol/L are shown in Table 2. The pumice have a high affinity for the metal ions studied and they are almost adsorbed in higher percentages from the aqueous solutions. The adsorption behavior at high initial metal concentrations shows a relative loss of adsorbed metal ions. The adsorption capacity of Pmc and (PAn/Pmc) composite was found as 0.055 and 0.065 mmol/g for Cu^{2+} and 0.031 and 0.268 mmol/g for Cr^{3+}, respectively from the calculation of adsorption isotherm equation. (PAn/Pmc) composite have more adsorption capacity than Pmc for Cu^{2+} and Cr^{3+}. It appears that, with increase of metal concentration in solution, a point is reached at which the solute is desorbed from the pumice surface and (PAn/Pmc) composite. This can be explained by the fact that at high metal concentrations, the adsorbed metal ions could begin to repel each other or that the effects of the ionic strength becomes more important [36].

Heavy metal adsorption on heterogeneous sorbents has been interpreted using both the isotherms viz. Langmuir Freundlich adsorption isotherms. Although the basic assumptions for these models were not fulfilled due to the heterogeneity of the sorbents surface, they were quite successful in predicting the experimental saturation capacities of the sorbents [36–38]. The Langmuir model is probably the best known and most widely applied sorption isotherm. It has produced good agreement with a wide variety of experimental data.

3.3.1. *Langmuir Isotherm*

The Langmuir isotherm applies to adsorption on completely homogenous surfaces with negligible interaction between adsorbed molecules.

Langmuir equation:

$$\frac{C_e}{q_e} = \frac{C_e}{Q_0} + \frac{1}{Q_0 b} \qquad (2)$$

Table 3
Comparison of the adsorption capacities of riverbed sand with various low-cost adsorbents

Adsorbent	pH	Temperature (°C)	Adsorption capacity, $Q°$ (mg/g)	Adsorption affinity (l/g)
Grafted silica	5.5	20	16.57	0.197
Perlite	6.0	30	361.60	0.069
Fly ash	6.5	30	1.39	–
Bagasse fly ash	4.0	30	2.40	18.9
Tree fern	–	20	13.90	0.038
Chitosan	3.5	–	2.83	13.14
Lemma minor L.	4.0	20	69.00	0.254
Prawn shell	6.0	25	92.40	0.083
Modified biomass	5.5	25	92.40	0.0024
Activated sludge	4.0	20	294.0	5.26
River bed sand	6.5	25	0.15	0.63
Pumice[a]	8.0	20	0.055	1990.76
Pumice/composite[a]	8.0	20	0.268	132.20

[a] This study.

where C_e is the equilibrium concentration (mmol/L), q_e the amount of adsorbed material at equilibrium (mmol/g), b the "affinity" parameter or Langmuir constant (L/mmol), and Q_0 is the "capacity" parameter (mmol/g) [39]. The Langmuir isotherm parameter was determined by least-squares fit of the sorption data. Q_0 and b were determined from the slope and intercept of the Langmuir plot and are presented in Table 2.

3.3.2. Freundlich isotherms

Freundlich adsorption isotherm is the relationship between the amounts of metal adsorbed per unit mass of the adsorbent (x/m) and the concentration of the metal at equilibrium (C_e).

Freundlich equation:

$$\left(\frac{x}{m}\right) = kC_e^{1/n} \tag{3}$$

where x is the amount of metal adsorbed (mmol), m the amount of sorbent (g), C_e the equilibrium concentration (mM) and k and n are Freundlich constants. The logarithmic form of the equation becomes:

$$\log\left(\frac{x}{m}\right) = \log k + \frac{1}{n}\log C_e \tag{4}$$

Here k and n are constants representing the adsorption capacity and intensity of adsorption, respectively. Analysis of the relationship between the adsorption capacity of pumice and the metal cation concentration at equilibrium was performed using the equations of Langmuir adsorption isotherm (Eq. (2)) and Freundlich (Eqs. (3) and (4)) [3,40]. The experimental results obtained for the adsorption isotherms were found to obey the Langmuir adsorption isotherms as seen in Table 2. It was found that more than 80% removal was achieved using these adsorbents. The adsorption data in the linearized forms gave satisfactory correlation coefficients for a part of the covered concentration range. The comparison of the experimental adsorption capacity obtained in this study with the data in the literature for various adsorbents shows that pumice and (PAn/Pmc) composite are effective sorbents for metal ions. A dosage of 0.5 g of pumice is sufficient to remove more than 80% of metal from the 1×10^{-4} M, 30 mL metal solution. These levels are similar to the achievable effluent levels of metals for a variety of wastewater treatment processes such as ion exchange and reverse osmosis.

3.3.2.1. Comparison of the adsorbent with other low-cost adsorbents. A comparison of the maximum adsorption, $Q°$ (mg/g) and adsorption affinity, b (L/mg) of pumice and the composite and other adsorbents has been made and is given in Table 3. Detailed account of the same has been published elsewhere by the authors [41]. Hydrous concrete particles [42] is also reported for the removal of Cr(VI).The adsorption capacity of the adsorbent used is relatively low as compared with other low-cost adsorbents. Values of adsorption affinity, b (L/mg), for the adsorbent used, are, however, higher than those of grafted silica, perlite, tree fern, Lemma minor L., prawn shell, and modified biomass (Table 3). Thus, the values of the two parameters for pumice and the composite are quite significant as compared with other adsorbents and value of adsorption affinity for pumice is highest. Pumice is available in plenty in all parts of world and it is non-toxic. Above all the adsorbent use is a part of the natural environment.

4. Conclusion

The adsorption of metal ions from aqueous solutions onto pumice powder and the composite been studied. The following conclusions may be drawn:

i. Removal of the selected cations copper and chromium was quite significant viz. 80 and 87% by Pmc and Pan/Pmc composite, respectively.
ii. Adsorption tests were carried out as function of pH, mixing time and metal ion concentration. The adsorption experiments indicated that pumice powder was effective in removing metal ions from aqueous solution.
iii. The adsorption is found to be highly dependent on pH. For the pumice–metal interaction, the adsorption takes place between pH values of about 5.0–8.0 and it would appear that adsorption, ion exchange and chelation reaction take place in the metal removal with the pumice. The experiments showed that metal ions were adsorbed at pH 5.0 and the maximum

adsorption is attained at around pH of solution 8.0. At pH between 2.0 and 5.0 adsorption of metals was negligible.

iv. The adsorption data was described by the Langmuir isotherm equation and the data in the linearized form of Langmuir equation gave satisfactory correlation coefficients for certain concentration ranges. The adsorption capacities are 3.495 mg/g for Cu^{2+}, 1.612 mg/g for Cr^{3+} for pumice and 3.368 mg/g for Cu^{2+}, 13.935 mg/g for Cr^{3+} for composite. More than 80% of studied cations were removed by pumice and 87% composite, respectively, from aqueous solution in single step.

Its efficiency is nearly same as expensive adsorbents, such as activated carbon and chitosan, and the cost equals to that of low-cost adsorbents, such as natural zeolites. The present study concludes that pumice powder could be employed as a low-cost adsorbent for the removal of metal ions from aqueous solutions. Continuing investigations will be focusing on application considerations such as multicomponent interactions, sorbent regeneration and disposal.

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