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Removal of Heavy Metals from Industrial Dye Effluent using Poly (N-tert-butyl acrylamide-co-Acrylamide/Maleic acid) Ferrogels

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ABSTRACT

In this paper, poly (N-tert -butylacrylamide-co-acrylamide/Maleic acid)ferrogels were prepared by in situ polymerization using NTB, AM and Maleic acid monomers in aqueous Methanol medium at 60° C . In the next step, Magnetic Fe_3O_4 particle was formed in the hydrogel via co-precipitation of Fe^{2+} and Fe^{3+} ions in alkaline medium at 70°C. Ferrogel adsorbs all type of heavy metals. Both the concentration of magnetic particles and cross linking density of the ferrogel plays an essential role in the adsorption. The adsorption rates for heavy metals are slow during first two minutes and it increases after 10 to 15 minutes.

Keywords : NTB, Maleic Acid, Acrylamide, Ferrogel Adsorbs , Polyacrylic Acid ,ICPMS

I. INTRODUCTION

Hydrogel is defined as two or multi component system consisting of a three dimensional network of polymer chain and filled with water in the space between macro molecules. Ferrogels were containing magnetic particles dispersed homo/ heterogeneously and confined in a polymer network. The ability to absorb aqueous solution without losing its shape and mechanical strength depends on the properties of the polymer, nature and the density of the network and accordingly they can absorb and retain various amount of water. Chains are connected by electrostatic force of hydrogen bond, hydrophobic interaction and such gels are not permanent. They can be converted into polymer by heating [**1**].

Many industrial manufacturing processes produce effluent containing heavy metals. Industries include metal finishing, automotive, semiconductor manufacturing, electroplated metal parts/washing, textile dye and steel etc. As a result of improper treatment prior to discharge many dissolved metals have been found in harmful concentration in ground water which are utilised for potable water. Wang et al[**2**]. , studied the Fe_3O_4 particles, which are superior in separation and recyclability, are widely applied in environmental remediation. Brundha et al. described the synthesis and swelling behavoir of poly(N-tert-amylacrylamide-co-Acrylamide/Maleicacid) hdrogels. These hydrogels were showed the increasing swelling behavoir with increasing amount of maleic acid content [**3**] . The poly(N-cyclohexylacrylamide -*co* –acrylamide / AMPS Na) Hydrogels were synthesized by Anbarasan et al.,[**4,**] which exhibited good swelling behavior. Sezgin et al., reported the removal of heavy metals (Cu(II), Ni(II), Zn(II), and total Cr) from real industrial wastewater being taken from galvanotechnic industry, by using polyacrylic acid (Aac) hydrogel[**5**] .

The metals most often linked to human poisoning causing disabilities, concerns and death are typically caused by copper, nickel, cadmium, chromium, arsenic, lead and mercury.[6] Many of them are required for human in trace amount, but larger dosage these heavy metals become toxic. Heavy metal toxicity can result in damage or reduced mental and central nervous system function, lower energy level and damage to blood composition, lungs, kidneys, liver and other vital organs. Nowadays there are serious concerns for the restricted usage of these heavy metals all over the world. The present study aimed to synthesis of N- tert-butylacrylamide based Ferrogels and to serve the alternative adsorbent for the removal of heavy metals from industrial effluents

II. EXPERIMENTAL

Synthesis of Ferrogels

The poly(NTB-co-AM/MA)Ferrogels were prepared by the free radical crosslinking copolymerisation of N-tert-butylacrylamide (NTB),Acrylamide (AM) and Maleic acid (MA) in 3:1 mixture of methanol water. Ammonium per sulphate was used as redox initiator system. The N-tert-butylacrylamide - 0.5g, Acrylamide – 0.5g, Maleic acid - 0.1g -0.7g, ammonium per sulphate 0.05g were dissolved in 20 mL of 3:1 mixture of methanol-water. The solution was purged with nitrogen for 30 minutes, after the addition of Methylenebisacrylamide (MBA) 0.045g. The reaction was carried out in the polymerization tube and it was immersed in the thermostat for one day at 60°C. Upon completion of the reaction the hydrogel was removed from the polymerization tube and immersed in the large excess of deionised water at room temperature for at least 72 hours. The water was replaced 3-4 times every day to remove the unreacted monomers[7]. After cleaning procedure the swollen gel was transferred in to an aqueous solution containing $FeCl_3.6H_2O$ (0.2M in 20mL) and $FeSO_4 \cdot 7H_2O$ (0.1M in 20L) at room temperature for 10 days. After adsorption procedure, the metal ion loaded hydrogel was treated with the mixture of KOH (0.5M in 50mL) and KNO_3 (2.0M in 50mL) at 70°C for an hour, which resulted in the precipitation of the corresponding oxide Fe_3O_4. The obtained Ferrogel was finally neutralised with deionised water by repeated washing. The chemical reaction of magnetite precipitation is considered to be as follows.

$$Fe^{2+} + Fe^{3+} + 8OH^- \xrightarrow{NO_3/\Delta} Fe_3O_4 + 4\,H_2O \quad \text{------ (1)}$$

Adsorption studies

The effluent samples were collected from the textile industry situated at Tirupur, Tamil Nadu. In this experiment the effect of various important parameters such as amount of adsorbent, immersion time of Ferrogel at different intervals were studied. The experiment was carried out at room temperature. The initial concentration of heavy metals in effluent was calculated by using ICPMS(Model : 7700 series Software: MassHunter). Table.1. The final concentration of heavy metals after immersing a particular amount of Ferrogel for a specified time interval such as 30m and 60 m were also calculated.

Effect of amount of adsorbent

The effect of amount of adsorbent on the removal of heavy metals was done by using 100mg and 200mg of Ferrogel. From this data the amount of adsorption at equilibrium time was calculated using the following equation

$$Qc = (C_0 - Ce)V/M \quad \text{---------- (2)}$$

Where,C_0 is the initial liquid phase concentration of the heavy metals, Ce is the final liquid phase concentration of heavy metals at equilibrium time mg/L. V represents the volume of effluent solution (L) and M is mass of the dry adsorbent (Ferrogel) used.

SEM Analysis

The Micro structures of Ferrogels were studied by Scanning electron Microscopy by using Hitachi, model-JSM-5000 imaging mode at 30 kV.

III. RESULTS AND DISCUSSION

1. IR spectral characterization of Ferrogel

The IR spectrum of poly(NTB-co-AM/ MA)Ferrogel was shown in Figure-1. The IR analysis of Ferrogel showed the presence of peaks corresponding to the functional group of all monomeric units present in the polymeric chain. The peaks were observed at 1227 cm^{-1} and 1336cm^{-1} tert-butyl group of NTB and at 1636 cm^{-1} C=O amide of both NTB and MA. Another peak at 1555 cm^{-1} due to C=O NH of AM unit. A broad at 3353 cm^{-1} corresponds to NH stretching of acrylamide. There was a new peak at 875 cm^{-1} for stretching vibration of Fe-O group of Fe_3O_4 particle and it conforms the formation of ferrate Nanocomposite Hydrogels.

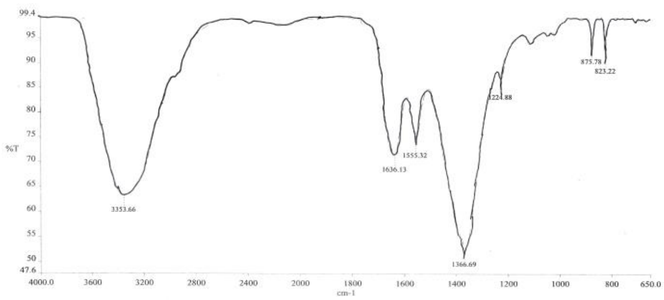

Figure-1. IR spectrum of poly (NTB-co-AM/MA)Ferrogel

2. Surface Morphology

The surface morphology of poly (NTB-co-AM/MA)Ferrogel is shown in Figure.-2 and the analysis conformed the ferrate(Fe_3O_4) particle were dispersed in the polymer matrix.

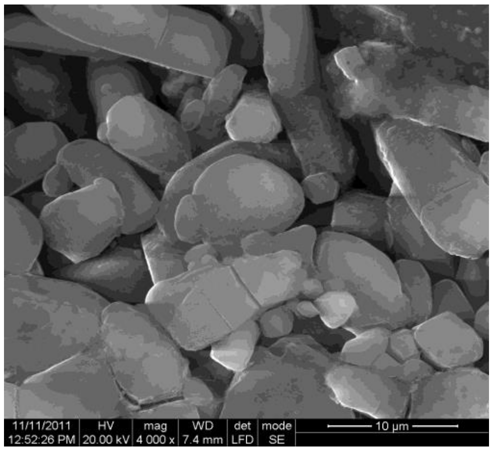

Figure 2. SEM image of the Ferrogel

3. Adsorption of Metals

Table-1. Removal of Metals from Industrial effluent using N-tert-amylacrylamide based Ferrogels

Metals	Removal of Metals at Time(Hrs)								
	Initial Conc.	2 Hrs	4Hrs	6Hrs	8Hrs	10Hrs	15hrs	20Hrs	24Hrs
Cr	2727.0	2027.0	1517.0	1021.0	865.8	769.9	526.8	521.9	501.2
Co	099.5	066.5	049.0	035.6	021.0	017.9	012.8	011.0	09.2
Ni	135.6	105.6	92.8	79.9	59.9	51.0	41.8	38.8	35.8
Cu	316.0	226.0	199.9	175.2	143.9	113.7	99.8	96.9	94.9
As	510.0	400.0	377.6	337.6	289.9	201.2	175.3	166.5	160.9
Se	355.5	305.5	271.2	241.8	195.2	166.4	134.2	129.3	126.4
Cd	376.6	286.6	258.2	208.9	185.9	155.8	101.9	97.9	95.9
Sn	574.5	485.2	435.9	395.8	346.9	326.3	281.0	275.0	269.5
Sb	515.8	485.6	426.8	385.9	315.0	295.0	245.9	239.8	232.9
Ba	2570.0	2021.0	1621.0	1221.0	885.3	735.3	585.4	580.2	569.6
Pb	065.7	056.0	048.7	035.7	025.9	020.9	016.8	015.2	014.1
Total Conc.	8246.2	6465.0	5298.1	4138.3	3334.7	2853.4	2221.7	2172.5	2110.4

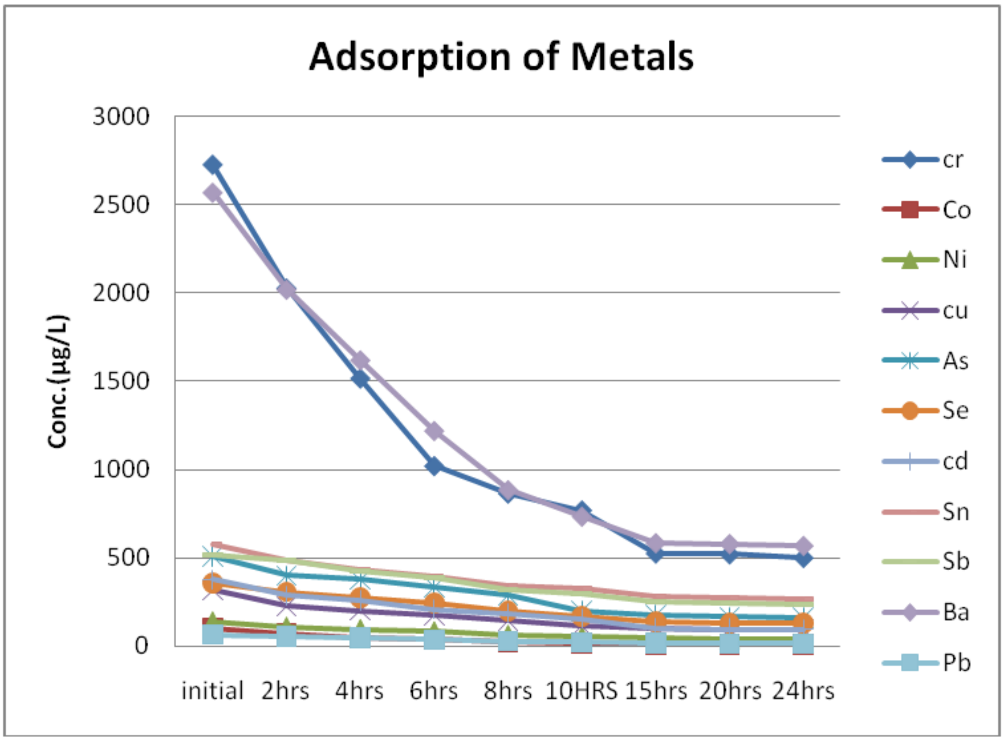

Figure-3. Removal of metal ions with time using Ferrogel

Table-2. Removal efficiency (RE %) of Metals from Industrial Dye effluent using N-tert- amylacrylamide based Ferrogels

Metals	Percentage of Removal (RE %)							
	2Hrs	4Hrs	6Hrs	8Hrs	10Hrs	15Hrs	20Hrs	24Hrs
Cr	25.7	44.4	62.6	68.3	71.8	80.7	80.9	81.6
Co	33.2	50.7	64.2	78.9	82.0	87.1	89.0	90.8
Ni	22.1	31.6	41.1	55.9	62.4	69.2	71.4	73.6
Cu	28.5	36.7	44.6	54.5	64.0	68.4	69.3	70.0
As	21.6	26.0	33.8	43.2	60.5	65.6	67.4	68.5
Se	14.1	23.7	32.0	45.1	53.2	62.3	63.6	64.4
Cd	23.9	31.4	44.5	50.6	58.6	72.9	74.0	74.5
Sn	15.5	24.1	31.1	39.6	43.2	51.1	52.1	53.1
Sb	5.9	17.3	25.2	38.9	42.8	52.3	53.5	54.8
Ba	21.4	36.9	52.5	65.6	71.4	77.2	77.4	77.8
Pb	14.7	25.9	45.7	60.6	68.2	74.4	76.9	78.5

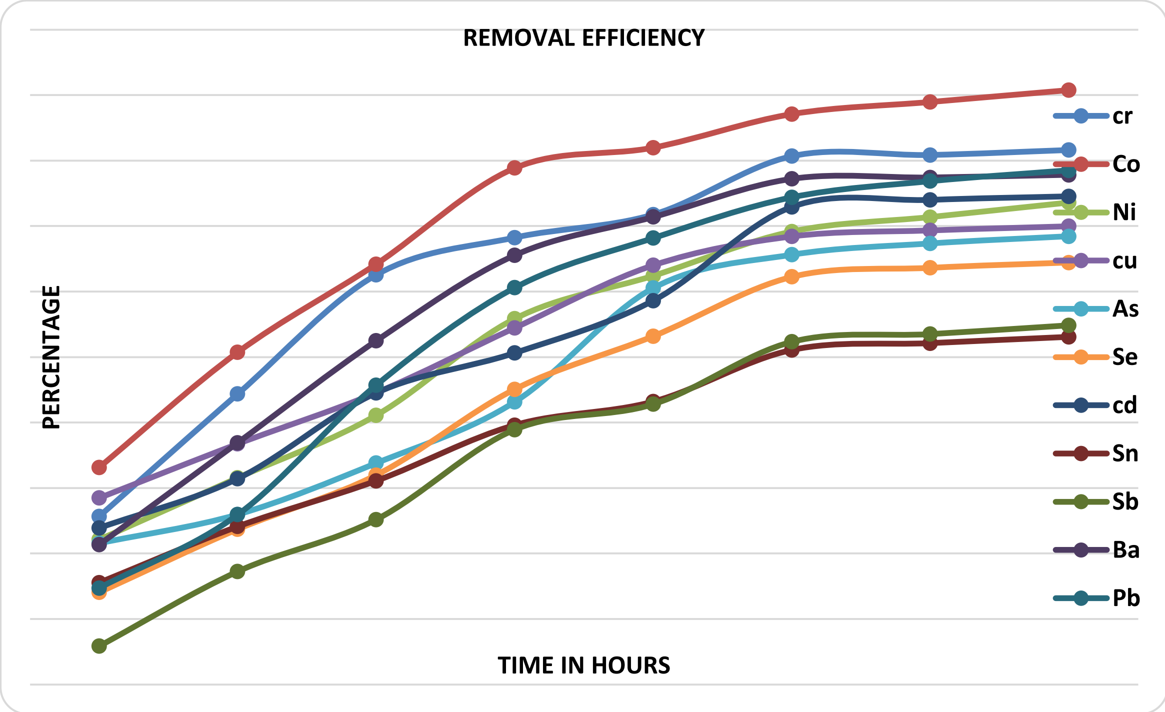

Figure-4. Removal efficiency of Ferrogel

The adsorptions of metals using ferrogel are shown in Table-1 and Figure-3 with respect to time. The adsorption of metals increased with increasing of time and it reached equilibrium v at 24 hrs. Individual metal adsorbed at various amount irrespective of its size, mostly heavy metals are adsorbed at maximum rate. The removal efficiency of Cobalt, Chromium and Lead are adsorbed at maximum of 90% (Figure-4). But the overall removal efficiency of all metal was 75%. Therefore these ferrogel material can be used as better adsorbent to remove the heavy metals from industrial effluent. Moreover the metals can be extracted from the adsorbed ferrogels by aqueous methanol and hence it can be reusable material.

IV. CONCLUSION

From this study, the removal efficiency of heavy metals from various textile industries effluent by adsorption on ferrogel has been found to be very useful for controlling water pollution due to hazardous heavy metals. It is indicated that the adsorption of heavy metal is influenced by amount of adsorbent (Ferrogel) and contact time. The removal of heavy metal is increased with increasing contact time. It is cheaply available. With this environment friendly adsorbent considerable heavy metal removal can be achieved. So it can be a better substitute for other adsorbent. With the experimental data it is possible to design and optimize the economical treatment process for heavy metal removal from the industrial effluent.

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