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Fabrication of PVA/Ag Nanocomposite Hydrogels using Sol Gel Technique: Swelling and Removal of Chromium Ion Studies

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Abstract: *The PVA/Ag nanocomposite hydrogel with 3D network has been fabricated via sol-gel method. The fabricated nanocomposites were well characterized by FT-IR, SEM, TEM and XRD. The swelling equilibrium has also been observed with various pH medium.*

Parallel adsorption studies have also been carried out with a different time, pH and dosage of adsorbent. The maximum removal of chromium for this sample is greater than other compound. Therefore, in this PVA/Ag nanocomposite hydrogel has a potential tendency for the removal of hexavalent chromium ion from industrial effluent wastewater.

Keywords: PVA, Swelling, Chromium ion removal, Composite, Waste water.

I. INTRODUCTION

Heavy metals are found in the environment due to various natural and anthropogenic activities. Heavy metal pollution is one of the most rapid pollution occurring due to industrialization is a serious threat to the environment and all forms of life [4]. Chromium is widely used by many industries and released into environment through various industrial processes including electroplating, metal finishing industry, iron, steel industries, inorganic chemicals production and tanneries etc[8].

Chromium exists in several oxidation forms ranging from -2 to +6 [9]. The most two common oxidation states which are III and VI. Cr (III) is hazardous only when it is found in high concentration, whereas Cr (VI) is toxic even at very low concentrations. Cr (VI) containing wastewater is treated by reducing to trivalent chromium in most cases before emitting into the environment [4]. The health hazards associated with exposure to chromium are dependent on its oxidation state.

Adverse effects of the hexavalent form on the skin may include ulcerations, dermatitis, and allergic skin reactions [1]. Inhalation of hexavalent chromium compounds can result in perforation of the mucous membrane of the nasal septum, irritation of the pharynx, larynx, asthmatic bronchitis, bronchospasms and edema as well as lung cancer [7]. In addition, Cr (VI) has a higher solubility and versatility than Cr (III) species expanding its perilous impacts. Accordingly, the evacuation of chromium, particularly the hexavalent chromium, is important before its discharge [5].

Various treatments employed for the removal of chromium from water include evaporation, deposition, reduction, precipitation, ion exchange and solvent extraction etc[11]. However, these methods are ineffective and require high energy for operation. Adsorption is considered as the most effective and widely used technique due to high removal efficiency, simplicity and low cost [6]. Adsorption is also an economical method and is very effective at low concentration of metal ions [4]. Hydrogel composites are one of the efficient adsorbents used in adsorption studies [2].

Polyethylene glycols are water soluble linear polymers and gel formation properties within them. Polyvinyl alcohol has excellent film forming, emulsifying properties which help in the formation of sol gel. Maleic acid is water soluble polymers which is a good binding agent. Nanocomposite and nano structured materials have unique properties such as large surface area, structure and bulk properties [12]. The properties nanomaterials have been an important application in environmental remediation and water purification [12].

In this study, the sol-gel method has been applied to synthesize silver nanocomposite for the adsorption of chromium from aqueous solution under batch conditions. The structure of the synthesized nanocomposite has been characterized by FT-IR, SEM, TEM and XRD. Moreover, the effects of pH, contact time and adsorbent weight in adsorption process were also investigated.

II. MATERIALS AND METHOD

All chemicals were of analytical grade. Chromium (VI) stock solution was prepared using potassium dichromate purchased. Silver nitrate (AgNO₃), Polyethylene glycol (PEG), Polyvinyl alcohol (PVA) and Maleic acid (MA) were used for the preparation of nanocomposite hydrogel. Distilled water was used for conducting the experiments.

A. Methods

Firstly, 2.835 g of Potassium dichromate was dissolved in 1000 ml of distilled water to obtain 1000 ppm stock solution. The concentration of chromium stock solution was measured using UV Spectrophotometer. The adsorbent gel was prepared by the sol gel method. 0.42 g of silver nitrate was dissolved in 1000 ml of distilled water. 0.25 g of polyethylene glycol (PEG), Maleic acid (MA) and polyvinyl alcohol (PVA) are dissolved in 20 ml distilled water. 50 ml of silver nitrate (AgNO₃) solution and 20 ml of the chemical solution are mixed and boiled in a hot plate at 100° Celsius until a colorless sol gel is formed. The formed gel is washed repeatedly using distilled water and then immersed in NaOH solution for about 30 minutes which then changes to brown color. The adsorbent gel is dried, washed, preserved and used for adsorption studies.

Table.1. Feed composition of adsorbent gel

S.NO	SAMPLE CODE	DISTILLED WATER (ml)	PVA (gm)	PEG (gm)	MA (gm)	AgNO ₃ (gm)
1.	PPM1	20	0.25	0.25	0.25	50
2.	PPM2	20	0.25	0.10	0.40	50
3.	PPM3	20	0.25	0.20	0.30	50
4.	PPM4	20	0.25	0.30	0.20	50

B. Experiment

The batch adsorption technique was adopted for adsorption studies. Swelling test was conducted for the adsorption studies. Adsorption studies were conducted for different parameters such as contact time, pH and dosage of adsorbent. About 20ml of the chromium stock solution was taken in a beaker at room temperature. Different dosage of adsorbent was taken in the beaker and after regular time interval and the concentration of the chromium solution is measured using UV spectrophotometer at 540nm [4].

III. RESULT AND DISCUSSION

A. Swelling Test

Swelling behavior was done by taking 0.1 g of dried adsorbent immersed in 100 ml of distilled water. Swollen gel was weighed at different time interval. The surface water was removed by filter paper [3]. The swelling equilibrium ratio was calculated using the following equation:

$$S_{eq}(g/g) = W_s - W_d / W_d \quad \text{----- 1}$$

where $S_{eq}(g/g)$ is equilibrium swelling ratio, $W_s(g)$ is the weight of swollen hydrogel sample, $W_d(g)$ is the weight of dry hydrogel sample.

Adsorption efficiency (E%) is given by the equation:

$$E = [(C_0 - C_t) / C_0] \times 100 \quad \text{-----2}$$

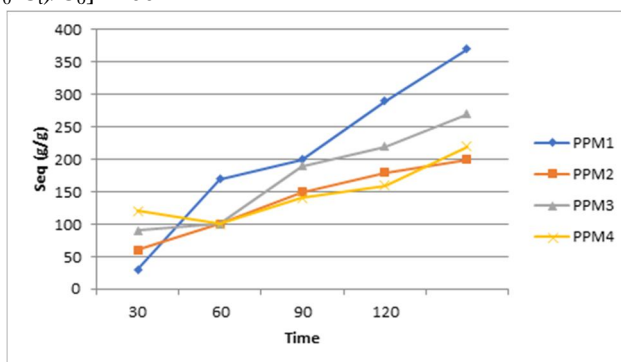


Figure-1: Swelling equilibrium of adsorbent at different time interval

B. Effect OF PH

Most extreme expulsion productivity of hexavalent chromium is higher at low pH. As the pH was enhanced from 2 to 9 effectiveness diminishes as appeared as shown in figure 1. Optimum pH was at 2 and every single other test were done at this pH [13]. As the pH value increases from 2 to 9 swelling was decreases due to weak interaction and the solubility of adsorbent. This causes reduction in adsorption between Cr (VI) and the adsorbent hydrogel network.

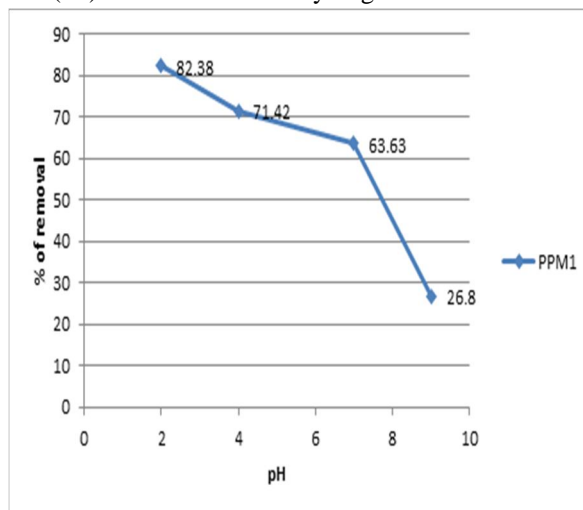


Figure- 2: Effect of pH of adsorbent at different pH

C. Effect of Dosage

As adsorbent dose increases, keeping the various parameters at steady esteem evacuation effectiveness first increases [4]. When the dosage of adsorbent has increases removal percentage also increases. Figure 3 clearly shows the percentage of removal of chromium ion depends on the dosage of adsorbent.

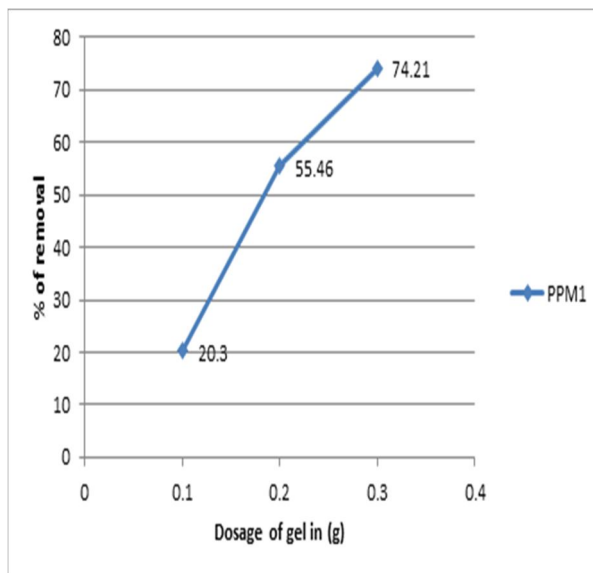


Figure- 3: Removal percentage of chromium ion at different dosage of adsorbent gel

D. Effect of Contact Time

As time of adsorption is changed from 15 to 90minutes, effectiveness originally diminished from 11.28 to 84.71%. As the time increases the surface inclusion of adsorbent is higher and further no adsorption happen. Figure 4 shows the impact of time on adsorption. Likewise change in Cr (VI) take-up with time is enhances the removal percentage also increases. Generally, when time increases swelling also increases [2].

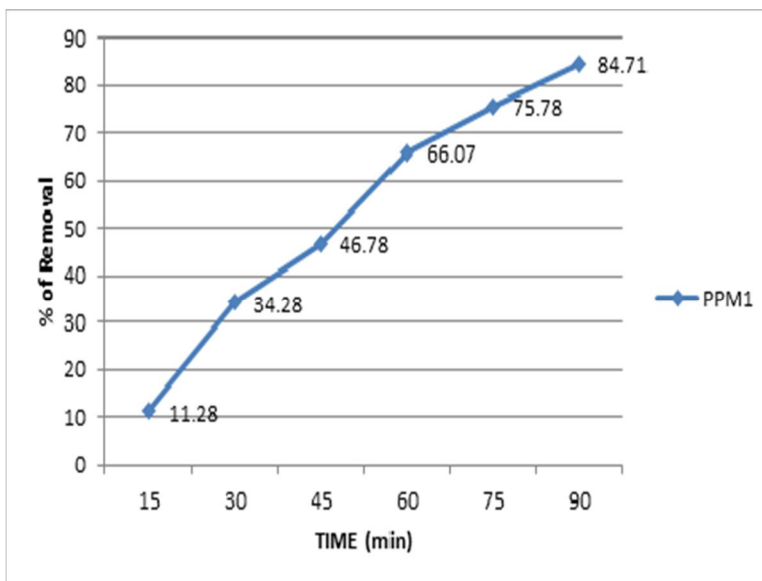


Figure- 4: Removal percentage of chromium ion at different time

E. TEM Analysis

The TEM image of silver nanocomposite is presented in figure 5 and it shows the porous surface of the adsorbent. It has spherical shape nanoparticle with 50 nm size of TEM image. It is confirmed by the formation of nanoparticles.

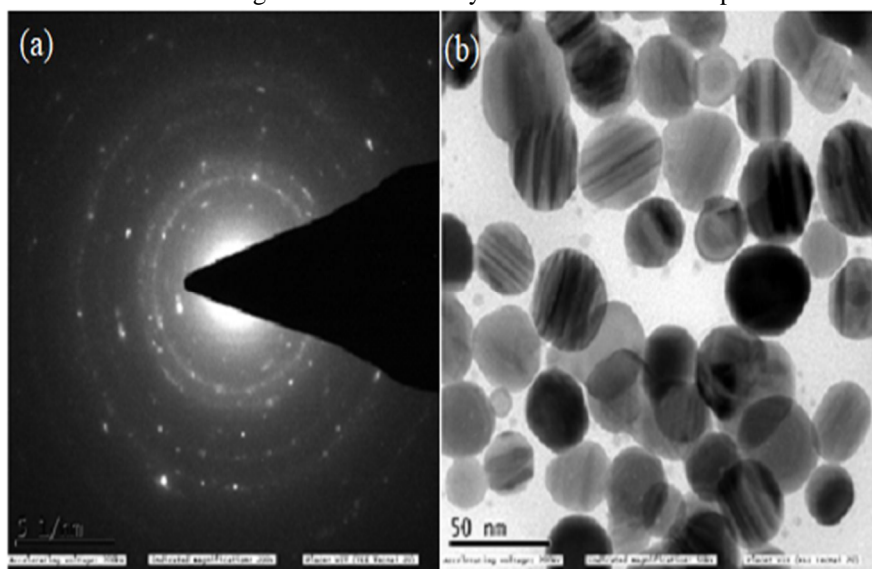


Figure- 5(a) &(b): TEM images of PVA/ Ag nanocomposite adsorbent

F. FT-IR Spectral Studies

In order to identify possible interaction and functional groups present on the surface of silver nanocomposite studies was performed by FT-IR studies. Before and after adsorption of Cr (+6) has been identified and confirmed by the changes of IR spectral. The FT-IR Spectra of PEG based hydrogel or adsorbent as shown in fig.6a. The peak has been observed at 3414 (OH stretching) 1647 (C=O Stretching) and 2881,2738 (C-H Stretching).

In PVA/ Ag nanocomposite, a peak at 1278.81 due to the C-O-C stretching vibration of PEG is observed, which indicates that PEG has been introduced into the composites. To identify the possible functional group on adsorbent involved in the Cr ion. FT-IR spectra were obtained before and after chromium metal ion adsorption. There was a small shift of the peak at 1643, 1427, 1091 and 840 cm^{-1} in PEG adsorbent to 3373, 1637, 1419, 1089 and 831 cm^{-1} for Cr^{+6} bonded adsorbent (Figure 6b). The peak 1419 cm^{-1} indicates the formation of PEG and Cr complex [10].

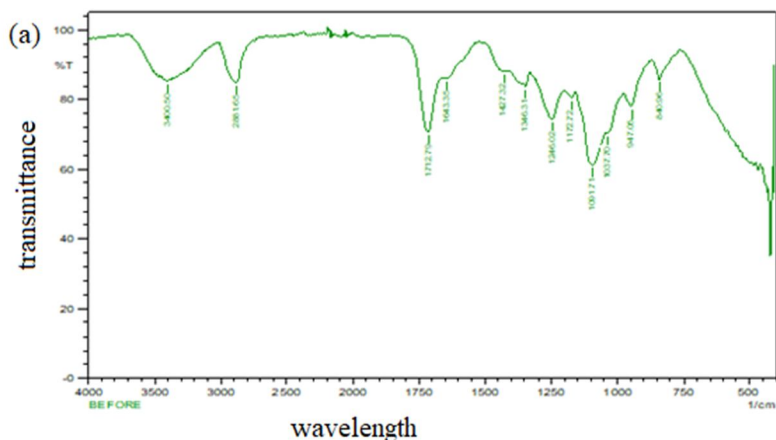


Figure- 6a: FT-IR spectra of the adsorbent before adsorption of chromium ion

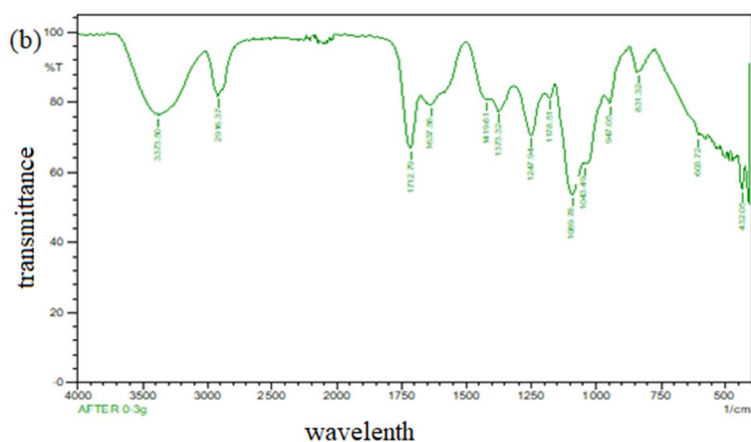


Figure- 6b: FT-IR spectra of the adsorbent after adsorption of chromium ion

G. PXRD Analysis

Fig 7 shows the XRD pattern of PVA/ Ag nanocomposites with comparative to before and after adsorption of chromium ions. The unloaded PVA/ AgNPs hydrogel has shown the peak position (2θ) 19.2, 24.3, 33.5, 39.1, 46.5, 55.1, 64.1 and 77.3 refers to the (101), (111), (420), (511), (202), (222), (242) and (311) crystal planes respectively. The coordination of PVA chain by addition of AgNPs inherent and develop PVA intermolecular chain network. However, the peaks due to the adsorption of chromium ion within the PVA/Ag nanocomposite network were not changed on shift or large variation. More or less most of the peaks were observed are similar. These results confirm that before and after adsorption of chromium ion in the PVA/Ag nanocomposite contains AgNPs strongly. In addition, it should be noted that even after the adsorption of Cr (VI) ion, the crystalline nature of the PVA/Ag nanocomposite was not significantly altered.

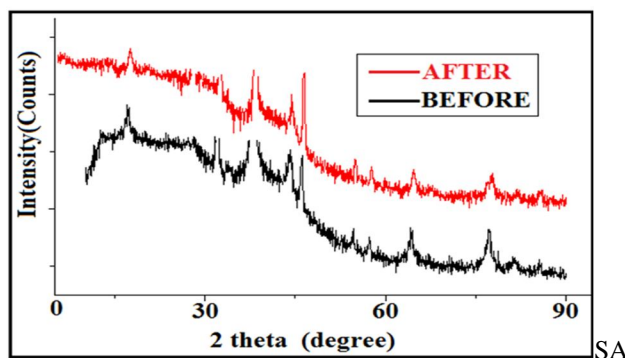


Figure- 7: XRD pattern of PVA/Ag nanocomposite for before and after adsorption of chromium ion

H. SEM Studies

The SEM images of adsorbent has shown rough and cracked surface morphologies provides excellent adsorption sites. It has been observed that surface of adsorbent after adsorption changed and the small cavities of the adsorbent had disappeared or filled up (Figure 8b).

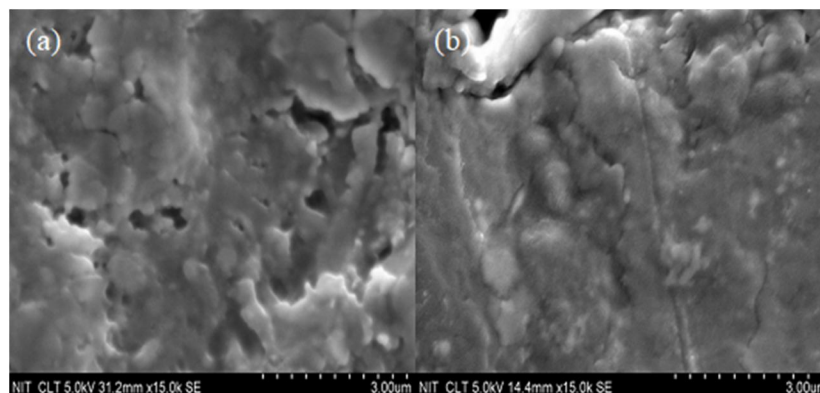


Figure- 8: SEM images of (a) before adsorption (b) after adsorption

IV. CONCLUSION

The nanocomposite hydrogel or adsorbent PVA/Ag was made using sol-gel technique. The structure of adsorbent has been developed. The PVA/Ag nanocomposite adsorbent is found to have an excellent capacity to adsorb chromium ions Cr^{+6} from wastewater and the removal percentage is found to be which is the greater when compared to other adsorbents. The synthesized adsorbent was characterized by FT-IR, SEM, XRD and TEM analysis. Hence, the present investigation reveals that the PVA/Ag nanocomposite might be used as promising material for heavy metal ion removal in different industries.

V. ACKNOWLEDGEMENT

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