Removal of cationic organic dye from their aqueous solutions efficiently and selectively by magnetic nanoparticles fabricated Hydrogel

A PROJECT WORK

SUBMITTED IN THE PARTIAL FULFILLMENT OF THE
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MASTER OF SCIENCE
IN

CHEMISTRY

SUBMITTED BY

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CANDIDATE'S DECLARTION

I Kanishka Kashyap (2K21/MSCCHE/61) student of M.Sc. (Chemistry) hereby declare that the project Dissertation titled " **Removal of cationic organic dye from their aqueous solutions efficiently and selectively by magnetic nanoparticles fabricated hydrogels** " which is submitted by us to the Department of Applied Chemistry, Delhi Technological University, Delhi in the partial fulfillment of the requirement for the award of the degree of Master of Science, is original and not copied from any source without proper citation. This work has not previously formed the basis for the award of any Degree, Diploma, and Associate ship. Fellowship or other similar title or recognition.

Place: Delhi

Date: 23/05/23

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CERTIFICATE

I/We hereby certify that the Project Dissertation titled " **Removal of cationic organic dye from their aqueous solutions efficiently and selectively by magnetic nanoparticles fabricated hydrogels** " which is submitted by Kanishka Kashyap (2K21/MSCCHE/61), Department of Applied Chemistry, Delhi Technological University, Delhi in partial fulfillment of the requirement for the award of the Master of Science, is a record of the project work carried out by the student under my supervision. To the best of my/our knowledge this work has not been submitted in part or full for any Degree or Diploma to this University or elsewhere.

Place: Delhi

Date: 23/05/2023 **Prof. Anil Kumar**

(Supervisor)

Dedicated

To

My Family

ACKNOWLEDGEMENT

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Kanishka Kashyap

ABSTRACT:-

In recent time, treatment of wastewater is the main concern for the life on earth. One of the major factors of waste water is organic dyes that lead to serious health and environmental threat to the ecosystem. Various methods were developed for removal of organic dyes from wastewater, adsorption is among the efficient and cost-effective technique. There are numerous varieties of adsorbents were used but I chose hydrogel Nano composites to enhance the adsorption capacity.

Using a hydrogel made of poly (acrylic acid) grafted onto Xanthan and guar gum (FeO-XG-GG-AA) and magnetic iron oxide nanoparticles (FeNp); a novel biocompatible magnetic iron oxide nanoparticle/hydrogel was created in this study. By using scanning electron microscopy with energy dispersive X-ray analysis (SEM-EDAX), X-ray diffraction, Fourier transform infrared spectroscopy (FT-IR), thermal gravimetric analysis (TGA), and UV spectroscopy, the structural, morphological, and magnetic properties of the optimized sample were characterized. The results show the hydrogel strength and stiffness are both increased by the addition of inorganic filler to the hydrogel polymer matrix which is more way powerful than that of a normal hydrogel. The magnetic Nano composite hydrogel has a way too much potential to be used as an adsorbent for the removal of CV dye from aqueous solution.

KEYWORDS-

Nanoparticles, Adsorption, Hydrogels, Dye removal.

CONTENTS

1. INTRODUCTION & LITERATURE REVIEW

As water is so crucial to our everyday existence, there is a constant need to preserve and improve its quality. In the forthcoming decades, the only alternatives for procuring fresh water considering the limited availability of our water resources are water treatment and recycling techniques[1]. Synthetic dye-related water contamination is now a major hazard on a global scale. Because of their coloring properties, synthetic dyes are utilized worldwide in a wide range of sectors including plastics, textiles, leather, paper, printing, pharmaceuticals etc[2]. Yet, these dyes are hazardous by nature and substantially pollute water when they are released into the environment by industries as waste products[3]. The majority of synthetic organic dyes are carcinogenic and toxic in nature, which has negative consequences on both marine life and human health and they can also cause allergic problems and skin irritations. Dyes are not biodegradable because of their higher water solubility and complex aromatic chemical structures, and are also resistant to aerobic oxidation[4].

It is essential to create dye treatment techniques that are simpler, more precise, and a little less expensive. Many methods, such as coagulation, ZoroCVa M. et al[5] developed chemical oxidation, membrane separation, catalytic degradation, activated carbon adsorption etc. are used for the removal of dyes from wastewater. Among them adsorption is one of the best techniques as it is simple and inexpensive to perform[6]. Huang et al.[7] developed a dye adsorbent made of graphene oxide modified zeolite to absorb cationic dyes (CV) in water. But due to its poor adsorption capability, it is not much effective as an adsorbent. The adsorption capability of these inorganic dye adsorbents, which have also been reported in numerous literatures, was not sufficient. As a result, they cannot be good choice for effective dye adsorbents[8].

Hydrogel are a class of polymeric materials that can hold plenty of water in their three-dimensional networks due to their hydrophilic structure. It is thought that this fact is extremely important for these items to be widely used in a variety of industrial and environmental application areas[9]. Many hydrophilic groups, including carboxylic (COOH), amide ($CONH_2$), amino groups (NH_2), sulfonic acid (SO_3H), and hydroxyl (OH), are present in hydrogels matrices. These hydrophilic groups offer plenty of active sites to accommodate foreign substances that are adsorbed on the surface of hydrogels[10].

These polymeric super-adsorbent hydrogels are specifically utilized in wastewater remediation via adsorption mechanisms as electrostatic, ion exchange, chemisorption, hydrogen bonding, hydrophobic, and complexation interactions between targeted dyes and adsorbents[11].

Exopolysaccharides, which are polysaccharides of microbial origin, were first identified in the 1950s. But over the past few decades, there has been a sharp rise in interest in polysaccharides for both food and non-food uses, largely because of their immense biotechnological application. Xanthan gum is an exopolysaccharide that is primarily derived from the strain X of the family Xanthomonas, which is a plant pathogenic microorganism. In order to lower production expenses, guar gum and locust bean gum are often combined with xanthan gum[12]. Guar gum, also known as Cymopsis tetragonolobus, is a significant agrochemical that is produced from the seed endosperm of the guar plant. It is a novel thickener and stabilizer because of its strong propensity to create hydrogen bonds in water[13].

Due to their high adsorption capabilities, low toxicity, and regeneration capacities, hydrogels are used as an adsorbent in numerous studies to remove pollutants from wastewater[14]. The hydrogel matrix can be made of synthetic[15], natural[16], or a mixture of the two types of polymers. For wastewater treatment, magnetite hydrogel Nano composites have undergone intensive research. A lot of focus has recently been placed on the Nano composite hydrogels used as adsorbents to remove harmful chemicals from water. By embedding the polymer networks of hydrogels with nanoparticles, hydrogel Nano composites can be created, which have improved water treatment performance. By adding organic or inorganic nanoparticles to hydrogels, it is possible to increase their mechanical strength, swelling characteristics, and ability to recover and reuse[10]. The drawbacks of traditional hydrogels would be resolved by research in this field. Inorganic nanoparticles like magnetite, charcoal, clay, and silica are just a few examples of the many that can be used to create NCHG. Depending on how the hydrogel chains and the nanoparticles' surfaces have been functionalized, the nanoparticles may serve as a physical or chemical cross linker to strengthen the hydrogel matrix[17]. Many methods like carbonaceous materials (viz., graphene and carbon nanotubes (CNTs)[18], metal chalcogenides[19] are reported.

A common form of magnetic iron oxide is magnetite (Fe_3O_4), which has a cubic inverse spinel structure with oxygen forming an FCC closed packing and Fe cations occupying the interstitial tetrahedral sites and octahedral sites[14] of adsorbent for the removal of organic dyes from aqueous solutions. M.Khairy et al.[23] synthesized TiO_2 nanoparticles on their photocatalytic activities toward removal of organic dyes.

Magnetite nanoparticle synthesis has been extensively researched and developed for a variety of biological technological applications, including the extraction of genomic DNA[20]. Applications in medical such as targeted drug delivery, labeling, separation[21].

Abbas afkhami et al.[22] synthesized Magnetic nickel zinc ferrite Nano composite as an efficient it is discovered that electrostatic interactions primarily promote dye adsorption on magnetic iron oxide nanoparticles that have opposing charges, and that this process is extremely sensitive to pH and ionic strength changes. Because to the combined effects of counter ion exchange and counter ion condensation, the effective electric charge on the magnetic iron oxide nanoparticles surface generally remains constant in a wide range of surface coverage's by dyes[24].

In this study, a quick and highly effective method for the removal of dyes using magnetic Fe_3O_4 hydrogel was proposed. Among other transition metals, Fe_3O_4 has the strongest magnetism and also has stable at ambient temperatures. A novel low-cost magnetic sorbent material was also created by incorporating magnetic iron nanoparticles into hydrogel matrix with affordable and environmentally friendly naturally occurring gums and iron salts.

By extensively cross-linking the polymer chains with nanomaterials, the addition of magnetic iron nanoparticles to hydrogels reduces the layer thickness (open space in the networks), resulting in larger pores and increased diffusion, high surface areas, and improved polymer water interactions to produce highly effective adsorbents[10].

There are numerous ways to create magnetic particles at the Nano scale, including ball grinding[25], sol-gel transformation[14], and hydrothermal synthesis. In this study synthesis of magnetite nanoparticles of iron salts by co-precipitation method is examined.

In light of this, we attempted to prepare MION with outstanding stability and size distribution into hydrogel in the current studies using a graft copolymer of poly (acrylic acid) onto xanthan and guar gum.Fourier transform infrared spectroscopy (FT-IR), scanning electron microscopy (SEM), and heat gravimetric analysis were used to characterize the optimized MION hydrogel. (TGA) and XRD technique is used for the Characterization of MION.

2. SYNTHESIS

2.1 CHEMICALS AND MATERIALS

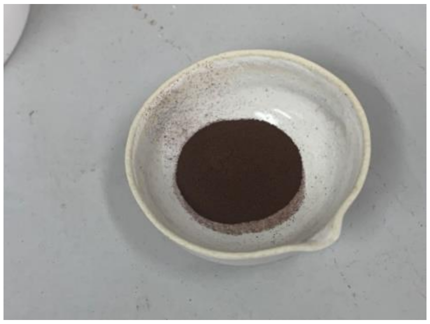
Iron (ll) Chloride tetra hydrate, anhydrous(lll) Chloride ($FeCl_3$), Sodium hydroxide (Fischer Scientific), Xanthan gum (S D fine chem. Ltd.), Potassium per sulfate (KPS, Fischer Scientific,), Acrylic acid (AA, CDH, New Delhi), and N, N'-methylene bis(acrylamide) (CVA, Merck, Germany), were consumed as provided.

2.2 SYNTHESIS OF MAGNETIC IRON OXIDE NANO COMPOSITE LOADED HYDROGELS

2.2.1 SYNTHESIS OF MAGNETIC IRON OXIDE NANOPARTICLES

Synthesis of magnetic iron oxide nanoparticles by co-precipitation method[26] was followed. $FeCl_2$ and $FeCl_3$ were dispersed in distilled water. Mole ratio was maintained 1:2 throughout the procedure. Temperature was maintained around 90^oC. Once the temperature reached stirring is given of approx. 30 min (stirring 300-500). Followed by the addition of 1M of NaOH drop wise and a color change from yellowish to black was observed immediately. pH was maintained 10-13 and stirring of two hours was given at temp 90^o resulting the formation of black precipitate. This mixture was then centrifuged at 4000 rpm for 15 min. To separate, precipitate was washed by ethanol and kept in oven for 24 hours for drying. The product was then calcined at 600^o C for 3 hours. After grinding these nanoparticles was stored in the absence of moisture, heat and light. The synthesized magnetic iron oxide nanoparticles are shown below:

(FeNp Before calcination)



(FeNp after Calcination)

2.2.2 SYNTHESIS OF XG-GG-AA HYDROGEL

A general procedure to synthesis hydrogels was conducted as follow: Xanthan gum (0.15) and Guar gum (0.15) was dissolved in 30 ml of distilled water and stirred until homogenous solution was formed. After the formation of homogenous mixture, 6 ml of acrylic acid was added into the mixture and stirred for 15 m followed by the addition of 0.5 g of NaOH in the mixture. Then 0.05 g of KPS and 0.065 g of MBA were added in the solution and stirring of 30 minutes was given. Then, the mixture was poured into test tube and kept in the water bath at 70^{o} C for 2 hours. After 2 hours hydrogel so formed was washed and chopped into pieces and dipped into distilled water overnight. On Next morning hydrogels was kept in oven for drying and kept in the absence of heat, light and moisture.

2.2.3 SYNTHESIS OF FEO-XG-GG-AA HYDROGEL

Xanthan gum (0.15) and Guar gum (0.15) was dissolved in 30 ml of distilled water and stirred until homogenous solution was formed. After the formation of homogenous mixture 6 ml of Acrylic acid was added into the mixture and stirring of 15 min was provided. After 15 min of stirring 0.05 of KPS initiator and various concentration (10 mg, 20mg, 30mg, 50mg) of iron oxide nanoparticles was incorporated via ex-situ in the solution and stirring of 30 min was given. The solution formed was poured into test tube and kept in the water bath at 70^{o} C for two hours. After two hours hydrogel so formed was washed and chopped into pieces and dipped into distilled water overnight. On Next morning hydrogels was kept in oven for drying and kept in the absence of heat, light and moisture.

3. EXPERIMENTAL SECTION

3.1 SWELLING STUDY

The well-known gravimetric approach was used to evaluate magnetic Nano-composite hydrogel of various concentrations (10mg, 20mg, 30mg, and 50mg). The dried hydrogel was sliced and weighed for this purpose. At the set times, the species were weighed after being submerged in deionized water. The bloated species were removed, the extra water on the surface was drained using filter paper, and the specimens were weighed once more. To check swelling ratio used equation is-

$$\boldsymbol{SR} = \frac{\mathbf{Wt - Wi}}{\mathbf{Wi}} \times \mathbf{100}$$

Where,

W_t is the swelled hydrogels at time t and W_i is the initial weight of the hydrogels.

3.2 ADSORPTION STUDY

The removal of the cationic dye CV was accomplished using the attempt-and-true batch procedure. The magnetic Nano-composite hydrogels has been immersed in 50 mL solutions of CV in a typical experiment with the concentration of 10 to 20 ppm. The removal procedures were carried out during contact intervals of 10 to 120 minutes. 10, 20, 30, and 50 mg iron loaded magnetic Nano-composite hydrogels was utilized to customize the sorbent dosage. A UV-vis spectrophotometer (UV-1800) was used to calculate the sorbent's removal effectiveness (R) and equilibrium adsorption capacity (qe) from 200 to 800 nm for CV by following equations-

$$\mathbf{R\% = \frac{Ci - Cf}{Co} \times 100}$$

$$\mathbf{qe = \frac{Ci - Cf}{W} \times V}$$

Where. Ci and Cf are the initial and final concentration of CV dye; V is the volume of dye solution (mL) and W is the mass of sorbent.

3.3 pH STUDY

The effect of pH on the adsorption process was studied at pH 5 to 9. For this 50 mg MION hydrogels was immersed in the 50 ml of 20ppm dye solution of pH 5 and 9. The removal procedures were carried out during contact intervals of 10 to 120 minutes. A UV-vis spectrophotometer (UV-1800) was used to calculate the sorbent's removal effectiveness (R) and equilibrium adsorption capacity (qe) from 200 to 800 nm for CV by above equations.

3.4 DESORPTION AND REUSABILITY STUDY

The desorption and reusability behavior of hydrogels loaded with magnetic iron oxide nanoparticles was investigated over the course of three consecutive cycles of adsorption and desorption .The magnetic Nano composite hydrogels that were dye-loaded were agitated in various and dye concentrations 10 to 20 ppm. As the outcome, the magnetic Nano composite hydrogels was separated from the solutions, and a UV-vis spectrophotometer was used to measure the dye concentration by the equation-

$$\mathbf{\%desorption = \frac{concentration\ of\ desorbed\ dye}{concentration\ of\ adsorbed\ dye}}$$

4. CHARACTERIZATION

4.1 UV SPECTROSCOPY

The (UV-1800 UV) spectrophotometer was used to provide the UV-visible spectra.

4.2 PARTICLE CHARACTERIZATION TECHNIQUES

With the use of Fourier Transform Infrared Spectroscopy (FTIR), X-Ray Diffraction (XRD), and Scanning Electron Microscopy (SEM), FeNp hydrogels produced under all processing conditions were systematically characterized in terms of functional groups, phase composition, crystallite size, and morphology structure.

4.2.1. Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectrometer was performed in Investigation of the functional groups in FeNp hydrogels using an FTIR spectrometer with a resolution of 4000-400 cm^{-1} was carried out. According to Herman S. Mansur et.al.[27] the iron oxide group (Fe-O), hydroxyl group (OH-), and atmospheric carbon dioxide (CO2) vibrations are the most distinctive functional groups in the FTIR spectrum of synthesized FeNp hydrogels. In order to get a more precise result, the powder sample was finely pulverized before being applied to the plate.

4.2.2. X-RAY DIFFRACTION (XRD)

X-Ray Diffractometer was used to determine the phase composition of FeNp hydrogels. The investigation of phase composition and crystallite size was produced using a wavelength of 0.15406 nm over a range between 5° and 80° and a slow scanning rate of 2. FeNp hydrogels' crystallite size was assessed using the peak broadening of XRD.

4.2.3. SCANNING ELECTRON MICROSCOPY (SEM)

SEM was employed for examining the FeNp hydrogel nanopowder surface. When secondary electrons were detected, a concentrated electron beam was traversed over a sample surface to produce an image.

4.2.4. THERMO-GRAVIMETRIC ANALYSIS (TGA)

In a nitrogen (N2) atmosphere, a thermogravimetric analyzer (TGA) was used to assess the thermal stability of FeNp hydrogels. For the examination of synthesized FeO-XG-GG-AA hydrogel, a Model-SDTQ600 TGA instrument from the USA was used. In order to avoid contamination, the analysis was carried out at a heating rate of 10°C/min in a nitrogen environment with a purging rate of 100 mL/min.

5. RESULTS AND DISCUSSION

5.1. PHZPC MEASUREMENT

The FeO-XG-GG-AA hydrogel's pHzpc value was determined to be 4.69 (Regression coefficient (R2) 0.987. The FeO-XG-GG-AA adsorbent's surface will therefore be negatively charged at pHzpc and positively charged below pHzpc. Due to effective physical interactions between the dyes and hydrogel matrix, FeO-XG-GG-AA hydrogel can effectively absorb cationic dyes at pH values higher than 4.65.

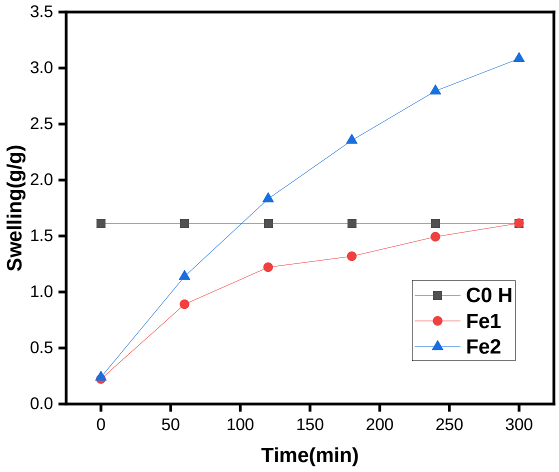
5.2. SWELLING BEHAVIOR

The magnetic Nano composite hydrogels' swelling characteristics were studied over a numerous time range. The volume of the sorbent considerably increased during swelling, which can significantly improve dye uptake. The swelling ratio in all loaded hydrogels increased throughout increasing time, reaching its peak after 5 hours. Maximum Swelling was observed in 50 mg magnetic iron oxide nanoparticles loaded hydrogel. The details are shown in table 1, figure 1 and shown by the images below.

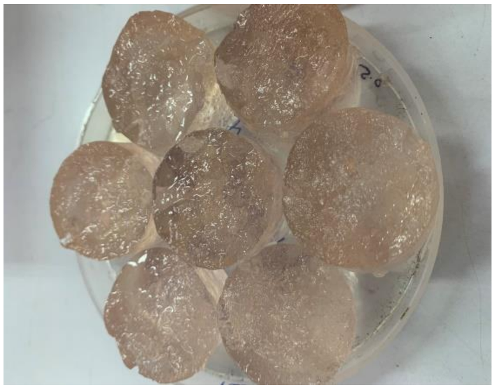
Table 1: Swelling Details of Various FeNp loaded Hydrogels.

Time	Controlled hydrogel(without FeNp)	20 mg FeNp hydrogels (Fe1)	50 mg FeNp hydrogels (Fe2)
Initial	0.246	0.298	0.311
1 hour	0.653	0.8903	1.139
2 hour	0.6818	1.2212	1.8319
3 hour	1.2403	1.3192	2.3545
4 hour	1.3515	1.4939	2.7949
5 hour	1.5099	1.6146	3.0836
24 hour	1.6483	1.7462	4.0764
48 hour	1.6616	1.7862	4.258

Figure 1:



(Initial Hydrogels)



(Swollen Hydrogels)

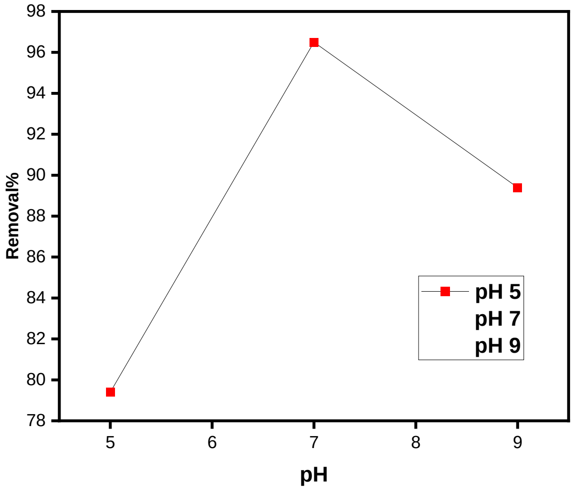
5.3 REMOVAL CONDITION OPTIMIZATION

The following are the most crucial variables that affect the removal process: pH, initial dye concentration, adsorbent dosage, and contact time.

5.3.1. EFFECT OF pH

In the pH range of 5 to 9, the impact of pH on CV dye removal effectiveness was investigated. Due to its positively charged surface, the sorbent was unable to show appropriate removal efficiency at acidic pH values. Contrarily, because of the excellent physical contacts (such as ionic and hydrogen bonding) between the dyes and the hydrogel matrix, the efficiency of dye removal above pH 8 was improved shown in figure 2 below.

Figure 2:



The highest removal efficiency for CV (96.25%) was obtained at pH 7.

Because of ionic interactions governed by the surface charges of the adsorbent and adsorbate in aqueous solution, cationic dyes bind to the FeNp-loaded hydrogels. The PZC predicts the charge on the adsorbent's surface. At pH levels greater than pHzpc, the FeNp loaded hydrogels has favorable removal efficiency for cationic dyes. The pHzpc of the hydrogels with FeNp loading is discovered to be (). As a result, cationic dyes are less effectively adsorbed by FeNp hydrogels at lower pH (pH < pHzpc) due to protonation of the functional groups present in the hydrogels loaded with FENp. Consequently, less adsorption is seen in acidic solutions.

5.3.2. EFFECT OF ADSORBENT DOSAGE

Adsorbent dosage optimization in the adsorption process is a crucial step because of its commercial and environmental benefits. As the adsorbent dosage was increased to 50 mg, the removal effectiveness of all dyes rose steadily.

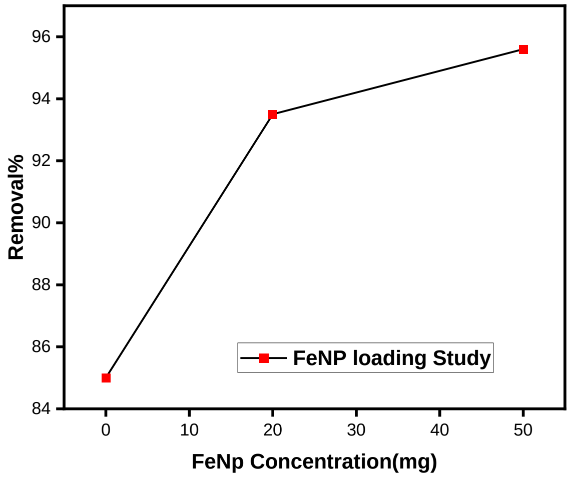
The maximum removal of CV in optimum sorbent dosage i.e. 25mg and 50 mg magnetic iron oxide hydrogels were obtained as 93.3% and 95.66% respectively.

5.3.3. EFFECT OF MAGNETIC IRON NANOPARTICLES LOADING IN HYDROGELS

The addition of magnetic iron oxide nanoparticles up to 50 mg constantly boosted the removal efficiencies of CV dye.

The maximum removal of CV dye in optimum loading of magnetic iron oxide nanoparticles hydrogels dosage i.e., 20, and 50 mg along with blank hydrogel (without iron np loaded) are 94.9%, 95.66% and 91% respectively shown by Figure 3.

Figure 3:



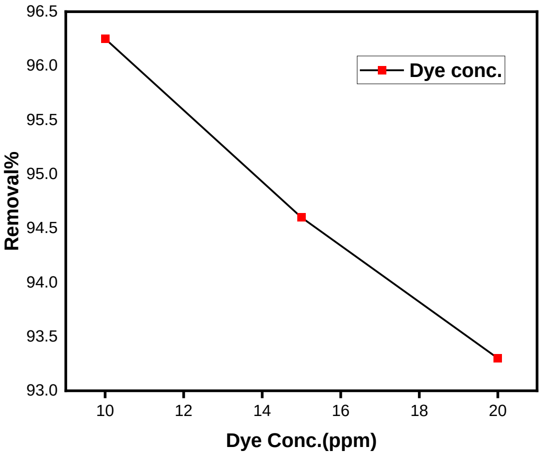
5.3.4. EFFECT OF INITIAL DYE CONCENTRATION

In most cases, dye-containing wastewater was concentrated before treatment. In order to maximize the effectiveness of adsorption processes, initial dye concentration optimization is very important. At greater concentrations, however, the saturation of the adsorbent active sites resulted in less dye being adsorbed. This is due to the fact that a significant number of binding sites are available for the initial adsorption of dye molecules due to the quantities of binding sites of a fixed amount of FeNp loaded hydrogels being fixed. Higher dye concentrations result in more cationic dye molecules in the

Solution, but fewer binding sites are accessible on the FeNp-loaded hydrogels for further adsorption, which leads to a decrease in dye adsorption.

In this study, the removal efficiency of magnetic iron oxide nanoparticles loaded hydrogels at 20 to 10 ppm was 96.25% to 93.3% shown in figure 4.

Figure 4:



5.3.5. EFFECT OF CONTACT TIME

The effect of contact time on removal efficiencies of CV dye was investigated. The highest removal efficiencies for CV dye were found to be 120 minutes, i.e. **96.25%**, beyond which time there is little change in the removal efficiencies of each dye. Therefore, the ideal contact time for CV dye was determined to be 120 minutes shown by UV data (Figure 5&6) and images below. The explanation is as follows: a large number of adsorption sites are initially available for interacting with the dye molecules, increasing the effectiveness of dye removal. Whereas after attaining equilibrium, the dye molecules cannot be added to the adsorbent to use the binding sites for adsorption there.

Figure 5:

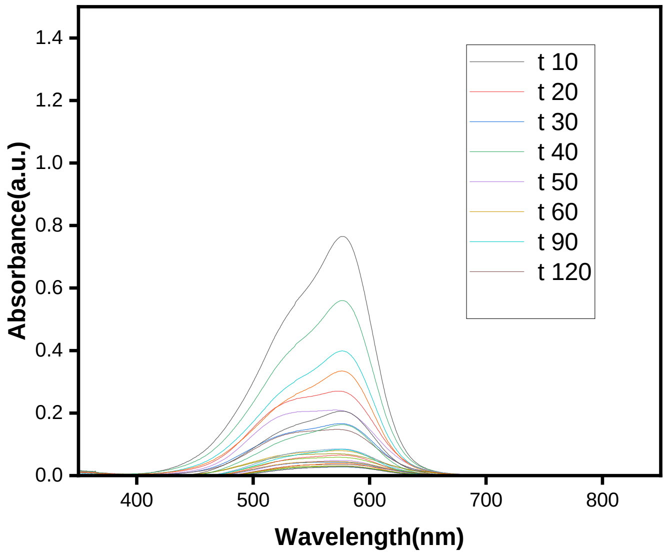
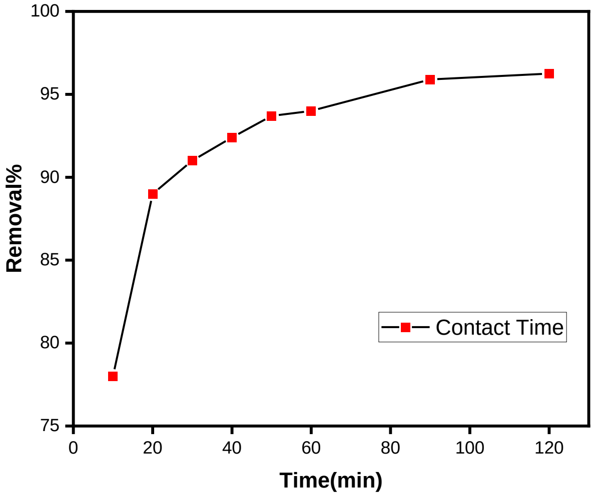
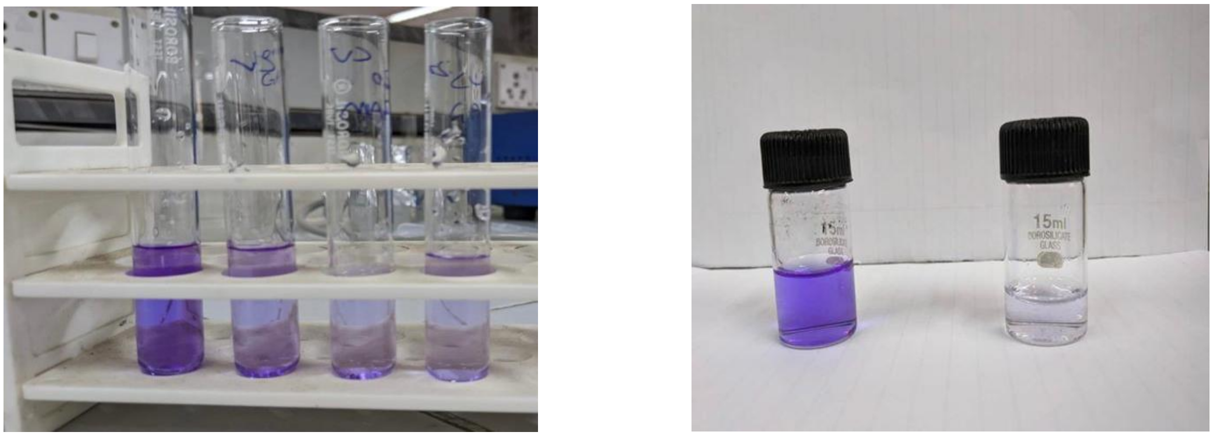


Figure 6:





(Removal of CV with contact time)

5.3.6. EFFECT ON DESORPTION AND REUSABILITY

Regeneration and reusability of an adsorbent in industrial processes are essential stages from an industrial and economic point of view. As experiment results, the 50 mg of FeNP hydrogels with 50 mg of FeNp loading showed the highest desorption efficiency i.e. 91%. The adsorbent was then taken for many adsorption-desorption cycles under optimal conditions in order to regenerate it after the desorption process described above. For the first three cycles, the removal efficiency of CV dye using FeNp hydrogel nearly remained constant; however, after that, the removal efficiency steadily declined due to saturation of the adsorbent surface, which reduced the availability of attachment sites.

6. FENP HYDROGELS CHARACTERIZATION ANALYSIS

6.1. FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

An essential method for examining the biomolecules found in FeNp hydrogels is FTIR spectrum analysis. To find the functional groups and organic compounds present in FeNp hydrogels, the FTIR spectra were examined. Using FTIR technology with a resolution of 400-4000cm^{-1}. Figure 7&8 displays the results of the tested ATR-FTIR spectrum of blank hydrogel without FeNp (H1) and FeNp incorporated hydrogel (H2) respectively.

Figure 7.

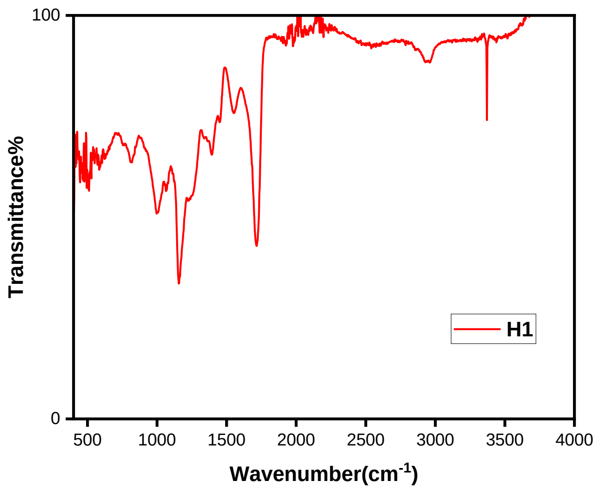
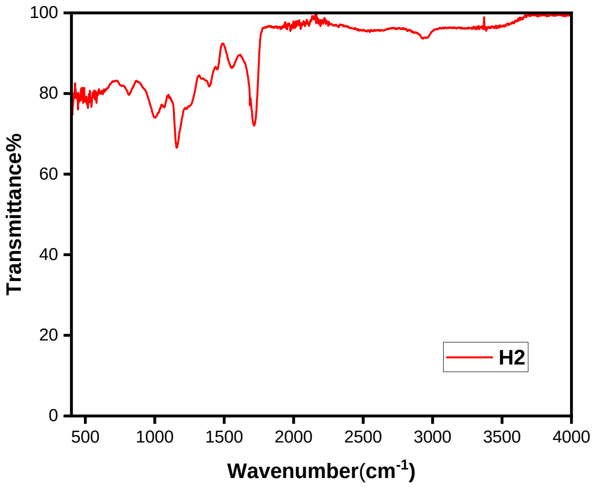


Figure 8.



From Figure 7, The bands at 3246 and 2881 cm^{-1} that were related to OH stretching vibrations with intermolecular hydrogen bonding and CH stretching, respectively, were shown by the pure XG was shifted to 2942 cm^{-1} due to crosslinking. Due to the polymer's O-H stretching vibrations combined with the C-H stretching vibrations, GG displays the distinctive absorption band at 3380 cm^{-1}. With this from figure 8, The oxygen in AA groups can interact with the surface of MION, as seen by the substantial decrease in intensity, slight shift of the C=O peak to 1,698 cm, and broadening of the OH peak in the FT-IR spectra of the FeNp hydrogel. The Fe-O bond peak is visible at 550–600 cm1, confirming the presence of Fe in the FeO–XG–GG–AA hydrogel.

6.2. X-RAY DIFFRACTION ANALYSIS

The structural organization and crystallinity of the materials were investigated by carefully examining and comparing the PXRD plots of FeNP and FeO-XG-GG-AA hydrogel Figures 9&10. The Structural analysis obtained from XRD figure 3&4 it shows that the particles are made of Fe_2O_3. It also shows that the particles with the smallest Z-average diameter were produced at a concentration of FeCl3 of 0.02 M. Consequently, 0.02 M was the ideal concentration for FeCl3. FeO-XG-GG-AA hydrogel did not exhibit any crystallinity in its PXRD pattern. Instead, the small, intense peaks that

Were visible in the FeO-XG-GG-AA hydrogel's graph were changed into a broad, narrow peak at 2θ = 25.98°, demonstrating that the combination of FeNp and XG-GG-AA was evenly distributed throughout the hydrogel matrix and pointing to a more disordered structure than controlled hydrogel. The formation and amorphous nature of the FeO-XG-GG-AA hydrogel were therefore confirmed.

Figure 9.

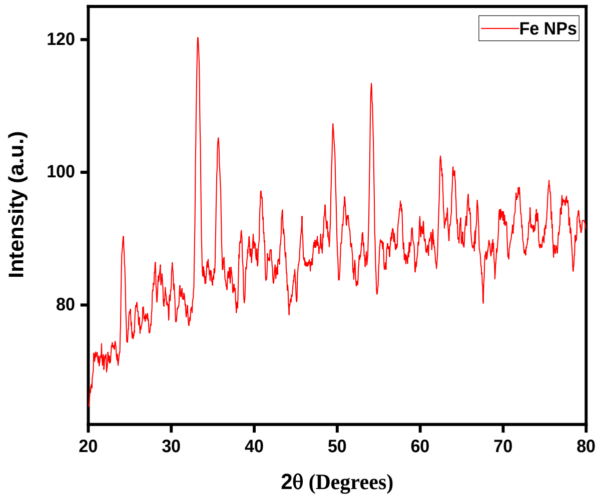
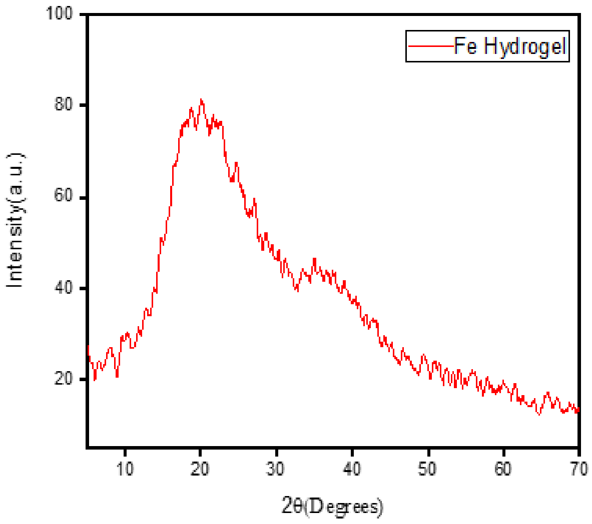


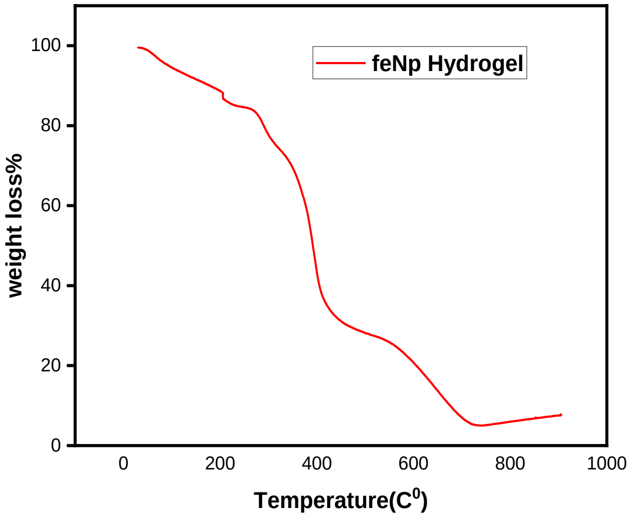
Figure 10.



6.3. THERMO-GRAVIMETRIC ANALYSIS (TGA)

The outcomes of a thermal investigation on the FeO-XG-GG-AA hydrogel are shown in Figure 11.It loses weight in four stages between 31 and 730 °C. The elimination of adsorbed water causes the first stage of weight loss to begin at 30 °C and last up to 205 °C, during which there was a 12% weight loss. The degradation of polymer chains may take place in the second step from 205 to 259 °C, the third step from 327 to 480 °C, and the fourth step from 524 to 720 °C.

Figure 11:



6.3 SCANNING ELECTRON MICROSCOPY (SEM) ANALYSIS

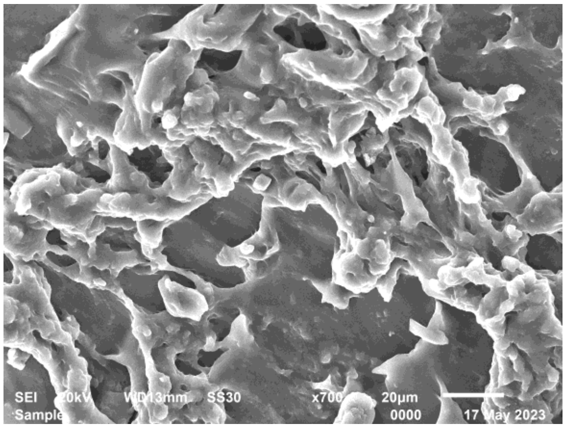


Fig.12 (Hydrogels without FeNp)

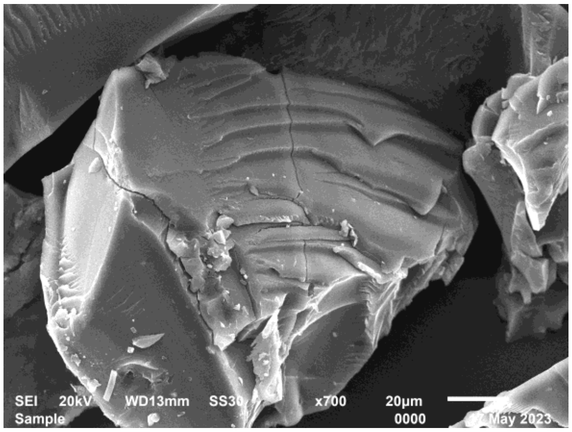


Fig.13 (FeNp incorporated Hydrogels)

SEM images of XG-GG-AA hydrogel (hydrogels without FeNp) and FeO-XG-GG-AA hydrogels (FeNp incorporated hydrogels) hydrogel were shown in Figures 12 and 13. The XG-GG-AA hydrogel (hydrogels without FeNp) has a very irregular and porous surface, which permit the hydrogel to effectively incorporate magnetic iron oxide nanoparticles. While in an SEM microgram of FeO-XG-GG-AA hydrogels (FeNp incorporated hydrogels), a regular surface and fewer pores are viewed, as magnetic iron oxide nanoparticles occupy the pores.

7. CONCLUSION-

To create an effective adsorbent for the removal of a cationic dye, CV, the synthesis of FeO-XG-GG-AA hydrogel matrix was studied. By analyzing the materials using various techniques like FTIR, XRD, TGA, SEM, and UV, the formation mechanism was examined. Nanoparticles appeared to be uniformly distributed inside the polymeric hydrogel network and produced a novel 3D structure with a high porosity and growing surface area, according to morphological and structural examination by SEM and FTIR. In order to determine the impact of various factors on the adsorption of CV, comparative batch tests using controlled hydrogel and FeO-XG-GG-AA hydrogel were conducted. These parameters included adsorbent dosage, initial dye concentration, contact time, pH, ionic strength, and temperature. The FeO-XG-GG-AA hydrogel's 96.5% CV adsorption capacity was raised by the increased surface area and active functional groups, which improved the availability of the active binding sites. Therefore, the FeO-XG-GG-AA hydrogel produced by the ex-situ incorporation of magnetic iron oxide nanoparticle into XG-GG-AA hydrogel provided an excellent cost-effective separating platform for long-term application of the materials in the wastewater purification process. It also demonstrated remarkable adsorption efficiency, dye removal ability in a wide variety of solution conditions, and reusability.

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9. LIST 0F CONFERENCES ATTENDED

1. Presented my own research work at the International Conference on **"*Chemical and allied Science and their applications*** "held at Delhi Technological University on 20th January, 2023.
2. Presented my own research work at International Conference on "***Recent Trends in Chemical Sciences-2023***" held at Miranda House, University Of Delhi on 14&15 February 2023.
3. Presented my own research work and in poster presentation at International Conference on *"**Recent Trends in Chemical Sciences and sustainable energy**"* held at National Institute of Technology, Delhi on 24&25 January 2023.

CERTIFICATE

This is to certify that ......Kanishka Kashyap.......................................from Delhi Technological University, Delhi.. has participated and presented the work (Oral Presentation) in International Conference on "**Chemical & Allied Science and their Applications**", organized by **Department of Applied Chemistry**, **Delhi Technological University** on Friday, 20th January, 2023.

Convener
Prof. Ram Singh

Richa
Co-Convener
Dr. Richa Srivastava

Prof. Anil Kumar
Head of Department

Department of Biotechnology

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INTERNATIONAL UNION OF PURE AND APPLIED CHEMISTRY

Miranda House
University of Delhi

International Conference

on

Recent Trends in Chemical Sciences-2023

This certificate is awarded to Kanishka Kashyap
from Department of Applied chemistry, D.T.U for participating in Oral Presentation in the International Conference on Recent Trends in Chemical Sciences-2023 held on 14-15 February 2023, organized by Department of Chemistry, Miranda House, University of Delhi, Delhi.

Prof. Mallika Pathak
Convenor

Prof. Bijayalaxmi Nanda
Principal

NITD/AS/RTCSSE/2023/69

Azadi Ka Amrit Mahotsav

G20

1st International Conference on
"Recent Trends in Chemical Sciences & Sustainable Energy"
(RTCSSE-2023)

CERTIFICATE OF PARTICIPATION

This is to certify that Prof./Dr./Mr./Ms. ***Kanishka Kashyap, Priyanka Yadav, Dr. Anil Kumar*** of Delhi Technological University, Delhi, India presented a oral/**poster** paper titled ***Removal of Cationic Organic Dye from their Aqueous Solutions Efficiently and Selectively by Magnetic Nanoparticles Fabricated Hydrogel*** the 1st International Conference on "Recent Trends in Chemical Sciences & Sustainable Energy" RTCSSE- 2023 held at National Institute of Technology Delhi during 24th - 25th March, 2023.

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Convener, RTCSSE- 2023