

Investigation of Heavy Metal Ions and Organic Dyes removal via Cellulose-Chitosan Hydrogel Coated Magnetite

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Abstract

This project investigated the removal of heavy metal ions and organic dyes using cross-linked cellulose-chitosan hydrogel coated magnetite for water purification. The experiment involved extracting cellulose from newspaper, then cross-linking it with commercially available chitosan to form hydrogel beads and then finally coating it on magnetite, which was synthesised via co-precipitation. The beads were then investigated on its properties in removing heavy metal ions (Copper(II) and Zinc ions) and organic dyes (Methylene Blue, Brilliant Green, Direct Red). In conclusion, magnetite, followed by the hydrogel coated magnetite beads and then hydrogel beads, was the most effective in the removal of the tested pollutants. The cellulose-chitosan hydrogel coated magnetite showed effective adsorption abilities to all pollutants tested. Out of the pollutants tested, the hydrogels had an affinity towards adsorbing Cu^{2+} and Brilliant Green.

1. Introduction

This project aims to investigate the removal of heavy metal ions and organic dyes using cross-linked cellulose-chitosan hydrogel coated magnetite for water purification. Recently, there has been increasing concern about water pollution all over the world, and these problems are expected to worsen in the coming decades, with water scarcity occurring globally (Shannon et al., 2008). The main concerns of water pollution are inorganic and organic materials such as heavy metal ions and organic dyes (Alo, 2020). Metals are introduced in aquatic systems as a result of the weathering of soils and rocks, from volcanic eruptions and a variety of human activities involving the mining, processing or use of substances that contain metal pollutants. Thus, when a high amount of metal ions are being released into the water and people drink such wastewater, toxic amounts of heavy metals enter the body and accumulate in their tissues, the resulting poisoning can cause serious damage, such as serious health problems and water-borne

diseases like cholera and kidney damage (Sana & Fazal-Ur-Rehman., 2018). Dyes on the other hand are highly toxic and pose a strong tendency toward eutrophication (Prevot et al., 2001).

Based on several studies, cellulose and chitosan are the two most abundant biopolymers available on earth and are environmentally friendly (Shen et al., 1995). Cellulose, when used as a blending polymer for chitosan can provide strong mechanical strength and improve the chemical stabilities of the hydrogel, whereas chitosan can prevent particle agglomeration, which can serve as an effective coating to stabilize the magnetite (Liu et al., 2012). As such, chitosan and cellulose were chosen to be combined to form the hydrogel. By making use of the abundant amine ($-NH_2$) and hydroxyl ($-OH$) functional groups of cellulose and chitosan, it is likely to enhance the adsorption capacity of the magnetite for heavy metal ions and organic dyes. By adding cross-links between polymer chains, it is likely to increase the adsorption capacity of the magnetite and the hydrogel's durability as this process results in the polymer becoming elastic, tougher, stronger and insoluble (Maitra & Shukla, 2020).

We plan to use the cellulose-chitosan hydrogel coated magnetite because it is relatively cheaper to make the hydrogel as other techniques of removing heavy metal ions and organic dyes are either expensive, energy-demanding, include chemical coagulation (Gecgel et al., 2013) or require advanced oxidation processes (Silva et al., 2013). Hence, this study would be one that investigates a cheaper, easier, and more environmentally friendly way to remove heavy metal ions and organic dyes. It also aims to compare the percentage removal of magnetite and magnetite coated with cellulose-chitosan hydrogel, as well as compare the percentage removal using hydrogel with and without magnetite.

2. Objectives and Hypothesis

2.1. Objectives

- To synthesise cross-linked cellulose-chitosan hydrogel beads and coat it on magnetite.
- To investigate the adsorption abilities of the cellulose-chitosan hydrogel coated magnetite beads on organic dyes (Methylene Blue, Brilliant Green, Direct Red) and heavy metal ions (Copper(II) and Zinc ions) in water.
- To compare the adsorption ability of cellulose-chitosan hydrogel coated magnetite beads

with that of magnetite and cellulose-chitosan hydrogel beads without magnetite.

- To investigate how contact time would affect the percentage removal of the pollutants.

2.2. Hypotheses

- Cellulose-chitosan hydrogel can be coated onto magnetite.
- Cellulose-chitosan hydrogel coated magnetite will yield a better adsorption of heavy metal ions and organic dyes than that of magnetite and the hydrogel without magnetite.

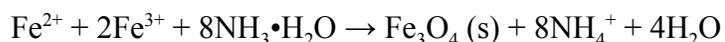
3. Materials and Methods

3.1. Extraction of Cellulose from Newspaper

Newspapers were obtained from the school's office and were cut into small pieces. 100 g of cut newspapers were treated in 700 ml of 5 % sodium hydroxide (NaOH) solution at 80 °C for 3 h. It was then removed and washed with deionised water (DI water) until pH 7. It was then treated in ethanol solution at room temperature overnight and then removed and washed again until pH 7. It was then soaked in hydrogen peroxide for 2 h and then removed and washed again until pH 7. Finally, the cellulose was then dried in an oven at 80 °C for 48 h before it was blended and stored for further use. The dried cellulose was then analysed by FTIR spectroscopy.

3.2. Synthesis of magnetite via co-precipitation

Iron (II) and Iron (III) salts were mixed in a molar ratio of 1:2 in DI water. 20 ml of aqueous ammonia (14 wt%) was added to the iron solution until pH 10 in a fume hood. The solution was then vacuum filtered for 24 h and allowed to cool to room temperature afterwards. The black precipitate of magnetite was collected and left to dry in an oven at 80 °C for 48 h before it was blended and stored for further use. The dried magnetite was then analysed by FTIR spectroscopy. The chemical reaction for the co-precipitation of magnetite is shown below:



3.3. Synthesis of Cross-linked Cellulose-Chitosan Hydrogel Beads

2 g of chitosan flakes were added into 20 ml 10 % acetic acid and 80 ml of DI water in a beaker. The contents in the beaker were then mixed on a hot plate stirrer at 70 °C at 200 rpm until the contents fully dissolved. 2 g of cellulose powder was then added into the chitosan solution and

the mixing was continued at 70 °C at 200 rpm on the stirrer until it fully dissolved, forming the stock solution. The blended solution was then dropped into 1 M NaOH solution to form hydrogel beads with a micropipette. The chitosan-cellulose hydrogel beads were allowed to stay in the NaOH solution with slow stirring for another 48 h for hardening. After 48 h, the crosslinked chitosan–cellulose beads were washed with DI water until pH 7. They were then dried in an oven at 80 °C for another 48 h and stored for further use.

3.4. Coating of Cellulose-Chitosan Hydrogel Beads on Magnetite

2 g of magnetite powder was added into 100 ml of the stock solution (from 3.3.) on the hotplate and the mixing was continued at 70 °C at 200 rpm for an hour. The blended solution was then dropped into a 1 M NaOH solution to form hydrogel beads using a micropipette. The chitosan-cellulose hydrogel coated magnetite beads were allowed to stay in the NaOH solution with slow stirring for another 48 h for hardening. After 48 h, the beads were washed until pH 7. It was then dried in an oven at 80 °C for another 48 h and stored for further use.

3.5. Adsorption studies

The percentage of pollutants removed is calculated using the following formula:

$$\text{Percentage removed} = \frac{\text{Initial concentration} - \text{Final concentration}}{\text{Initial concentration}} \times 100\%$$

3.5.1. Removal of heavy metal ions

50 ppm of Cu²⁺ and Zn²⁺ ions solutions were prepared. To 20 ml of each metal ion solution, 0.5 g of the hydrogel beads/hydrogel coated magnetite beads/magnetite was added. The solutions with the respective adsorbents were then placed on a magnetic stirrer and stirred at different time intervals of 2, 4 and 24 h at 600 rpm. After the designated time, they were centrifuged at 9500 rpm for 15 min to obtain product water. The product water was then tested for the remaining heavy metal ion concentration using a colorimeter. 5 replicates were conducted for each test.

3.5.2 Removal of organic dyes

50 ppm of Direct Red, Methylene Blue and Brilliant Green dyes were prepared. To 20ml of each solution of the dye, 0.5 g of the hydrogel beads/hydrogel coated magnetite beads/magnetite was added. The solutions with the respective adsorbents were then placed on a magnetic stirrer and

stirred at different time intervals of 2, 4 and 24 hours at 600 rpm. After the designated time, they were centrifuged at 9500 rpm for 15 min to obtain product water. The product water was then tested for the remaining dye concentration using the UV-Vis spectrophotometer. 3 replicates were conducted for each test due to time constraint.

4. Results and Discussion

4.1. Characterisation by Fourier-transform infrared spectroscopy (FTIR)

4.1.1. FTIR (Cellulose extracted from newspaper)

Figure 1 (**Appendix**) shows the broad peak at 3693 cm^{-1} which is a characteristic for stretching vibration of the $-\text{OH}$ group in polysaccharides which includes both inter- and intra- molecular hydrogen bond vibrations in cellulose. The band at 1509.16 cm^{-1} and the various peaks between 1428 to 898 cm^{-1} are attributed to CH stretching vibration of all hydrocarbon constituents in polysaccharides, matching the bands reported in literature (Oh et al., 2004).

4.1.2. FTIR (Magnetite synthesised via co-precipitation)

Figure 2 (**Appendix**) shows the values obtained for magnetite which are associated with the Fe-O bond stretching of the tetrahedral and octahedral sites of the magnetite crystalline phase, where the bands shown matches the adsorption bands reported in literature (Silva et al., 2013). However, as the magnetite was not dried enough, there were disruptions to frequency.

4.2. Batch Adsorption Studies of Hydrogel Coated Magnetite Beads

The cellulose-chitosan hydrogel beads and magnetite are the 2 controls for this experiment.

4.2.1. Adsorption of Copper (II) and Zinc ions

4.2.1.1. Magnetite adsorption mechanism

Magnetite forms surface complexes between the functional groups ($\equiv\text{FeOH}$) and the Cu^{2+} and Zn^{2+} with the possible reaction given as follows: (Marmier et al., 1999)



, where $(\equiv\text{FeO})_q \text{Cu}_r(\text{OH})_s / \text{Zn}_r(\text{OH})_s (2-q-s)$ symbolises surface complexes and s , q and r are stoichiometric coefficients (Tartaj et al., 2003; Cotton & Wilkinson, 1988). Magnetite also has large surface areas, high number of surface active sites and high magnetic properties, which

possibly provides more adsorption sites for the Cu^{2+} and Zn^{2+} . This leads to Cu^{2+} and Zn^{2+} uptake as the number of sites are present in the deprotonated form (Kalpaklı, 2015). As shown in Figure 6 (Appendix).

4.2.1.2. Hydrogel adsorption mechanism

The dissolved pollutant ions or molecules possibly penetrated easily into cellulose or chitosan hydrogels and established bonds with the $-\text{NH}_2$ and/or $-\text{OH}$ groups through three different kinds of interactions, either through complexation (or chelation) between the lone pair electrons of N and/or O and the metal ions, through the crystallization of the metal ions with the complexed metals as nucleation sites, or through electrostatic attraction (or ion exchange) between the protonated amino groups and various anions allows hydrogel to easily adsorb the Cu^{2+} and Zn^{2+} (Liu et al., 2012).

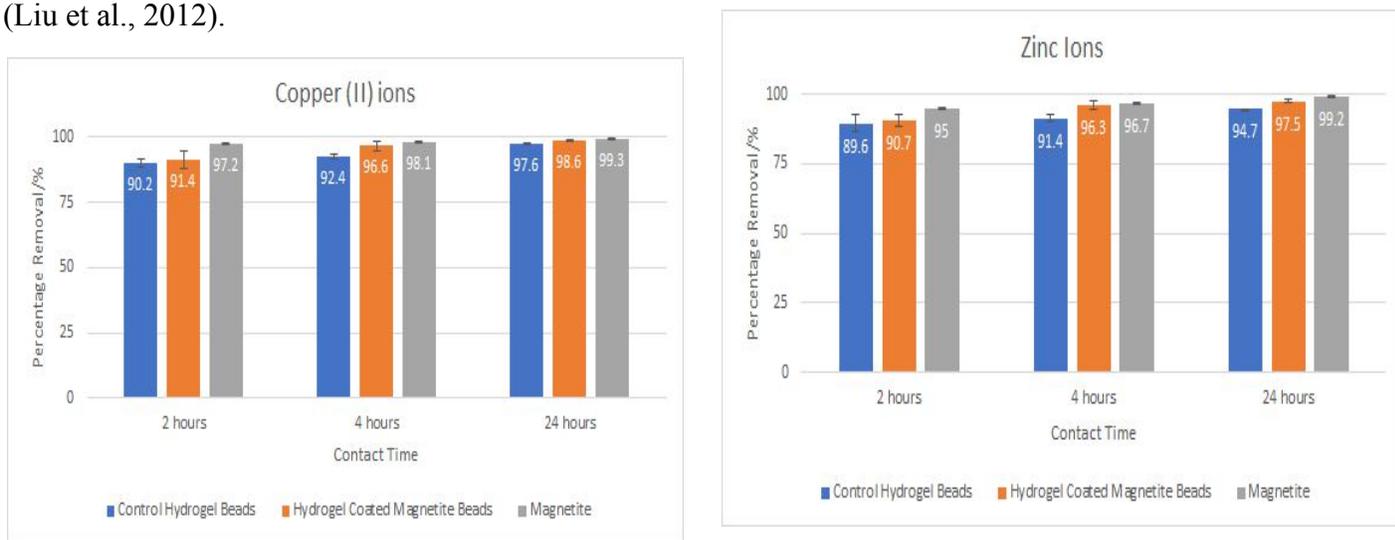


Fig. 3: Removal of Cu^{2+} and Zn^{2+} respectively by different adsorbents and contact times

Figure 3 shows that magnetite has the highest adsorption capacity, followed by hydrogel coated magnetite beads and hydrogel beads. The percentage removal of the metal ions at 24h contact time were chosen to be analysed via the Mann-Whitney U Test to obtain the p-values as at 24h, the percentage removals were the highest.

Table 1: Mann-Whitney U Test on heavy metal ions (contact time 24 hours)

Metal ion tested	Sample comparison	p-value	Conclusion
Copper (II) ion	Hydrogel against hydrogel coated magnetite	0.021 (<0.05)	Significant difference
	Hydrogel against magnetite	0.012 (<0.05)	Significant difference
	Hydrogel coated magnetite against magnetite	0.059 (>0.05)	Insignificant difference
Zinc ion	Hydrogel against hydrogel coated magnetite	0.008 (<0.05)	Significant difference
	Hydrogel against magnetite	0.012 (<0.05)	Significant difference
	Hydrogel coated magnetite against magnetite	0.094 (>0.05)	Insignificant difference
Copper (II) ions against Zinc ions	Hydrogel	0.012 (<0.05)	Significant difference
	Hydrogel coated magnetite	0.116 (>0.05)	Insignificant difference
	Magnetite	0.753 (>0.05)	Insignificant difference

Table 1 shows that the hydrogel beads are more effective in removing Cu^{2+} than Zn^{2+} (p-value 0.012), possibly because Cu^{2+} can easily form metal-chelate with chitosan than Zn^{2+} , which could be adsorbed by the hydrogels (Liu et al., 2012). The hydrogel coated magnetite beads are also shown to be better at removing and ions compared to the control hydrogel beads (p-value Cu^{2+} 0.021, Zn^{2+} 0.008), possibly due to the presence of the magnetite which improved adsorption capacity of the hydrogel.

4.2.2. Adsorption of Methylene Blue, Brilliant Green and Direct Red dyes

4.2.2.1. Magnetite adsorption mechanism

Similar to 4.2.1.1., dyes can be adsorbed to the surface of magnetite via electrostatic interactions (Keyhanian et al., 2016).

4.2.2.2. Hydrogel adsorption mechanism

Dye adsorption occurred possibly due to adsorption transport mechanism into swollen polymer networks. The higher water content and porous structure networks allows for solute diffusion through the hydrogel structure. When dipped into an aqueous media, it absorbs water, resulting in considerable changes of its polymer structure, allowing the dye molecules to easily penetrate into our hydrogel and establish bonds with the $-\text{NH}_2$ and/or $-\text{OH}$ groups (Paulino et al., 2006).

4.2.3. Standard Curve

Figure 4 (**Appendix**) shows how the final concentration of the dyes were determined. However the calibration curve used for Brilliant Green would lead to inaccuracies in determining the final concentration as Beer-Lambert's law assumptions break down at high concentrations due to electrostatic interactions between molecules in close proximity. The last three readings on the calibration curve exceed the suitable absorbance value of 1.0.

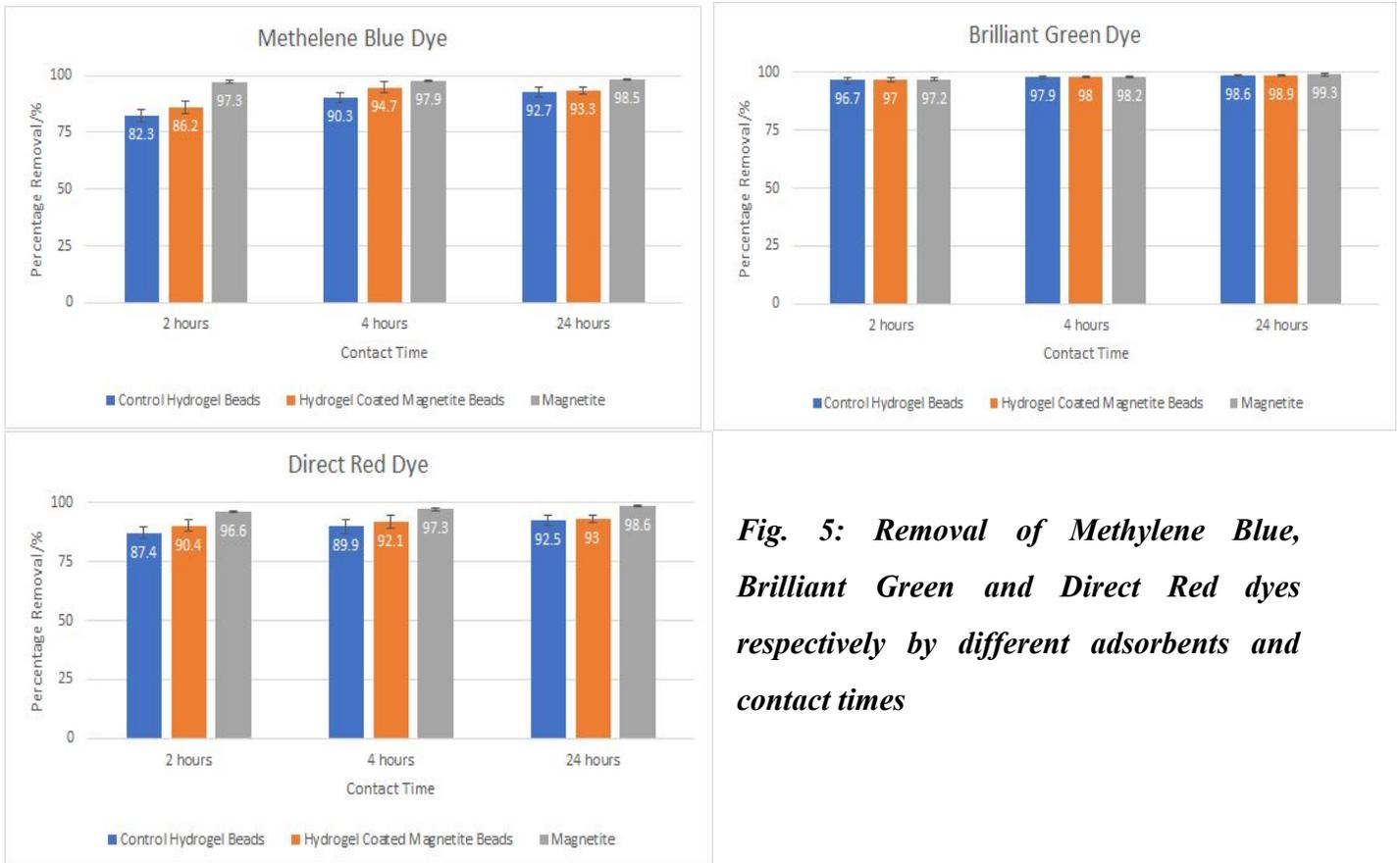


Fig. 5: Removal of Methylene Blue, Brilliant Green and Direct Red dyes respectively by different adsorbents and contact times

Figure 5 shows that magnetite has the highest adsorption capacity, followed by hydrogel coated magnetite beads and hydrogel beads. The percentage removal of the dyes at 24h contact time were chosen to be analysed via error bar overlap as only 3 readings were taken for each test.

Table 2: Error bar overlap test on organic dyes (Contact time 24h)

Organic Dyes	Sample Comparison	Error bar overlap	Conclusion
Direct Red	Hydrogel against hydrogel coated magnetite	Overlap	Insignificant difference
	Hydrogel against magnetite	No overlap	Significant difference
	Hydrogel coated magnetite against magnetite	No overlap	Significant difference
Methylene Blue	Hydrogel against hydrogel coated magnetite	Overlap	Insignificant difference
	Hydrogel against magnetite	No overlap	Significant difference
	Hydrogel coated magnetite against magnetite	No overlap	Significant difference
Brilliant Green	Hydrogel against hydrogel coated magnetite	No overlap	Significant difference
	Hydrogel against magnetite	No overlap	Significant difference
	Hydrogel coated magnetite against magnetite	No overlap	Significant difference
Brilliant Green against Methylene Blue	Hydrogel beads	No overlap	Significant difference
	Hydrogel coated magnetite beads	No overlap	Significant difference
	Pure magnetite	Overlap	Insignificant difference
Brilliant Green against Direct Red	Hydrogel beads	No overlap	Significant difference
	Hydrogel coated magnetite beads	No overlap	Significant difference
	Pure magnetite	Overlap	Insignificant difference
Methylene Blue against Direct Red	Hydrogel beads	Overlap	Insignificant difference
	Hydrogel coated magnetite beads	Overlap	Insignificant difference
	Pure magnetite	Overlap	Insignificant difference

Table 2 shows that both the hydrogel beads were more effective in adsorbing Brilliant Green than Methylene Blue and Direct Red (error bar No overlap), possibly due to the porous structure and the strong interaction between the $-OH$ and $-NH_2$ groups of our hydrogel. When they were placed into the Brilliant Green solution, through a swelling process, Brilliant Green could have easily diffused into the hydrogels and be caught by the $-OH$ group, therefore improving the dynamics rate and adsorption capacity of Brilliant Green (Huang et al., 2018).

4.2.4. Overall

Originally, we hypothesized that the hydrogel when coated on magnetite will increase the adsorption capacity of the magnetite. However, according to our data, the adsorption capacity of the magnetite was reduced instead. One possible explanation is that when the hydrogel was coated on the magnetite, the pH of the hydrogel could have been altered. This changes the rate of swelling due to the presence of $-NH_2$ and $-OH$ groups in the polymers. However, this will only alter the adsorbing capacity of the magnetite slightly (Shah et al., 2015). As such, there were insignificant differences for heavy metal ions removal between the hydrogel coated magnetite beads and magnetite (*Table 1*). Another explanation could be that the hydrogel blocked and

reduced the number of available surface active sites of the magnetite for the pollutants to bind to, hence decreasing the adsorption capacity.

5. Conclusion

Cellulose-chitosan hydrogel has been successfully synthesised and coated on magnetite, and is better at removing Cu^{2+} , Zn^{2+} and Brilliant Green as compared to the control hydrogel beads, but for Direct Red and Methylene Blue, they have comparable adsorption capacities. Furthermore, the hydrogel decreases the adsorption capacity of magnetite. The hydrogel beads are also more effective in removing Cu^{2+} than Zn^{2+} . Magnetite is more effective in removing Cu^{2+} , Zn^{2+} Methylene Blue and Direct Red than both hydrogel beads (with and without coating on magnetite), but they have comparable adsorption capacity for Brilliant Green with the hydrogel coated magnetite beads. Finally, the longer contact time, there is a higher adsorption capacity of the hydrogel beads and magnetite.

For the applications of the hydrogel coated magnetite beads, it can be used to purify wastewater in third world countries where such pollutants are found in large concentrations, and can be used for recycling of ions by using the beads to absorb and desorb the ions attracted.

6. Future Work

Due to time constraints arising from the virus, we were unable to gather much data to prove our explanations, and could only come up with possible explanations for our data. Further work can therefore be done by getting more data for the removal of dyes, as well as sending the magnetite for SEM to prove our explanations. More work can also be done by increasing the initial concentration of the pollutants till magnetite has less than a 90 % percentage removal and repeat the experiment to see if the hydrogel coating will increase the adsorption capacity of magnetite this time around. Testing the effect of pH on the hydrogel beads can also be considered. Mixing the heavy metal ions and organic dyes together to mimic real-life situations, where various types of pollutants are found in wastewater can also be worked upon further. The desorption ability of the hydrogel may also be tested for its reusability.

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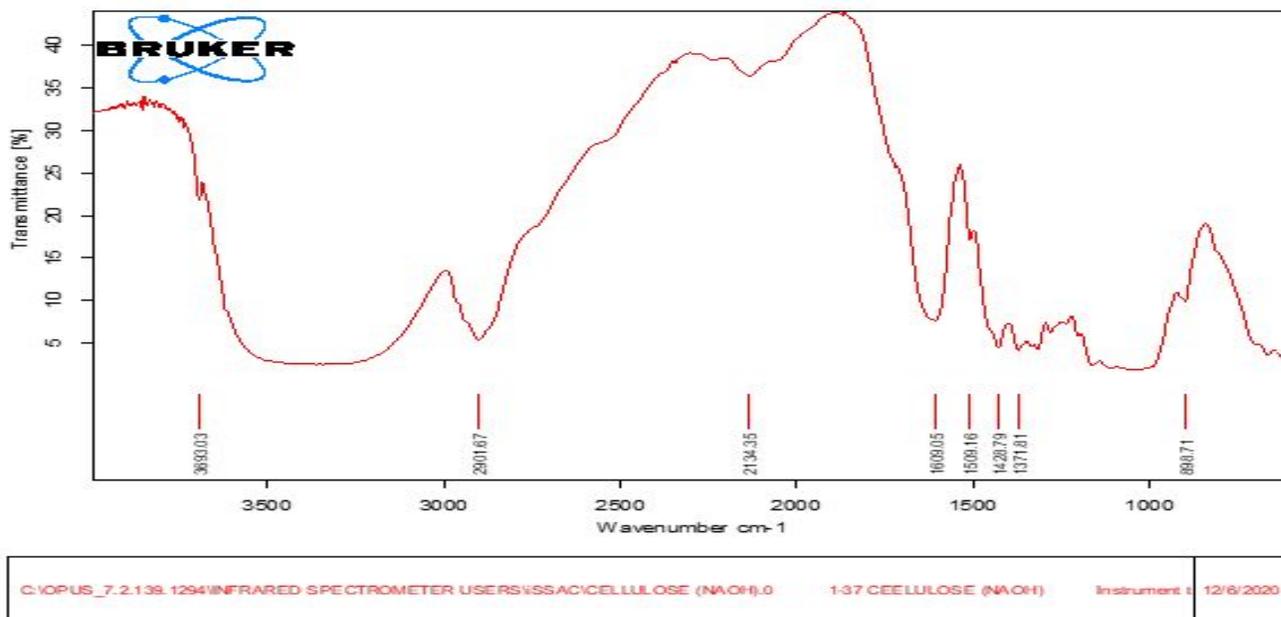
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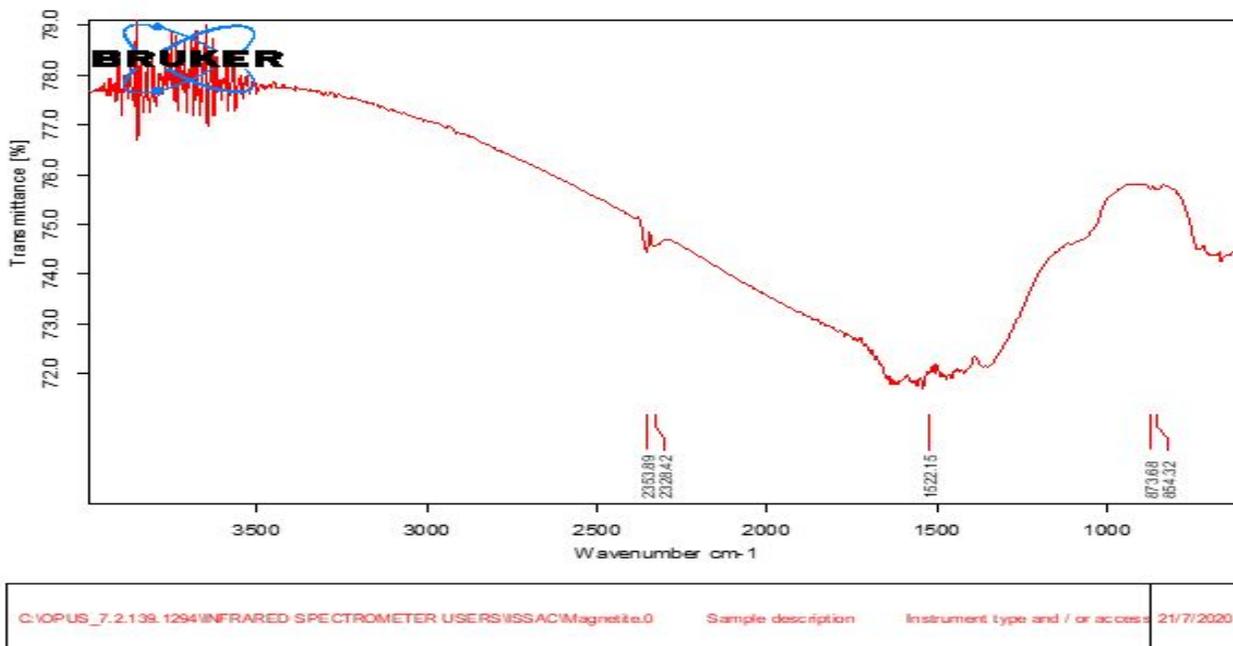
Appendix

FTIR Spectrums



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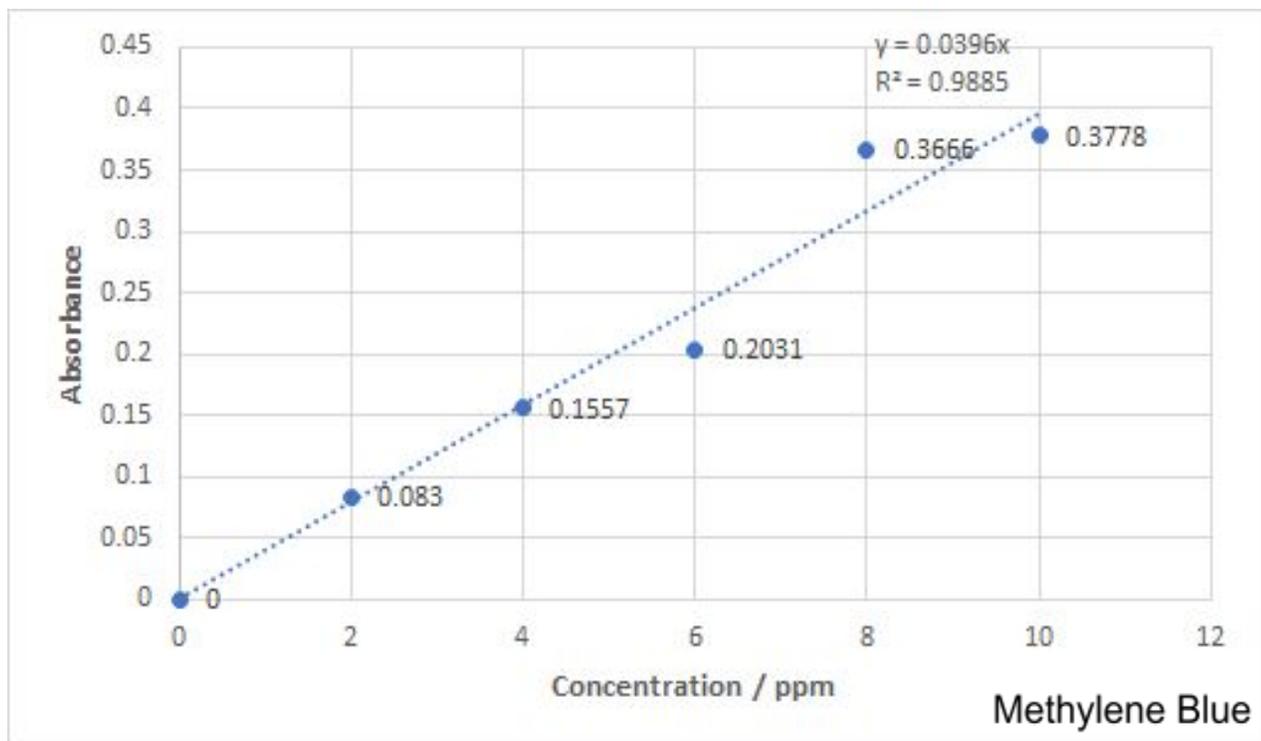
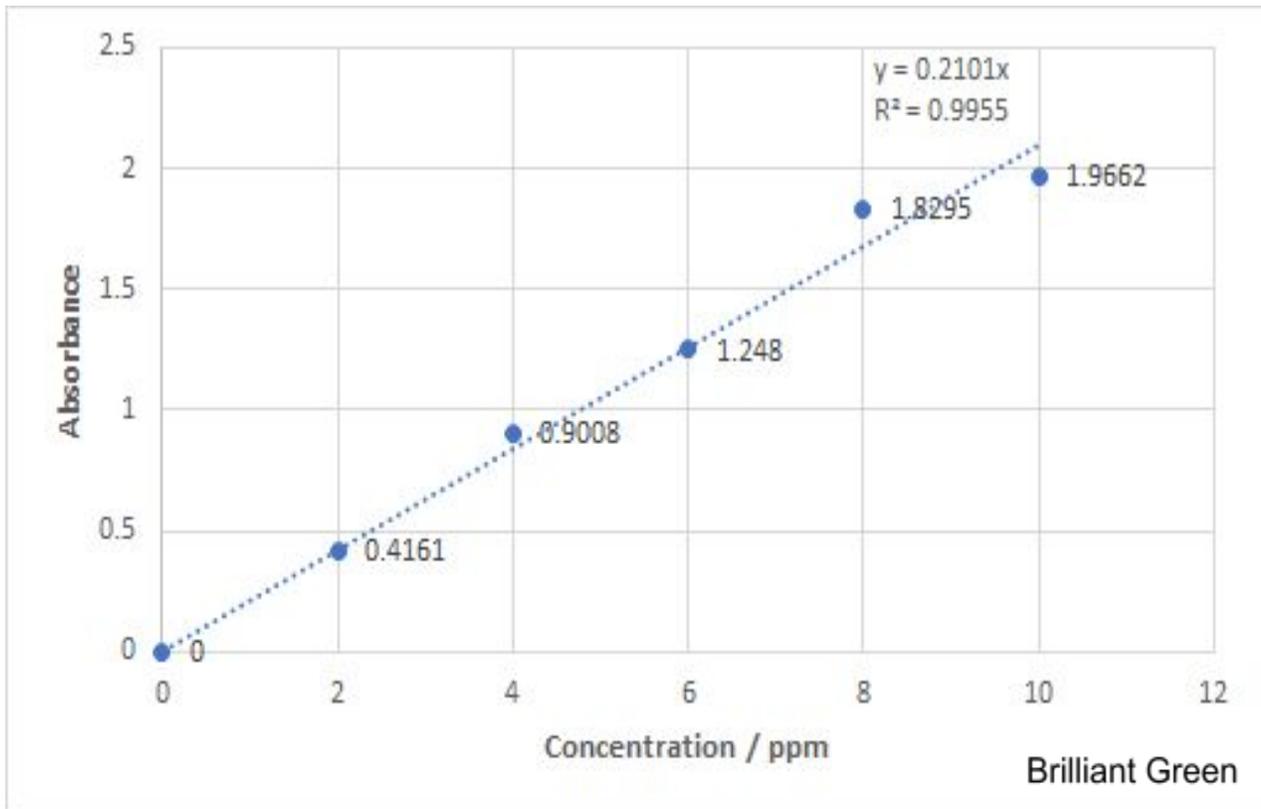
Fig. 1: FTIR spectrum of cellulose extracted from newspapers



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Fig. 2: FTIR spectrum of magnetite synthesized via co-precipitation

Calibration Curves



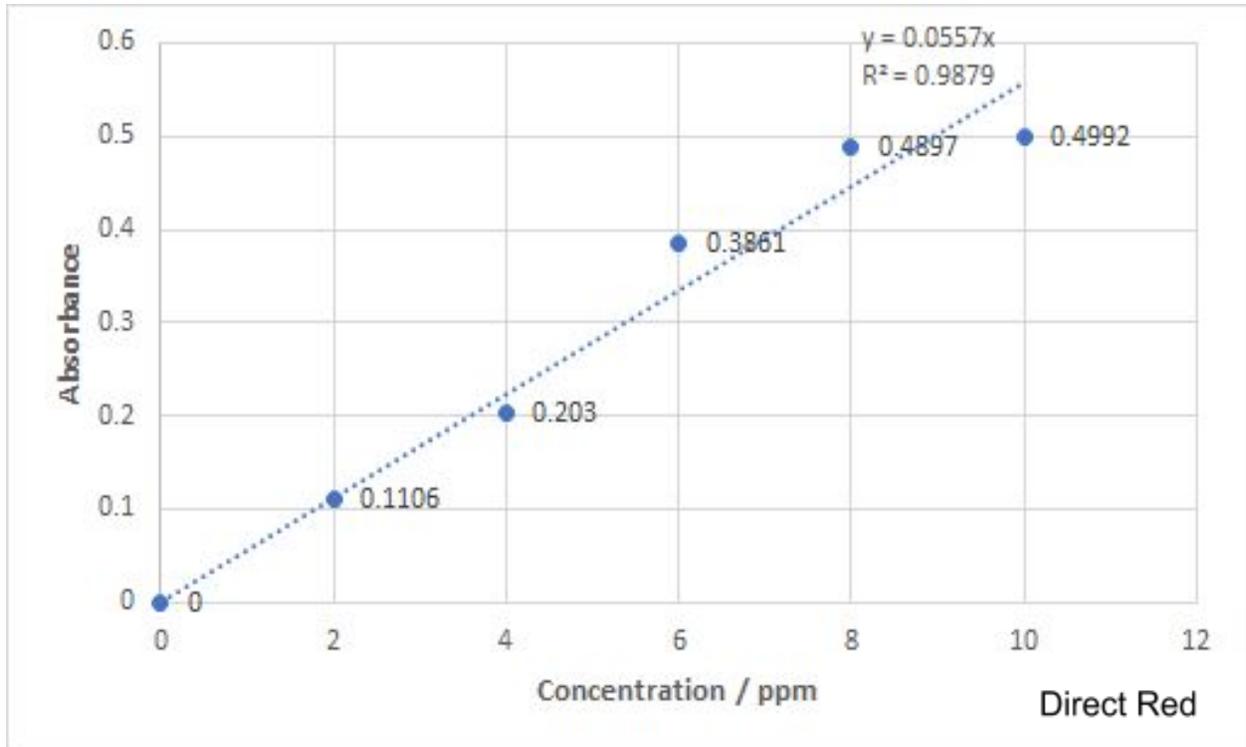
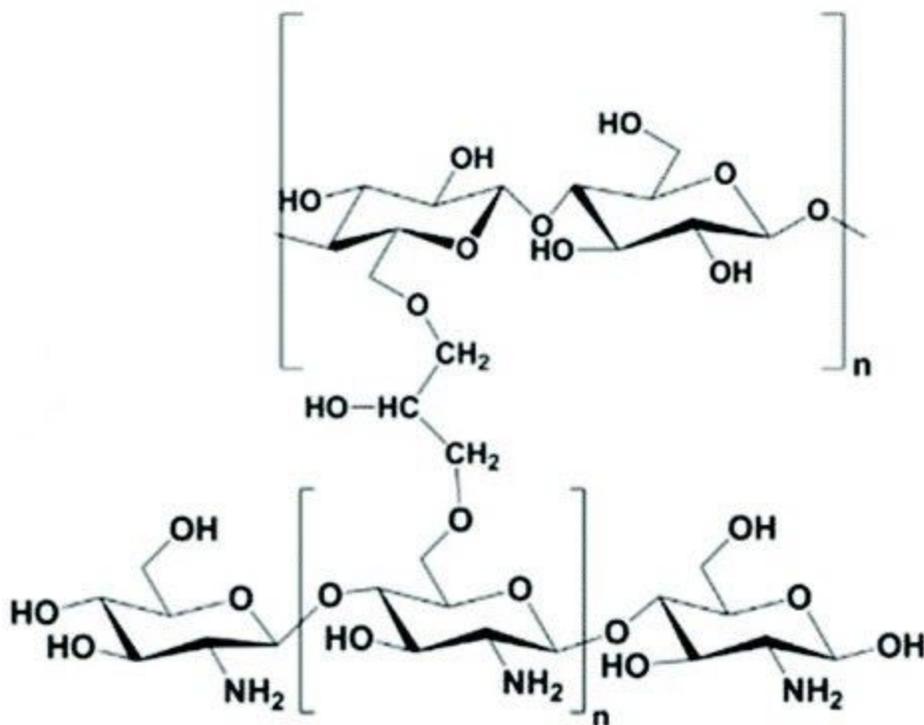


Fig. 4: Calibration curves showing the concentrations corresponding to absorbance of Brilliant Green, Methylene Blue and Direct Red respectively

Structure of Cellulose-Chitosan Hydrogel



The $-NH_2$ and the $-OH$ are the main groups in the polymers.

Magnetite adsorption mechanism (heavy metal ions)

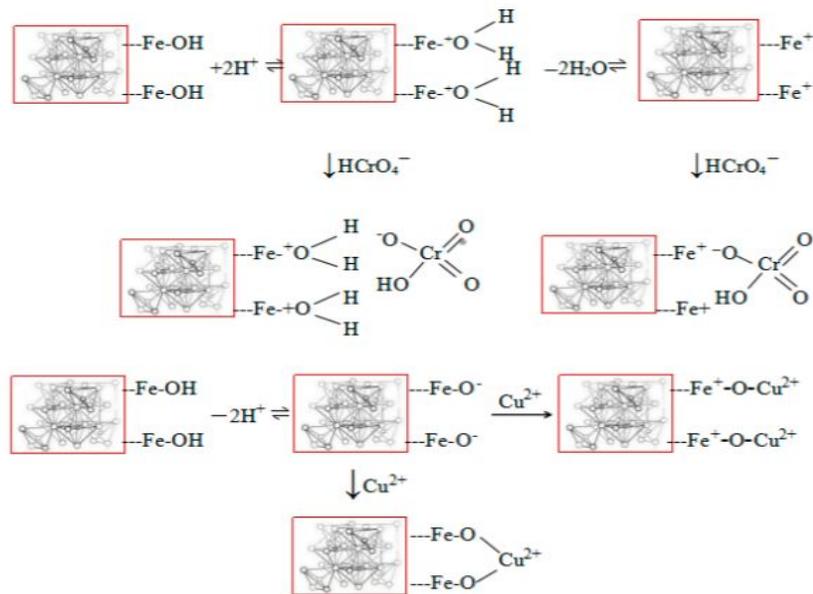


Fig. 6: Visual representation of magnetite adsorption mechanism for heavy metal ions (Cu^{2+})

Heavy metal ions forming metal-chelate with chitosan in the hydrogel

