Synthesis of magnetised sawdust strontium nanocomposites for enhanced water purification

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Abstract

In recent years, there has been a rise in occurences of eutrophication, which is the excessive enriching of water bodies with nitrate and phosphate ions. Furthermore, heavy metal ions such as copper(II) and zinc(II) ions may only be mildly toxic compared to other heavy metal ions, however they still can pose serious risks to aquatic life. In this project, we have investigated the effectiveness and potential of magnetised sawdust-strontium (MSD-Sr) nanocomposites in water purification, as compared to magnetised sawdust (MSD). We focused on the adsorption of phosphates and copper(II) ions in this study. In phosphate adsorption tests, MSD-Sr removed 75.57% of phosphate ions from water, while MSD removed 70.18% of phosphate ions from water. In copper(II) ion adsorption tests, MSD-Sr and MSD removed 97.10% and 37.94% of copper(II) ions from the water respectively. It can be seen that MSD-Sr has retained the phosphate adsorption capability of MSD while greatly improving its copper(II) ion adsorption capability, showing the great versatility and potential of MSD-Sr in water purification.

1 Introduction

1.1 Literature review

Nitrate and phosphate eutrophication in marine ecosystems is a global problem. Marine eutrophication has a negative impact on food security, ecosystem health and economy through disruptions in tourism, fisheries and health industries. (Ngatia, Grace, Moriasi, & Taylor, 2019)

Aquatic organisms are sensitive to Cu^{2+} and Zn^{2+}. Even though internal concentrations of these required elements are homeostatically controlled, toxic effects can occur at the fish gill surface.

As such, Cu^{2+} ion pollution in water bodies is something which must not be ignored. (Fu et al., 2016)

Sawdust is abundantly available from the timber and forest industry and has been studied in the recent past as an adsorbent. Sawdust is actually an efficient adsorbent that is effective to many types of inorganic and organic pollutants. (Sahmoune & Yeddou, 2016)

Strontium-based materials have relatively high electrical value, chemical stability, thermal and corrosion resistivity. Since strontium-based nanoparticles are positively charged (Sr^{2+}), they are successfully used for removal of various types of ionic contaminants especially phosphate compounds. (Sereshti, Afsharian, Bidhendi, & Nodeh, 2019)

1.2 Objectives

- To investigate the effect of the presence of strontium ions on the adsorption capacity of MSD-Sr nanocomposites
- To investigate the effect of sonication on the adsorption capacity of MSD-Sr nanocomposites
- To investigate the effect of the concentration of strontium nitrate solution on the adsorption capacity of MSD-Sr nanocomposites
- To investigate the effect of the concentration of strontium nitrate solution on the adsorption capacity of MSD-Sr nanocomposites
- To investigate the reusability of modified sawdust

1.3 Hypotheses

- Strontium nanoparticles can adhere to the surface of MSD
- $PO4^{3-}$ and Cu^{2+} can be removed from water using the MSD-Sr nanocomposites
- The MSD-Sr nanocomposites can be reused for a number of times

2 Materials and Methods

2.1 Materials

2.1.1 Chemicals

- Iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$)
- Iron(II) sulfate heptahydrate ($FeSO_4 \cdot 7H_2O$)
- Concentrated aqueous ammonia
- Potassium dihydrogen phosphate (KH_2PO_4)
- Copper(II) sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$)
- Sawdust
- Sodium hydroxide (NaOH)
- Strontium nitrate ($Sr(NO_3)_2$)

2.1.2 Apparatus

- Standard filtration apparatus
- Büchner funnel
- Oven
- Magnet
- Volumetric flasks (2 *l*, 10 m*l*)
- Blender
- Sonicator
- Orbital shaker
- Colorimeter
- Fourier-transform infrared spectroscopy (FTIR)

2.2 Methodology

2.2.1 Synthesis of Magnetised Sawdust (MSD)

Sawdust was washed with deionised water, dried and blended into smaller particles. Smaller particles were then separated using a sieve. 6.1 g of iron(III) chloride hexahydrate and 4.2 g of

iron(II) sulfate heptahydrate were dissolved in 100 cm^3 of deionised water. Subsequently, 1 g of sawdust was added to the solutions and mixed. Concentrated aqueous ammonia was then added into the solution until the solution reaches pH 10, to induce co-precipitation of magnetite onto the sawdust. The mixture was left to stand for 15 minutes before being filtered using vacuum filtration. The residue was washed until neutral pH and dried in an oven. The chemical reaction for the co-precipitation of magnetite is shown below:

$$Fe^{2+}\,(aq) + 2\,Fe^{3+}\,(aq) + 8\,OH^{-}\,(aq) \rightarrow Fe_3O_4\,(s) + 4\,H_2O\,(l)$$

2.2.2 Synthesis of Magnetised Sawdust - Strontium nanocomposites (MSD-Sr)

0.1 & 0.2 mol dm^{-3} strontium nitrate solutions were made by dissolving 2.116 g and 4.232 g of strontium nitrate ($Sr(NO_3)_2$) respectively in 100 cm^3 of deionised water, 2 solutions of each concentration were made. 1 g of MSD was added to each of the solutions. The samples were sonicated or stirred on an orbital shaker for 30 minutes to achieve homogeneous solutions, before being adjusted to pH 11 using concentrated aqueous ammonia. The mixtures were then stirred on an orbital shaker for 1 hour before being filtered using regular filtration apparatus. The residues were washed until pH 7 and were dried in an oven.

The MSD-Sr produced were labeled A, B, C and D for simplicity. Each letter represents a different method of preparation and concentration of strontium nitrate solution used, as shown in Table 1:

Sample ID	Concentration of $Sr(NO_3)_2$ / mol dm^{-3}	Method of preparation
MSD-Sr Type A	0.1 M	Sonicated
MSD-Sr Type B	0.1 M	Orbital Shaker
MSD-Sr Type C	0.2 M	Sonicated
MSD-Sr Type D	0.2 M	Orbital Shaker

Table 1: Sample ID of Magnetised Sawdust - Strontium (MSD-Sr) nanocomposites

2.2.3 Adsorption Studies of MSD and MSD-Sr

0.1 g of the various MSD-Sr samples mentioned in Table 1 was each added to 25 ml of phosphate ion solution (50 ppm) or copper(II) ion solution (50 ppm) and the samples were shaken on an orbital shaker at 150 rpm for 24 hours. The adsorption studies were also conducted on MSD without strontium for comparison with MSD-Sr. Five replicates were conducted for each type of adsorbent on each pollutant. Pollutant solutions which do not contain any adsorbent served as the controls for the experiments. A magnet was used to separate the magnetic adsorbent and the supernatant was obtained. Final concentration of phosphate and copper(II) ions were analysed using a colorimeter. The percentages of phosphate and copper(II) ions removed were calculated using the following formula:

$$Percentage\ removal = \frac{Initial\ concentration - Final\ Concentration}{Initial\ Concentration}\ x\ 100\%$$

2.2.4 Investigating the Reusability of MSD-Sr

10 cm^3 of 2 mol dm^{-3} sodium hydroxide solution was added to each sample of adsorbent. The adsorbents were allowed to soak for 1 hour. After that, the sodium hydroxide solutions were poured away and the adsorbents were washed until pH 7. The adsorbents were then dried in the oven, after which they could be used for the second or subsequent adsorptions.

3 Results and Discussion

3.1 Results for adsorption tests

3.1.1 Phosphate ions

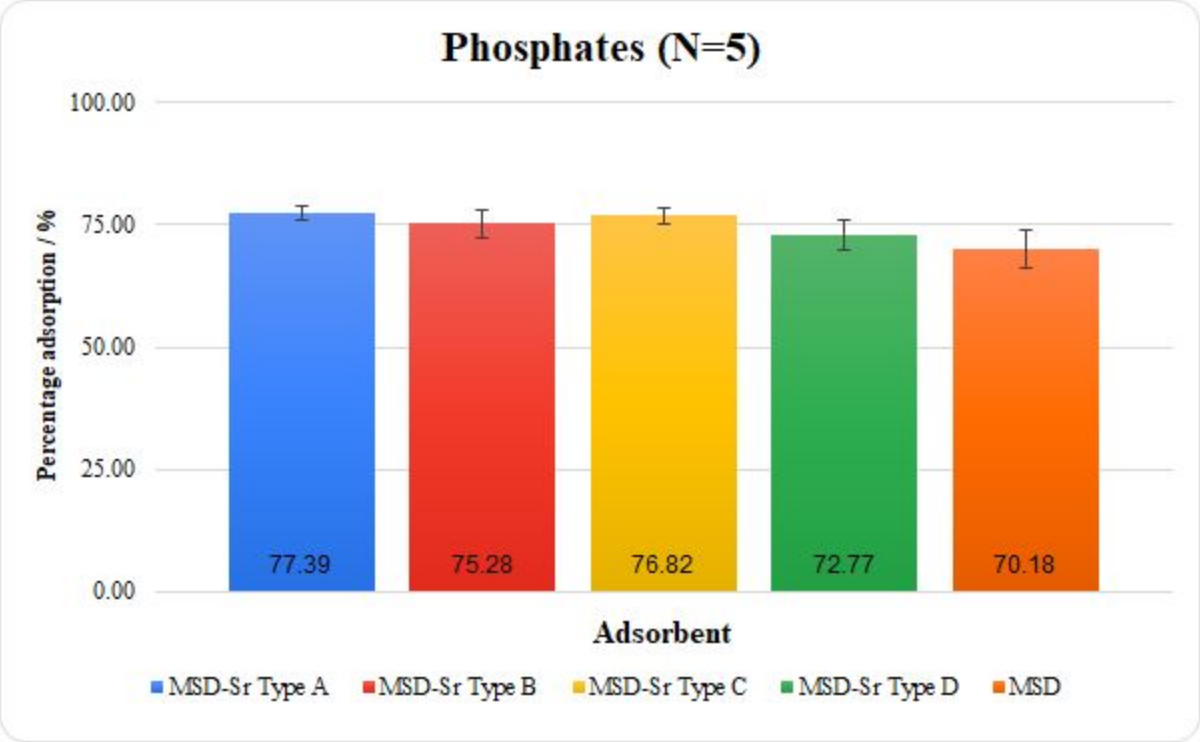

Figure 1: Phosphate ion percentage adsorption for the 4 types MSD-Sr and MSD, N=5

As seen from Fig. 1, all the adsorbents were able to adsorb at least 70% of the phosphate ions after 24 hours, and all the 4 types of MSD-Sr performed slightly better than MSD.

Variable tested	Samples concerned
Presence of strontium nanoparticles	A vs. MSD B vs. MSD C vs. MSD D vs. MSD
Method of preparation	A vs. B C vs. D
Concentration of strontium nitrate solution	A vs. C B vs. D

Table 2: Summary of the different independent variables to be tested and the samples concerned

As seen from Table 2, we will be comparing the phosphate adsorption capabilities of the different adsorbents based on the different variables listed. We will be conducting the Mann-Whitney U Test on each pair of adsorbents at a significance level of 5% to see if the differences in their adsorption capabilities are statistically significant.

Samples concerned		**P-value**	**Significant?**
MSD-Sr Type A (0.1 mol dm^{-3} MSD-Sr Sonicated)	MSD	0.07215	No
MSD-Sr Type B (0.1 mol dm^{-3} MSD-Sr Shaker)	MSD	0.20045	No
MSD-Sr Type C (0.2 mol dm^{-3} MSD-Sr Sonicated)	MSD	0.08692	No
MSD-Sr Type D (0.2 mol dm^{-3} MSD-Sr Shaker)	MSD	0.33724	No

Table 3: Comparison of phosphate adsorption based on the presence of strontium nanoparticles

We can see from Table 3 that there is no significant improvement in the phosphate adsorption capability of MSD with the addition of strontium nanoparticles.

Samples concerned		**P-value**	**Significant?**
MSD-Sr Type A (0.1 mol dm^{-3} MSD-Sr Sonicated)	MSD-Sr Type B (0.1 mol dm^{-3} MSD-Sr Shaker)	0.41683	No
MSD-Sr Type C (0.2 mol dm^{-3} MSD-Sr Sonicated)	MSD-Sr Type D (0.2 mol dm^{-3} MSD-Sr Shaker)	0.20045	No

Table 4: Comparison of phosphate adsorption based on the method of preparation

Similarly, there was no significant difference in the phosphate adsorption capability of MSD-Sr when sonication was incorporated into the synthesis process.

Samples concerned		P-value	Significant?
MSD-Sr Type A (0.1 mol dm^{-3} MSD-Sr Sonicated)	MSD-Sr Type C (0.1 mol dm^{-3} MSD-Sr Sonicated)	0.50000	No
MSD-Sr Type B (0.2 mol dm^{-3} MSD-Sr Shaker)	MSD-Sr Type D (0.2 mol dm^{-3} MSD-Sr Shaker)	0.30153	No

Table 5: Comparison of phosphate adsorption based on the concentration of $Sr(NO_3)_2$ solution

Likewise, there was no significant difference in the phosphate adsorption capability of MSD-Sr when the concentration of strontium nitrate solution used to synthesise MSD-Sr was varied.

3.1.2 Copper(II) ions

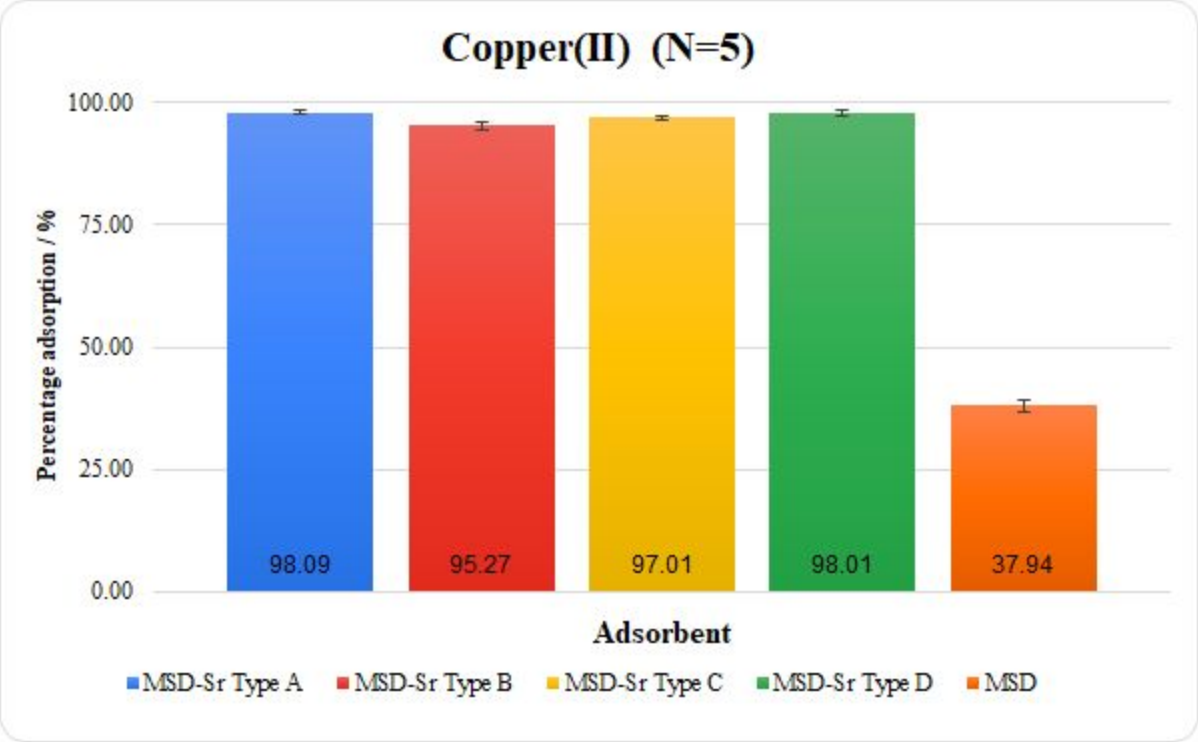

Figure 2: Copper(II) ion percentage adsorption for the 4 types MSD-Sr and MSD, N=5

As seen from Fig. 2, there is a drastic improvement in the copper(II) ion adsorption capabilities of all the 4 types of MSD-Sr compared to MSD.

Again, we will be comparing the different adsorption capabilities of the different adsorbents based on the same set of variables as seen from Table 2. We will also be conducting the Mann-Whitney U Test on each pair of adsorbents at a significance level of 5% to see if the differences in their adsorption capabilities are statistically significant.

Samples concerned		**P-value**	**Significant?**
MSD-Sr Type A (0.1 mol dm^{-3} MSD-Sr Sonicated)	MSD	0.00604	Yes
MSD-Sr Type B (0.1 mol dm^{-3} MSD-Sr Shaker)	MSD	0.00604	Yes
MSD-Sr Type C (0.2 mol dm^{-3} MSD-Sr Sonicated)	MSD	0.00604	Yes
MSD-Sr Type D (0.2 mol dm^{-3} MSD-Sr Shaker)	MSD	0.00604	Yes

Table 6: Comparison of phosphate adsorption based on the presence of strontium nanoparticles

As seen from Table 6, the addition of strontium nanoparticles to MSD has proven to improve its copper(II) ion adsorption capability significantly.

Samples concerned		**P-value**	**Significant?**
MSD-Sr Type A (0.1 mol dm^{-3} MSD-Sr Sonicated)	MSD-Sr Type B (0.1 mol dm^{-3} MSD-Sr Shaker)	0.01831	Yes
MSD-Sr Type C (0.2 mol dm^{-3} MSD-Sr Sonicated)	MSD-Sr Type D (0.2 mol dm^{-3} MSD-Sr Shaker)	0.07215	No

Table 7: Comparison of phosphate adsorption based on the method of preparation

From Table 7, we can see that for MSD-Sr synthesised with 0.1 M strontium nitrate solution, sonication has made a significant improvement in its copper(II) ion adsorption capability. However, for MSD-Sr synthesised with 0.2 M strontium nitrate solution, sonication did not make a significant improvement in the copper(II) ion adsorption capability of MSD-Sr.

Samples concerned		**P-value**	**Significant?**
MSD-Sr Type A (0.1 mol dm^{-3} MSD-Sr Sonicated)	MSD-Sr Type C (0.1 mol dm^{-3} MSD-Sr Sonicated)	0.07215	No
MSD-Sr Type B (0.2 mol dm^{-3} MSD-Sr Shaker)	MSD-Sr Type D (0.2 mol dm^{-3} MSD-Sr Shaker)	0.03005	Yes

Table 8: Comparison of phosphate adsorption based on the concentration of $Sr(NO_3)_2$ solution

From Table 8, we can see that for the sonicated samples of MSD-Sr, there was no significant difference in their adsorption capability when the concentration of strontium nitrate solution used to synthesise them was varied. However, for the 2 samples which were not sonicated, an increase in the concentration of strontium nitrate solution used to synthesise them increased their copper(II) ion adsorption capability significantly.

A possible explanation has been proposed for the two sets of data seen above in Tables 7 and 8 which might seem a little erratic. When sonication is used to synthesise MSD-Sr, the degree of adherence of strontium ions to the MSD surface is roughly similar for any concentration of strontium nitrate solution used in the synthesis, likely due to the fact that there is a limit to the degree of strontium ion adsorption to the MSD surface, hence a variation in the concentration of strontium nitrate solution used to synthesise MSD-Sr does not affect its copper(II) ion adsorption capability as seen in Table 8. However, when sonication was not used in the synthesis, the strontium nanoparticles tend to be more loosely connected to the MSD, hence there is not really a limit to the amount of strontium nanoparticles that can be incorporated into the nanocomposite, and an increase in the concentration of strontium nitrate solution used in the synthesis increases the copper(II) adsorption capability significantly as seen from Table 8. Such phenomena are

supported by results obtained in Table 7. When 0.1 mol dm^{-3} strontium nitrate solution was used in the synthesis of MSD-Sr, the degree of adherence of strontium ions to the MSD surface was higher when sonication was used in the synthesis, and the copper(II) adsorption capability of the sonicated MSD-Sr was significantly higher as seen in Table 7. However, when the concentration of strontium nitrate used was increased to 0.2 mol dm^{-3}, the degree of adherence of strontium ions for the sonicated MSD-Sr remained roughly the same, while the degree of adherence of strontium ions to the MSD surface increased significantly for the non-sonciated MSD-Sr, such that the difference in the adsorption capabilities of MSD-Sr synthesised with 0.2 mol dm^{-3} strontium nitrate solution became insignificant as seen in Table 7.

3.2 Results for reusability tests

3.2.1 Phosphate ions

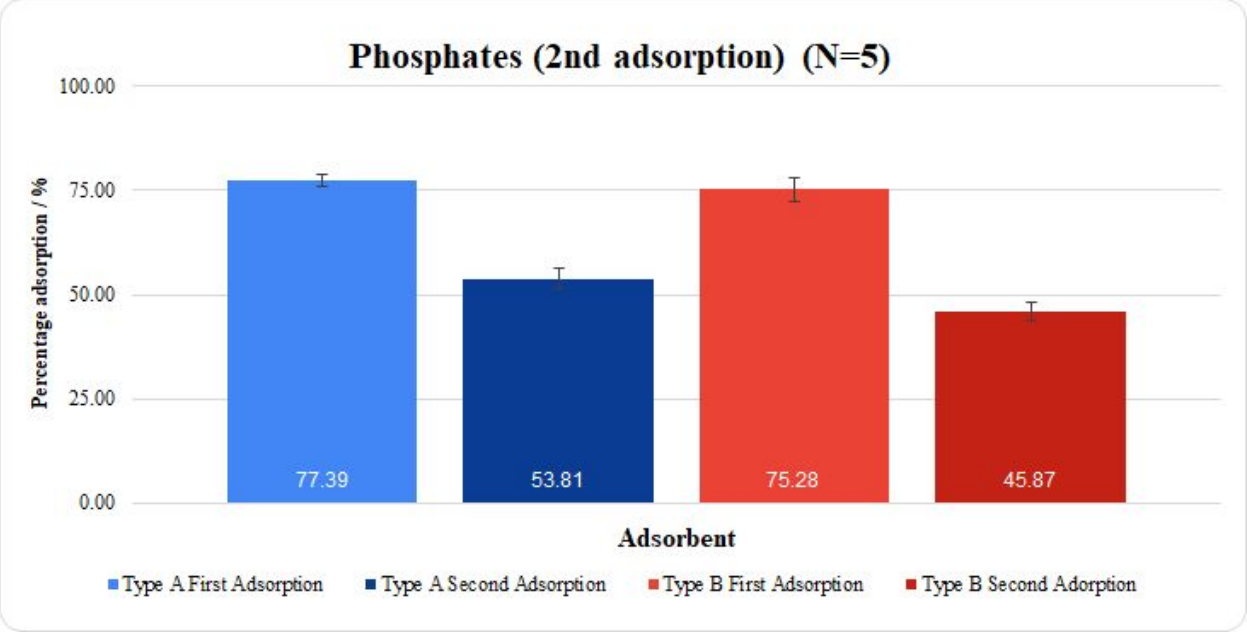

Figure 3: Phosphate ion percentage adsorption for the 4 types MSD-Sr and MSD, N=5

As seen from Fig. 3, there is a large drop in adsorption capacities between the two cycles, with 69.5% and 60.9% of the adsorption capacity being retained during the second adsorption cycle for Type A and Type B respectively.

Variable tested	Samples concerned
Method of preparation	A (2nd adsorption) vs. B (2nd adsorption)
Number of times adsorbent is reused	A (1st adsorption) vs. A (2nd adsorption) B (1st adsorption) vs. B (2nd adsorption)

Table 9: Summary of the different independent variables to be tested and the samples concerned

As seen from Table 9, we will be comparing the phosphate adsorption capabilities of the different adsorbents based on the different variables listed. We will also be conducting the Mann-Whitney U Test on each pair of adsorbents at a significance level of 5% to see if the differences in their adsorption capabilities are statistically significant.

Samples concerned		P-value	Significant?
MSD-Sr Type A (Second adsorption)	MSD-Sr Type B (Second adsorption)	0.03005	Yes

Table 10: Comparison of the 2nd phosphate adsorption based on method of preparation

From Table 10, we can see that sonication has significantly improved the phosphate adsorption capability of MSD-Sr in the second adsorption, suggesting that sonication is more effective in the long run as more adsorption cycles are conducted.

Samples concerned		P-value	Significant?
MSD-Sr Type A (1st adsorption)	MSD-Sr Type A (2nd adsorption)	0.00604	Yes
MSD-Sr Type B (1st adsorption)	MSD-Sr Type B (2nd adsorption)	0.00604	Yes

Table 11: Comparison of the 2nd phosphate adsorption for each type of adsorbent

From Table 11, we can see that there is a significant decrease in adsorption capacity as more adsorption cycles are conducted.

3.2.2 Copper(II) ions

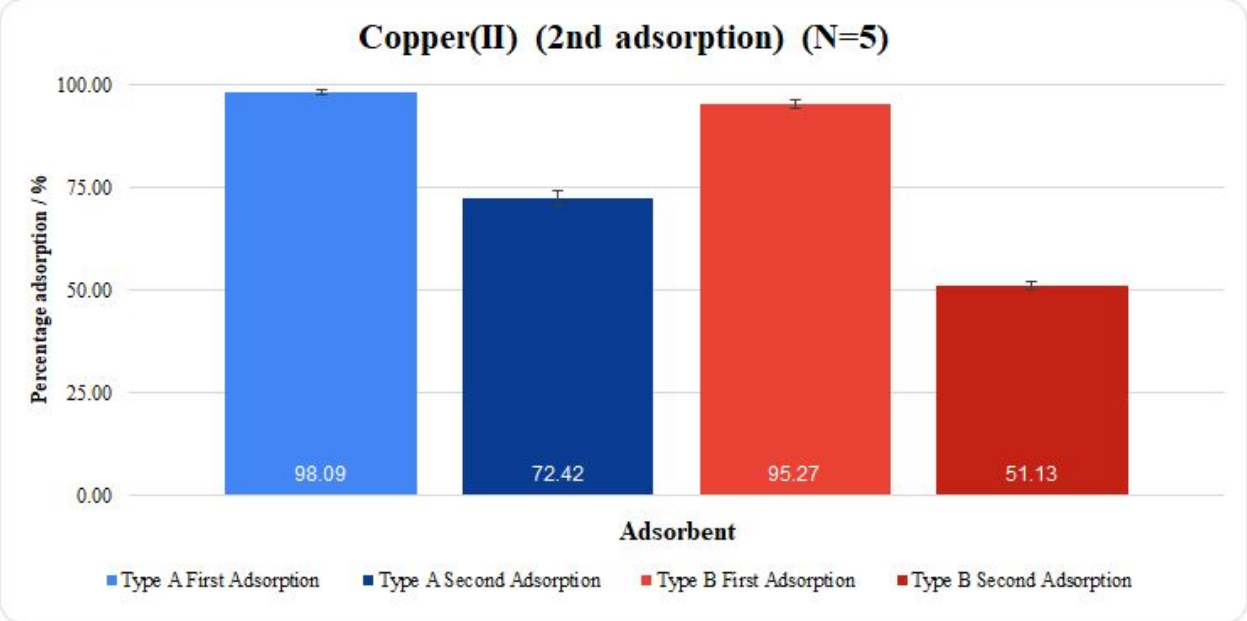

Figure 4: Copper(II) ion percentage adsorption for the 4 types MSD-Sr and MSD, N=5

As seen from Fig. 4, there is a large drop in adsorption capacities between the two cycles, with 73.8% and 55.7% of the adsorption capacity being retained during the second adsorption cycle for Type A and Type B respectively.

Again, we will be comparing the different adsorption capabilities of the different adsorbents based on the same set of variables as seen from Table 9. We will also be conducting the Mann-Whitney U Test on each pair of adsorbents at a significance level of 5% to see if the differences in their adsorption capabilities are statistically significant.

Samples concerned		**P-value**	**Significant?**
MSD-Sr Type A (Second adsorption)	MSD-Sr Type B (Second adsorption)	0.00604	Yes

Table 12: Comparison of the 2nd copper(II) ion adsorption based on method of preparation

From Table 12, we can see that sonication has significantly improved the copper(II) ion adsorption capability of MSD-Sr in the second adsorption, suggesting that sonication is more effective in the long run as more adsorption cycles are conducted.

Samples concerned		**P-value**	**Significant?**
MSD-Sr Type A (1st adsorption)	MSD-Sr Type A (2nd adsorption)	0.00604	Yes
MSD-Sr Type B (1st adsorption)	MSD-Sr Type B (2nd adsorption)	0.00604	Yes

Table 13: Comparison of the copper(II) adsorption for each type of adsorbent

From Table 13, we can see that there is a significant decrease in adsorption capacity as more adsorption cycles are conducted.

A possible explanation for our aforementioned results is that the 1 hour desorption time allocated to the adsorbents after they have completed their first round of adsorption is not sufficient to remove all the phosphate or copper(II) ions, clogging the adsorbents' active sites and preventing complete adsorption of pollutant particles in the second adsorption. Thus, in our future work, we aim to extend the desorption time to test whether it improves the reusability of the nanocomposites. Furthermore, sonication has proven to improve the adsorption capability of MSD-Sr when it is reused as shown from our results, a possible reason for this is that sonication allows the constituents of MSD-Sr to be more strongly bound together, hence more strontium ions remain adhered to the MSD after the desorption and washing process for the adsorbent to be reused.

3.3 Summary of Findings

Summarising our findings for adsorption tests, we found that:

- MSD is already efficient at PO_4^{3-} ion adsorption, but it is ineffective at Cu^{2+} ion adsorption
- MSD-Sr is not significantly better than MSD as an adsorbent for PO_4^{3-} ions. However, MSD-Sr is significantly better than MSD as an adsorbent for Cu^{2+} ions
- Sonicated MSD-Sr does not show significant improvements in adsorption capacity during the first adsorption cycle. However, as the MSD-Sr is reused, sonication has proven to make significant improvements to the adsorption capabilities of MSD-Sr

3.4 Characterisation

3.4.1 Fourier Transform Infrared Spectroscopy (FTIR)

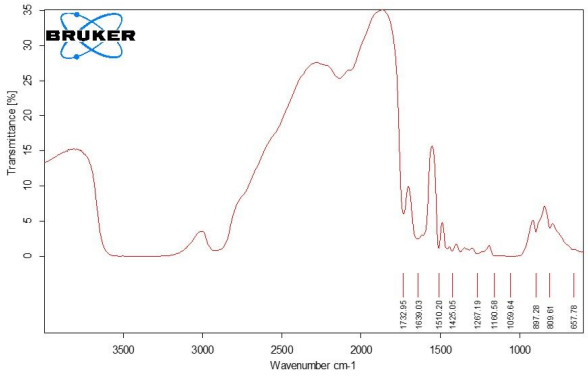

Figure 5: FTIR spectra of sawdust

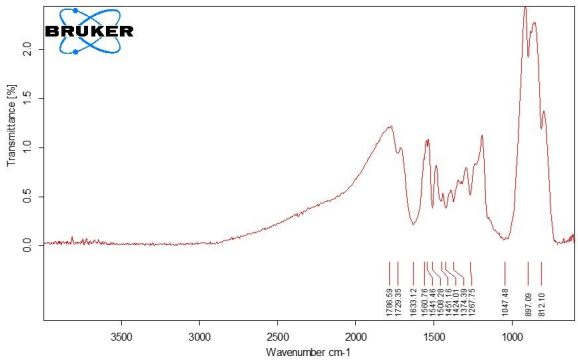

Figure 6: FTIR spectra of MSD

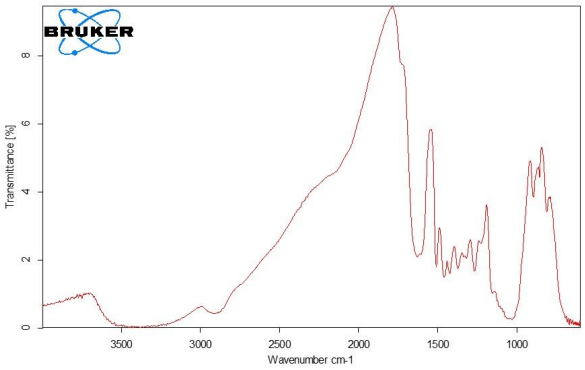

Figure 7: FTIR spectra of MSD-Sr

We can see the similarities in the peak wavenumbers from the 3 spectra. This suggests that all 3 samples contain the same functional groups, namely those which are present in sawdust such as the O-H functional group. However, there is an evident drop in the transmission spectra. This can be explained by different amounts of IR radiation being absorbed by each sample. One possible explanation for the different levels of IR radiation absorption is the presence of additives to the sawdust such as Strontium and magnetite. Hence, we can speculate that magnetite and strontium nanoparticles have successfully adhered to the sawdust structure, although further tests with Scanning Electron Microscopy (SEM) and X-Ray Diffraction (XRD) would have to be

conducted to confirm the successful binding of magnetite and strontium nanoparticles to our MSD-Sr.

4 Conclusion and Future Work

4.1 Conclusion

MSD is already effective at removing $PO4^{3-}$ ions, but not very effective at removing Cu^{2+} ions. MSD-Sr does not show significant improvements in $PO4^{3-}$ adsorption, but shows significant improvements at removing Cu^{2+} ions. This suggests that MSD-Sr is more versatile and can remove more types of pollutants including heavy metal ions which regular MSD cannot.

Furthermore, sonicated MSD-Sr shows significantly greater adsorption capabilities as compared to shaken MSD-Sr, during the second adsorption, suggesting that sonicated MSD-Sr is a better adsorbent of pollutants than shaken MSD-Sr in the long run due to its ability to retain its adsorption capacities after multiple adsorption runs.

4.2 Future Work

In the future, the adsorption isotherm studies and further reusability tests can be done on our MSD-Sr. The effect of pH on the adsorption capacity of MSD-Sr, as well as the anti-bacterial properties of MSD-Sr can be studied, while thermodynamic studies can also be conducted. The adsorption capacities of MSD-Sr can be compared to commercial options for water purification such as activated carbon. MSD-Sr can also be applied in a real-life context to test its effectiveness in adsorbing pollutants from industrial wastewater and polluted water sources.

5 References

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