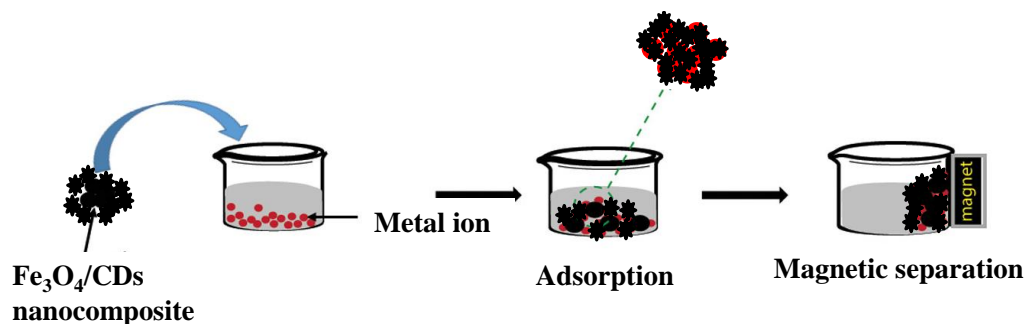# Removal of Hg and Pb in aqueous solution using magnetite carbon dots nanocomposite

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GRAPHICAL ABSTRACT

Fe₃O₄/CDs nanocomposite — Metal ion — Adsorption — Magnetic separation

ABSTRACT

Hg and Pb metals are major concerns because of their high degree of toxicity in public environment, which also pollute aquatic systems. Nanocomposite adsorbents have been developed for cleaning polluted water at low. The study aims to determine the removal efficiency of magnetite carbon dots nanocomposite in case of Hg and Pb in the aqueous solution. In this paper, magnetite carbon dots (symbolized as Fe_3O_4/CDs) nanocomposite was synthesized through a two-step process of co-precipitation and pyrolysis methods. The synthesized nanocomposite was analyzed using Utraviolet visible (UV-vis), Fourier transform infrared spectroscopy (FTIR), scanning electron electron microscopy (SEM), electron dispersive X-ray fluorescence (EDXRF), X-ray diffraction (XRD) and atomic absorption spectrophotometer (AAS). According to the results, the synthesized (Fe_3O_4/CDs) nanocomposites were found to be 20 nm in diameter, superparamagnetic property, surface roughness and deformations, important functional groups, iron-rich nanocomposite and favorable removal efficiency for Hg (82.70 %) and Pb (72.91 %), respectively. Therefore, the results indicated that these Fe_3O_4/CDs nanocomposites are potentially attractive agents for the removal of heavy metals ions, especially Hg and Pb from industrial wastewater.

1. Introduction

The aquatic environment is being polluted by heavy metals contamination and man-made chemical compounds such as multicolored dyes through the food chain, which negatively impacted in health terms because those are not capable of being decomposed and accumulate in our body (Nouri and Montazer Faraj, 2022). Also, adverse effects are a major threat to humans which can cause cancer. Copper, chromium, mercury and lead ions are several health risks for human health. Recently, a study has reported that water used by several industries get polluted by toxic elements (Cu, Cr, Pb, Fe, and Zn) with excessive concentrations against WHO guidelines (Elgaraby et al., 2021).

The exposure of living beings to toxins of elements is by means of several roots including drinking water, working in toxic metal-based workplace and eating unclean food. Then, they accumulate in the human body, reaching toxic concentrations. Some heavy metals are essential for both plant nutrients and biological importance, but there is a negative impact on human because they are nonbiodegradable

and have a lengthy half-live (Ali et al., 2019). For example, high-dose exposure to lead has been linked with hypertension, stroke and cell degeneration. High dose concentration of chromium exposure has been associated with vital organs (liver and kidney) damages, nervous tissue and blood circulation in disorder. Also, high dose of Hg is one of major concerns to nerve cells in the brain and disturb kidneys' function (Engwa et al., 2019).

Ferrite-based nanoparticles (Fe_3O_4 NPs) are the most widely used adsorbents for water treatment to remove toxic metals from wastewater. The Fe_3O_4 NPs were synthesized using many approaches i.e., coprecipitation, sol-gel, hydrothermal, electrochemical, and microemulsion. Of these methods, the co-precipitation is a promising one which was not tedious, simple and low-cost, low-temperature processing and the easy separation. Several studies have used Fe_3O_4 NPs for toxic metals adsorption such as arsenic, lead and chromium. In addition, Fe_3O_4 NPs are also used for dye removal against methylene blue, bromophenol blue, methyl red, bromocresol green, and eriochrome black-T. The Fe_3O_4 NPs have a beneficial use as an adsorbent due to the easiest separation

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process from the solution after the adsorption process (Dave *et al.*, 2014). Therefore, many researchers get inspiration on exploring Fe_3O_4 NPs through using several water purification methods for heavy metals removal.

However, colloidal Fe_3O_4-based magnetic NPs tend to form agglomeration easily in water owing to larger surface area to volume ratio and dipole-dipole interaction between nanocrystals (Nouri, 2022). Surface modification of Fe_3O_4 NPs can provide better colloidal stability in water, which also enhance biocompatibility. The surface of Fe_3O_4 NPs modification or coating was proposed with multiple materials like gold, silicon compounds, carboxylates, polymers, sulphonates, phosphates, and carbon dots. Thus, modifying its physical and chemical features is an essential tool to suppress their cytotoxicity, and decrease its diameter size and stay stable in water. A previous study has demonstrated that citric acid (CA) functionalized Fe_3O_4 magnetic NPs surface in which carboxylate groups on citric acid might bind the surface of magnetic NPs and propagate their negativity and hydrophilicity (Kim *et al.*, 2001). In this regard, a new nanocomposite with higher activity in aqueous solution was presented (Qiao *et al.*, 2016).

Carbon dots (CDs) as a latest generation of carbon-based materials have emerged as excellent characteristics including chemical inertness, low toxicity, hydrophilicity, simple preparation, ease of functionalization and low cost. Also, the CDs include precious nanostructures to give intrinsic peroxidase-like nature. Owing to the aforementioned virtues and hydrophilicity, CDs are being chosen for a newly compatible magnetic nanocomposite. Moreover, Fe_3O_4/CDs nanocomposites were synthesized for the purpose of making photocatalysts (Li *et al.*, 2022).

For example, chitosan, activated carbon, polymers, graphene quantum dots, graphene oxide, and multiwall carbon nanotubes are promising precursors of carbon based nanomaterials in cooperating with magnetic NPs to synthesize Fe_3O_4/CDs nanocomposite for solving water pollution problem. Moreover, Fe_3O_4/CDs nanocomposite was reported for determining the removal capability against methyl blue dye, which is contaminated in water. In this phenomenon, CDs acted as reactive amine and hydroxyl groups which are responsible for surface functionalization and modifications. Therefore, in nanocompositing Fe_3O_4 with CDs, hydroxyl and carboxyl groups existed on CDs can provide modification to the surface of Fe_3O_4 and cannot easily aggregate. Furthermore, CDs can stand as its stable structure of Fe_3O_4 NPs as capping agent. CDs can enhance the prepared nanocomposite surface with important functional groups which are useful for potential attraction of negatively charged nanocomposite with positively charged chemical pollutants in the environment (Soltys *et al.*, 2021).

To the best of our knowledge, there is no report for synthesis of Fe_3O_4/CDs nanocomposites as adsorbents for Hg and Pb removal. In this study, the magnetite carbon dots (Fe_3O_4/CDs) were successfully synthesized using coprecipitation and pyrolysis method for the removal of selected toxic heavy metals in aqueous solution. The reaction method is significantly simple, fast and cost-effective. The main objectives of this paper are; to prepare Fe_3O_4/CDs nanocomposite, to measure its physicochemical properties, to evaluate elemental contents, and to assess adsorption efficiency of prepared sample. The physicochemical properties of the prepared nanocomposites were determined by using analytical techniques such as SEM, UV-vis spectroscopy, FTIR, EDXRF, XRD and TGA. In addition, the removal efficiency of Hg and Pb was performed through AAS analysis. The result indicated that the percentage of selected heavy metal removal efficiency was found to be 71.91 % for Hg (II) cation and 82.80 % for Pb (II) cation. Therefore, this research is expected that it would contribute to removing heavy metals from industrial wastewater to some extent.

2. Materials and methods
2.1. Chemical and reagent

Iron (III) chloride (97%; $FeCl_3$), ammonium iron (II) sulphate (99.97%; $(NH_4)_2 Fe(SO_4)_2 6H_2O$), ammonium hydroxide (25% NH_4OH), and ethanol (99.5%; C_2H_5OH) were purchased from laboratory chemicals and equipments store, Myanmar Supply Co., Ltd., Yangon. Citric acid (99.5%; $C_6H_8O_7$) and hydrochloric acid (37%; HCl) were available at Department of Chemistry, Pakokku University, Myanmar. Deionized water was used throughout the experiments. The used chemicals and reagents for this research are analytical grade and were used directly without any further modifications.

2.2. Synthesis of Fe_3O_4

The synthesis of Fe_3O_4 NPs was done in accordance with the following procedure. First, $FeCl_3$ (8.1 g) and Mohr's salt (9.8 g) were mixed together in a beaker with 25 mL of deionized (DI) water and then directly put into 20 mL of ammonia followed by vigorous stirring. After that, the mixture was taken to ultrasonication for 30 min until adjusting pH 9 and continued stirring at 70 ℃ for 30 min. The resulting black mixture solution was centrifuged at 10 rpm and cleaned with DI water and ethanol. The obtained Fe_3O_4 was transferred to 100 mL of DI water with stirring and ultrasonication for 30 min. After pH 9 was adjusted, the mixture was stirred again at 80 ℃ for 1 hour. Finally, Fe_3O_4 NPs were obtained and they were conveniently separated via an external magnet field for further use (Jannah and Onggo, 2019).

2.3. Synthesis of Fe_3O_4/ CDs nanocomposite

Fe_3O_4/CDs nanocomposite was synthesized through a two-step of the co-precipitation and pyrolysis. To the reaction flask, 12 g/L of CDs was added into 12 g/L of Fe_3O_4 NPs and continued reaction for stirring for 30 min. Then, 0.1 M NaOH (20 mL) was put drop-wise. The reacting process further went continuously under stirring for 2 hour at 80 ℃ and it was centrifuged at 3000 rpm for 20 min to isolate the clean mixture solution from impurities. After that, the pure black precipitate of Fe_3O_4/CDs nanocomposite was collected by decantation using common external magnet. Finally, they were washed several times with deionized water, and kept by air dried vacuum (Kwee, 2023).
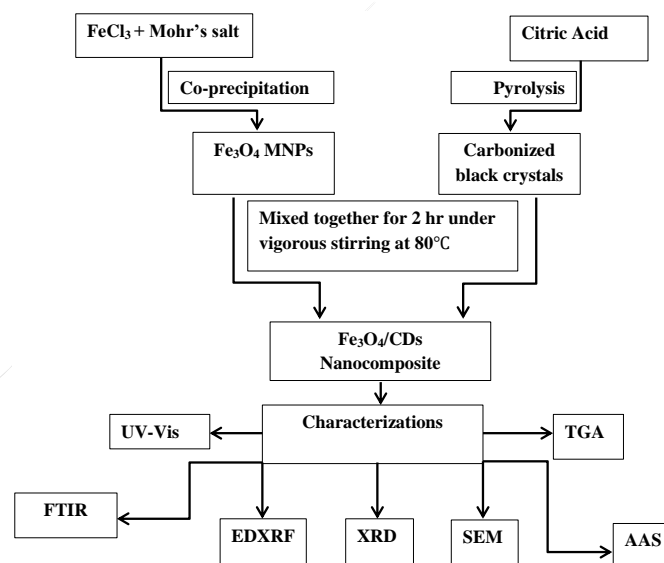

Fig.1. The schematic diagram of research flow chart.

2.4. Characterization of Fe_3O_4/CDs nanocomposite

The prepared Fe_3O_4/CDs were further characterized by using different analytical techniques such as SEM, UV-vis, FTIR, EDXRF, XRD and TGA, respectively. SEM analysis was carried out for a visual inspection of external porosity and morphology using FESEM, Zeiss Ultra60 in the Department of Chemistry, Yatanapon University, Myanmar. The development of magnetic nanoparticles was observed by appearance of the solution using UV-vis spectrophotometer of LAMBDA™ 265 (PerkinElmer). The FTIR spectrum was recorded using FTIR spectrophotometer (IRPrestige-21, Shimadzu, Japan) within the range of 400-4000 cm^{-1} and EDXRF (Shimadzu EDX-700 Spectrometer) analysis was determined in the Department of Chemistry, Monywa University, Myanmar to find elemental contents in prepared sample. X-ray diffractogram of prepared sample was recorded using Rigaku Multijlex 2KW Powder X-ray, Akishima, Tokyo, Japan in the Universities' Research Centre of Yangon University (URC-YU). The diffraction patterns were recorded in the 2θ range of 10º to 70º. TGA results of Fe_3O_4/CDs nanocomposite were measured using DG-DTA (DTG 60/60 H, (SHIMADZU), Japan), in the University of Research Center, Yangon University, Myanmar to record the weight losses of prepared sample. The heat temperatures were changed from 30 to 600 ℃.

2.5. Adsorption Efficiency of Hg and Pb by Fe_3O_4/CDs nanocomposite

Firstly, 10 mg of Fe_3O_4 magnetic nanoparticles were put into 16 mL of solution (DI water and 10 mg/L of each of selected heavy metals cation solution), its time range was from 1 to 45 min. After that, the Fe_3O_4

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magnetic nanoparticles were separated from the solution with a permanent magnet. The residual heavy metal ions in the solution were studied by using atomic absorption spectrophotometer. Similarly, the evaluation of the removal efficiency of selected heavy metal cations were done by Fe_3O_4/CDs nanocomposite using the above-mentioned procedure. Removal efficiency of cations was computed by using of the following equation (Masoudi and Honarasa, 2018).

$$Removal\ efficiency\ (\%) = \frac{C_o - C_r}{C_r} \times 100 \qquad (1)$$

where, C_0 and C_r are before and after residual concentrations of the cations in the solution (mg/L), respectively.

3. Results and discussion
3.1. Synthesis of magnetite carbon dots nanocomposite

Fe_3O_4/CDs nanocomposite was successfully synthesized using coprecipitation and pyrolysis methods. For preparing Fe_3O_4 MNPs, bulk magnetite (Fe_3O_4) was first obtained from the mixture of the ionic iron sources by a co-precipitation method. The prepared magnetite was collected using the external magnet, indicating their magnetic properties which were in accordance with other reports (Fahmi et al., 2022).

After that, the citric acid (CA) was singly subjected to reacting itself through pyrolysis which made it carbonized. Then, the synthesized magnetite was further cooperated with CA-derived CDs to make the magnetite covered and enhance its stability. In this reacting route, any hydroxyl moieties on citric acid were interacted by hydrogen or other hydroxyl spikes, coating magnetite and becoming a carbon-like structure of CDs onto magnetite (Fe_3O_4/CDs) nanocomposite as a new layer as shown in Fig. 2c.

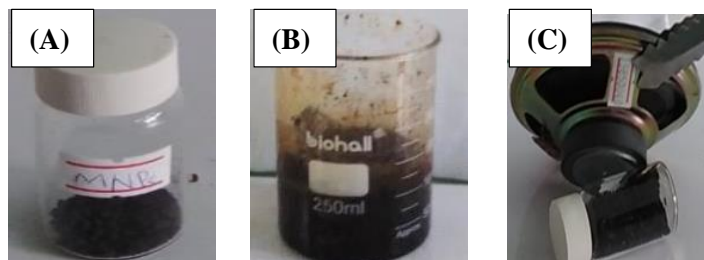**Fig. 2.** (a) The synthesized Fe_3O_4 magnetic nanoparticles, (b) The synthesized carbon dots produced from citric acid (c) and The produced Fe_3O_4/CDs nanocomposite attracted using external magnet.

3.2. Physicochemical properties of Fe_3O_4/CDs nanocomposite analysis

After the synthesis of the Fe_3O_4/CDs nanocomposite, the results of physicochemical characteristics were studied by using of SEM, UV-vis, FTIR, EDXRF, XRD and TGA in order to verify the structural formation of produced magnetite carbon dots nanocomposite.

Firstly, SEM was applied to determine the structure and spherical morphology of the synthesized Fe_3O_4/CDs nanocomposite. In Fig. 3, Fe_3O_4 and CDs could connect roughly and randomly to each other that brought with stable coating structures of microscopic deformations, which have a larger specific surface area. This phenomenon is conducive to improve the adsorption activity for cationic heavy metals on Fe_3O_4/CDs.

Fig. 3. The SEM photograph of Fe_3O_4/CDs nanocomposite.

Furthermore, the absorption of Fe_3O_4/CDs nanocomposite was also recorded by UV-vis spectrophotometry to determine the maximum wavelength absorbed by the prepared sample. From this analysis, it was confirmed that the synthesized Fe_3O_4/CDs nanocomposites depicted a maximum absorbance at a significant peak of 202.5 nm, assigned the $\pi \rightarrow \pi^*$ electron transition of the C=C bond in the CD core as shown in Fig. 4, which is in correspondence with other reports of magnetic nanocomposite (Jlassi et al. 2020).

Moreover, functional groups of produced Fe_3O_4/CDs nanocomposite were confirmed by FTIR to identify the incorporation of magnetite and CDs at wave numbers of 400-4000 cm^{-1} as shown in Fig. 5. According to the obtained results, the characteristic peak at 547 cm^{-1}, which confirmed the presence of Fe–O bending groups due to the interconnection of iron oxide and CDs. The absorption bands were 1092, 1394, 1590 and 1633 cm^{-1}, which were assigned by C-O stretching, O-H bending, C=C stretching, C=O stretching. Moreover, broad two bands at 3129 and 3400 cm^{-1} which corresponded to C-H

sp^2 stretching bands and O-H stretching as shown in Figure 3.2. Especially, existing carboxylate moieties and hydroxyl groups on the surface of Fe_3O_4/CDs nanocomposite played a key role in the interaction of heavy metal cations. These functional groups could be helpful for heavy metal adsorption ability on Fe_3O_4/CDs nanocomposite through electrostatic interaction. The Fe_3O_4/CDs nanocomposite was further analyzed by technique to evaluate the composition of elements contained in Fe_3O_4/CDs nanocomposite. The spectrum of Fe_3O_4/CDs nanocomposite was shown in Fig. 6. The presence of significant peaks for iron revealed the formation of iron-rich magnetite nanocomposites, which was corresponding to the stretching band of Fe-O in the FTIR spectrum. According to the result, the major elemental component of iron is (97.29%). Sulphur was detected to be 1.95%. Furthermore, other trace amounts of Mn, Mo, Cr, Ca, Cu, and Zn were detected to be 0.22, 0.16, 0.14, 0.11, 0.05 and 0.04%, respectively. Thus, the formation of iron-rich nanocomposites and its superparamagnetic properties was confirmed.
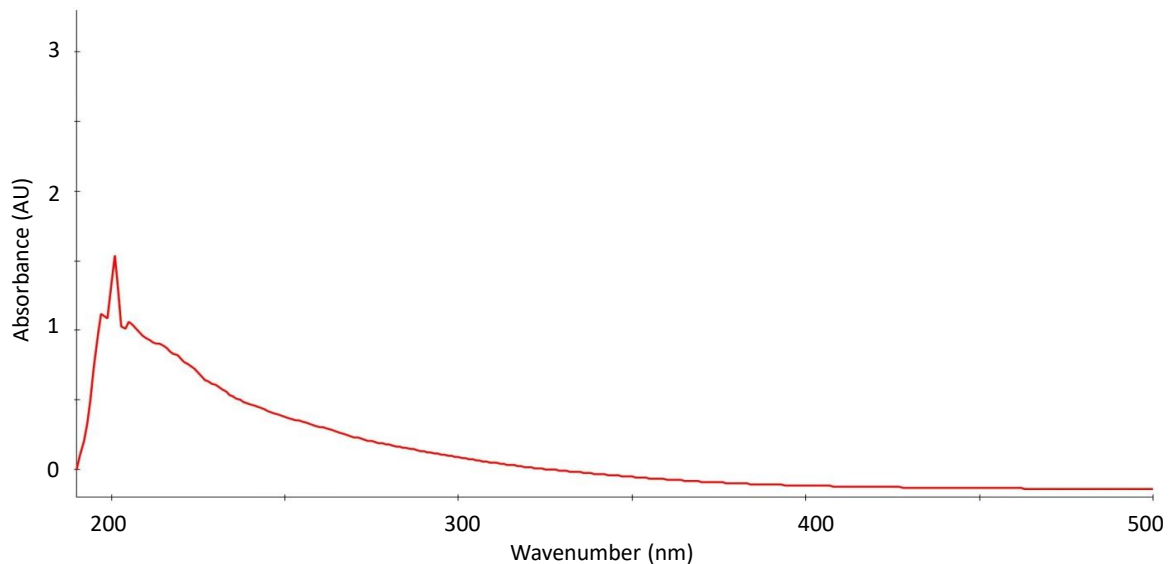
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Fig. 4. The UV-visible peaks of Fe_3O_4/CDs nanocomposite.

Finally, XRD technique was also used to provide detailed information about structural information of nanomaterials such as lattice parameters, phase nature, and crystalline structure. In Fig. 7, peaks at 29.8.°, 34.8°, 44.1°, 57.1°, 61.9° and 71.9° were observed, which are in correspondence with Miller indices of Fe_3O_4 in the nanocomposite (Ali, A., *et al.* 2022). The peaks can be indexed by (220), (311), (400), (511), (440), (533) lattice planes. The diameter of Fe_3O_4/CDs nanocomposite was evaluated using the following Scherrer formula.

$$D = \frac{K\lambda}{BCos\theta} \qquad (2)$$

where, D is represented by its diameter, K is a Scherrer constant (K = 0.9), λ indicates XRD wavelength, B is represented by full width at half maximum (FWHM) measured in radians, and θ is diffraction angle, respectively. According to the calculation from using the Scherrer equation, the obtained nanoparticle diameter was to be 17 nm, which was in agreement with the above mentioned report (Ali *et al.*, 2022). Moreover, the maximum diffraction peak of Fe_3O_4/CDs nanocomposite which emerged at 30-40⁰ was observed, which might be representative of the moderate crystallinity of the Fe_3O_4/CDs) nanocomposite. These findings observed that the diameter of prepared magnetic carbon dots nanocomposite was correctly ranged in the nanoscale.
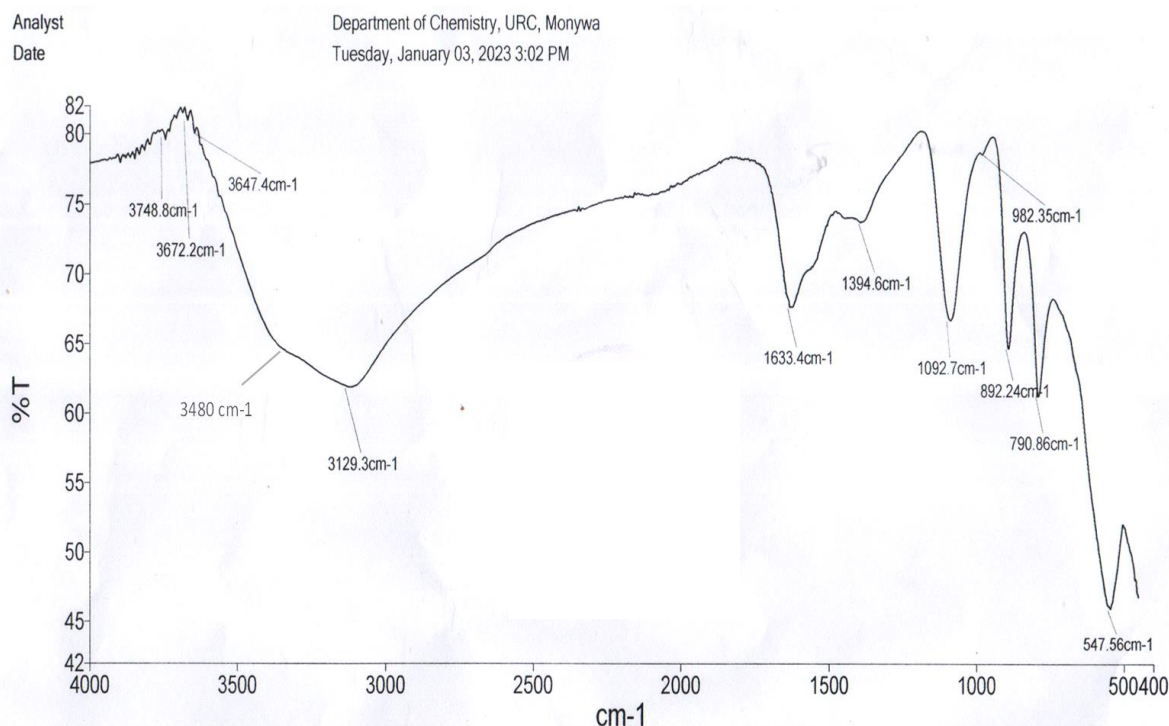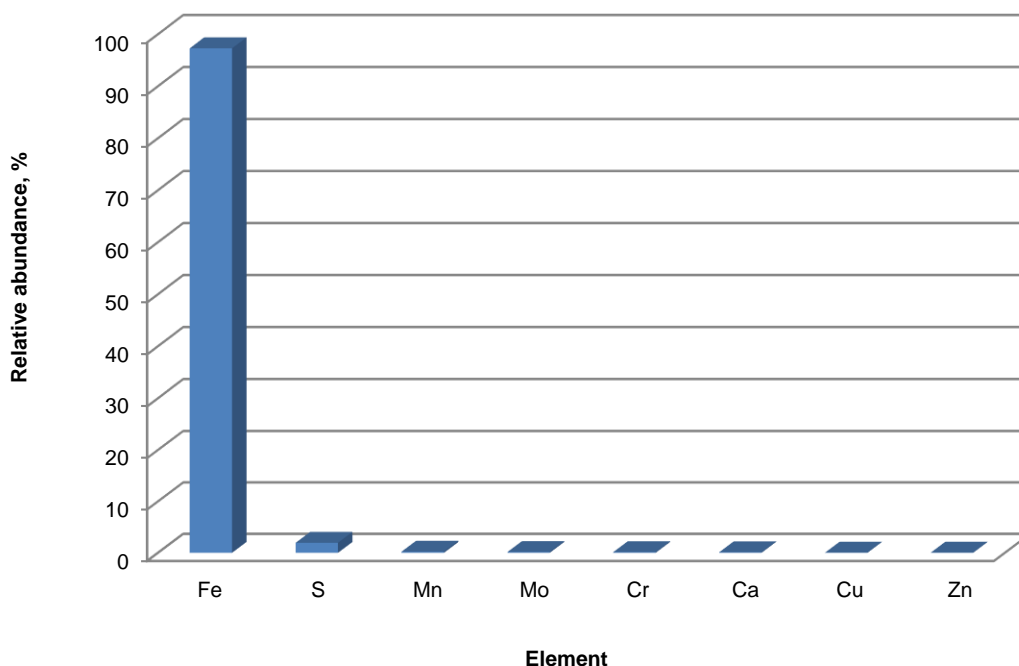

Fig. 5. The FTIR spectrum of Fe_3O_4/CDs nanocomposite.

Fig. 6. Chart of elemental composition of Fe_3O_4/CDs nanocomposite.
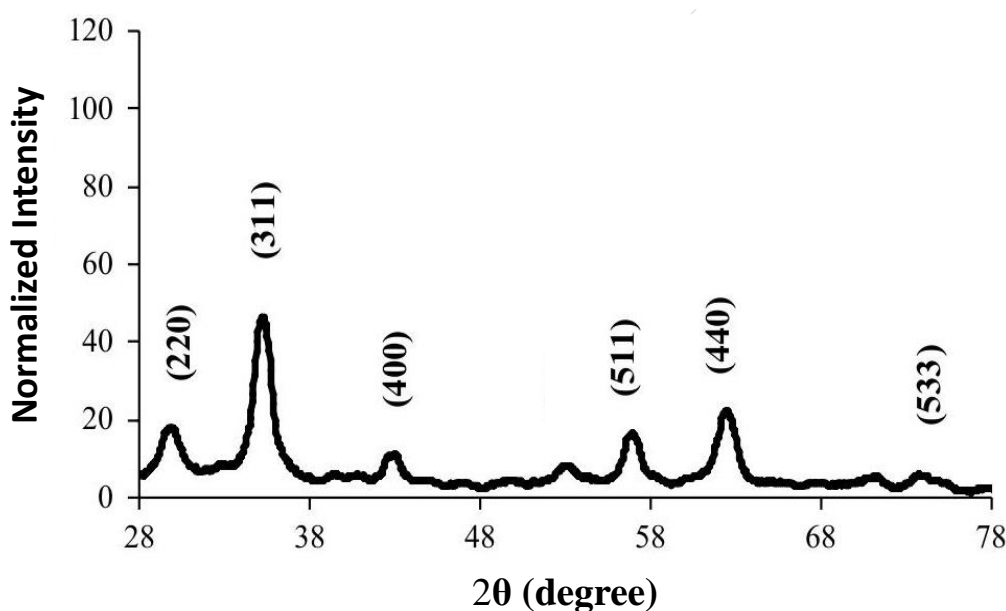

Fig. 7. The XRD peaks of the prepared Fe_3O_4/CDs nanocomposite.

3.2. Thermogravimetric analysis

The decomposition behaviors of Fe_3O_4/CDs nanocomposite has been observed by TGA analysis, which were treated with 10 °C/min (heating rate) from 20 °C to 550 °C in air. As indicated in Fig. 8, the red line indicated the curves 81.78 °C, 267.43 °C, 400.96 °C, 471.79 °C, 519.60 °C and 576.49 °C, respectively. The thermal stability of the Fe_3O_4/CDs nanocomposite is good and remains stable, but at a temperature of about 576 °C, the thermal stability of Fe_3O_4/CDs nanocomposite was with little decrease, it is likely due to the decomposition of carboxylic and release of CO_2 gas.

3.3. Determination of Hg^{2+} and Pb^{2+} removal efficiency by Fe_3O_4/CDs nanocomposite

A well-known preparation of MNPs and carbon based nanomaterials has gained deep attention for the use of nanoadsorbent to remove heavy metals in wastewater. Because both carbon dots and Fe_3O_4 nanoparticles have a potential performance to synthesize magnetite carbon dots nanocomposite, which was possibly implemented to remove heavy metals by adsorption. Thus, the investigation of the prepared Fe_3O_4/CDs nanocomposite effect on the removal of Hg^{2+} and Pb^{2+} was studied in aqueous solution. Afterward, the adsorption ability of Fe_3O_4/CDs nanocomposite for Hg^{2+} and Pb^{2+} removal was revealed and results were compared to the bare Fe_3O_4 nanoparticles. Table 1 and 2 indicated the removal efficiency (%) of 10 mL^{-1} of each cation in the presence of 10 mg of prepared Fe_3O_4/CDs nanocomposite. Significantly, removal efficiency (%) of Hg^{2+} and Pb^{2+} for the Fe_3O_4/CDs nanocomposite is higher than that of Fe_3O_4 magnetic nanoparticles as shown in Fig. 9. After combination of MNPs and CDs, there are carboxylate moieties and carbonyl groups are positioning on the surface of the prepared nanocomposite. So, the negatively charged –COOH or OH on the nanocomposite can interact with Hg and Pb cations for adsorption process. The phenomenon is that Hg^{2+} and Pb^{2+}could efficiently coordinate onto the surface of Fe_3O_4/CDs nanocomposite. Accordingly, carbon dots covering nanocomposite significantly enhanced the adsorption capability of MNPs.
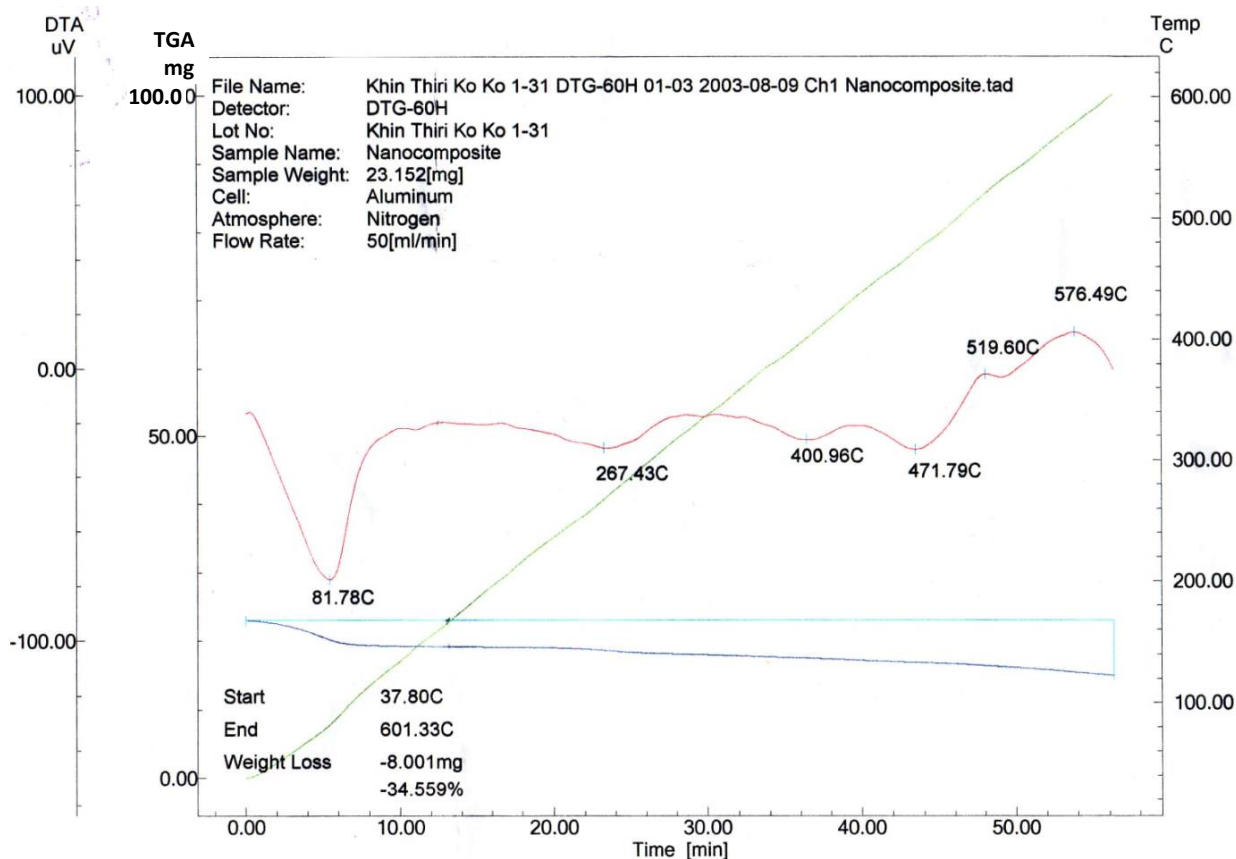
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Fig. 8. Thermogravimetric analysis (TGA) curve of prepared Fe_3O_4/CDs nanocomposite.

Table 1. Comparison of 10 mg Fe_3O_4 MNPs and Fe_3O_4/CDs nanocomposites for the removal of 10 mL^{-1} of Hg^{2+}.

No.	Samples	Heavy metal	Removal efficiency (%)
1	Fe_3O_4	Hg	46.11
2	Fe_3O_4/CDs Nanocomposite	Hg	82.70

Table 2. Comparison of 10 mg Fe_3O_4 MNPs and Fe_3O_4/CDs nanocomposites for the removal of 10 mL^{-1} of Pb^{2+}.

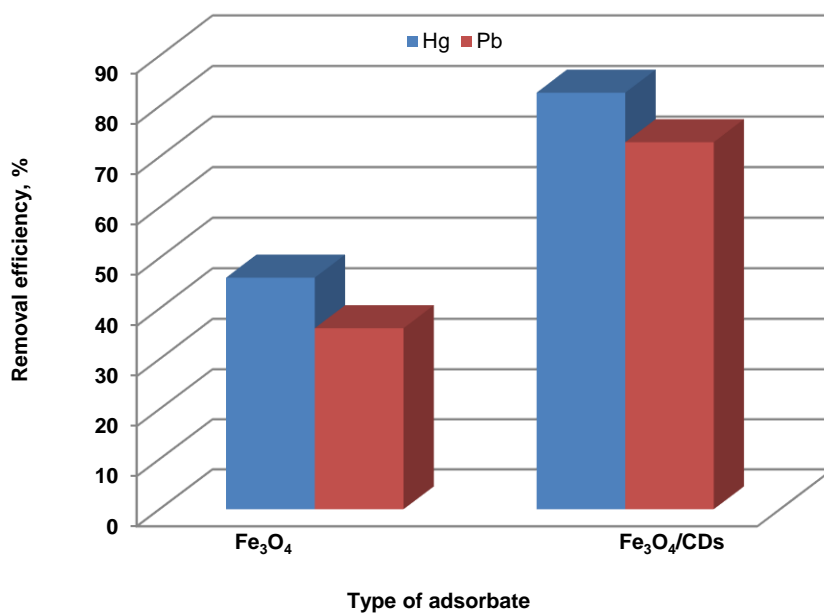| No.	Samples	Heavy metal	Removal efficiency (%)
1	Fe_3O_4	Pb	36.11
2	Fe_3O_4/CDs Nanocomposite	Pb	72.91

Fig. 9. The chart of removal of Hg and Pb by adsorption of Fe_3O_4 and Fe_3O_4/CDs.

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Table 3. The comparison of removal of heavy metals by iron oxide-based nanomaterials in adsorption with present study.

Nanoadsorbent	Heavy Metal	Removal efficiency	Ref.
Fe_3O_4-ZrMOF@GSH	Hg(II), Cd(II), and Pb(II)	95–99%	Ragheb *et al*., 2022
Fe_3O_4@Z-NCNT/PC	Pb(II)	99%	Jafari *et al.* 2020
Fe_3O_4@SiO_2@GLYMO(S)-en	Pb(II)	90%	Masjedi *et al*., 2020
Magnetic chitosan nanocomposites	Pb(II)	94%	Liu *et al*., 2009
PANI-F-S	Hg, As and Pb	~80%	Hashemi *et al*., 2019
Fe_3O_4/CDs Nanocomposite	Pb(II) & Hg (II)	72.91% & 82.70%	Present study

4. Conclusions

In summary, low-cost magnetite carbon dots (Fe_3O_4/CDs) nanocomposite was successfully synthesized by a two-step process of co-precipitation and pyrolysis methods in this work. The prepared Fe_3O_4/CDs nanocomposite possessed some important features like superparamagnetic property, surface roughness and deformations with large surface area, potential functional groups and iron-rich nanocomposite. In particular, the synthesized (Fe_3O_4/CDs) nanocomposites had satisfying removal efficiency of Hg^{2+} (72.91%) and Pb^{2+} (80.70%), respectively in comparison with that of the bare Fe_3O_4 magnetic nanoparticles. Moreover, the reaction is simple and cost-effective. The nanocomposite demonstrated better superparamagnetic properties at basic condition (pH 9). However, the effect of pH, temperature and sample dosage was still required to perform so as to show environmental tolerance. And then, nanocomposites are still required to treat it with wastewater in real life using adsorption kinetics. To sum up, it is hoped that the Fe_3O_4/CDs

nanocomposite can be used effectively as a removal agent of Hg^{2+} and Pb^{2+} in the environment according to the findings and explored method.

Author Contributions

Yaung Kwee: Conceptualization, investigation, methodology, and writing, review, and editing the manuscript.
Khin Thiri Ko Ko: Laboratory work.

Conflict Interest

The authors declare that they have no known competing financial interests that could have appeared to influence the work reported in this research.

Acknowledgments

Data Availability Statement

The datasets used in the current study are available on request.

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