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Comparative Study of Concrete Wastes as Low-Cost Adsorbent for Copper Removal from Aqueous Solution

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ABSTRACT

The presence of copper in water sources poses significant environmental and health risks, requiring effective removal strategies. Adsorption is one of the most efficient processes to treat contaminated water due to its advantages such as availability, low cost, and eco-friendly nature. This study explores the potential of utilizing concrete wastes as a low-cost adsorbent for the copper ions removal from aqueous solutions. Two types of concrete wastes have been used as low-cost adsorbents in this study: i) normal concrete waste (NCW) with mix proportion of Cement, water, aggregates 20, aggregates 10, sand and ii) modified concrete waste (MCW) with Cement, water, aggregate 10, sand, superplasticizer (2% cement), silica fume (10% cement). The uptakes of copper ions on NCW and MCW were studied at different initial metal Cu2+ ion concentrations, pH, adsorbent dosages and contact time. The maximum removal efficiency of copper removal for both types of concrete wastes achieved 99.99% at pH 5.0 for both NCW and MCW at equilibrium conditions. The maximum metal uptakes capacity achieved at 0.6 mg/g for both NCW and MCW exhibits good adsorption characteristics. The NCW adsorption data fitted well with Langmuir isotherm (R^2= 1), whilst the MCW data obeyed Freundlich isotherm (R^2 = 0.9889).

Keywords: Normal Concrete Waste (CW), Modified Concrete Waste (MCW), adsorption isotherms, Atomic Absorption Spectrometry (AAS)

INTRODUCTION

The most valuable resource is water for all life on Earth, which is usually contaminated by various contaminants produced from human activities. In recent years, heavy metal-induced water pollution has presented conservationists with a unique issue. The astounding amounts of heavy metals used in recent decades have unavoidably led to a rise in the amounts of metallic materials entering water. Adsorption is one of the most efficient processes to clean contaminated water. The adsorption process presents advantages such as availability, low cost, and eco-friendly nature (Hussain et al. 2021). Low-cost adsorbents could be used to remove hazardous heavy metals from wastewater in an economical manner compared to commercial activated carbon. Varieties of industrial solid wastes, agricultural wastes, and random products have an affinity for heavy metal adsorption (Ho et al. 2021a).

Copper, as one of the most extensively utilized industrial metals, poses a significant ecological and toxicological threat due to its growing accumulation in organisms. Industrial wastewater originating from various processes such as cardboard manufacturing, metal cleaning and smelting, paint and pigment production, mining operations, fertilizer usage, electroplating baths, petroleum refining, brass rinses, wood pulp processing, printed circuit

board production, and etching contains substantial quantities of copper metal ions (Khan et al. 2021). Copper toxicity can cause irritation, corrosion, liver damage, irritation of the central nervous system and depression (Jain 2013). A threshold limit for Cu discharge by treated industrial effluent to the river must not exceed concentration of 0.20 (Standard A) and 1.0 mg/L (Standard B) under the Malaysian Environmental Quality Act 1974 (Jabatan Alam Sekitar 2009). The permissible limit of Cu in drinking water 2.0mg/L stated by WHO is 2.0mg/L (C. Abbot et al. 2022; Manne et al. 2022)

Concrete wastes contribute to 5% of the overall construction and demolition (C&D) wastes that originate from construction, demolition, and renovation operations. Environmental Protection Agency (EPA) Australia classifies C&D wastes as solid waste generated during construction activities, including waste from building and demolition operations, as well as excavated materials (Pallewatta et al. 2023). The construction material waste is not easily biodegradable and incorrect disposal of these materials can result in land, air and wastewater pollution, contributing to a great hazard to the environment. This has put construction industries under pressure to consider suitable methods to protect the environment across all industries including construction activities (Ho et al. 2021b) Countries such as Australia, Japan and Singapore have demonstrated the recycling of C&D wastes practices by >90% (Mah et al. 2018). However, the recovery of C&D wastes in Malaysia was 13.7 % in 2018 and still low (CIDB Malaysia, 2020). Cement brick waste can be considered as an ideal media for the adsorption of heavy metals due to its structural and chemical properties such as high bulk density, various reactive minerals from cement, sand, crushed rock which favors the adsorption of heavy metals (Mokokwe & Letshwenyo 2022). Furthermore, a nano-lamellar structure of silicate minerals in clay or cementous materials has been reported to adsorb metals on broken surfaces due to its low swelling and water dispersion (Barati et al. 2013; Yoo et al. 2018; Ali & Abd 2019). These studies utilised concrete sludge and crushed concrete demolition waste (CCDW) respectively to remove Cu, Pb, and Zn, with the maximum removal efficiency achieving 95 to 100%. Therefore, in this study concrete wastes were used as an alternative for low-cost adsorbent to remove copper ions (Cu^{2+}) in the aqueous solution. Two types of concrete wastes, namely normal concrete waste (NCW) and modified concrete waste (MCW) were employed as low-cost adsorbents and the performance of both adsorbents were compared in the current study. To attain an appropriate removal efficiency of Cu^{2+}, the effect of four physicochemical factors including concrete dosage, initial pH, initial Cu^{2+} concentration, and contact time were studied in batch system. The adsorption isotherms were also applied to understand the adsorption phenomena for both types of adsorbents in this study.

MATERIALS AND METHODS

PREPARATION OF NCW AND MCW AS LOW-COST ADSORBENT

NCW and MCW were obtained from Concrete Laboratory, UiTM Shah Alam, Selangor. Concrete wastes were in the form of concrete cubes, which were left unused after undergoing concrete strength testings in the laboratory. These concrete cubes were used as adsorbents and may contribute to another achievement in the waste management process. NCW (Cement, water, aggregates 20, aggregates 10, sand) and MCW (Cement, water, agg 10, sand, superplasticizer-2% cement, silica fume-10% cement) were collected and crushed using jaw crushing separately. Then the crushed concrete was sieved to the desired size which is 4.75 mm to 2.00 mm. After that, both crushed NCW and MCW were soaked in distilled water for 24 hours to remove any dust or dirt particles and dried using a muffle furnace for approximately 60 minutes at temperature of 850 °C (Figure 1) (Ma et al. 2021). Both concrete wastes were allowed to cool down to room temperature and kept in dry storage prior use.

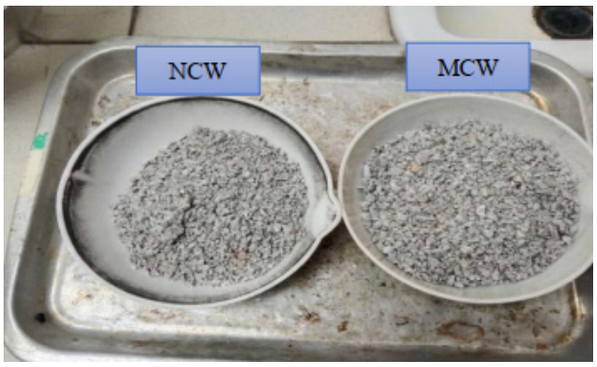

FIGURE 1. Dried concrete wastes

PREPARATION OF STOCK SOLUTION AND SYNTHETIC WASTEWATER

Copper Sulphate Pentahydrate (CuSO4.5H2O) was used to prepare synthetic wastewater by dissolving 3.93 g of $CuSO_4.5H_2O$ in 1 litter of distilled water, resulting a stock solution with concentration of 1000 mg/L of Cu^{2+} ion (Malacas et al. 2019). Subsequently, the required concentration of synthetic wastewater solutions was prepared by diluting the stock solution with distilled water

to achieve the desired concentration range of 5 mg/L to 30 mg/L. 0.1 M hydrochloric acid (HCl) and 0.1 M sodium hydroxide (NaOH) were used to adjust the pH of the synthetic wastewater solutions.

BATCH MODE STUDIES

The entire set of experiments was conducted through batch mode investigations. 100 ml of synthetic wastewater samples containing Cu^{2+} ions were added with a known quantity of the adsorbent, in conical flasks. The flasks were agitated at 100 rpm using an orbital shaker at room temperature (27ºC) for specific time intervals. The impact of pH, agitation duration, initial metal ion concentration, and the quantity of adsorbent were examined in relation to the removal of Cu^{2+} ions (Table 1). All the experiments were run in duplicates. The study range of these parameters were selected based on the previous studies (Lim 2020). Following the designated contact time, the flask content was withdrawn and filtrated using Whatman filter paper. The resulting filtrate was then utilized for estimating Cu^{2+} ions through Atomic Absorption Spectrometry (AAS) (AAnalyst 800, Perkin Elmer), equipped with a Cu hollow cathode lamp as a radiation source. The acetylene flow rate and the burner height were adjusted in order to obtain the maximum absorbance signal (energy level output of 68mA). The batch adsorption process is summarized in Figure 2 (Khaled et al. 2019).

Control experiments were implemented without the use of absorbent to account for any metal ion adsorption on the container walls, and the observed effect was found to be negligible. Upon obtaining the result from AAS analysis, the removal percentage of individual sample was calculated and analysed. The formula to calculate the removal percentage of Cu^{2+} (Eq.1) and adsorbent uptake at equilibrium (Eq. 2) are as follows:

$$\%R = (Co-Ce)/Co \times 100 \quad (1)$$

$$qe = (Co-Ce)/MV \quad (2)$$

where, qe = metal uptake capacity (mg), Co = initial metal concentration (mg/L), Ce = equilibrium metal concentration (mg/L), V = volume of stock solution (L), M = dry mass of adsorbent added (g).

TABLE 1. Summary of different parameters varied in this study

Initial Cu^{2+} Concentration (mg/L)	pH	Adsorbent Dosage (g)	Contact Time (min)
5, 10, 15, 20, 25, 30	2	5	60
NCW: 20 MCW: 30	2, 3, 5, 7, 9, 11	5	60
NCW: 20 MCW: 30	9 5	5, 10, 15, 20, 25, 30	60
NCW: 20	9	10	5, 10, 15,20, 25, 30, 35, 40, 45,50, 55, 0, 65, 70, 75
MCW: 30	5	25	

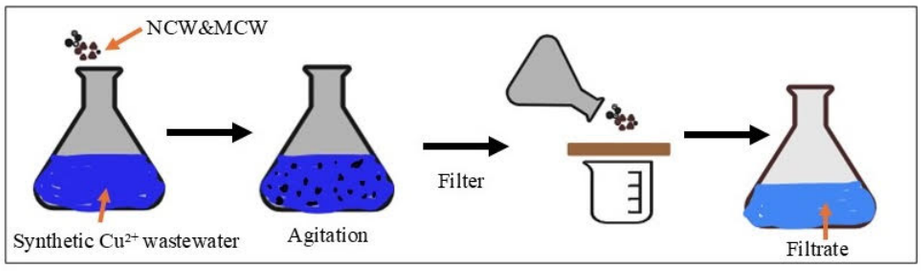

FIGURE 2. General procedure of batch experiment of NCW or MCW for Cu^{2+} removal and agitated at 100 rpm

ISOTHERMS STUDY

After conducting batch adsorption, the adsorption data were fitted into Langmuir and Freundlich isotherms. The metal ions removal was determined using Equation below.

$$qe=((Co-Ce)\ V)/m \quad (3)$$

where;
qe = amount of metal ions removal at equilibrium, mg/g
Co = initial concentration of metal ion in liquid sample, mg/L
Ce = equilibrium concentration of metal ion in liquid sample, mg/L
V = volume of liquid sample, L
m = mass of adsorbent, mg

For Langmuir isotherm, the Ce and Qe obtained from Equation below will be fitted into the linearly Langmuir equation:

$$Ce/Qe = Ce/Q_{max} + 1/(K_{Lmax}) \quad (4)$$

where;
Q_{max} = maximum adsorption capacity, mg/g
K_L = adsorption energy, L/mg

A graph of Ce/Qe against Ce was plotted. The slope of the graph could be used to find the Q_{max} and the intercept could be used to find K_L. Meanwhile, for Freundlich isotherm, the Ce and Qe obtain from Equation (3) could also be insert into the linearly Freundlich isotherm equation:

$$\log Qe = logK_F + (1/n) \log Ce \quad (5)$$

where;
K_F = Freundlich constant representing the adsorption capacity
n = Freundlich constant representing the adsorption intensity

RESULTS AND DISCUSSION

EFFECT OF INITIAL CU^{2+} CONCENTRATION

The effect of initial concentrations of Cu^{2+} (solution (5 – 30 mg/L) onto 0.5g/L of NCW and MCW adsorbents was conducted in 60 minutes is shown in Figure 3. The removal of Cu^{2+} by both concrete wastes achieved >90%, regardless of the initial concentration. The removal of Cu^{2+} achieved 99% at concentration ≥ 20 mg./L for both concrete wastes. The removal efficiencies of Cu^{2+} by NCW and MCW were lower (<95%) at concentrations <15 mg/L may be due to the lower of Cu^{2+} ion concentration than the active adsorption sites. decreased as the initial concentration of Cu^{2+} (Ab et al. 2022a). As the concentration of Cu^{2+} increases, more metal ions become available for interaction with the active sites on the on concrete waste and achieved ~0.6 mg/g of metal uptake at initial concentration of Cu^{2+} 30mg/L for both concrete wastes.

EFFECT OF PH

The pH of the synthetic wastewater solution plays a crucial role in the adsorption process, with removal efficiency hinging on this parameter. In pursuit of the optimum pH for Cu^{2+} removal, experiments involved equilibrating 5g/L of NCWs adsorbent in 100 mL solutions containing 20 mg/L of Cu^{2+} at various pH ranging from 2.0 to 11.0 for 60 minutes contact time. The highest removal efficiency of Cu^{2+} was almost 100% at pH 2-11 for both concrete wastes, showing that these adsorption processes were pH independent (Figure 4). The high surface area, porous structure, and active binding sites on the concrete waste could contribute for this pH independence. These binding sites consistently interact with Cu^{2+} ions through mechanisms such ion exchange and complexation. Concrete waste and other materials with a high calcium content have built-in buffering capabilities that stabilize the adsorption sites and sustain high removal effectiveness at different pH levels. This demonstrates how resilient concrete waste is as an adsorbent, able to function at a high level without requiring pH adjustments, which streamlines the treatment procedure and lowers operating expenses (Ab et al. 2022b; Su et al. 2024). The metal uptake capacity for MCW is higher than NCW, which is 0.6 mg/g (Table 3). This may be due to the higher adsorbent surface in MCW compared to NCW (0.4 mg/g). In addition, the addition of silica fume in the MCW may add more active surface area for adsorption (Kalkan et al. 2012). Given the objective of establishing a new method for industrial use, it was decided to consistently maintain the pH at 5.0 for all subsequent experiments.

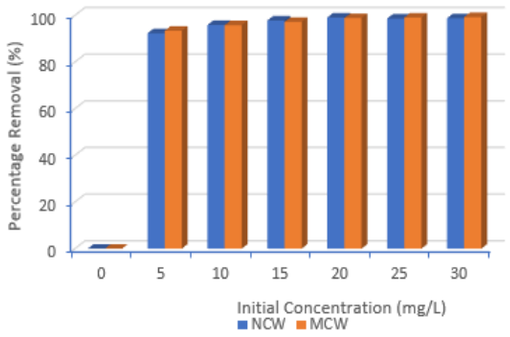

FIGURE 3. Percentage removal of Cu^{2+} at different initial concentrations using NCW and MCW as adsorbent (constant parameters: adsorbent dosage = 0.5g/L, contact time = 60 mins, agitation speed =100 rpm)

TABLE 2. Cu^{2+} uptake capacity by NCW and MCW at different initial Cu^{2+} concentrations

	NCW	MCW
Initial Concentration, Co (mg/L)	Metal Uptake Capacity, (mg/g)	Metal Uptake Capacity, (mg/g)
0	0	0
5	0.0923	0.0934
10	0.1918	0.1916
15	0.2934	0.2916
20	0.3964	0.3954
25	0.4933	0.4954
30	0.5927	0.5956

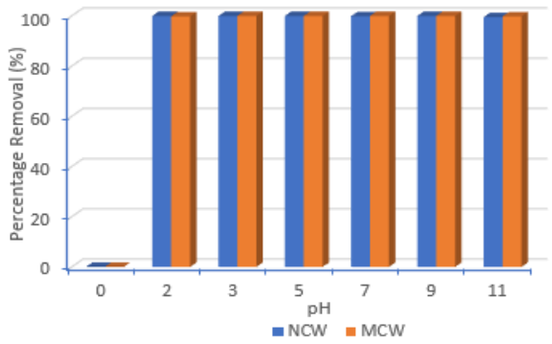

FIGURE 4. Percentage Removal of Cu^{2+} at different pH using NCW and MCW as adsorbent (constant parameters: 20 mg/L, 0.5g/L NCW and MCW, 60 mins, 100 rpm)

TABLE 3: Cu^{2+} uptake capacity by NCW and MCW at different pH (initial Cu^2 concentration of 20 mg/L)

pH	NCW	MCW
	Metal Uptake Capacity, (mg/g)	Metal Uptake Capacity, (mg/g)
0	0	0
2	0.3999	0.5987
3	0.3999	0.5999
5	0.3999	0.5999
7	0.3994	0.5999
9	0.3999	0.5999
11	0.3980	0.5987

EFFECT OF ADSORBENT DOSAGE

The removal of Cu^{2+} at different adsorbent dosages from 5 g to 30 g (0.5-3g/L) is detailed in Table 4. The optimal removal of Cu^{2+} occurred at a dosage of 5 g (0.5g/L), achieving a 99.99% Cu^{2+} removal for both NCW and MCW, which are comparable with the results from previous studies (Table 5) This phenomenon is attributed to the augmented number of binding sites with the increase in adsorbent dosage and reached saturation point (Hemalatha 2014). Higher dosages, when used beyond the saturation point, do not significantly improve adsorption performance and can lead to practical drawbacks such as costly, inefficient resource utilization and excess adsorbent for disposal (Badawi et al. 2021). The variation in adsorption capacity is linked to the adsorbent dosage, influencing the abundance of active sites and the available surface area for adsorption. This dependency arises from the augmentation in surface area, enhancing the likelihood of collision and interaction between the particles of NCW and MCW with Cu^{2+}(Kumar et al. 2010).

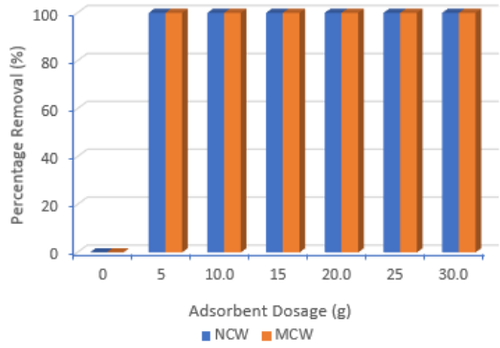

FIGURE 5. Percentage removal of Cu^{2+} for different adsorbent dosage using NCW and MCW as adsorbent (constant parameters: initial concentration = 20 mg/L, pH = 5.0, contact time = 60 mins, agitation speed = 100 rpm)

TABLE 4. Cu^{2+} uptake capacity by NCW and MCW at different adsorbent dosage (initial pH constant at 5)

Adsorbent Dosage (g)	NCW	MCW
	Metal Uptake Capacity, (mg/g)	Metal Uptake Capacity, (mg/g)
0	0	0.000
5	0.4999	0.6000
10.0	0.2499	0.3000
15	0.1667	0.2000
20.0	0.1249	0.1500
25	0.0999	0.1200
30.0	0.0833	0.1000

TABLE 5. Performance of different low-cost adsorbent materials to remove Cu^{2+} and other heavy metals

Material	Type of metals	Initial concentration of metal mg/L	Max Adsorption capacity mg/g	Max Removal efficiency	Reference
Excess sludge	Cu (II)	100	47.62	92%	(Mu et al. 2018)
Thermally modified waste concrete powder	Pb (II)	500	46.02	92.96%	(Ma et al. 2021)

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Crushed concrete demolition waste	Cu (II)	100	39.286	100%	(Ali & Abd Ali 2019)
Dragon fruit peel Rambutan peel Passion fruit peel	Cu (II)	100	92.593 192.308 121.951	53.80% 97.70% 66.00%	(Phuengphai et al. 2021)
Concrete sludge	Cu (II) Pb (II) Zn (II)	100	-	98.3% 99.9% 95.2%	(Yoo et al. 2018)
Granules of brick waste (GBW)	Nickel (Ni+2)	50	1.153	39.4%	(Mhawesh & Abd Ali 2020)
Normal concrete waste (NCW) Modified concrete waste (MCW)	Cu (II)	30	NCW=0.5 MCW=0.6	99.9% 99.9%	This Study

EFFECT OF CONTACT TIME

To explore the impact of contact time and the removal percentage of Cu^{2+} by both types of concrete wastes, the adsorption experiment was conducted by agitating 100 mL of Cu^{2+} solutions (20 mg/L, pH 5.0) with 10 g of adsorbent (1 g/L) for 75 minutes. Every interval time of 5 minutes, the flask was withdrawn, filtrated and the Cu^{2+} content was analyzed using AAS.

The adsorption of Cu^{2+} as a function of time and concentrations is presented in Figure 6 and Table 6. The initial phase of the adsorption process exhibited a rapid rate of uptake (the first 5 minutes), which later slowed down, ultimately reaching saturation. In the initial stages, metal ions of the adsorbate preferentially occupied numerous active sites in a random manner, resulting in the swift uptake of Cu^{2+} ions. Over time, these active sites became blocked, leading to a gradual decrease in the rate of uptake. For achieving maximum removal of Cu^{2+} ions by NCW and MCW, it was determined that solutions equilibrated within 5 minutes (Table 5). This may be due to the rapid adsorption that causes efficient diffusion of adsorbate molecules across the surface of the adsorbent, indicating chemisorption as the primary mechanism (following pseudo-second-order model) (Badawi et al. 2021). This rapid adsorption, achieving 99.99% removal within 5 minutes, is comparable to other studies involving high-surface-area adsorbents like activated carbon, biochar, and metal-organic frameworks, which also report significant metal ion removal within a short time frame (Ba et al., 2023; Yang, 2022)

The graph depicted in Figure 6 are singular, smooth, and continuous for both concrete wastes, suggesting the formation of a monolayer coverage of the adsorbate on the adsorbent surface (Hemalatha 2014). Complete removal was attainable with a contact time as short as 5 minutes, achieving 99.99% removal.

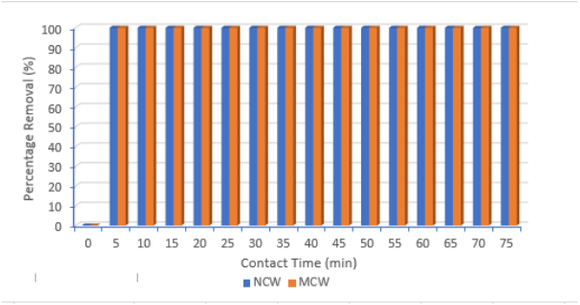

FIGURE 6. Percentage Removal of Cu^{2+} for different contact time using NCW and MCW as adsorbent (constant parameters: initial concentration = 20 mg/L, pH = 5.0, adsorbent dosage = 10 g, agitation speed =100 rpm)

TABLE 6. Cu^{2+} uptake capacity by NCW and MCW at different contact time (adsorbent dosage constat at 10g)

	NCW	MCW
Contact time (min)	Metal Uptake Capacity, (mg/g)	Metal Uptake Capacity, (mg/g)
0	0	0
5	0.2499	0.1199
10-75	0.2499	0.1199

ADSORPTION ISOTHERMS

The adsorption data were fitted to both Freundlich and Langmuir adsorption isotherms. The graph (Figure 7) show that the adsorption isotherm for Cu^{2+} removal using NCW fitted well with Langmuir isotherm as a perfectly linear line with correlation coefficient, R^2 =1 compared to Freundlich isotherm (R^2 = 0.9195, Figure 8). Langmuir Isotherm is for monolayer adsorption on adsorbent surface containing finite number of identical sites. The Langmuir constants q_{max} and K_L for both concrete wastes were

calculated from the slope and intercept of the plots. The q_{max} and K_L for NCW (Figure 7) is 0.24979 mg/g and 400336.28 L/mg respectively. The calculated R_L was 0.09992×10^6. The adsorption process of Cu^{2+} was favourable as it was within the range of $0< R_L <1$(Ayawei et al., 2017). The plot of Ce/qe vs Ce in Figures 9 and 10 shows Cu^{2+} adsorption onto MCW obeys Langmuir and Freundlich Isotherms. The graphs show a perfectly linear line with correlation coefficient, $R^2 =1$ and $R^2 = 0.9889$ for NCW and MCW respectively. The Langmuir constants q_{max} and K_L for MCW (Figure 9) are 0.1199 mg/g and -834028.357 L/mg respectively. Since the value of adsorption capacity (q_{max}) and the equilibrium constant (K_L) on the Langmuir isotherm are negative, meaning that this isotherm does not meet the requirements to be used as an appropriate isotherm model to describe the process of adsorption. Whilst the Freundlich constants Kf and n for MCW are 0.9993 and 1.0852 respectively. Since the Freundlich isotherm has a positive Freundlich equilibrium constant (Kf) and a positive adsorption intensity (1/n), thus, Freundlich isotherm is more suitable to describe the Cu^{2+} adsorption onto MCW. The Freundlich isotherm shows that the adsorption process forms in several physical layers on the surface of the adsorbent (multilayer), which involves the entrance of adsorbate onto the surface of the adsorbent through Van Der Waals bonds (weak bonds) and this reaction is reversible (Raji et al. 2023). In addition, the value of 1/n is less than 1, indicating that condition of the MCW in the adsorption process (Cu^{2+}) is favourable to be used as an adsorbent due to the strong interaction of the adsorbent and adsorbate. When both the Langmuir and Freundlich models fit the experimental data, and the equilibrium concentration (Ce) is the same at a certain time point due to a short time range (5 min), it shows that both monolayer adsorption (described by the Langmuir model) and adsorption or multilayer adsorption on heterogeneous surfaces (described by the Freundlich model) occur rapidly and simultaneously during the early stages of the adsorption process (Perwitasari et al. 2021).

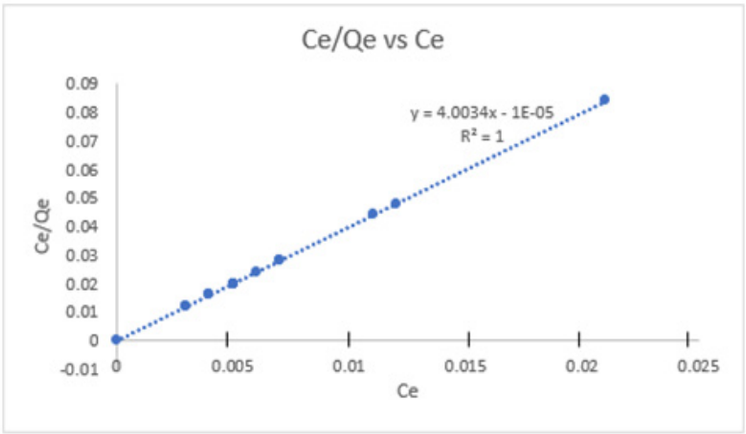

FIGURE 7. Graph of Ce/Qe vs Ce for Langmuir Isotherm using NCW as adsorbent

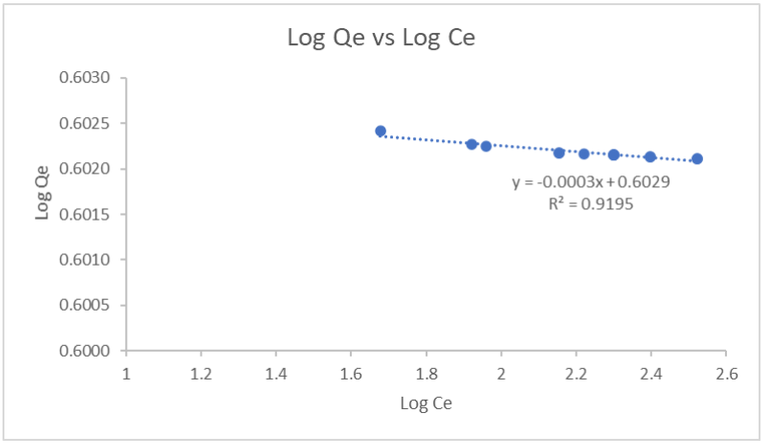

FIGURE 8. Graph of log Qe vs log Ce for Freundlich Isotherm using NCW as adsorbent

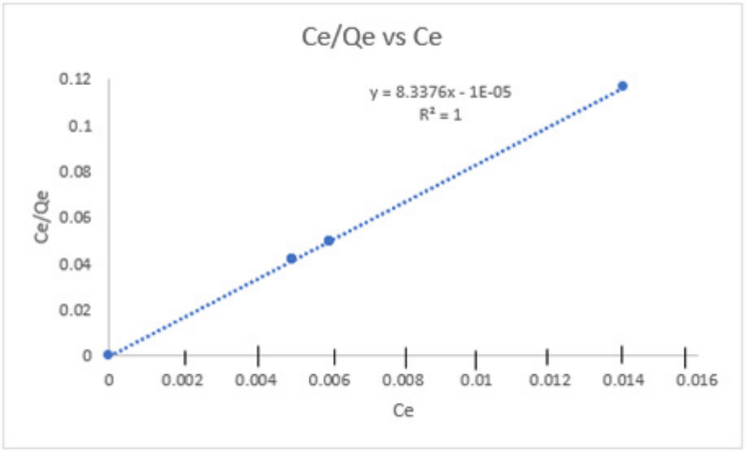

FIGURE 9. Graph of Ce/Qe vs Ce for Langmuir Isotherm using MCW as adsorbent

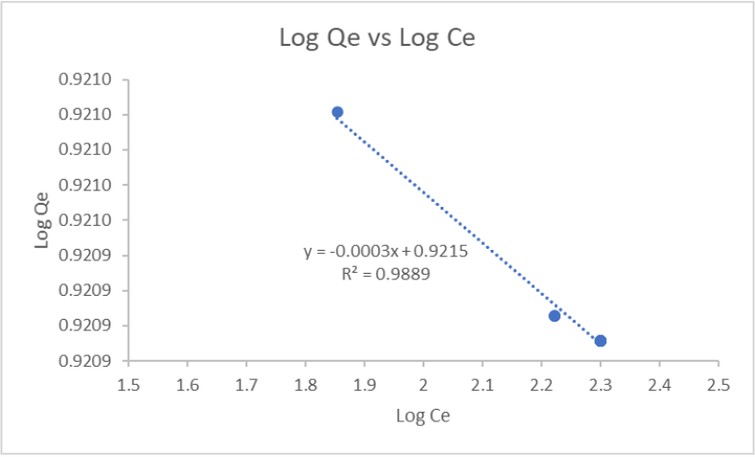

FIGURE 10. Graph of log Qe vs log Ce for Freundlich Isotherm using MCW as adsorbent

CONCLUSION

In this study adsorbents used were concrete wastes (NCW and MCW) which are available in large quantities and can be used as an alternative to existing commercial adsorbents for removal of Cu^{2+}. The removal of these carcinogenic toxicants was found to depend on dosage, pH, initial concentrations of Cu^{2+} ions and contact time. The adsorption capacity of both NCW and MCW for Cu^{2+} was not shown significantly different as they showed the same absorption rate (99.99%). The contact time for the maximum adsorption required is 5 minutes. The pH was independently affected by the adsorption of Cu^{2+} onto both types of concrete wastes. However, the higher Cu^{2+} uptake capacity was achieved by MCW. The equilibrium adsorption data are satisfactorily fitted Freundlich (MCW) and Langmuir (NCW) isotherms. The calculated values of the dimensionless separation factor from the Langmuir constant also confirmed favorable adsorption of Cu^{2+} onto NCW and MCW. Some further studies could be carried out to further understand the properties of concretes wastes as adsorbent in the future by using higher concentration of Cu^{2+}, evaluating regeneration, reuse possibilities and long-term stability. It can be concluded that NCW and MCW adsorbent appears to be technically feasible and eco-friendly, thus reducing waste generation.

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DECLARATION OF COMPETING INTEREST

None.

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