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Adsorption of Cu^{2+} and Fe^{2+} From Single Metal Ion Solution Using Unmodified and Formaldehyde Modified Kola-Nut (*Cola Nitida*) Testa

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Abstract: *The modification of kola-nuttesta with 0.2M sulphuric acid and 39.9%(v/v) formaldehyde was carried out at 50^{0}C. The formaldehyde modified kola-nut testa (FMKT) and unmodified kola-nut testa (UKT) were characterized by specific gravity, point of zero charge, melting point, solubility and FTIR spectroscopy. The adsorption capacity of the formaldehyde modified kola-nut testa(FMKT)for Cu^{2+} and Fe^{2+} ions in aqueous solution at equilibrium conditions and room temperature were: 2.24 mg/g for Cu^{2+}, 1.97 mg/g for Fe^{2+}.The unmodified kola-nut testa(UKT) gave adsorption capacities of 2.05 mg/g for Cu^{2+} and 2.12 mg/g for Fe^{2+}metal ions. Percentage removal of the metal ions from a single metal ion aqueous solution were carried out under various adsorption conditions such as temperature, pH, contact time, adsorbent dose and initial metal ion concentrations. The Fe^{2+} metal ion gave better results with optimal average percentage removal above 90%, using the formaldehyde modified kola-nut testa when compared to Cu^{2+} metal ion with an average of 70% percentage removal. Similarly, the unmodified kola-nut testa followed a similar trend. Thus an indication that both unmodified and formaldehyde modified kola-nut testa holds promising potentials in the treatment of industrial effluents containing heavy metals.*

Keywords: *Adsorption capacity, formaldehyde modified kola-nut testa, single metal ion, percentage removal*

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I. Introduction

The removal of heavy metals from aqueous solution has been studied extensively (Bandelaet. *al.*,2016; Mohammed *et. al.*,2014; Dhabab 2011; Johnson *et. al.*, 2008). Industrially, there are varied techniques available in the removal of heavy metals from wastewaters. Distillation, reverse osmosis, electro-dialysis, evaporation, chemical precipitation, ion-exchange, ultra-filtration, nano-filtration, flocculation and coagulation are some of the methods frequently used in the treatment of wastewaters containing heavy metals, (Chukwu*et. al.*, 2017;Uchechukwu*et. al.*, 2016; Bhatnagar*et. al.*, 2015; Mohammed *et. al.*, 2014; Srivastava and Goyal, 2010). However, these techniques have some drawbacks which limit their usefulness. Some of the demerits of these technologies include: high cost, maintenance cost, high energy requirements and non-biodegradability. These drawbacks gave impetus for the search for locally available and cost-effective alternative methods for the treatment of wastewaters.

Bio-sorption technique is an emerging technology that utilizes low cost materials such as: agricultural wastes (Sharma *et. al.*, 2013; Abia and Igwe 2006; Ahalya*et. al.*, 2003), activated carbon from natural sources (Youssef *et. al.*, 2013), industrial waste materials (Mohammed *et. al.*,2014); etc., to remove heavy metals from aqueous medium. The use of agricultural wastes such as: kola-nut testa, coconut coir, sugarcane bagasse, beans husks, rice husks, etc., as bio-sorbent materialor heavy metal removal from aqueous solution can be attributed to their chemical properties (Bhatnagar*et. al.*, 2015; Garcia-Reyes and Rangel-Mendez, 2009; Sud*et. al.*,2008; Mohan and Pittman, 2007).

The utilization of agricultural waste materials in the remediation of wastewaters containing heavy metals has shown that they are effective in removing heavy metals from aqueous solutions. Mohammed *et. al.*,(2014) reported that coconut shell, mango skin, rice husk, palm oil fuel ash and corn cob are effective in the treatment of industrial effluents laden with heavy metals. Giwa*et. al.*,(2013), used melon husk (*Citrulluslanatus*) in the adsorption of cadmium from waste water. In addition, Kumar (2006), showed that agricultural products and by products could be used as low-cost adsorbents for heavy metal removal from wastewaters. Similarly, Shah *et. al.*,(2016) demonstrated the removal of nickel from aqueous solutions using agricultural waste while Kyzas*et. al.*,(2013) removed copper ions using coffee waste from a local café.These findings however indicated that adsorption of metal ions from aqueous solutions with agro waste materials is favourable and cost effective.

However, further research has been carried out on the modification of theses agro-wastes materials to improve their performance. Thi*et. al.*,(2015) modified Sugarcane bagasse with H_2O_2, NaOH and citric acid;

from his findings the modified sugarcane bagasse exhibited the highest adsorption capacity. In their work, Chukwu*et. al.*,(2017) and Uchechukwu*et. al.*,(2016) demonstrated the removal of heavy metal ions in produced water using cation exchange resins from formaldehyde polymerized peanut testa (*Arachishypogaea L*) extract. Results obtained from their study showed that the modified resins compared favourably with commercial ion exchange resin (Bio-Rex 70).

In their review, Nilanjana*et al.,* (2008); reported that biosorption represented a biotechnological innovation which was cheaper for the treatment of aqueous solution contaminated with heavy metals. The emergence of this promising technique was due to the shortcomings of conventional methods of removing heavy metals from aqueous streams. It is hoped that bio-sorption will compete and eventually replace conventional methods when fully established in the nearest future. Hence, this study demonstrates the adsorption of Cu^{2+} and Fe^{2+} from single metal ion solution using unmodified and formaldehyde modified kola-nut (*cola nitida*) testa.

II. Experimental Materials And Methods

Sample Preparation and Extraction

Kola-nut samples were obtained from Nkwo Market at Omuma Local Government Area in Rivers State, Nigeria. The samples were subsequently identified at the University of Port Harcourt Herbarium as *Cola nitida*. The kola-nuts were then hulled and the testa washed, sundried and sieved to 100μm particle size, this formed the unmodified kola-nut testa (UKT).Subsequent treatment with formaldehyde (CH_2O) was done to give the formaldehyde modified kola-nut testa (FMKT). All reagents used were of analytical grade from BDH and used without further purification.

Extraction and modification procedure reported by Kumar *et. al.,* (2012) and Adediran*et. al.,* (2007) was adopted. Here, kola-nut testa (100g) of particle size 100μm was dissolved in a mixture of 400 ml formaldehyde (39% v/v)and 1600 ml of 0.2M H_2SO_4. The mixture was homogenised and heated at 50^{0}C in a water bath for 120 minutes. Thereafter, the mixture was filtered and washed several times with deionised water until the pH of the filtrate became neutral. The formaldehyde modified kola-nut testa extract (FMKT) was oven-dried at 50^{0}C for 24 hours.

Characterization of FMKT and UKT

The modified and unmodified kola-nut testa was characterized by the determination of their solubility, melting point, specific gravity, point of zero charge and functional groups.Solubility of FMKT and UKT in water, ethanol, acetone and n-hexane were determined by dissolving 1.000 gram each of the adsorbents (FMKT and UKT) in 100 ml of each of the aforementioned solvents. The mixture was gently swirled for 20 minutesat room temperature (RT) and observed for any dissolution.

The melting point of FMKT and UKT was determined by capillary tube experiment, while the specific gravity of the products was determined with the aid of a pycnometer. The pH at point of zero charge of the adsorbents was determinedaccording to previouslyreported method (Chukwu*et. al.*,2017 and Uchechukwu 2017)using a calibrated pH meter (Hanna pH meter model H12211). The functional groups present in FMKT and UKT were determined using Alpha FTIR spectrophotometer (model 12028429). All weight measurements were carried out using the Radwag Analytical Balance (model AS220/C/2).

Preparation of Metal Ion Solutions

The metal ion concentration (100ppm) was prepared from each of the metal saltsby weighing appropriate masses (0.3174g of $CuSO_4.5H_2O$ and 0.6448g $(NH_4)SO_4.FeO_4.6H_2O$)into separate 1000ml volumetric flasks. Small quantity of deionised water was used to dissolve the metal salt which was gently swirled until the salt crystals dissolved. Deionised water was added to the solution in the flask to the scribed line. Each of the metal ion solution in the volumetric flask was labelled 100 ppm M^{2+} (M = Fe or Cu).

Adsorption Studies

Adsorption studies of FMKT and UKT by the heavy metal ions Cu^{2+} and Fe^{2+} from aqueous solutions was studied under different equilibrium conditions (temperature, pH, contact time, initial metal ion concentration and adsorbent dose.

A 10 ppm of Fe^{2+} and Cu^{2+}solution was prepared by dilution of the stock solution. The pH of each metal stock solution was adjusted to 6.0 with either 0.1 M HCl or 0.03 M NaOH. Thereafter, 0.201 gram each of FMKT and UKT was then added to each of the metal ion solution and the mixture agitated for two hours at room temperature. The mixture was filtered afterwards and the residue dried in the oven at 80^{0}Cwhile the final metal concentration in the filtrate was determined using an atomic absorption spectrophotometry (Bulk Scientific Model 210/211VGP). The amount of heavy metals adsorbed by FMKT and UKT was evaluated form the expression:

$$Qe = \left[\frac{Co - Ce}{m}\right] x\, V \;\; \dots\dots\dots\dots 1$$

Where *Qe* is the amount of metal ion removed from aqueous solution by the adsorbents (FMKT and UKT) at equilibrium in mg/g; *Co* and *Ce* are the initial and final equilibrium metal ion concentration of each metal ion solution respectively in ppm units, *m* is the mass of the adsorbents (FMKT and UKT) in grams and *V* is the volume of the solution in litres.

Effect of temperature on adsorption was carried out by adding a known concentration of the metal ions (5 ml of Fe^{2+} and Cu^{2+} stock solution was diluted to 50 ml) to 0.201 gram of FMKT and UKT at different temperatures (40.0^0C to 80.0^0C). The samples were agitated for two hours and transferred to a thermostat water bath pre-set at the respective temperature for 10 minutes. At each set temperature, the appropriate sample container was removed from the water bath and filtered,the residue oven-dried at 80.0^0C while the metal ion concentration in the filtrate was determined usingAAS. The amount of heavy metals removed from aqueous solution by the adsorbents (FMKT and UKT) was calculated from the equation:

$$Removal\ \% = \left[\frac{(Co - Ci)}{Co}\right] x\, 100 \;\dots\dots\dots 2$$

Where Removal (%) is the amount of heavy metals removed by the adsorbents (FMKT and UKT) at various temperatures in percentage, *Co* and *Ci* are the initial and final concentration of the metal ion solution.

The effect of pH on Adsorption was carried out between pH 2.0 – 10.0. After varying the pH of each metal ion (10ppm of Fe^{2+} and Cu^{2+})solution, 0.201 gram of the adsorbents (FMKT and UKT) was added to each of the metal ion solution. The mixture was equilibrated for 12 hours and filtered afterwards. The final metal concentration at each pH was determined from the calibration curve of the metal.

The contact time was varied from 20-60 minutes at room temperature and initial metal ion concentration of 10 ppm. A 50 ml of 10 ppm Fe^{2+} and Cu^{2+} solution was prepared for each of the metal ion solution. Similarly, 0.201gram of the adsorbents (FMKT and UKT) was added to each of the metal ion solution at different contact time.

The metal ion concentration was varied from 10-50 ppm. This was achieved by drawing 5 ml, 10 ml, 15 ml, 20 ml and 25 ml from each metal ion stock solution (Fe^{2+} and Cu^{2+}) and made up to 50 ml with deionised water. A 0.201 gram of the adsorbent (FMKT and UKT) was added to each of the metal ion solution at the various concentration ranges. The mixture was agitated and allowed to equilibrate for one hour. After this time, the mixture was filtered, the residue dried and the final metal ion concentration determined from the calibration curve of the metal.

The effect of adsorbent dosage on the adsorption of the metal ion (Fe^{2+} and Cu^{2+}) unto the adsorbents (FMKT and UKT) was carried out as follows: 10 ppm of each metal ion solution (Fe^{2+} and Cu^{2+}) was prepared by appropriate dilution with deionised water. Various quantities of the adsorbents (FMKT and UKT) were added to each of the metal ion solution. The quantities of the adsorbents (FMKT and UKT) added ranges from 0.200-1.000 grams (i.e. 0.200, 0.400, 0.600, 0.800 and 1.000gram). The mixture was shaken for 30 minutes and filtered. The final metal ion concentration of the filtrate was determined in Buck Scientific Atomic Absorption Spectrophotometer from the calibration curve of the metal of interest.

III. Results And Discussion

Results for the adsorption of Cu^{2+} and Fe^{2+} from a single metal ion solution using unmodified kola-nut testa (UKT) and formaldehyde modified kola-nut testa (FMKT) showed that both UKT and FMKT are sparingly soluble in water, ethanol and acetone but insoluble in hexane and benzene. This indicates that they contain substances that arepolar and are able to associate with polar solvents through hydrogen bonding. The incomplete solubility of the adsorbents (UKT and FMKT) in polar solvents indicates that they contain substances of different molecular weight and the high molecular weight substances were probably responsible for their partial solubility in water, ethanol and acetone. The obvious high value of specific gravity for FMKT (1.264) when compared to that of the unmodified testa (0.742) can be attributed to the presence of formaldehyde as a modifying agent.

Other characterization studies such as pH at point of zero charge, melting point and FTIR were found to be in conformity with the report by Nwafor(2017). Figure 1 presents the pH at point of zero charge for the adsorbents. FMKT occurred at pH 4.0 and 13.8; while UKT occurred at pH 8.5 and 13.8. The indication of this finding is that at these pH values, the surface charge distributions on the adsorbents were generally neutral. The point at which the line crosses the initial pH (pHi) axis at ΔpH = 0 was chosen as the point of zero charge for the adsorbents as reported by Cardenas-Peña *et.al.*,(2012).

The adsorption capacity of the adsorbents (UKT and FMKT) for each of the metal ion in aqueous solution was carried out at room temperature and pH 6.0 to get 2.24 mg/g for Cu^{2+} and 1.97 mg/g $Fe^{2+}$for FMKT while UKT was 2.05 mg/g for Cu^{2+} and 2.12 mg/g for Fe^{2+}.

Furthermore, adsorption studies for the two metal ions (Cu^{2+} and Fe^{2+}) were carried out at various conditions ranging from changes in pH, temperature, initial metal ion concentrations, contact time and adsorbent dose.Figure 2 presents the percentage removal of Cu^{2+} and Fe^{2+} from aqueous solution by UKT and FMKT at different temperatures. The results revealed that the metal ion Fe^{2+}had a higher percentage removal of 85%-95% by both formaldehyde modified (FMKT) and unmodified (UKT) adsorbentwhen compared to $Cu^{2+}$with average percentage removal of 50% to 75%. The high percentage removal of Fe^{2+} by the adsorbentcan be attributed to its relatively small ionic radius (0.61Å) when compared to that of Cu^{2+} (0.73Å) (Lee 1996). Optimum percentage removalof 94% was recorded for Fe^{2+} throughout the temperature range of study (40^0C to 80^0C) using formaldehyde modified adsorbent (FMKT) while Cu^{2+} recorded 79% at 50^0C for the same adsorbent FMKT. Additionally; the formaldehyde modified adsorbent had a higher percentage removal than the unmodified adsorbent similar results had been reported by Adediran*et. al.,*(2007) in their work on adsorption of Pb, Cd, Zn, Cu and Hg ions on Formaldehyde and Pyridine Modified Bean Husks..

From figure 3, pH greatly influenced the percentage removal of Fe^{2+} and Cu^{2+}metal ions from the aqueous solution. As much as 96% of Cu^{2+} was removed from the aqueous solution at pH 8.0 by FMKT while 95% of Fe^{2+} was removed from the aqueous solution at pH 4.0 by FMKT. Increase in pH resulted to the gradual decrease in percentage removal of Fe^{2+} metal ion and an increase in the percentage removal of Cu^{2+} metal ion. Similar observation was made for the unmodified adsorbent UKT, where 81.6% of Cu^{2+} and 87.9% of Fe^{2+} were removed from the aqueoussolution at pH 4.0 and 6.0 respectively. These finding indicated that the metal ions Fe^{2+} and Cu^{2+}were favourably removed from aqueous solutions within the low pH range of 4.0 and 6.0.

The amount of Fe^{2+} and Cu^{2+} removed from the aqueous solution by the adsorbents at various contact time of 20mins to 100mins isshown in figure4. As usual, percentage removal of the metal ions (Fe^{2+} and Cu^{2+}) wasrelatively higher in the presence of the adsorbent FMKT when compared to the unmodified adsorbent UKT.Generally, percentage removal of the metal ions was quantitative all through the contact time of study in both adsorbents.

The effect of initial metal ion concentration on the percentage removal of the metal ions ($Fe^{2+}$and Cu^{2+}) was very distinct as presented in figure 5.0. Both the unmodified (UKT) and formaldehyde modified adsorbents (FMKT) optimally removed 95.97% and 96.98% of Fe^{2+}respectively at 20mg/L to 50mg/L metal ion from the aqueous solution. Conversely, percentage removal of Cu^{2+} metal ion was 57.80% at 20mg/L for the adsorbent FMKT and 43.43% at 40mg/L for UKT. This further consolidates the fact that adsorption of Fe^{2+} onto the adsorbents UKT and FMKT is more favourable than the metal ion Cu^{2+}. Again, the formaldehyde modified adsorbent (FMKT) gave higher and relatively optimum percentage removal when compared to the unmodified adsorbent (UKT). This same trend has been recurrent in the study so far.

Figure 6 presents the effect of adsorbents dosage on the percentage removal of the metal ions Fe^{2+} and Cu^{2+}. Only 0.200 gram of UKT and FMKT was needed to remove over 90% of Fe^{2+} from aqueous solution as shown in the plots. Increase in adsorbent dosage of FMKT gave a corresponding slight decrease in the amount of the metal ion Fe^{2+} removed from the aqueous solution. Adsorption of Cu^{2+} metal ion followed a different trend. In the presence of the unmodified adsorbent (UKT) only 0.200grams was required to achieve over 90% metal ion removal and thereafter decreased with a corresponding increase in adsorbent dosage. However, in the presence of the formaldehyde modified adsorbent (FMKT), increase in adsorbent dose gave a corresponding increase in percentage metal ion removal from 0.200grams to 0.800grams before decreasing drastically when 1.000g was introduced. Optimal percentage removal of 92% was obtained using 0.800gram adsorbent (FMKT) dose. This trend was completely opposite when the unmodified adsorbent was used.

IV. Conclusion

Kola-nut testa as agro waste has been successfully modified using 0.2M sulphuric acid and 39.9% (v/v) formaldehyde at 50^0C. The formaldehyde modified kola-nut testa (FMKT) and unmodified kola-nut testa (UKT) were equally characterized and further used for adsorption studies in the removal of Cu^{2+} and Fe^{2+} ions in aqueous solution.Adsorption capacity of the formaldehyde modified kola-nut testa (FMKT) for Cu^{2+} and Fe^{2+} ions in aqueous solution at equilibrium conditions and room temperature were: 2.24 mg/g for Cu^{2+}, 1.97 mg/g for Fe^{2+}. The unmodified kola-nut testa (UKT) gave adsorption capacities of 2.05 mg/g for Cu^{2+} and 2.12 mg/g for Fe^{2+} metal ions. Percentage removal of the metal ions from a single metal ion aqueous solution showed that Fe^{2+} metal ion gave better results with optimal average percentage removal above 90%, using the formaldehyde modified kola-nut testa when compared to Cu^{2+} metal ion with an average of 70% percentage removal. This could be attributed to the relatively small ionic radii of Fe^{2+} when compared to the ionic radii of Cu^{2+}. Similarly, the unmodified kola-nut testa followed a similar trend. Thus Formaldehyde modified kola-nut testa and unmodified kola-nut testa has proven to be good and alternative source for the remediation of industrial effluents containing these heavy metals.

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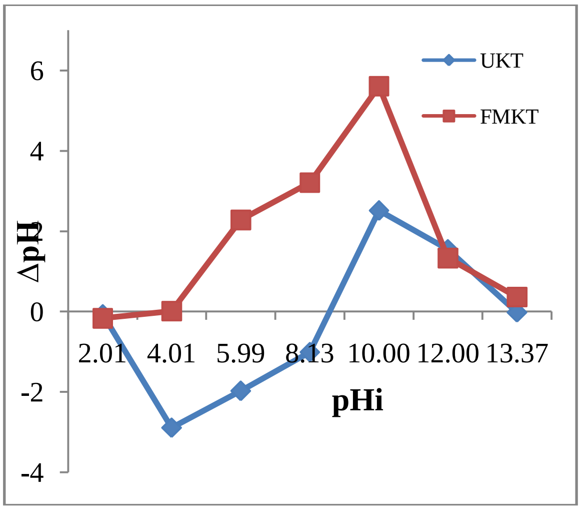

Figure 1: pH at Point of Zero Charge for UKT and FMKT

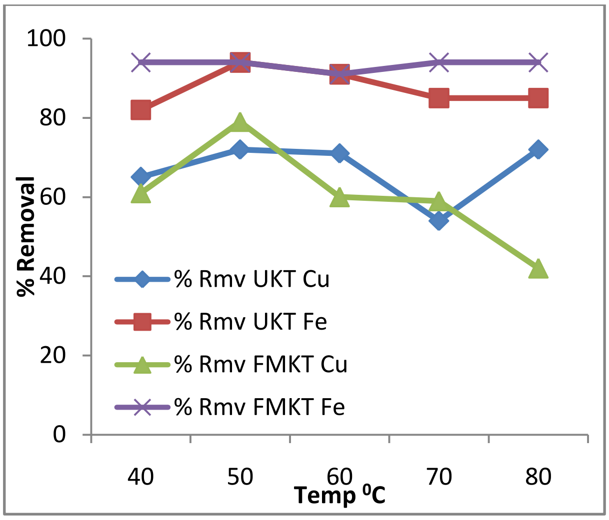

Figure 2: Effect of Temperature on the Adsorption of Heavy Metals by UKT and FMKT.

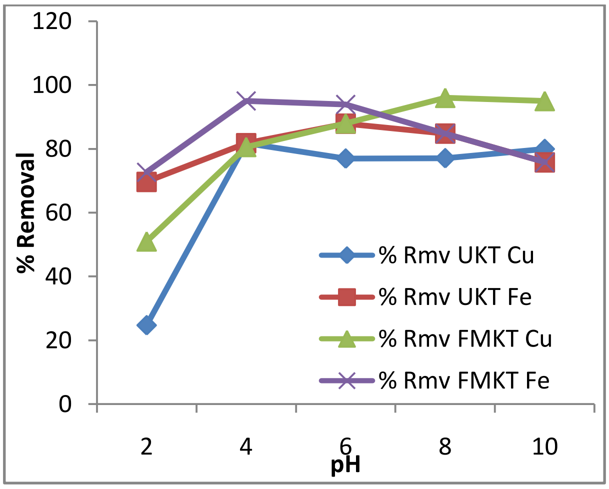

Figure 3: Effect of pH on adsorption of heavy metals by UKT and FMKT.

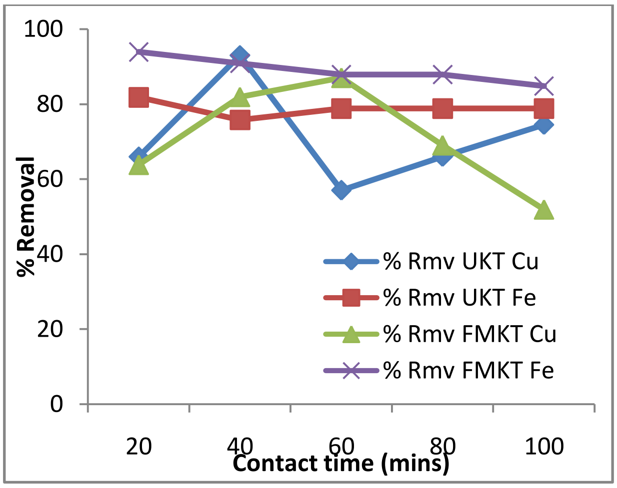

Figure 4: Effect of contact time on the adsorption of heavy metals by FMKT and UKT.

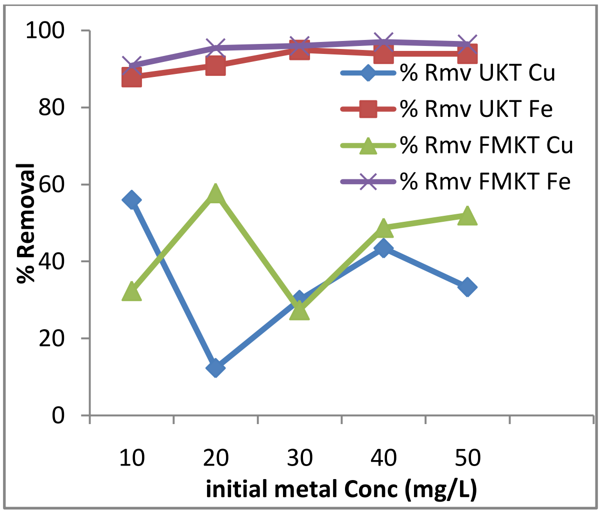

Figure 5: Effect of initial metal ion concentration on the adsorption of heavy metals by FMKT and UKT.

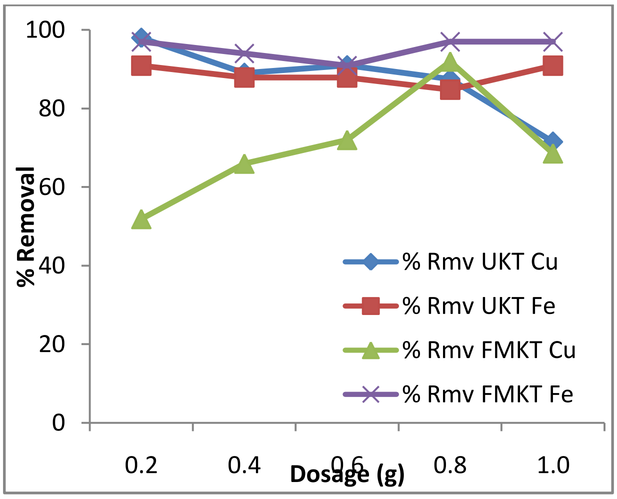

Figure 6: Effect of FMKT and UKT dosage on the adsorption of heavy metals.

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