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Equilibrium Isotherm Studies on the Adsorption of Malachite Green and Lead Ion from Aqueous Solution Using Locally Activated *Ugwaka* Clay (Black Clay)

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Authors' contributions

This work was carried out in collaboration between all authors. Author ACO designed the study, performed the statistical analysis, wrote the protocol, and wrote the first draft of the manuscript. Authors OIM and ACA managed the analyses of the study and the literature searches. All authors read and approved the final manuscript.

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ABSTRACT

The efficiency of Malachite green dye and Lead ion removal from aqueous solution and industrial waste effluents by adsorption using activated clay made from Ugwaka clay (Black Clay) have been investigated. Pilot-scale activation of the clay was performed at a temperature of 100ºC for 1.5 hours with 3.0 M Phosphoric acid. The clay to acid ratio was 1:10 (w.w%). The results of the adsorption studies showed that activated clay produced from Ugwaka Clay is a good adsorbent. The adsorption data were analyzed using the Langmuir, Freundlich and Temkin model at different temperatures. The Freundlich isotherm model fitted best among the temperature variables, thereby

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signifying a monolayer adsorption. Statistical analysis conducted at 99% confidence level ($P \leq 0.01$) showed that the optimum conditions for the adsorption of the malachite green were 50ºC, 65 minutes, 0.60 g of adsorbent and pH 6.8, 45ºC, 35 minutes, 0.80 g of adsorbent and pH 2.35 for lead ion adsorption. The adsorption resulted in 90.57% and 89.7% removal of Malachite green dye and Lead ion respectively. The results of the effects of process parameters showed that they positively influenced the adsorption process.

Keywords: Adsorption; activated clay; lead ion; malachite green.

1. INTRODUCTION

Environmental pollution increases proportionally with an increase in industrial technology [1,2]. Industries discharge different types of heavy metal wastes and dyes into the environment at an unprecedented and increasing rate. These heavy metal wastes and dyes may be discharged into the streams, rivers and lakes. The continuous enrichment of this water with these metal wastes and dyes beyond the healthy levels may cause poisoning, leading to various sicknesses. Consequently, the remediation of polluted industrial wastewater before they are released into the environment remains a topic of global concern [3,4]. Moreover, the contamination of groundwater and management of water is today a major concern in the management of water resources [5].

Contamination of the environment from a variety of sources has become an increasingly serious problem in recent years. Heavy metal pollution is an environmental problem of worldwide concern. The heavy metals such as lead, copper, cadmium, zinc and nickel are among the most common pollutants found in industrial effluents. Even at low concentrations, these metals can be toxic to organisms, including humans. According to the World Health Organization (WHO), the most toxic metals are aluminium, chromium, magnesium, iron, cobalt, nickel, copper, zinc, cadmium, mercury and lead [6,7].

The removal of heavy metals from waters and waste-waters is important in terms of protecting public health and environment owing to their accumulation in living tissues throughout the food chain as non-biodegradable pollutants. Heavy metal pollution is found to occur in various types of industrial wastewater such as those produced by metal plating facilities, mining operations, metallurgical engineering, battery manufacturing processes, the production of paints and pigments, electroplating, nuclear power plants, and ceramic and glass industries [8,9]. There are various methods of removing heavy metals including chemical precipitation, membrane filtration, ion exchange, liquid extraction or electrodialysis. However, these methods are not widely used due to their high cost and low feasibility for small-scale industries. In contrast, the adsorption technique is one of the preferred methods for removal of heavy metals because of its efficiency and low cost [10]. Adsorption is also recognized as an economic method to remove metal ions from aqueous solution [11]. The used economical and effective methods of removing heavy metals from wastewater have resulted in the search for unconventional materials that may be useful in reducing the levels of accumulation of heavy metals in the environment [12].

Metals exist in wastewater in many forms, including soluble, insoluble, inorganic, metal organic, and reduced, oxidized, free metal, precipitated, adsorbed and complexes. The presence of these destroy the natural sources especially groundwater and also gives health problems to the human being such as allergy, skin diseases, respiratory system disorders, heart disease and many others [13]. Unlike organic pollutants, the majority of which are susceptible to biological degradation, heavy metals will not degrade into harmless end products. Therefore, elimination of heavy metals from water and wastewater is important to protect public health [14]. Effluents from the production of batteries, gasoline additives, pigments, alloys and sheets among others often contain high concentrations of lead ions [15].

Dyes and pigments are widely used by several industries like plastics, textile, cosmetic, pharmaceutical, paper etc. to color their final products [16,17]. The textile dyeing process is an important source of contamination responsible for the continuous pollution of the environment. The volume of wastewater containing processed textile dyes is on steady increase. Over Six hundred million tones and approximately 12,000 different types of dyes and pigments are produced worldwide annually [18]. A

considerable percentage of these dyes go into the effluent during the dyeing process as they are highly soluble in water. It is estimated that 10–15% of the dye is lost in the effluents during the dying process [19]. These coloured compounds are not only aesthetically displeasing but also inhibiting sunlight penetration into the stream and affecting aquatic ecosystem. Dyes usually have complex aromatic molecular structures which make them more stable and difficult to biodegrade. Furthermore, many dyes are toxic to some microorganisms and may cause direct destruction or inhibition of their catalytic capabilities.

Majority of these dyes are recalcitrant and usually take a very long time for biodegradation. Moreover, it is reported that the intermediates formed during biodegradation of the dyes are more toxic than the original molecules [20]. These industrial discharges are highly colored with high Biochemical Oxygen Demand (BOD) as well as Chemical Oxygen Demand (COD) [21]. The dyes make penetration of sunlight to reach the lower layers impossible thus affecting the possibility for aquatic plants to perform photosynthesis. Many of these dyes are classified as toxic or even carcinogenic [22]. The main sources of wastewater generated by the textile industry originate from the washing and bleaching of natural fibers and from the dying and finishing steps. Given the great variety of fibers, dyes and process aids, these processes generate wastewater of great chemical complexity and diversity, which are not adequately treated in conventional wastewater treatment Plant.

There are various conventional methods of removing dyes including coagulation and flocculation, oxidation or ozonation and membrane separation. However, these methods are not widely used due to their high cost and economic disadvantage. Chemical and electrochemical oxidations, coagulation are generally not feasible on large scale industries. In contrast, an adsorption technique is by far the most versatile and widely used. The most common adsorbent materials are: alumina-silica, metal hydroxides and activated carbon. As proved by many researchers, removal of dyes and heavy metals by activated carbon is economically favorable and technically easier. Activated carbon is widely used as an adsorbent due to its high adsorption capacity, high surface area, microporous structure, and high degree of surface respectively. This work was aimed at studying the efficiency of removal of Malachite green dye and lead (ii) ion from aqueous solution using locally activated clay**.**

2. MATERIALS AND METHOD

2.1 Raw Materials and their Sourcing

The main raw materials used in this work include: Ugwaka Clay (Black clay), Malachite green dye and aqueous solution containing lead ion. Others are distilled water, Phosphoric acid, sodium hydroxide etc. The natural clay sample was obtained from the construction site were water inspection carried out 45 ft beneath the ground.

2.2 Clay Activation

Raw Ugwaka (Black clay) clay material was prepared for activation by first drying it under the sun to make them amenable to grinding. The clay sample was then reduced to small particle size by grinding with a laboratory mortar and pestle and sieved to a particle size of 300 µm. 100g of the clay sample was mixed with 500 ml of the prepared H_3PO_4 acid. The resulting suspension was heated on a magnetically stirred hot plate at a temperature of 100ºC for 1.50 hours and was allowed to cool after which the clay and acid were separated by pouring the resulting slurry in a funnel with a filter paper to aid the separation. The clay residue was washed free of the acid several times with distilled water until a neutral point is obtained with pH meter. The clay was then dried at a temperature of 115ºC for 2.5 hours, after which the clay was ground again, sieved to 75 µm particle size and stored in airtight container.

2.3 Waste Water Effluent for Heavy Metal Removal

The wastewater effluent was collected from Oriental Textile Industry Aba in Abia State Eastern Provence of Nigeria. It was then analyzed to establish the presence of any heavy metal and the presence of Lead ion was confirmed by using Atomic absorption spectrophotometer model 2010.VGP manufacturer USA. Then adsorption was carried out using the activated clay.

2.4 Batch Adsorption Studies

The activated clay was characterized by its adsorption capacity of Malachite green dyes and

Lead ion. The adsorption experiments were performed by taking 100 ml stock solution of the wastewater effluent and dye (10 mg/l) and treated with 0.2 g of the adsorbent. After the adsorption, the solution was centrifuged and the absorbance measured at their wavelengths. The parameters of interest studied were particle size, pH, adsorbent dose, solution temperature, initial adsorbate concentration and contact time. The experiment was conducted at ambient temperature of 25ºC. The samples were equilibrated for about 2 hours and then withdrawn, filtered using the Wattman filter paper and then analyzed. Each of the parameters was singled out and varied while the others were kept constant and their results were taken. The amount of dye and ion adsorbed was calculated based on mass balance equation as given by the equation;

$$q_e = \frac{(C_o - C_e)\,V}{W}$$

Where;

Q_e = The equilibrium adsorption capacity per gram dry weight of the adsorbent (mg/g).
C_o = Initial concentration of dye/lead ion in the solution (mg/l).
C_e = Final or equilibrium concentration of dye/lead ion in the solution (mg/l).
V = Volume of the solution (L)
W = The dry weight of adsorbent (g)

The percentage removal (%) was calculated for each equilibrium state as:

$$\text{Percentage removal (\%)} = \frac{C_o - C_e}{C_o} \times \frac{10\ 0}{1}$$

3. RESULTS AND DISCUSSION

The result showed that the surface area increased with the acid activation as shown in Table 1. The unactivated clay has the lowest surface area of 46m^2/g while activated ugwaka clay gave a surface area of 199 m^2/g. Similar results on the surface area of unactivated clay (39.7 m^2/g) and activated clay (140 m^2/g) were obtained by Musa et al., (2007). It is seen that the pH of natural clay is slightly alkaline (pH of 7.2) almost tending to neutral pH. After activation, the pH of the clay solution became acidic (of about 3.8) mainly because the acid that was used in the acid activation penetrated into the clay molecules during the activation. Acid activation increased the oil retention capabilities of the clay from 11.34% to 31.15%.

From the result of Table 2, it was observed that the major elements in clay are alumina, iron and sodium while trace elements such as potassium, manganese, calcium, nickel were also observed. The biggest element in terms of composition was aluminium.

3.1 Effect of Dosage

Effect of dosage was studied on the range of adsorbent dosage as shown in Fig. 1. and Fig. 2. for the adsorption of malachite green dye and lead ion respectively. The results showed that the percentage of adsorption increased with increase in adsorbent dosage. The increase in the percentage removal of dyes and lead ion with the increase in adsorbent dosage could be as a result of increased surface area which facilitated the availability of more adsorption sites. A similar report was given by [23,24].

3.2 Effect of pH

Effects of pH on the adsorption of malachite green and lead ion were studied over a range of values as shown in Fig. 3 and Fig. 4 respectively. The result indicated that maximum removal of lead and malachite green dye occurred at pH 10 respectively. The adsorption was observed to have increased and decreased over a range of pH. This could be due to the degree of protonation of the surface of the activated clay which resulted in increase in diffusion and subsequently increases in adsorption due to electrostatic attraction as reported by [25]. Also, the number of negatively charged adsorbent sites decreased as the pH decreased. This

Table 1. Physical properties of the clay (Ugwaka clay)

Property	Raw Ugwuaka clay	Clay activated with H_3PO_4
Surface area (m^2/g)	46	187
pH	7.2	3.8
Moisture (%)	14.1	15.62
Oil retention (%)	11.34	31.15

causes the number of positively charged surface sites to increase which did not favor the adsorption of positively charged dye cations due to electrostatic repulsion, lower adsorption of Malachite green at acidic pH is due to the presence of excess H^+ ions competing with dye cations for the adsorption sites of the adsorbent. This agrees with the report by [26,27].

Table 2. Atomic Absorbance Spectrophotometer (AAS) analysis of the raw clay

Elements	Concentration (mg/l)
Mn	0.4234
Cu	0.0176
Na	12.321
K	0.5478
Al	26.456
Ca	0.1678
Ni	0.1435
Mg	0.0192
Fe	19.443
Zn	0.0167

3.3 Effect of initial Ion Concentration

Effect of initial ion concentrations was studied for the initial ion concentration ranging from 20 to 110 mg/l as shown in Fig. 5 and Fig. 6. The results showed that the amount of malachite green dye and lead ion adsorbed increased as their initial concentrations increased but the percentage of adsorption decreased with increase in time. This could be attributed to the fact that, at lower concentration, the ratio of the initial number of the molecules to the available surface area is low. A similar report was given by [28,29].

Table 3. Characteristics of wastewater effluents

Property	Raw sample	Activated sample
pH	3.08	
Temperature	28 C	
Conductivity	18.65	
Total solids	2.34	
Total dissolved solids	0.02 mg/l	
Dissolved oxygen	12.34 mg/l	
BOD	560.00 mg/l	
COD	1034.47 mg/l	
Lead	28.96 mg/l	2.97 mg/l
Fe	1342 mg/l	
Cu	0.072 mg/l	
Cr	1.72 mg/l	

3.4 Effect of Temperature

Effect of temperature was studied at a range of 303K and 333K as shown in Fig. 7 and Fig. 8. The results showed that the percentage removal of malachite green dye and lead ion increased with increase in temperature. This buttressed the fact that, as the temperature increased, the rate of diffusion of adsorbate molecules across the external boundary layer and internal pores of the adsorbent particles increased as reported by [30].

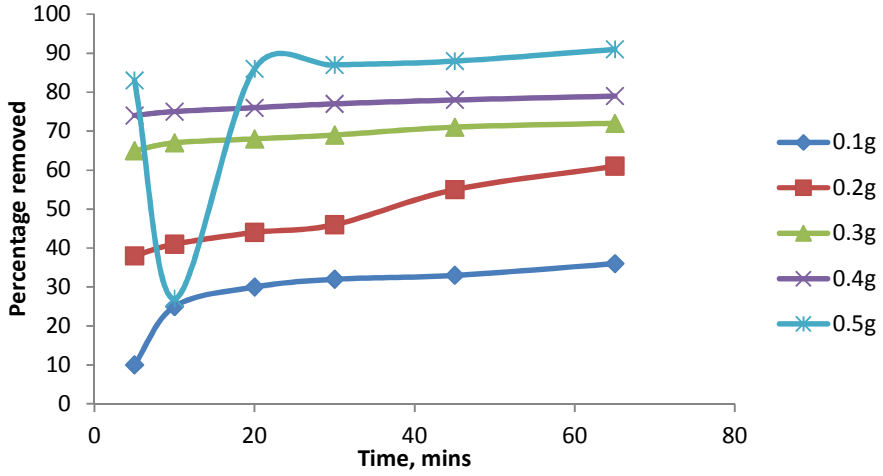

Fig. 1. Effect of adsorbent dosage on the adsorption of Malachite green dye

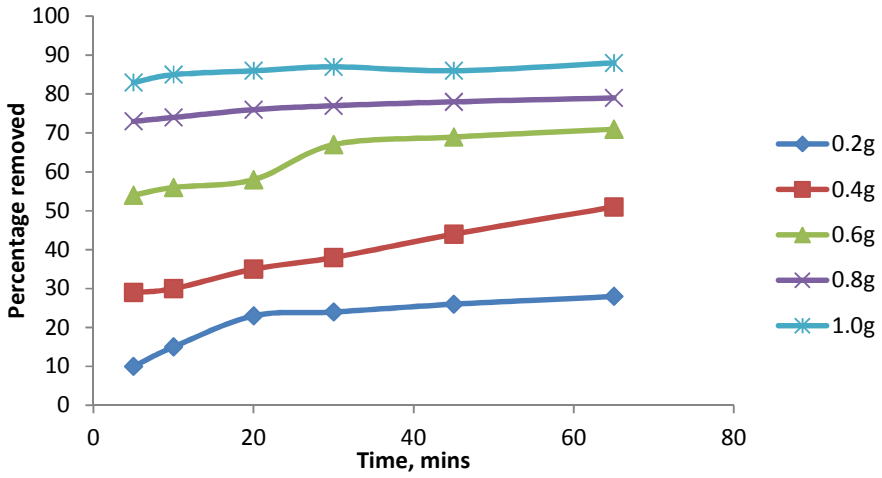

Fig. 2. Effect of adsorbent dosage on the adsorption of lead ion

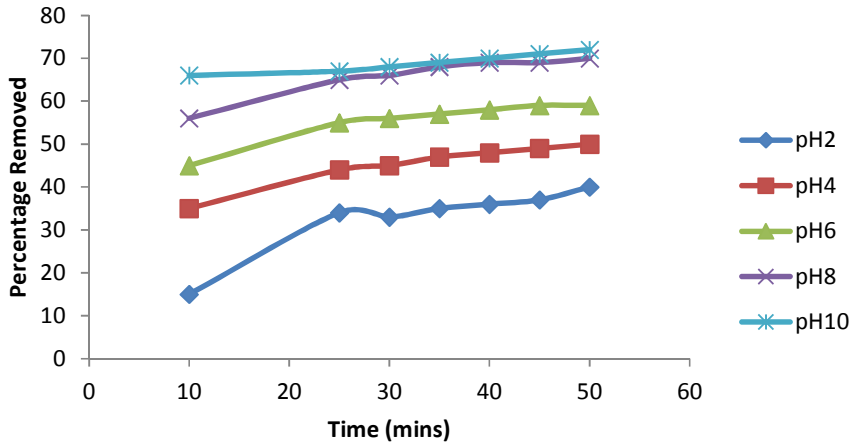

Fig. 3. Effect of pH on the adsorption of malachite green dye.

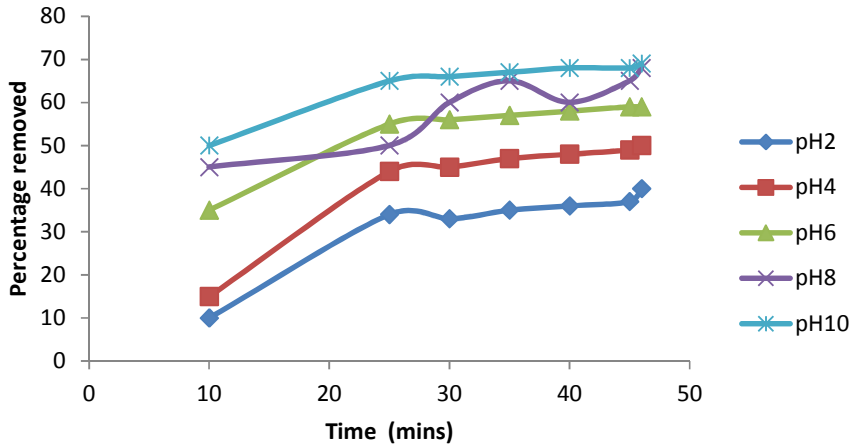

Fig. 4. Effect of pH on the adsorption of lead ion.

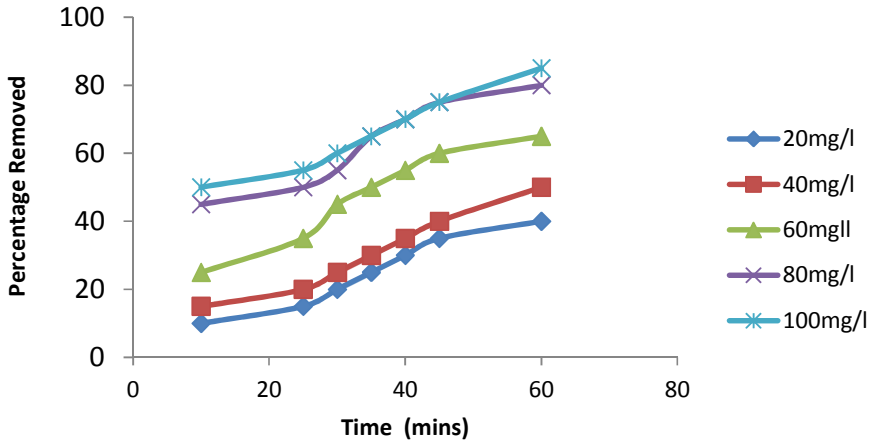

Fig 5. Effect of Initial ion concentration on the adsorption of malachite green dye

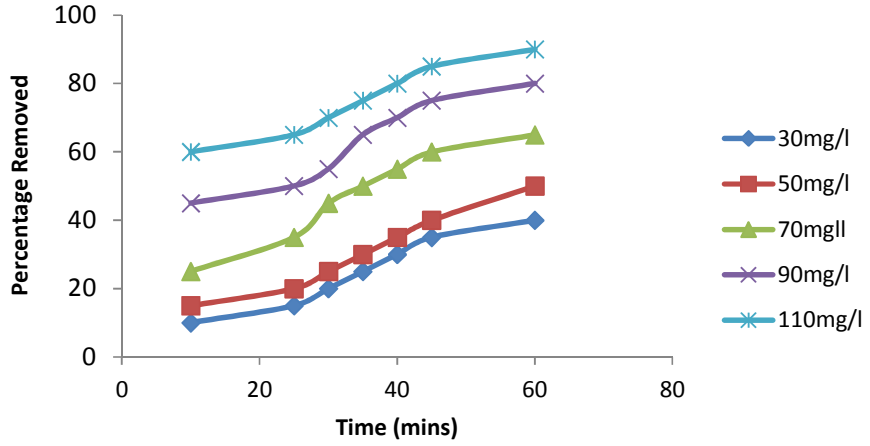

Fig. 6. Effect of initial ion concentration on the adsorption of lead ion

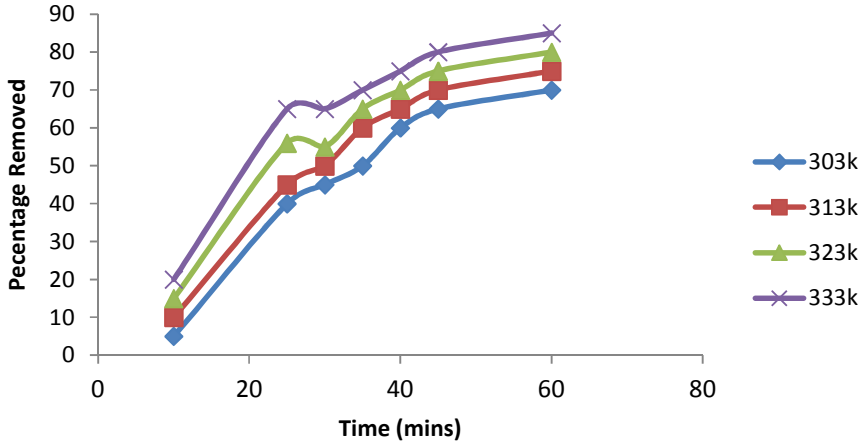

Fig. 7. Effect of temperature on the adsorption of malachite green dye

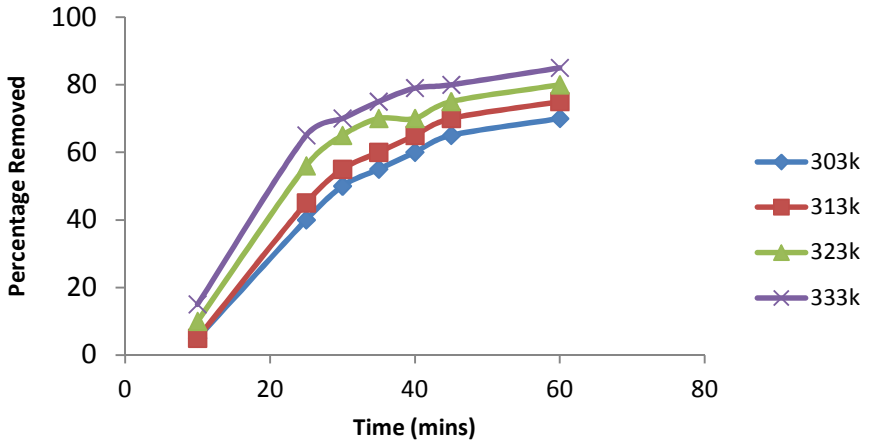

Fig. 8. Effect of temperature on the adsorption of lead ion

3.5 Isotherm Studies

3.5.1 The langmuir isotherm

The Langmuir isotherm model is valid for monolayer adsorption onto a surface containing a finite number of identical sites. The linear form of Langmuir isotherm is represented by the following equation [31].

$$\frac{C_e}{q_e} = \frac{1}{Q_o b} + \frac{C_e}{Q_o}$$

Where;

C_e = the equilibrium concentration of the adsorbate (malachite green dye and heavy metal Pb^{2+}) (mg/l).
q_e = The adsorption capacity at equilibrium (mg/g).

The constant Q_o signifies the adsorption capacity (mg/g) when monolayer is complete and **b** is related to the affinity of the binding sites. When C_e / q_e was plotted against C_e, a straight line graph with slope of $1/Q_o$ was obtained. The Langmuir constants **b** and $\boldsymbol{Q_o}$ were calculated using the slope and intercept (constant) from the graph.

An essential dimensionless constant of the Langmuir isotherm used in identifying the feasibility and favorability of the adsorption process called "Separation factor" **(RL)** was adopted. The separation factor (RL) was calculated in each case using the following equation:

$$RL = \frac{1}{1 + bC_o}$$

Where Co is the initial adsorbate concentration (mg L^{-1})
b = the Langmuir constant.

Equally, the regression coefficient $\boldsymbol{R^2}$ was used to further analyze the suitability and conformity of the data on how they fit into the isotherms. The closeness of the $\boldsymbol{R^2}$ value to 1.0 determines its level of fit.

3.5.2 Freundlich isotherm

This isotherm is an empirical equation employed to describe heterogeneous adsorption system. Freundlich isotherm is also applied to plot the equilibrium data of the adsorption.

The linear form of Freundlich equation can be expressed as;

$$\ln q_e = \ln k_f + (1/n) \ln C_e$$

Where;

q_e= the amount of adsorbate adsorbed per unit mass of adsorbent (mg/g)
C_e = the equilibrium concentration of the adsorbate (mg/l)
k_f and **n** are Freundlich isotherm constants; where k_f indicates the adsorption capacity of the adsorbent. This can be defined as the adsorption or distribution coefficient, and it represents the quantity of the adsorbate adsorbed onto the activated carbon for a unit equilibrium concentration.

The Freundlich isotherm is determined by plotting the graph of In q_e against In C_e. Ink_f and n are determined by the Freundlich constant and slope respectively. The slope 1/n ranging between 0 and 1 is a measure of the adsorption intensity or surface heterogeneity.

3.5.3 The Temkin isotherm model

The Temkin isotherm equation suggests a linear decrease of sorption energy as the degree of completion of the sorptional center of an adsorbent is increased. This model takes into account the presence of indirect adsorbate/adsorbent interaction and suggests that because of these interactions, the heat of adsorption of all molecules in the layer would decrease linearly with coverage.

The Temkin isotherm has been used in the form

$$q_e = \left(\frac{RT}{bt}\right) L_n K_T C_e$$

Where $BT = \left(\frac{RT}{bt}\right)$

R = Universal gas constant (8.314 J/mol.K)
T = Absolute temperature (K) (25 C + 273K)

To linearise the Temkin isotherm model, a graph of q_e versus Inc_e gives a straight line which enables the determination of the isotherm constants K_T and B_T. K_T is the equilibrium binding constant (L/mg) corresponding to the maximum binding energy and the value increased with increase in temperature for both the adsorbents, suggesting the corresponding increase of maximum binding energy and

Table 4. Calculated isotherm parameters for the adsorption of Malachite green dye on activated clay

Isotherm	Temperature ℃		
model	**30**	**40**	**50**
Langmuir			
Q (mg/g)	156.39	168.54	177.28
b (L/mg))	0.0613	0.699	0.0987
R_L	0.0384	0.0312	0.0176
R^2	0.9123	0.9342	0.9267
Freundlich			
n	3.823	3.850	3.923
K_f (L/g)	25.75	25.84	25.86
R^2	0.9821	0.9817	0.9911
Temkin			
b (J/mg)	138.62	139.01	138.69
K (L/g)	4.996	5.034	5.001
R^2	0.7041	0.7115	0.7233

Table 5. Calculated Isotherm parameters for the adsorption of Lead ion on activated clay

Isotherm	Temperature ℃		
model	**30**	**40**	**50**
Langmuir			
Q (mg/g)	136.12	162.12	171.32
b (L/mg))	0.00712	0.00767	0.00772
R_L	0.234	0.219	0.286
R^2	0.9092	0.9003	0.8976
Freundlich			
n	1.428	1.456	0.8241
K_f (L/g)	1.891	1.967	2.108
R^2	0.9878	0.9864	0.9931
Temkin			
b (J/mg)	121.102	108.981	107.901
K (L/g)	0.1278	0.1244	0.1235
R^2	0.9123	0.9212	0.9154

constant B_T is related to the heat of adsorption. The increase in temperature increases the heat of adsorption from 30°C to 50°C. The correlation coefficient, R^2 from this work confirms that the adsorption of malachite green does not follow Temkin isotherm while the adsorption of lead ion does in this work as shown in Table 4 and 5. Similar report was given [32,33]

4. CONCLUSION

This study showed that activated clay prepared from *Ugwaka clay* is a promising adsorbent for the removal of malachite green dye and heavy metal from aqueous solution over a wide range of concentrations. The amount of Malachite green and lead ion adsorbed was found to depend on pH, adsorbent dosage, temperature, initial ion concentration and contact time.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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