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Comparative Analysis on the Efficiencies of Unmodified Clay and Microorganism-Modified Clay as Adsorbent for the Adsorption of Lead (II) Ions from Wastewater

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Abstract

Comparative study of the efficiencies of clay adsorbents (unmodified and microorganism- modified clay) in the adsorption of lead (II) ion from wastewater was studied as a function of some operating conditions, such as initial concentration, pH, agitation time, and temperature. The maximum adsorptive capacity of unmodified clay and the clay modified with microorganisms were found to be 30.40 mg/g and 42.22 mg/g respectively. Thermodynamics parameters evaluation gave a negative value of ΔH^o of -4967.4 J/mol and -4655 J/mol for UMC and MMC respectively, indicating the exothermic nature of the process. Also, the negative value of ΔS^o of -20.87 J/mol and -14.77 J/mol for UMC and MMC respectively reflect the increasingly orderliness of the adsorption system as the metal ions were been attached to the adsorbent surface. The adsorption of lead (II) ions onto unmodified and microorganism modified clay was favoured by Freundlich isotherm model. The heterogeneity factor (n) for UMC and MMC were less than one ($n < 1$) which indicates the process of adsorption was chemical and non-linear. Furthermore, Freundlich constant (K_F) related to adsorption capacity for UMC and MMC was 2.01 mg/g and 2.16 mg/g respectively, confirming that the adsorption capacity of microorganism-modified clay is greater than that of the unmodified clay.

Keywords: *Clay, lead (II) ion, adsorption, microorganism, isotherms.*

1.0 Introduction

There is a rise in clean water demand both for agricultural and domestic use as a result of rapid spread of industrial development and population growth. Correspondingly, the continuous discharge of toxic and poisonous substances into the body of water has worsened. Two-third of the world population was predicted to suffer from the effects of continuous discharge of toxic waste into water bodies [10]. The major source of these contaminants is usually industrial effluent containing pesticides, herbicides, fertilizers, antibiotics, dyes, phenols, proteins, and detergents. This will result to the depletion of the level of oxygen available for aquatic lives [13]. The focus of recent research work is on the treatment and degradation of these pollutants using technologies such as floatation-coagulation, electro-coagulation, membrane and biological treatment [15]. However, some of these techniques are associated with high capital and cost and scale-up challenges despite their high efficiencies. Consequently, adsorption and bioremediation techniques are now being utilized greatly due to their low cost, simplicity, efficiency and environmental friendliness.

The investigation of the adsorptive performance of clay has been done extensively. The unique characteristics of clay and clay minerals such as their excellent swelling ability, ion exchange capacity, surface area and charge enable them to adsorb both organic and inorganic contaminants. The high affinity of clay in the removal of cationic inorganic and organic contaminants like lead and dyes is due to their ability to exchange their cat-ions with other cat-ions [12].

Due to the widespread availability of clay soil and the need for the development of technologies for pollution control in recent times, it is not surprising that clay are now being used to control of toxic materials even in small quantities. It can be shown that microorganisms such as parasites, bacterial and viruses are removed from polluted water by the addition of clay. Clay possesses some unique properties that made it an excellent adsorbent for some applications such as control of pesticides, liners for waste disposal and for nuclear waste management and control. This study will bring to light, an improved bioremediation method for effective remediation of surface water contaminated with toxic metals by employing the combined adsorptive properties of clay and microbes.

2.0 Methodology

2.1 Materials and Equipment

The primary materials utilized in this research include: Silver trioxonitrate (v) solution ($AgNO_3$), Bromocresol green, de-ionized water, distilled water, Hydrochloric acid (HCl), Lead (II) Nitrate, Sodium hydroxide, trioxonitrate (v) acid (HNO_3), phenol disulphonic acid, Staphylococcus cohnii (GC subgroup A). Furthermore,

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the following equipment were utilized in this research, and these are: Atomic Absorption Spectrophotometer (Varian AA240), Burette, Centrifuge (KUBOTA6500), conical flasks, digestion tube (50 Cl), electronic weighing balance (Shimadzu UX4200H), Erlenmeyer flask, New York, NY, USA), Heating mantle (EW-04805), muffle furnace, Nessler's tube, oven (Gallenkamp OHA-010), pH meter (Hanna HI 2211), pyrometer bottle, and refrigerator,

2.2 Sampling and Sample Preparation

2.2.1 Preparation of Unmodified (natural) Clay Sample

100 g of clay soil (montmorillonite) was suspended in 0.5 litre of de-ionized water and left without disturbance for 24 hrs. This was followed by careful decantation and then centrifugation at 4000 rpm for duration of 20 minutes to obtain pure clay. The resulting clay was dried at 80^{o}C temperature, and then grounded. The clay sample was stored in an airtight sample bottle. 0.5 litre.

2.2.2 Preparation of Clay Modified with Microorganisms

Natural clay sample (montmorillonite clay) was washed several time using distilled water to make it neutral, which was then sieved using a 200-mesh screen. The solid clay fraction was further washed in distilled water followed by decantation. The washed clay was then dried at about 105^{o}C, grounded and sieved using a 200 mesh screen. 100 g of the sample of clay was combined with 10 ml, 10^{8} CFU /ml of staphylococcus cohnii and shaken for about 1 hour, and stored.

2.2.3 Preparation of Metal ion Standard Solution

1 g of lead (II) nitrate was dissolved in 1 litre of distilled water to obtain 1000 mg/l of lead (II) ion. NaOH was used to adjust the pH of the solution.

2.3 Adsorption Experiment

Four different batches of experiment were conducted each for the two clay samples to study the effect of pH (2-12), concentration (5 mg/l to 50 mg/l), time intervals (30 -180 mins.) and temperature (20 - 100^{0}C) on the efficiency of unmodified clay and microorganism modified clay for the adsorption of lead (II) ion from contaminated water using. Batch experiments were conducted using 250ml volumetric flask. Adjustment of pH of the aqueous solution was done using sodium hydroxide. At equilibrium point, the aqueous solution was filtered using Whatman No 42 filter paper while the analysis of the free metal concentrations was determined using AAS.

The amounts of the adsorbed metal ions were calculated using Equation (1)

$$qe = \frac{(C_o - C_e)V}{m} \quad (1)$$

q_e is the quantity of adsorbate ion adsorbed in mg/g of the adsorbent. C_o is the initial concentration of the heavy metal ion while C_e is the equilibrium concentration of the metal ion after adsorption, m is the mass of the adsorbent, and V is the volume of the solution in cm^{3}.

The percentage of metal ion removal by the adsorbent is calculated using Equation (2).

$$\% \text{ Removed} = \frac{Co - Ce}{Co} \times 100 \quad (2)$$

2.4 Adsorption Equilibrium Studies

Equilibrium experiment for the adsorption of Pb^{2+} was conducted at different concentrations (5 – 50mg/l) at pH of 4.5 and temperature of 30^{o}C for 90 minutes using 50 g of the adsorbents. The following adsorption isotherms were utilized to describe the adsorption processes:

2.4.1 Langmuir Adsorption Isotherm

Langmuir adsorption isotherm model was used to explain how adsorbate molecules adhere to the solid surface of the adsorbent. It is calculated using Equation (3),

$$\frac{C_e}{q_e} = \frac{1}{KQ^o} + \frac{C_e}{Q^o} \quad (3)$$

where, C_e is the equilibrium concentration, q_e is the adsorptive capacity of the adsorbent, K is the equilibrium constant, and Q^o is the amount adsorbed per unit mass of adsorbent at equilibrium (mg/g).

2.4.2 Freundlich Adsorption Isotherm

Freundlich isotherm was utilized to describe how adsorbates are adsorbed onto the adsorbent surface. The model is expressed in Equation (4),

$$\text{Log } q_e = \log K_f + \frac{1}{n}\log C_e \quad (4)$$

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where, q_e is the amount heavy metal ions adsorbed per unit mass of the adsorbent (mg/g), K_f is the adsorptive capacity constant, n is the Freundlich equilibrium constant, and C_e is the equilibrium concentration.

2.4.3 Temkin Isotherm Model

The linear form of the Temkin isotherm equation is expressed in Equation (5),

$$q_e = B_1 \ln K_T + B_1 \ln C_c \quad (5)$$

where K_T is the equilibrium binding constant (1/mg), while B_1 is the constant related to heat of adsorption.

2.4.4 Dubinin-Radushkevich (D-R) Isotherm

The linear form of D-R isotherm equation is expressed in Equation (6),

$$\ln q_e = \ln q_d - \beta\varepsilon^2 \quad (6)$$

where q_d is the D-R constant (mg/g), β is the free energy (mol^2/KJ^2), and ε the Polanyi potential.

2.5 Thermodynamic Study

Using the results obtained adsorption experiments, the equilibrium constant (K_c) is determined using the Equation (7),

$$K_c = \frac{C_a}{C_e} \quad (7)$$

where,

$C_a = C_o - C_e$ = the concentration of the adsorbate adsorbed at equilibrium (mg/l)

ΔH^o *and* $\Delta S°$ are related, as expressed in Equation (8).

$$In K_c = \frac{-\Delta H°}{RT} + \frac{\Delta S°}{R} \quad (8)$$

Where, ΔH^o is the standard enthalpy change, ΔS^o is the standard enthalpy change, T is the temperature (K), and R is the universal gas constant (8.314 $Jmol^{-1}$ K^{-1}).

3.0 Results and Discussion

3.1 Effect of pH on the Adsorption on the Adsorption of Pb^{2+} onto UMC and MMC

C_0 = 44.70 mg/l ; V = 100 ml ; m = 50 g

Figure 1 showed that there was constant rise in the amount of lead ions adsorbed as pH is increased, reaching maximum point when the pH is at 6 as seen in Table 1. This is due to the rise in the number of negative charges on the adsorbents' surfaces which is likely to attract the positively charged lead (II) ions, to bind to its surface [3], hence the rise in the uptake. However, at very low pH, the adsorption of lead (II) cations will be hindered as a result of overall surface positive charge caused by protons hence restricting the access to the ligands by the ions due to repulsive forces [5]. The adsorptive capacity of the modified clay was improved by more than 30mg/g compared to that of unmodified clay.

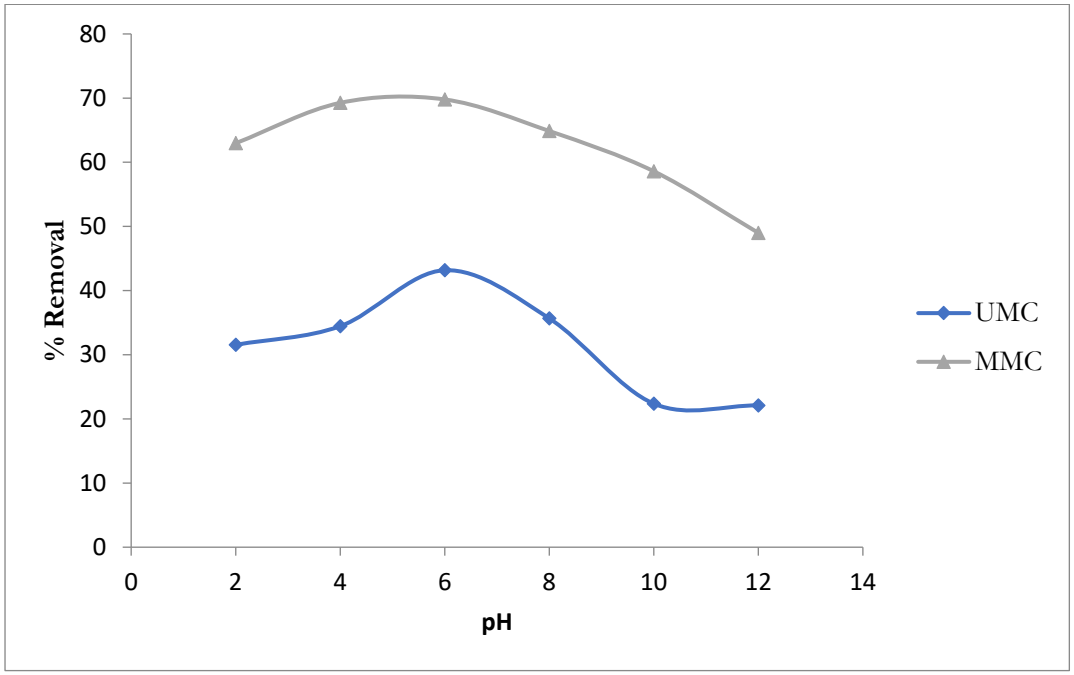

Figure 1: Effect of pH on the Adsorption of Pb^{2+} onto Umodified Clay (UMC), and Microorganism-modified Clay (MMC)

Table 1: Effect of pH on the removal of Pb^{2+} onto UMC and MMC

pH	q_t (mg/g)		% Removal	
	UMC	MMC	UMC	MMC
2	28.20	56.30	31.54	62.97
4	30.80	61.92	34.45	69.26
6	38.60	62.40	43.17	69.79
8	31.88	58.00	35.66	64.87
10	20.00	52.38	22.37	58.59
12	19.76	43.80	22.10	48.99

3.2 Effect of Initial ion Concentration on the Adsorption on the Adsorption of Pb^{2+} onto UMC and MMC

The effect of initial concentration on the amount adsorbed is indicated in Figure 2. The uptake of the metal ion increased as the concentration of increases, until equilibrium concentration of 30 mg/l is reached. The higher adsorption rate is as a result of availability of active sites at lower concentrations [8]. Hence, available sites become fewer at higher concentrations resulting in the reduction of adsorption rate [11].

Furthermore, the adsorptive capacities of the clay samples increased steadily as the concentration of the metal ion is increased. This is as a result of the rise in the driving force to break down all mass transfer resistance existing between the solid and aqueous phase as the concentration rises [9]. We can show from Table 3.2 that the percentage of metal ion adsorbed by microorganism-modified clay was higher than that of the unmodified sample by more than 10%.

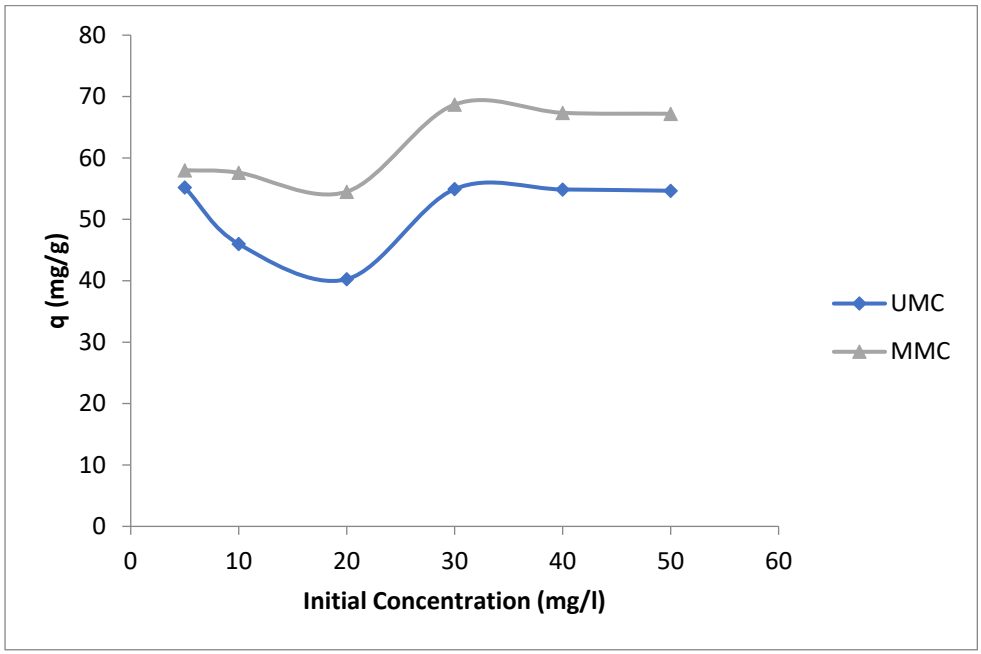

Figure 2 Effect of initial ion concentration on the removal of Pb^{2+} onto UMC and MMC

Table 2: Effect of Initial ion concentration on the Removal of Pb^{2+} onto UMC and MMC

Initial concentration (mg/l)	q_t (mg/g)		% Removal	
	UMC	MMC	UMC	MMC
5.00	5.52	5.80	55.20	58.00
10.00	9.20	11.52	46.00	57.60
20.00	16.10	21.80	40.25	54.50
30.00	32.96	41.22	54.93	68.70
40.00	43.90	53.88	54.87	67.35
50.00	54.66	67.20	54.66	67.20

3.3 Effect of Contact Time on the Adsorption on the Adsorption of Pb^{2+} onto UMC and MMC

The adsorption of Pb^{2+} from aqueous solution occurred rapidly for the first 30 minutes, and then slower until the equilibrium time of 150 minutes was reached. Microorganism-modified clay efficiently adsorbed more than 40% of the pollutant more than those of unmodified clay as shown in Table 3. It can be deduced from Figure 3.3 that the adsorption process progressed in two stages: the initial rapid phase where the adsorbents (modified and

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unmodified) removed over 50 % of the adsorbate and the final slower phase where the adsorption progressed gradually [9]

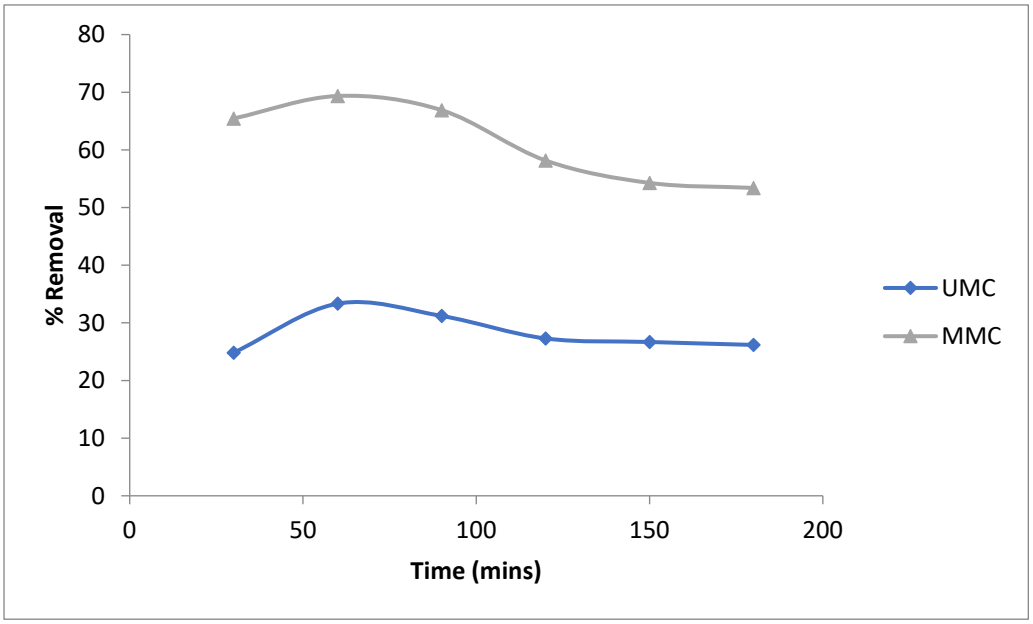

Figure 3: Effect of contact Time on the Adsorption of Pb^{2+} onto Umodified Clay (UMC), and Microorganism-modified Clay (MMC)

Table 3: Effect of Contact Time on the Adsorption of Pb^{2+} onto UMC and MMC

Time (Mins)	q_t (mg/g)		% Removal	
	UMC	MMC	UMC	MMC
30	22.20	58.52	24.83	65.45
60	29.80	62.00	33.33	69.35
90	27.90	59.80	31.20	66.89
120	24.40	52.00	27.29	58.16
150	23.86	48.52	26.68	54.27
180	23.40	47.74	26.17	53.40

3.4 Effect of Temperature on the Adsorption of Pb^{2+} onto UMC and MMC

The effect of temperature on the adsorption of Pb^{2+} onto modified and unmodified clay was investigated, and the results are seen in Figure 4 and Table 4. The amount Pb^{2+} onto clay samples increased in direct proportion with the temperature, with the maximum uptake of Pb^{2+} occurring at a temperature of 40^{o}C as a result the increase in the sites available for adsorption as the temperature rises [6]. Thermodynamic parameters such as enthalpy change and the standard entropy of the adsorption were obtained from the Van't Hoff plot of InK_c against 1/T. Van't Hoff thermodynamic parameters are shown in Table 5. The enthalpy changes for the clay samples (modified and unmodified) were found to be negative (see Table 6). Hence, we can deduce that the adsorption of lead (II) ions onto unmodified and microorganism-modified clay adsorbents is exothermic. Furthermore, the standard entropies of UMC and MMC were also negative, describing how the adsorption system is becoming more ordered as solute particles are attached to the surface of adsorbent [7].

Table 4: Effect Temperature on the Adsorption of Pb^{2+} onto Umodified Clay (UMC) and Microorganism-modified Clay (MMC)

Temperature (^{o}C)	C_e (mg/l)		q_e (mg/g)		% Removal	
	UMC	MMC	UMC	MMC	UMC	MMC
20	31.22	24.30	26.96	40.80	30.15	45.63
40	25.80	21.60	37.80	46.20	42.28	51.68
60	26.30	22.00	36.80	45.40	41.16	50.78
80	30.76	25.70	27.88	38.00	31.18	42.50
100	34.50	26.50	20.40	36.40	22.81	40.71
120	34.80	26.73	19.80	35.94	22.14	40.20

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Table 5: Van't Hoff Parameter for Adsorption of Pb^{2+} onto Unmodified Clay (UMC) and Microorganism-modified clay (MMC)

	UMC		MMC	
T(K)	$\frac{1}{T}$	InK_c	$\frac{1}{T}$	InK_c
293	0.003413	-0.8398	0.003413	-0.1749
313	0.003195	-0.3112	0.003195	0.4055
333	0.003003	-0.3572	0.003003	0.0313
353	0.002833	-0.7916	0.002833	-0.3021
373	0.002681	-0.2184	0.002681	-0.3757

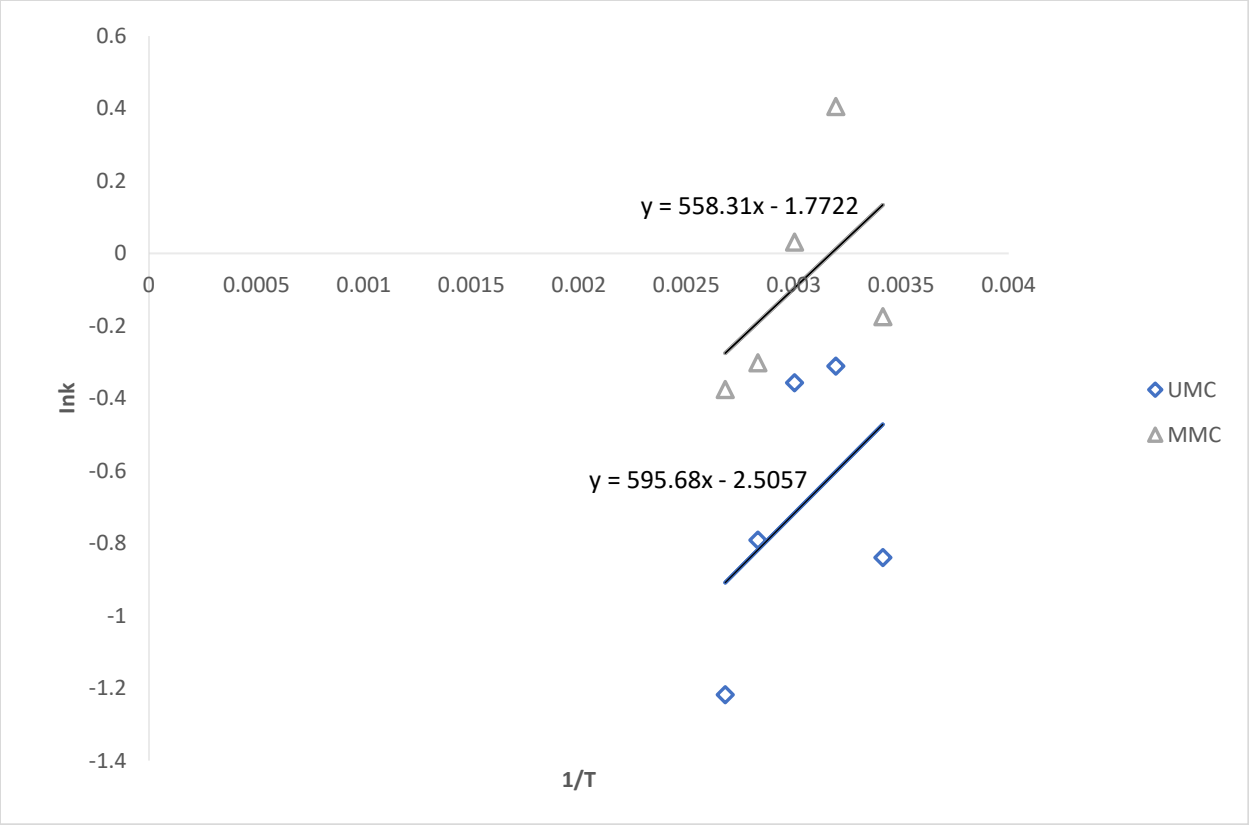

Figure 4: Van't Hoff Plot for Adsorption of Pb^{2+} onto Umodified Clay (UMC) and Microorganism-modified Clay (MMC)

Table 6: Thermodynamic Parameters for the Removal of Pb^{2+} onto unmodified clay (UMC), organically modified clay (OMC), and microorganism modified clay (MMC).

	Unmodified Clay			Microorganism-modified Clay		
T(K)	ΔG^o (J/mol)	ΔH^o (J/mol)	ΔS^o (J/mol)	ΔG^o (J/mol)	ΔH^o (J/mol)	ΔS^o (J/mol)
293	2.05			0.43		
313	0.81			-1.06		
333	0.99	-4967.4	-20.87	-0.087	-4655	-14.77
353	2.32			0.89		
373	3.78			1.17		

3.5 Adsorption Isotherms

The Langmuir, Freundlich, Temkin, and Dubinin-Radushkevich(D-R) isotherm models were employed to explain the removal of Pb^{2+} by clay samples. The summary is seen in Table 7 Adsorption isotherms are of fundamental importance when designing adsorption system, since they help to show how the metal ions are mobilized between the liquid phase and the adsorbent at equilibrium point. The value of linear correlation coefficient (R^2) obtained from Langmuir isotherm is indicated in Figure 5 for unmodified clay and microorganism-

modified clay are 0.1244 and 0.4275 respectively. This confirms that Langmuir isotherm is not well-fitting to the experimental data. Hence, Langmuir isotherm is not a good representation of this adsorption experiment [1].

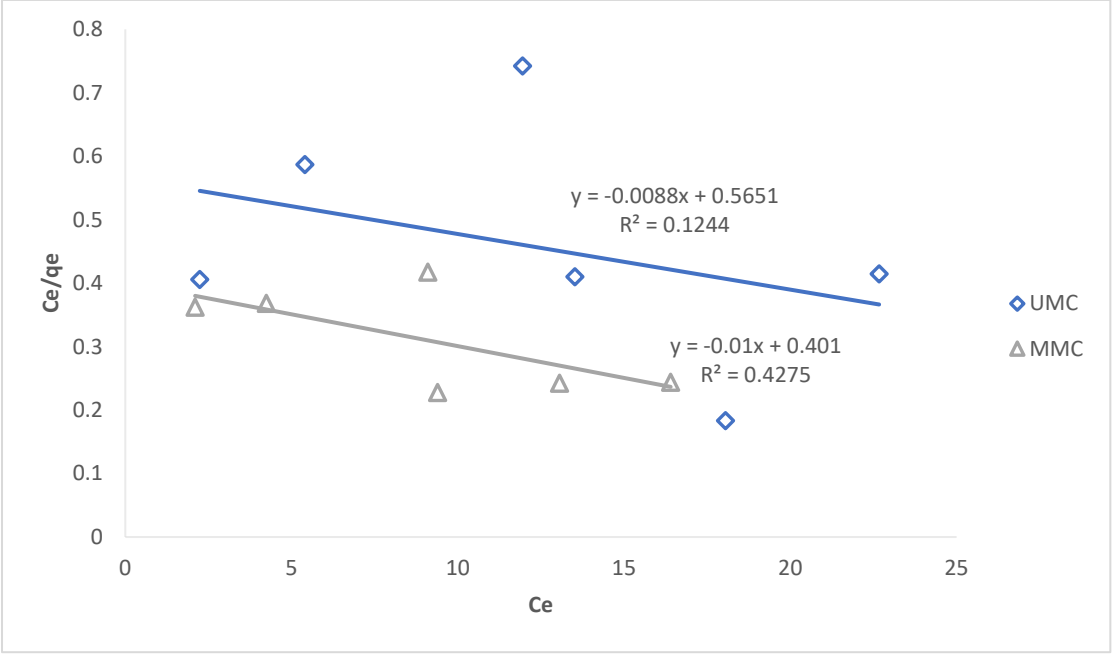

Figure 5: Langmuir Adsorption Isotherm for adsorption of Pb^{2+} onto Unmodified Clay (UMC) and Microorganism-modified Clay (MMC)

Furthermore, Freundlich equilibrium constant (n) was gotten from the plot of log q_e against log C_e in Figure 3.6. If n = 1, then the adsorption is linear, if n < 1, then the adsorption is a chemical process, and if n > 1, the adsorption is a physical process [1]. The values of n for unmodified clay and microorganism-modified clay are 0.99 and 0.82 respectively. This implies that the adsorption process is chemisorption. The R^2 for UMC and MMC using Freundlich isotherm are 0.9218and 0.9532 respectively. These show that Freundlich isotherm favours the adsorption experiment, and can describe this adsorption process. The Freundlich constant, K_f obtained for UMC and MMC are 2.01and 2.16dm^3/g respectively. Freundlich constant is an indicator of adsorbent capacity [2]. Hence, the adsorptive capacity of microorganism- modified clay is higher than that of unmodified (natural) clay.

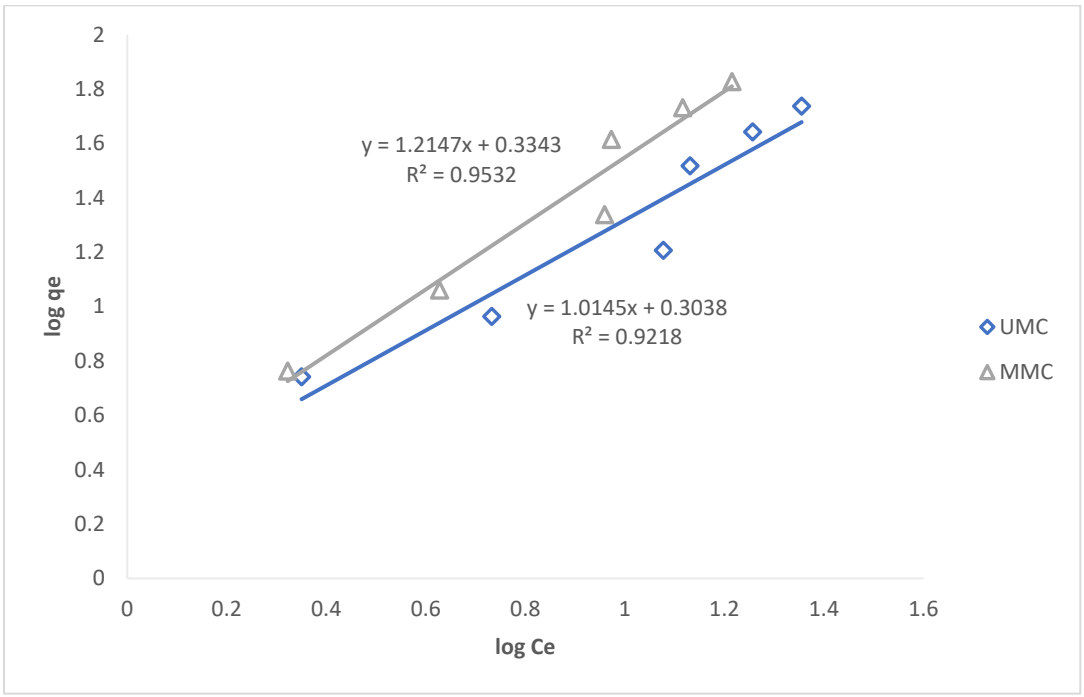

Figure 6: Freundlich Adsorption Isotherm for the Adsorption of Pb^{2+} onto Umodified Clay (UMC), and Microorganism-modified Clay (MMC)

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The plot of q_e against In C_e known as Temkin isotherm model (see Figure 7) was also applied to determine the isotherm constants K_T (equilibrium binding constant which corresponds to the binding energy). The Temkin isotherm constant for UMC and MMC are 0.40 and 0.43 lit/mg respectively. Higher K_T means a more favorable adsorption [4]. This further confirms the improved adsorption capacities of pre-treated clay sample.

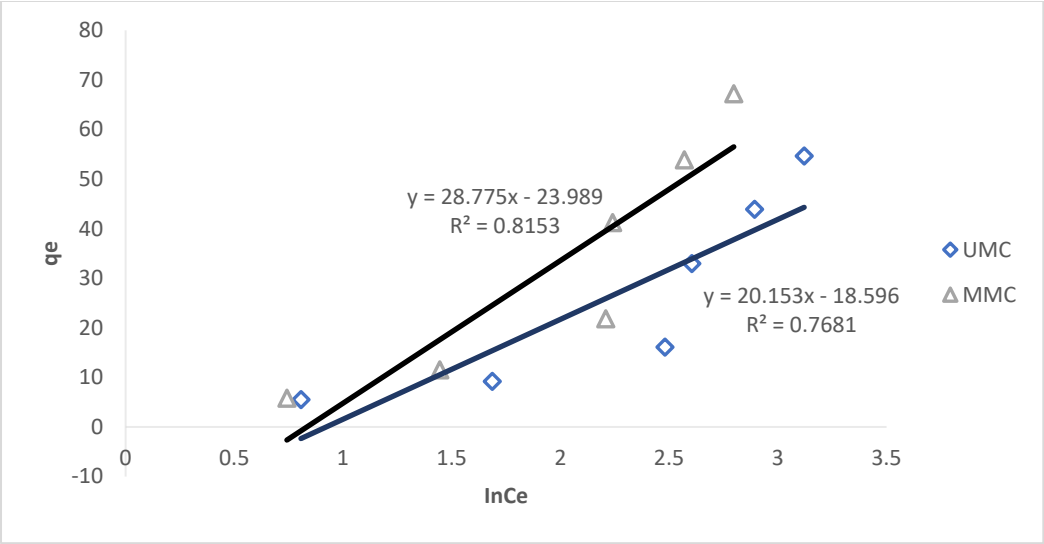

Figure 7: Temkin isotherm for the Adsorption of Pb^{2+} onto Umodified Clay (UMC and Microorganism-modified Clay (MMC)

From the plot of Inq_e against ε^2 in Dubini-Radushkevich isotherm model as seen in Figure 8, the energy of adsorption obtained for all the clay samples were all the same and are equal to 0.5kJ/mol.

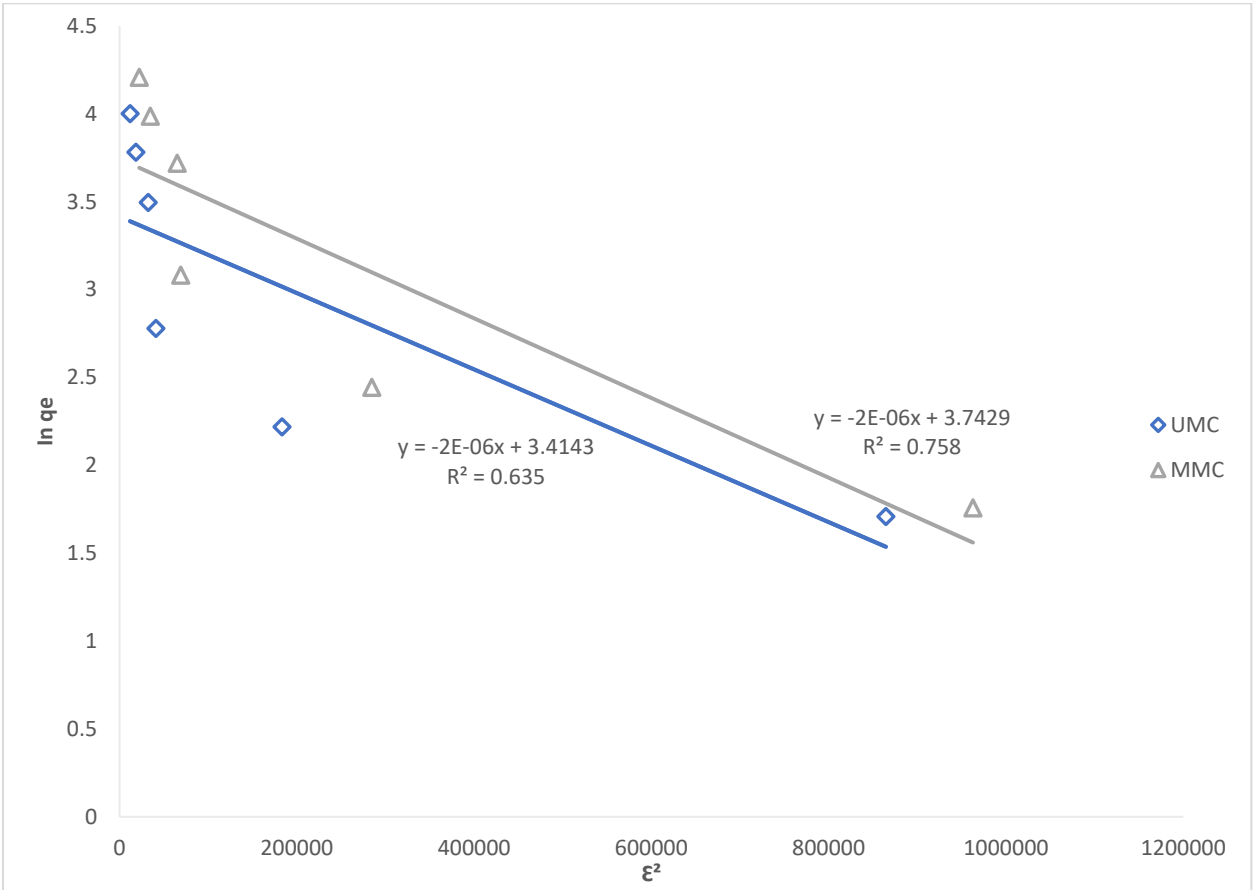

Figure 8: Dubinin-Radushkevich(D-R) adsorption isotherm for Umodified Clay (UMC) and Microorganism-modified Clay (MMC)

Table 7: Isotherm Constants for the Adsorption of Pb^{2+} onto UMC and MMC

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Isotherm Model	UMC	MMC
Freudlich	R^2 = 0.0.9218	R^2 = 0.9532
Temkin	n = 0.99 K_f (mg/g) = 2.01 R^2 = 0.7681 B_1 = 20.153 K_T (l/mg) = 0.40	n = 0.82 K_f (mg/g) = 2.16 R^2 = 0.8153 B_1 = 28.775 K_T (l/mg) = 0.43
D-R	R^2 = 0.635 q_d (mg/g) = 30.40 E (kJ/mol) = 0.50	R^2 = 0.758 q_d (mg/g) = 42.22 E (kJ/mol) = 0.50

4.0 Conclusion

The adsorptive capacity of microorganism-modified clay is greater than that of unmodified clay adsorbent. Adsorption of Pb^{2+} onto UMC and MMC were found to be dependent on agitation time, pH, temperature and initial concentration. The modified clay adsorbent adsorbed 30% more of the metal ions than the unmodified clay adsorbent. Freundlich isotherm model fits the adsorption of Pb^{2+} onto UMC and MMC, and can be utilized to explain the adsorption process. This was based the value of R^2 from the isotherm models. From the parameters obtained from the thermodynamic study, the adsorption of Pb^{2+} onto montmorillonite clay is feasible and exothermic process.

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