

# Kinetics and Equilibrium Thermodynamic Studies of the Adsorption of Phenolphthalein and Methyl Orange onto Muscovite Clay

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**Abstract** A batch adsorption method for the removal of methyl orange and phenolphthalein from aqueous media onto muscovite clay has been assessed and proven to be successful. The adsorption studies were performed at 303, 323 and 343 K. Factors such as temperature and pH were evaluated. Equilibrium adsorption for all the adsorbates was attained after 30 minutes. Investigation of the adsorption isotherm at 303 K using the Langmuir and Freundlich isotherm models showed that the adsorption of both indicators obey the Langmuir isotherm model with monolayer adsorptive capacities of 13.00 and 2.48 mg/g for methyl orange and phenolphthalein, respectively. The pseudo-second-order kinetic model best describes both adsorption processes with  $R^2 > 0.99$  and negative activation energies indicating physisorption processes. Assessment of the thermodynamic parameters showed that although the adsorption processes were endothermic ( $\Delta H$  values of +8.77 kJ/mol and +15.62 kJ/mol for methyl orange and phenolphthalein respectively) over the range of temperatures studied, the relatively high entropy changes (+38.05 kJ/(molK) and +52.52 kJ/(molK) for methyl orange and phenolphthalein respectively) gave an overall negative change in Gibbs free energy making the processes spontaneous. Generally, the adsorption of both dyes was found to increase steadily within the pH range of 3.3 to 7.0 but decreased drastically from pH = 8.0 to 10.0, a phenomenon which can be attributed to electrostatic repulsion between anionic sites on the dyes and negatively charged active sites on the surface of the adsorbent.

**Keywords:** Muscovite clay; adsorption; thermodynamics; kinetics; dyes.

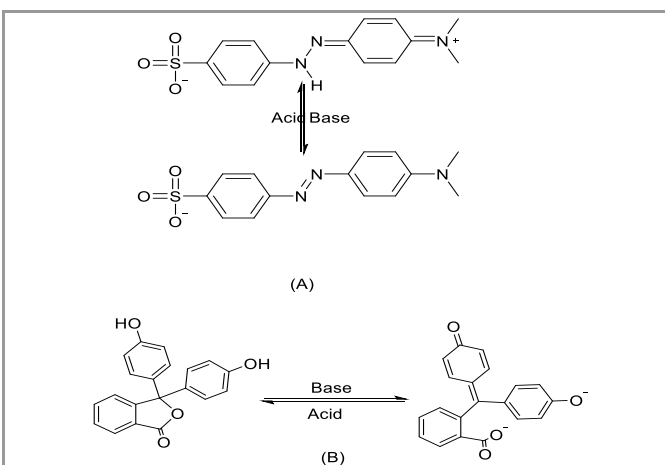
## 1. Introduction

Methyl orange (MO) and phenolphthalein (phph) are important dyes commonly used as endpoint indicators in acid-base titrations. They have the ability to give different colours in solutions of different pH and as such are used as visual indicators in titrimetry. The main problem with the use of these indicators is that they are usually discarded together with the solution after the titrimetric process. These effluents then enter the environment where they act as carcinogens, mutagens and teratogens on aquatic life and on humans [1]. Methyl orange also serves as a model azo dye for the study of the fate of a series of water soluble dyes used in textile, leather and paper industries [2] whereas phenolphthalein is a suitable representative for both phenols and phthalein dyes [3]. Globally, about 700,000 tons of these dyes are produced each year and about 15% of them end up in wastewaters which are discharged as effluents into the environment [4].

Adsorption has been found to be one of the most effective techniques successfully employed for colour removal from wastewater [5]. The use of clay minerals is advantageous because of its low cost and ready availability [6]. Kaolinite, a 1:1 aluminosilicate comprising layers of tetrahedral silica sheets linked to octahedral alumina sheets by shared oxygen atoms, has been successfully employed in the removal of dyes such as maxilon red GRL, yellow 4GL [6], methylene blue [7], reactive blue 221 [8] and rhodamine B [9]. Other clay materials include (a) montmorillonite for the adsorption of Congo Red [10], rhodamine B [11] and acid red G [12], (b) bentonite for the removal of diazo [13], Congo red and acid dyes [14] and (c) the removal of Coomassie blue [15], acid blue 25 [16] and methyl orange using muscovite clay [17]. Although all clay materials have the same fundamental structure; made up of tetrahedral  $\text{SiO}_4$  and octahedral  $\text{AlO}_6$  units, the differences in their adsorptive properties are largely due to the

differences in surface charge [18]. The hydrolysis of the Si-O-H and Al-O-H bonds along the surfaces or interstices of the clay structure produces these surface charges. These charges are usually positive or negative depending on the pH. Clay materials with excess negative charges have high anion exchange capacities and vice versa [19]. The high adsorptive properties of clay minerals can also be attributed to factors such as swelling ability, ion exchange ability and large surface complexation ability [20].

The structures of MO and phph are pH sensitive. As shown in figure 1A, methyl orange is present as the hydrazine form in an acidic medium with the reduction of the azo group. This dipolar ion has a positive charge on the  $-\text{N}(\text{CH}_3)_2^+$  group and a negative charge on the  $-\text{SO}_3^-$  with nucleophilic centers on the phenyl rings. In a basic medium however, the azo form is the predominant species with the negative charge on the  $-\text{SO}_3^-$  group.



**Figure 1.** Ionic species of (A) methyl orange and (B) phenolphthalein in acidic and basic media.

These differences in charge distribution on MO in different media can be regarded as interesting phenomena affecting adsorptive properties on different surfaces. According to Jiang et al [21], the maximum adsorption capacity for MO on a chitosan/ $\beta$ -cyclodextrin polymer is optimum from pH 4 to 9 but drops sharply above pH of 9 as a result of the saturation of the active sites by  $\text{OH}^-$  at high pH. Similarly, the relatively less polar anhydride form of phenolphthalein is dominant in an acidic medium with the highly polar carboxylate species being present in the basic medium. Recently, we reported the purification and characterization of Ghanaian muscovite clay as a cheap and readily available adsorbent for the removal of substituted phenols and methyl orange from aqueous solutions [17]. This material has a point of zero charge ( $\text{pH}_{\text{pzc}}$ ) of 5.6 which is slightly acidic and a high level of exchangeable cations such as  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Na}^+$  and  $\text{K}^+$ . Due to its ready availability and favorable surface characteristics, this work will investigate the feasibility of using muscovite clay to adsorb methyl orange and phenolphthalein (widely used acid-base

indicators) from aqueous media under different temperature and pH conditions. Currently, there is no data in the literature on the adsorption studies of these dyes using muscovite clay.

### Materials and methods

**Adsorption experiment:** A previously reported method [17] for the batch adsorption experiment was employed with slight modifications as follows; Exactly 50 mg/L standards of methyl orange and phenolphthalein were prepared and diluted with double distilled water. For the batch adsorption studies, 0.5 g of the clay was added to 50 mL of the adsorbate. The mixture was stirred with a magnetic stirrer at 300 rpm. Exactly 5 mL of the mixture was withdrawn after 30 minutes, filtered and the absorbance measured on a Shimadzu T70 UV-Vis spectrometer at predetermined wavelengths of maximum absorptions (methyl orange = 275 nm and phenolphthalein = 463 nm). The experiments were carried out in a temperature controlled water bath at 303, 323 and 343 K. The pH was also adjusted with 0.1 M HCl and 0.1 M NaOH.

### Results and discussion

The adsorption capacity of muscovite clay for MO and phph was estimated using the equation 1.

$$q_t = \frac{(C_o - C_t)V}{m} \quad 1$$

Where  $q_t$  is the amount of indicator adsorbed at any given time,  $C_o$  is the initial concentration of the dye,  $C_t$  is the concentration that remains after time  $t$ ,  $V$  is the volume of the solution and  $m$  is the mass (0.5 g) of the used clay. At equilibrium, the measured concentration is  $C_e$  and the amount of indicator adsorbed is  $q_e$ . As shown in figure 2A, 3.070 mg of phenolphthalein was adsorbed per gram of clay at 303 K from an initial concentration of 50 mg/L after 30 minutes of stirring. This corresponds to an adsorption efficiency of 38.4% after the first 30 minutes of the adsorption process. This dropped to 2.71 mg/g after the first hour until finally 2.74 mg/g was adsorbed after three hours. The equilibrium adsorbed amount was found to be 3.07 mg/g. Relatively, the adsorption capacity of the clay for phenolphthalein was higher at 323 K as compared to the amount adsorbed at 303 K suggesting an endothermic adsorption process [22].

The adsorbed amount of methyl orange, however, showed little variation with temperature. As shown in figure 2(B), the adsorption process equilibrated after 30 minutes with 7.66% of the initial 50 mg/L being adsorbed onto the surface of the clay at 303 K. The rate of the adsorption process was further studied by the Lagergren's [23]; first-order reaction model was given in equation 2.

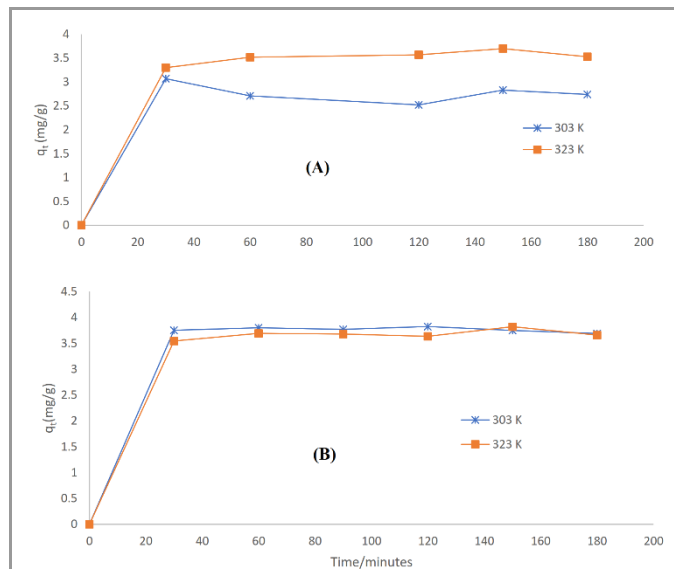
$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \quad 2$$

The rate constant,  $k_1$ , can be estimated from the slope of a straight line plot of  $\log(q_e - q_t)$  against  $t$ . The kinetics was also investigated using the pseudo-second order kinetic-model described by Ho and McKay [24,25] given as;

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad 3$$

Where  $k_2$ , the rate constant of the pseudo-second order adsorption model, can be estimated from the slope and intercept of the linear plot of  $t/q_t$  against  $t$ . From Table 1, the R-squared values for all the adsorption processes suggest a second-order kinetic model with values ranging from 0.994 to 0.999. This suggests that the adsorption process strongly depends on the availability of polar

(attachment) sites on the dyes as well as available sites on the surface of the clay. The  $k_2$  values show that the rate of adsorption of methyl orange is more than ten times faster than that of phenolphthalein. An estimation of the activation energy ( $E_a$ ) [25] shows that both processes occur by physisorption [26] with  $E_a$  values of  $-30.12 \text{ Jmol}^{-1}$  and  $-3.3 \text{ kJmol}^{-1}$  for methyl orange and phenolphthalein, respectively.



**Figure 2.** Amount of (A) phenolphthalein and (B) methyl orange adsorbed by 0.5 g of muscovite clay at 300 and 323 K respectively from an aqueous medium.

The negative value of the  $E_a$  shows that increasing the temperature leads to a reduced probability of the dyes being adsorbed onto the clay. However, the lower values recorded for MO indicate that its adsorption is relatively more favorable than that of phph. Similar results have been reported for the adsorption of basic dyes from aqueous solutions onto sepionite [25] and the adsorption of methylene blue onto water-hyacinth [27].

**Table 1.** First and second order kinetic parameters for the adsorption of methyl orange and phenolphthalein onto muscovite clay at 303 and 323 K.

Sample designation	Blank Temperature/K	Kinetics			
		First order $k_1/\text{min}^{-1}$	$R^2$	Second order kinetics $k_2/\text{g.mg}^{-1}.\text{min}^{-1}$	$R^2$
Methyl orange	303	0.009	0.304	13.514	0.9
	323	0.002	0.002	13.501	0.9
Phenolphthalein	303	0.011	0.678	0.368	0.9
	323	0.020	0.351	0.277	0.9

As shown in Figure 1, the azo form of MO is the predominant species in both basic and neutral media. The probable sites of attachment to the surface of the clay are the  $-\text{SO}_3^-$ , phenyl rings, azo and the  $(\text{N}(\text{CH}_3)_2)$  nucleophilic groups. Although these interactions generally occur by physisorption and require energy (endothermic), they can be spontaneous if there is a high entropy

change at the solution adsorbent interface [28]. As reported elsewhere [17], the positive  $\text{pH}_{\text{pzc}}$  and the exchangeable cations on the surface of the muscovite clay also make it suitable for the adsorption of methyl orange. Similarly the anhydride form of phph is dominant in acidic and neutral solutions. This relatively uncharged species has weaker attachment sites as compared to MO. Thus, MO shows a higher adsorption capacity relative to phph. According to Murray [29], one-fourth of the surface  $\text{Si}^{4+}$  in the tetrahedral phyllosilicate sheets of muscovites are replaced by  $\text{Al}^{3+}$  ions. The excess negative charge [30] is balanced by cations such as  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$  and  $\text{K}^{+}$  [18]. These cations then serve as possible attachment sites for polar dye molecules.

The mode of adsorption of the respective dyes onto the surface of the clay was assessed isothermally at 303 K using the Langmuir and Freundlich isotherm models. These models have been successfully employed to explain the adsorption properties of dyes [19,31,32], heavy metals [33,34] and various environmental pollutants [35] onto suitable solid phase adsorbents. According to the Langmuir isotherm model:

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{K_L q_m} \quad 4$$

where  $C_e$  is the equilibrium concentration of the dye (mg/L) and  $q_e$  is the amount of dye adsorbed (mg) per gram of muscovite,  $q_m$  is the monolayer adsorption maximum (mg/g) and  $K_L$  is the equilibrium constant (L/mg). A plot of  $C_e/q_e$  against  $C_e$  for different equilibrium dye concentrations can be used to determine  $K_L$  and  $q_m$ . From the linear form of the Freundlich isotherm model:

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e \quad 5$$

Where  $K_f$  and  $n$  are constants related to the adsorption capacity and the adsorption strength respectively for a multilayer adsorption model. From table 2, the correlation coefficients ( $R_2$  values) show that the adsorption of the dyes onto muscovite clay can best be described using the Langmuir isotherm model which means that at the working concentration (4 mg/L – 20 mg/L) using 0.5 g of muscovite clay at a temperature of 303 K, the surface of the clay particles is covered by a monolayer of phenolphthalein and methyl orange. It can be observed that the amount of monolayer adsorption capacity ( $q_m$ ) for the removal of methyl orange from aqueous media was found to be 13 mg/g which is higher than that of phenolphthalein (2.48 mg/g). This shows that the  $-\text{SO}_3^-$  group in methyl orange interacts strongly with the adsorption sites on the clay surface than that of the carbonyl and hydroxyl groups in phenolphthalein. The favorability of the adsorption process can be estimated from the  $K_L$  value. According to Hameed [36],  $K_L > 1$  indicates that the process is unfavorable;  $K_L = 1$ , linear;  $0 < K_L < 1$ , favorable and  $K_L = 0$ , irreversible. As shown in table 2, the  $K_L$  values for both adsorption processes were greater than 1 confirming the relatively small adsorption capacity of both adsorbates at 303 K.

The effect of temperature on the adsorption process was further investigated at 323 and 343 K using equation 6,

$$\ln K = -\frac{\Delta H}{RT} + \frac{\Delta S}{R} \quad 6$$

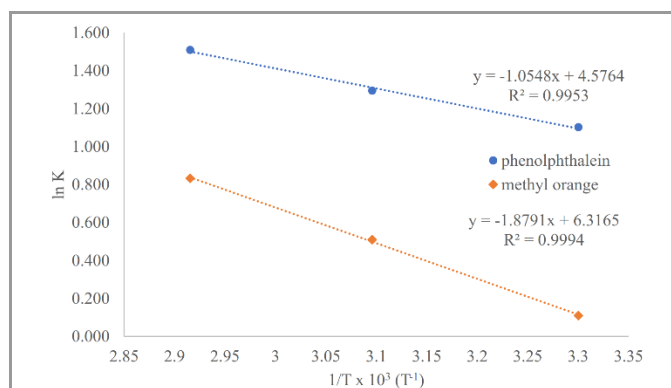
Where  $R$  (gas constant) is equal to  $8.314 \text{ JK}^{-1}\text{mol}^{-1}$ , a plot of  $\ln K$  against  $1/T$  gives a linear relation from which  $\Delta H$  (enthalpy change) and  $\Delta S$  (entropy change) of the processes can be estimated from the slope and intercept respectively. As shown in fig. 3, the strong correlation coefficients (0.995 and 0.999) obtained indicate the reliability of the plot to estimate these thermodynamic parameters. The change in Gibbs free energy ( $\Delta G$ ) was also evaluated at each temperature using the relation,

$$\Delta G = -RT \ln K \quad 7$$

**Table 2.** Langmuir and Freundlich isotherm parameters for the adsorption of methyl orange and phenolphthalein onto muscovite clay at 303 K.

Dye	Langmuir isotherm constants			Freundlich isotherm constants		
	$q_m$ (mg/g)	$K_L$ (L/mol)	$R^2$	$n$	$KF$ ( $\text{mg}^{(1-1/n)}\text{L}^{1/n}/\text{g}$ )	$R^2$
Methyl orange	13.00	2.737	0.987	0.73	8.42	0.543
Phenol phthal ein	2.48	1.043	0.859	2.23	17.69	0.155

The calculated values for the changes in enthalpy, entropy and Gibbs free energy for the adsorption processes are shown in table 3. The negative values of  $\Delta G$  recorded over the temperature range indicate that the adsorption process is spontaneous and the feasibility of removal of the dyes from aqueous solution increases with temperature.



**Figure 3.** Plot of  $\ln K$  against  $1/T$  for the adsorption of phenolphthalein and methyl orange onto muscovite clay.

Generally, the adsorption of phenolphthalein is highly dependent on temperature as compared to methyl orange. An enthalpy change of 8.77 kJ/mol and 15.62 kJ/mol for methyl orange and phenolphthalein respectively show that both adsorption processes are endothermic and require excess thermal energy to occur. However, the high  $\Delta S$  values (38.05 kJ/molK and 52.52 kJ/molK respectively) which have been recorded make  $T\Delta S$  higher than the required enthalpy. Therefore according to the Gibbs equation,

$$\Delta G = \Delta H - T\Delta S \quad 8$$

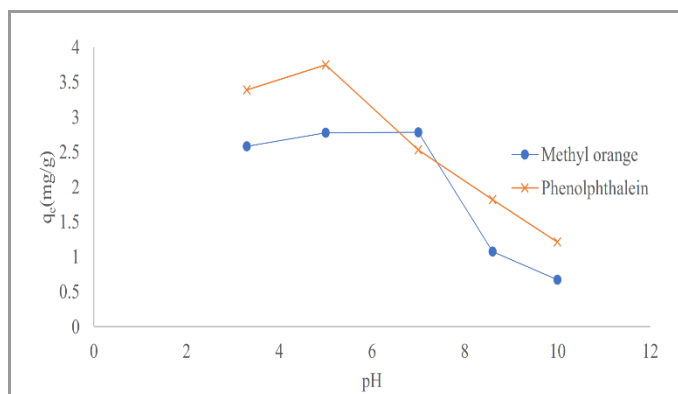
there is a resultant decrease in the change in Gibbs free energy which makes the process spontaneous. Similar results have been reported by Almeida et al who worked on the removal of methylene blue from coloured effluents using montmorillonite clay. This behavior was attributed to the potential expandability of the interlayer spacing of the clay material which allowed more dye to be adsorbed as the temperature increased [37].

The effect of pH on the adsorption of dyes onto clay surfaces has been found to be influenced by factors such as the surface charge on the adsorbent as well as the structure of the dye molecules [38]. As evident from figure 4, the adsorption capacity of the clay was assessed from a pH of 3.3 (acidic) to a pH of 10 (basic). At low pH, the excess  $\text{H}^+$  reacts with the surface Si-OH groups to form Si-

$\text{OH}_2^+$ . This increases the propensity of the clay for the adsorption of anionic dyes [18]. Similarly, the excess acid protonates specific groups such as the azo, tertiary amine and the  $-\text{SO}_3^-$  on methyl orange. As shown in figure 4, the equilibrium adsorbed amount of MO increased slightly from 2.581 mg/g at pH = 3.3 to 2.774 mg/g at pH = 5. It then peaked at 2.782 mg/g at neutral pH. This clearly shows that the lower the pH, the lower the equilibrium adsorption capacity of MO onto the surface of the adsorbent. At high pH, deprotonation mainly occurs at the surface; Si-OH yielding Si-O<sup>-</sup> in order to make the surface charge more negative and attractive to positive attachment sites on the adsorbate. As shown in Figure 1, the predominantly azo form of the MO present in basic solution has the anionic  $-\text{SO}_3^-$  group as the charged site.

**Table 3.** Thermodynamic parameters for the adsorption of methyl orange and phenolphthalein onto muscovite clay at three different temperatures.

Dye	+ΔH(kJ/mol)	+ΔS(kJ/molK)	-ΔG(kJ/mol)		
			303 K	323 K	343 K
Methyl orange	13.00	2.737	0.987	0.73	8.42
Methyl orange	8.77	38.05	11.52	12.28	13.00
phenolphthalein	15.62	52.52	15.90	17.00	18.00



**Figure 4.** Effect of pH on the adsorption of phenolphthalein and methyl orange onto muscovite clay.

The repulsion between this group and the anionic surface of the clay is responsible for the decline in the adsorption capacity of muscovite for MO observed from pH = 8.6 to pH = 10. A similar trend was observed for phenolphthalein within the studied pH range. As illustrated in figure 1, the carboxylate form of phph in basic medium has a higher charge density than that of the azo form of MO. As a result, it is expected that there will be higher repulsion between the phenolphthalein and the surface of the clay. But relatively, the adsorption capacity was higher than that of MO. This could be attributed to electrostatic attraction between the

phenolphthalein species and exchangeable cations on the surface of the clay [39].

Although thermodynamic and kinetic data have shown that the adsorption of methyl orange and phenolphthalein onto muscovite clay is largely by physisorption, factors such as temperature and pH can be controlled to increase the adsorption capacity of the adsorbent for these dyes.

## Conclusion

In this study, the adsorption of methyl orange and phenolphthalein onto muscovite clay was assessed and found to be successful. Batch adsorption studies showed that the sorption process equilibrated after 30 minutes and the pseudo-second-order kinetic model can best be used to explain the rate of both adsorption processes with  $R^2 > 0.99$ . The Langmuir and Freundlich isotherms were evaluated for both dyes at 303 K and it was observed that both processes obey the Langmuir isotherm model with monolayer adsorption capacities of 13.00 and 2.48 mg/g for methyl orange and phenolphthalein, respectively.

Assessment of the thermodynamic parameters at 303, 323 and 343 K showed that the adsorption processes are generally endothermic ( $\Delta H$  values of 8.77 kJ/mol and 15.62 kJ/mol for methyl orange and phenolphthalein respectively); but, the high entropy changes resulted in negative values of the changes in Gibbs free energy explaining the spontaneity of the processes at the working temperatures. Generally, the adsorption of both dyes was found to steadily increase within the pH range of 3.3 to 7.0; but, decreased drastically from pH = 8.0 to 10.0. Moreover, it can be regarded as a phenomenon which can be attributed to electrostatic repulsion between anionic sites on the dyes and the negatively charged active sites on the surface of the adsorbent.

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