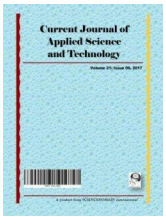

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Adsorption and Optimization Studies of Congo Red from Solution Using Montmorillonite-Silica Nanocomposite

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Authors' contributions

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

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ABSTRACT

Congo red was removed on Montmorillonite-silica nanocomposite from solution by adsorption process. The adsorption parameters studied were adsorbent dose, initial concentration and contact time. The optimum adsorption parameters were found to be 2 mg/L, 1.5 g and 40 minutes for initial concentration, adsorbent dose and contact time respectively with removal percentage of 84.10%. Pseudo first and second order kinetics were used for the studies. Pseudo second order best fit the adsorption process with R^2 = 1 than the Pseudo first order which has R^2 = 0.933. Experimental data were best fitted by the Langmuir Isotherm with R^2 =0.9024 other than the Freundlich Isotherm, R^2 =0.568. The R_L of 0.994 of the Langmuir isotherm shows the favourability of the adsorption process. The maximum adsorption capacity by Langmuir isotherm was found to be 172. 40 mg/g.

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1. INTRODUCTION

Dyes are used in various industries such as pharmaceutical, cosmetic, paper, leather, food stuff, plastics, and textile etc. The dye effluents are discharged into environment by either water or land. The discharge of these effluents into the receiving environments results in hazardous health problems as most of these dyes have carcinogenic effects on the living organisms [1-6]. Due to the toxicity (carcinogenic or mutagenic) of the dye pollutants, when discharge into the environments it causes a serious problem with respect to aquatic life and human health [7]. In other words, the toxic nature of the dye effluents leads to the death of soil microorganisms when they are used for irrigation purposes and this affects agricultural productivity [8]. Dye contamination in wastewater causes problems in several ways: the presence of dyes in water, even in very low quantities, is highly visible and undesirable; colour interferes with penetration of sunlight into waters; retards photosynthesis; inhibits the growth of aquatic biota and interferes with gas solubility in water bodies. There are different methods of treatment of dye contaminated wastewater which includes; Biological treatment, Chemical oxidation, Ozonation, membrane filtration, electrochemical method, Coagulation and flocculation, Liquid-liquid extraction, and Adsorption. But adsorption has been shown to be an effective way of removing organic matter from aqueous solutions in terms of initial cost, the simplicity of design, ease of operation and insensitivity to toxic substances [9-11]. Nanocomposite is a multiphase material where one of the phases has one, two, or three dimensions of less than 100 nanometers (nm) or structures having nano-scale repeat distances between the different phases that make up the material [12]. Rice husk (RH) is an agricultural waste which is abundantly available in rice producing countries like China, India, Bangladesh, Brazil, US, Cambodia, Vietnam, Myanmar, and South East Asia. The burning of RH produces the rice husk ash (RHA). The main composition of rice husk ash is amorphous silica constituting about 83–90%. Thus, it is suggested that the amorphous silica rich RHA could become a potential resource of low cost precursor for the production of value-added silica based materials for practical applications [13].

The aim of the present work was the optimisation of adsorption conditions for the effective removal of Congo red using Montmorillonite-Silica Nanocomposite. Thus, the adsorption conditions evaluated includes adsorbent dose, contact time and initial concentration of Congo red. Also, kinetic and isotherm models were applied to establish the rate of adsorption and adsorption capacity.

2. MATERIALS AND METHODS

2.1 Preparation of Solution/Reagents

1 M H_2SO_4: A 5.3 ml of concentrated sulphuric acid was pipetted and poured into 30 ml distilled water in a 100 ml volumetric flask and diluted with distilled water to the mark.

1MHCl: A measuring cylinder was used to measure 30.9 ml of concentrated HCl. It was then transferred into a 100 ml volumetric flask and diluted to the mark with distilled water.

2.5M NaOH: An accurately weighed 100 g of sodium hydroxide was placed in 1000 ml volumetric flask, and distilled water was added before making up to the mark.

1000 mg/L Congo Red stock solution: One gram Congo Red was weighed and placed in 100 ml volumetric flask. Distilled water was added and mixed thoroughly until the Congo Red dissolved. It was then made up to the mark with distilled water.

2.2 Sample Collection and Preparation

Both montmorillonite clay and rice husk samples were collected at Talasse, Balanga Local Government Area of Gombe state, Nigeria. The 1000 g rice husk was washed thoroughly with tap water six times and rewashed with distilled water three times to remove the adhesive dirt. It was dried overnight and subsequently, oven dried at 95°C and kept for ashing [14].

The clay (1000 g) was washed several times with tap water and rewashed further with distilled water four times. The washed sample was air dried and then oven dried for four hours at 110°C. The clay was ground and sieved using a 250 µm sieve. A weighed of 250 g of the powdered clay was refluxed with 500 ml of 1M

HCl for 1 hour at 120°C. It was then washed with distilled water five times to remove the acid. The clay was then dried in an oven at 105°C for six hours until a constant weight was obtained. It was then ground and kept for further synthesis of the montmorillonite–silica nanocomposite, [15-17].

2.3 Preparation of Rice Husk Ash

Rice husk ash was prepared according to Mohammad et al. [4]. A 500 g portion of dried rice husk was refluxed with 1000 ml hydrochloric acid for 1 hour at 95°C to remove some metallic impurities. After completion, it was washed thoroughly with distilled water five times and dried in an oven for five hours at 110°C. The treated rice husk was calcined in a muffle furnace for 7 hours at 650°C. The white rice husk ash was obtained and kept for further preparations.

2.4 Synthesis of Montmorillonite-silica Nanocomposite

A 20 g of white rice husk ash (WRHA) was refluxed with 2.5M NaOH solution for 1 hour with constant stirring at 200°C and then filtered, where a clear sodium silicate solution was obtained. A weighed amount of 80 g of acidified clay was added into 200 ml solution of the sodium silicate obtained with constant stirring. It was then precipitated with 1M H_2SO_4 drop wise and measuring the pH until it is equal to 8.9, it was then kept for 24 hours and then washed five times with distilled water until the pH was observed to be 7. It was then oven dried for five hours, ground and sieved with a 250 µ sieve, [18-20].

2.5 Adsorption Studies

All the experiments in these studies were carried out in 500 ml conical flask containing 200 ml Congo Red. After adding the amount of adsorbent, the flasks were agitated using magnetic stirrer while studying the parameters such as; the effect of adsorbent dose, initial concentration and contact time.

After the adsorption process, the flash content was filtered using Whatman filter paper. The filtrate obtained was analyzed using UV/Visible spectrophotometer (Model JENWAY 6300) at the wavelength of 497 nm. The adsorption percentage of the adsorbate and the amount of adsorbed, q_e (mg/g) were calculated according to the following equation;

$$Removal\ \% = \frac{(C_o - C_e)x\ 100}{C_o} \quad (1)$$

$$Amount\ adsorbed, q_e = \frac{(C_o - C_e)xV}{m} \quad (2)$$

Where, C_o = initial concentration of Congo red solution in mg/L, C_e =equilibrium concentration of Congo red solution in mg/L, m = mass of the adsorbent in grams, V = volume of Congo red test solution in liters(L) and q_e = amount adsorbed in mg/g.

3. RESULT AND DISCUSSION

3.1 Effect of Adsorbent Dose

The effect of adsorbent dose for Congo red dye, is shown in Table 1, it was observed that there was an increase in the removal percentage from an adsorbent dosage of 0.5 g – 1.5 g which decreases thereafter, Fig. 1. This scenario is against the report by several authors [21-24] where the removal percentage increases with increase in adsorbent concentrations. However, a similar case was reported by Olayinka et al. [25], in which removal percentage increases with increase in adsorbent dose and thereafter has a sharp decrease. And the authors reported that this may be due to aggregation of adsorption sites resulting in a decrease in the total adsorbent surface area of particles available to adsorbate and an increase in diffusion path length. This observation was in agreement with the previous investigation by Adebayo et al. [26]. Thus, the optimum adsorbent dose was found to be 1.5 g with a percentage removal of 74%.

Table 1. Effect of adsorbent dose table for Congo Red

Adsorbent dose(g)	Absorbance	Co (mg/L)	Ce (mg/L)	Co-Ce (mg/L)	% Removal
0.5	0.027	2	0.780	1.220	61.00
1.0	0.024	2	0.694	1.306	65.30
1.5	0.018	2	0.520	1.480	74.00
2.0	0.019	2	0.549	1.451	72.55
2.5	0.021	2	0.607	1.393	69.65

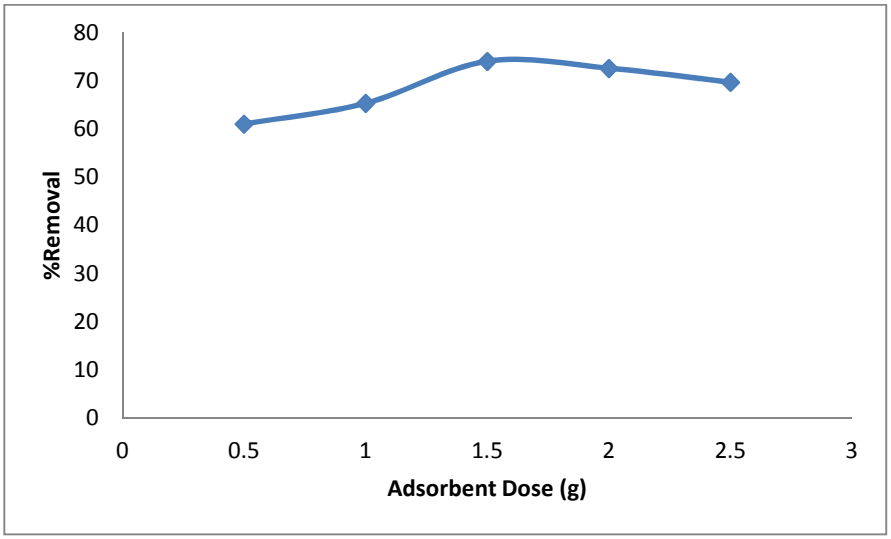

Fig. 1. Effect of adsorbent dose graph for congo red at adsorbate conc. of 2 mg/L, contact time of 60 (min), and varied dosage of 0.5, 1.0, 1.5, 2.0 and 2.5 g

3.2 Effect of Initial Concentration of Adsorbate

The adsorption of Congo red dye on montmorillonite-silica nanocomposite was studied at different initial concentrations from 1 mg/L to 5 mg/L. The result was represented in Table 2 and Fig. 2, it was observed that there was a high removal percentage increase from 1 mg/L to 2 mg/L. Beyond this concentration, the percentage removal decreases very slightly. This scenario is against the report by several authors [20-23] where the removal percentage increases with increase in initial concentrations. However, a similar case was observed in an investigation by Moosa et al., Olayinka et al. [24,25] where removal percentage increases with increase in initial concentration and thereafter have a sharp decrease. The optimum initial concentration was observed to be 2mg/L with percentage removal of 79.80%.

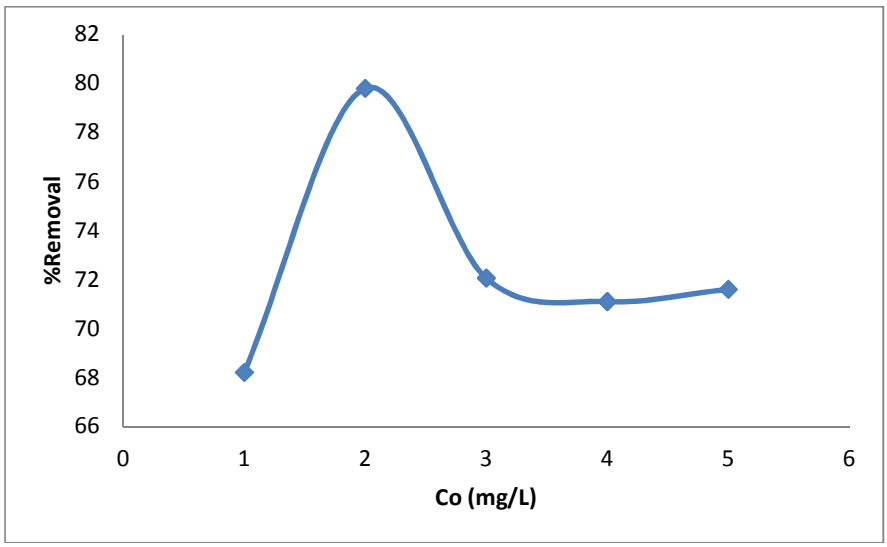

Fig. 2. Effect of initial concentration graph for congo red at various concentrations of 1, 2, 3, 4 and 5 mg/L, Contact time of 60 (min) and an adsorbent dose of 0.5 g

3.3 Effect of Contact Time

The experimental results of the effect of contact time obtained from the batch adsorption process of Congo red dyes; Table 3 and Fig. 3, these show that the effect of contact time plays an important role as the percentage removal of the dye increases rapidly with the increase of the contact time from 10 to 40 minutes. The percentage removal increases rapidly and then decreases significantly as the contact time for the adsorption increase. A similar observation was reported by Adebayo et al. [26] with even formation of plateau. Also, two stage sorption mechanism with the first rapid and quantitatively predominant and the second slower and quantitatively insignificant has been extensively reported in literature [27-29]. These two stage sorption mechanisms lead to plateau formation and thus, this behaviour gives away a slow approach to equilibrium [29]. Hence, the optimum contact time was obtained to be 40 minutes with the percentage removal of 84.10%.

Table 2. Effect of initial concentration for congo red

Adsorbent dose(g)	Absorbance	Co (mg/L)	Ce (mg/L)	Co-Ce (mg/L)	%Removal
1.5	0.011	1	0.3179	0.6821	68.21
1.5	0.014	2	0.4046	1.5954	79.80
1.5	0.029	3	0.8381	2.1619	72.06
1.5	0.040	4	1.1560	2.844	71.10
1.5	0.049	5	1.4161	3.5839	71.67

Table 3. Effect of contact time for Congo red

Time (min)	Adsorbent dose (g)	Co (mg/L)	Ce (mg/L)	Co-Ce (mg/L)	% Removal
10	1.5	2	0.6936	1.3064	65.32
20	1.5	2	0.6358	1.3642	68.21
30	1.5	2	0.4046	1.5954	79.77
40	1.5	2	0.3179	1.6821	84.10
50	1.5	2	0.3757	1.6243	81.22
60	1.5	2	0.5202	1.4798	73.99

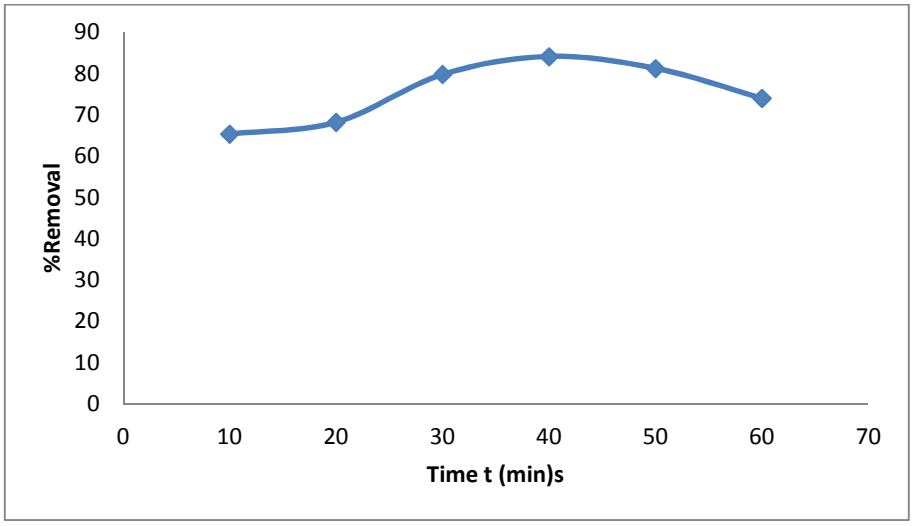

Fig. 3. Effect of contact time graph for Congo red at adsorbent dose of 1.5 g, varied contact time of 10, 20, 30, 40, 50 and 60 min and adsorbate concentration of 2 mg/L

3.4 Adsorption Isotherm Study

The experiment data studied in this adsorption process were analysed using only two models namely: Langmuir isotherm model and Freundlich isotherm model.

3.4.1 Langmuir isotherm

This describes quantitatively the formation of a monolayer adsorbate on the outer surface of the absorbent, and after that no further adsorption takes place. Thereby, the Langmuir represents the equilibrium distribution of metal ions between the solid and liquid phase, the Langmuir isotherm is valid for monolayer adsorption on to a surface containing a finite number of identical sites. The model assumes uniform energies of adsorption onto the surface and no transmigration of adsorbate in the plane of the surface. For liquid adsorbate, however, the Langmuir isotherm is usually expressed as follows;

$$q_e = \frac{(q_{max} C_e)}{(K_l + C_e)} \quad (3)$$

For correlation purposes, the equation is rearranged as follows;

$$\frac{C_e}{q_e} = \frac{1}{K_l q_{max}} + \frac{1}{q_{max}} . C_e \quad (4)$$

Where,

C_e = the equilibrium concentration of adsorbate (mg/L).
q_e = Is the equilibrium value of adsorbate adsorbed per unit weight of adsorbent (mg/g).
q_{max} = Is the maximum amount of adsorption corresponding to monomolecular layer coverage (mg/g).
K_L = Is the Langmuir constant and is related to measure of affinity of the adsorbent (l/mg).

A linearized plot of $\frac{Ce}{qe}$ against C_e yields a straight line graph which has an intercept and slope which correspond to $R_l = \frac{1}{1+ C_o K_l}$ and $\frac{1}{q_{max}}$ respectively, from which the q_{max} and K_l can be calculated.

To confirm the favourability of an adsorption process to Langmuir isotherm, the essential features of the isotherm can be expressed in terms of a dimensionless constant known as the separation factor or equation parameter R_L, which can be calculated by the following equation;

$$R_l = \frac{1}{1+ C_o K_l} \quad (5)$$

Where, C_o is the initial concentration.

The value of R_l indicates whether the isotherm is irreversible (R_l=0), favourable (0<R_l<1), linear (R_l=l), or unfavourable (R_l>1). Hence, from Table 4, the R^2 of 0.9 shows the fitness of the experimental data and the maximum adsorption capacity was found to be 172.40 mg/g. The R_L of 0.994 of the Langmuir isotherm shows the favourability of the adsorption process.

3.4.2 Freundlich isotherm

The Freundlich isotherm is an empirical equation employed to describe the heterogeneous system, it assumes that the adsorption energy of a solution or ion binding to a site on an adsorbent depends on whether or not the adjacent sites are already occupied.

The Freundlich equation is written as;

$$Log q_e = LogK + \frac{1}{\mathrm{n}} \mathrm{Log C_e} \quad (6)$$

Where, q_e and C_e are the equilibrium adsorption capacity of the adsorbent and the equilibrium concentration in the aqueous solution respectively.

Table 4. Langmuir isotherm data table

Dye	Isotherm	Constant	Values (mg/g)
Congo Red	Langmuir	q_{max}	172.40
		R^2	0.9024
		K_L	0.0002
		R_L	0.994

K and n are the Freundlich constants related to adsorption capacity. And the plot of $Logq_e$ against $LogC_e$ gives the Freundlich isotherm graph. And the parameters; n and K were also obtained from the slopes and intercept of the graph.

For the Freudlich isotherm Table 5, the parameters were also obtained from the slope and intercept of the graph of the plot of log q_e against log C_e and R^2 value obtained was observed to be 0.568.

3.5 Kinetic Adsorption Study

The pseudo-first order and the pseudo-second order kinetics are the only kinetics studied for this experimental adsorption process.

The pseudo-first order equation is represented as follows:

$$\log(q_e - q_e) = logq_e - \frac{K_1 t}{2.303} \quad (7)$$

Where, K_1 is the Lagergren rate constant of adsorption (1/min).

The plot of log (q_e-q_t) against t, gives linear relationship from which q_e and K_1 are determined from the intercept and slope of the plot respectively. The data for pseudo first order is shown in Fig. 4 and the R^2 values indicate that the data fit the process.

The pseudo-second order equation can be represented by the following linear form below.

$$\frac{t}{q_e} = \frac{1}{K_2 q_e^2} + \frac{t}{q_e} \quad (8)$$

Where, K_2 is the second order rate constant of adsorption (g/mg min). The values of K_2 and q_e are determined from the intercept and slope of the plot of $^t/_{q_t}$ againt t. The data for pseudo second order is shown in Fig. 5 and the R^2 values indicate that the data fit the process than the pseudo first order.

Table 5. Freundlich isotherm

Dye	Isotherm	Constant	Value
Congo Red	Freundlich	K_F	0.39
		R^2	0.568
		N	0.49

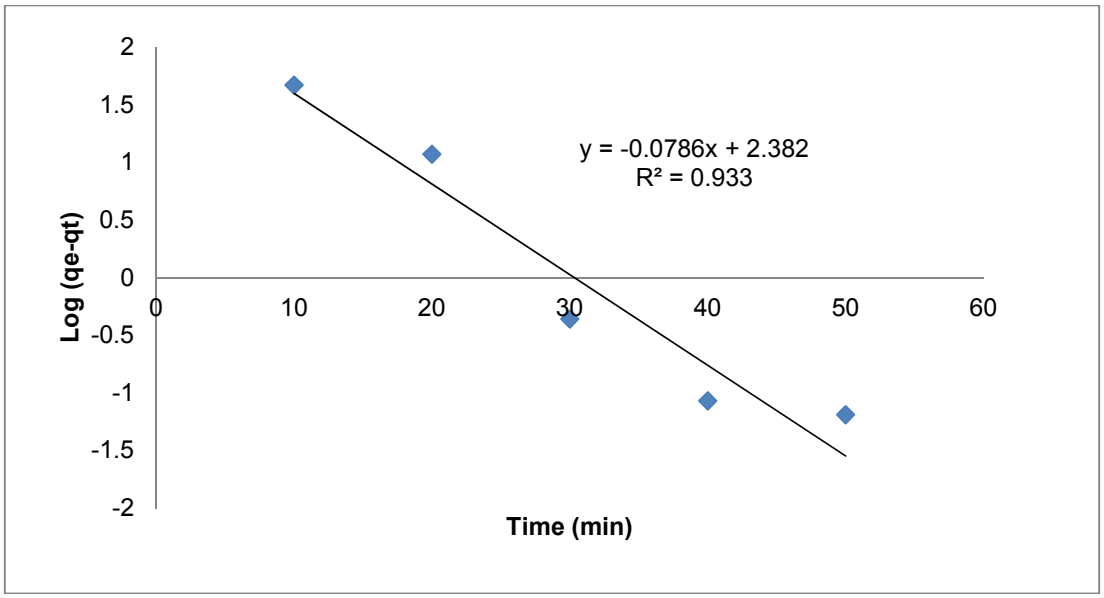

Fig. 4. Pseudo first order kinetic graph

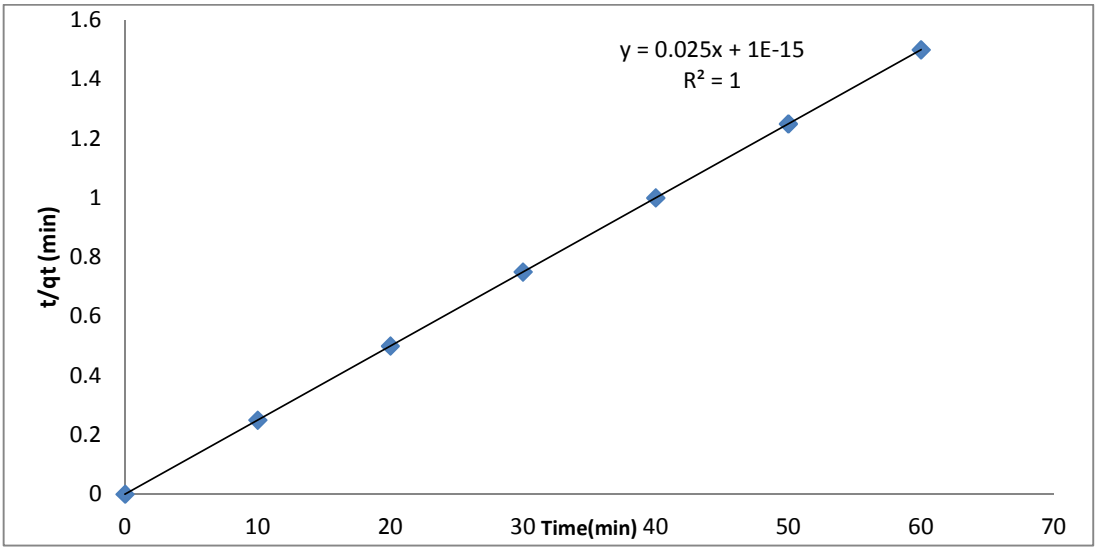

Fig. 5. Pseudo second order kinetic graph

4. CONCLUSION

The adsorption process of Congo red was carried out using Montmorilonite silica nanocomposite. From the experimental data the percentage removal of 84.10% obtained at an optimum initial concentration of 2 mg/l, an optimum adsorbent dose of 1.5 g and optimum contact time of 40 minutes. Experimental data were best fitted by the Langmuir Isotherm with R^2 =0.9024 other than the Freudlich Isotherm, R^2 =0.568. The pseudo second order kinetic with R^2 = 1 best fit the adsorption process other than the pseudo second order kinetics, R^2 =0.933.

Therefore, montmorillonite-silica nanocomposite obtained from montmorillonite clay and Rice husk ash can serve as a cost-effective adsorbent in the removal of Congo red dye.

5. RECOMMENDATION

The authors recommend that further work should be carried with full characterisation of the adsorbent With FTIR, SEM, TEM, and XRD etc to understand the adsorption mechanism for this particular work as well as to derive a good conclusion.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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