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Adsorption of anionic dye (Reactive black 5) using macadamia seed Husks: Kinetics and equilibrium studies

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ABSTRACT

Adsorption of Reactive black 5 dye currently used in textile industries was investigated using Macadamia Seed Husks (MSH) from a local Macadamia nut processing industry. Adsorption increased with increase in contact time, adsorbent dose and dye concentration. Conversely, the percentage dye adsorbed decreased with increase of solution pH and size of MSH particles. Sorption increased with an increase in temperature signaling an endothermic process. Batch adsorption data best fitted to Freundlich adsorption isotherm model with K_f value of 4.20679 and $n = 2.3375$. The adsorption kinetics followed a pseudo-second-order kinetic model with high correlation coefficients ($R^2 > 0.98$). These results demonstrated that MSH is an environmentally friendly, locally available, effective and economical adsorbent for the removal of RB5 dye from industrial wastewaters.

Background information

Dyes play vital role in colouring of fabrics and garments in textile industries. Currently, more than 1.0×10^5 different kinds of dyes and pigments are manufactured annually worldwide [1]. Industrialization, population increase and need for color variety are some of the factors responsible for increased demand of dyestuffs. As a result enormous quantities of wastewater polluted with synthetic dyes and chemicals are released into environment. Up to 2.0×10^5 tons of dyes are lost in wastewater annually during dyeing and finishing operations as a result of inefficiency in the dyeing process [2]. Discharge of wastewater contaminated with dye into water bodies causes significant problems, such as amplified COD, TDS, BOD, toxicity, turbidity and reducing light penetration, which negatively affect aquatic plants [3]. Public health concern and environmental pollution have forced governments to establish rigid laws on permissible pollutants concentration to be released into the environment by industries.

Dye removal from effluent is a complex and expensive process because dyes are made up of complex aromatic structure stable to light, heat, oxidation and bio-degradation [4]. Wastewater treatment methods including chemical, physical and biological systems have been developed and evaluated in dye treatment from wastewaters [5]. However, these methods face limitations such as high cost, long operation time, production of high amounts of sludge and incomplete dye removal [6].

Adsorption has been lauded as one of the most predominant treatment technique due to its simplicity, inexpensive, efficiency and ability to utilize locally available bio-material. Activated carbon is frequently utilized in many industries as

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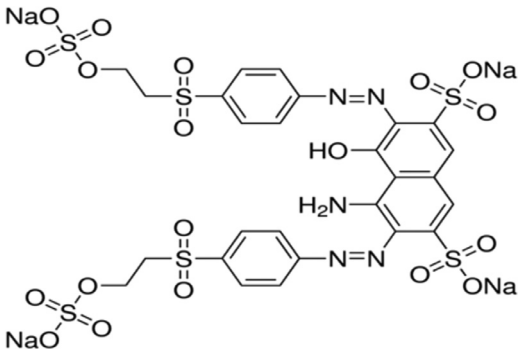

Fig. 1. Chemical structure of reactive black 5 Dye.

adsorbent due to its high efficiency. However, its high cost and losses during regeneration limit its use [7]. Search for an alternative eco-friendly, affordable and cost effective adsorbent materials has been the focus by many researchers. Materials such as Barbados shells [8], palm kernel fiber [9], cactus [10], sawdust [11], spirogyra [1], *Eichhoria crassipes* roots [12], pea shells based activated carbon [13], flamboyand pods (*Delonix regia*) activated carbon [14] among others have been successfully studied.

Macadamia nut production has been rising in the past years. Macadamia farming have captured the interest of many famers in East Africa due to its ability to fetch better prices compared to other cash crops [15]. Macadamia nuts have several applications including whole nut, crushed nut as a food ingredient (biscuit, cake or ice cream, etc.), cooking vegetable oil and also as an alternative fuel in diesel engine [16]. The rise has led to production of high amounts of unutilized Macademia Seed husks (MSH) which pollute the environment. Disposed MSH in the landfills takes long to decompose hence act as breeding places for pathogens as well as causing air pollution when burned.

Reactive black 5 (RB5), ($C_{26}H_{21}N_5Na_4O_{19}S_6$,) (Fig. 1) is a tetrasulphonated disazo dye is popular in textile industries for dying materials such as cellulose and cotton fibres. The discharge of RB5 to the environment posed major threat to public health since exposure to the dye causes impaired respiratory system, acute bronchitis, skin irritations, mutations, bladder cancer among other effects [17,18]. Therefore, it's paramount for wastewaters containing RB5 to be cleaned before disposing them to the environment. Different low-cost agricultural by-products such as banana peel powder [19] spent tea leaves [20] pumpkin seed hulls and eggshells [21] have been evaluated for removal of RB5dye from wastewater with varied success. This study investigates the practicability of utilizing MSH as affordable biomaterials for sorptive removal of RB5dye from contaminate wastewater.

Materials and methods

Materials

Macadamia seed husks (MSH) were collected from the dumpsite of the Jungle factory (Macadamia nut processing industry) Thika, Kenya. The husks were mechanically crushed into smaller particles and sieved into different homogenous particle sizes. Tap water was used to wash the particles severally to remove soil and other impurities. Meshed MSH was further soaked in 10% H_2O_2 solution for 24 h at 25°C to remove soluble impurities. The clean MSH were repeatedly washed using distilled water till a neutral pH was attained before sun drying. RB5 dye was obtained from Sigma Aldrich, Nairobi, Kenya and utilized without further purification. All reagents used in the study were of analytical grade quality.

Batch sorption studies

Batch adsorption experiments were conducted in 100 mL conical flask using 50 mL of RB5 solution. Flasks were shaken on a KJ-201BD oscillator operating at 140 rpm running at diverse time intervals. The effect of agitation time was studied by adding 2.5 g of MSH adsorbent of particle size 300–600 µm into 50 mL of RB5 dye at 60°C and pH 3. Effect of dosage on sorption of RB5 dye was investigated using 1.0, 1.5, 2.0, 2.5 and 3.0 g of MSH at 50°C. The influence of particle size was examined by adding 2.5 g of each particle size 150–300, 300–600, 600–1180, 1180–2360 and 2360–4750 µm in different conical flasks containing 50 mL of 91.1247 mg/L RB5 at pH 3. Initial dye concentration effect was evaluated by varying the RB5 dye concentration from 9.1247 mg/L to 91.2474 mg/L. Effect of temperature was also examined at a temperature range of 25 °C to 80 °C while pH was investigated by varying the RB5 dye pH from 1 to 14. All experiments were carried out for 600 min. The pH of the solutions was adjusted as required using 0.1 M NaOH and 0.1 M HCl. Aliquots from the supernatant solution were drawn and placed in 10 mm cuvettes and the absorbance of the solution measured against a blank reagent using a UV–VIS spectrophotometer (HALO RB-10) and returned into the flask to maintain the original volume. The amount

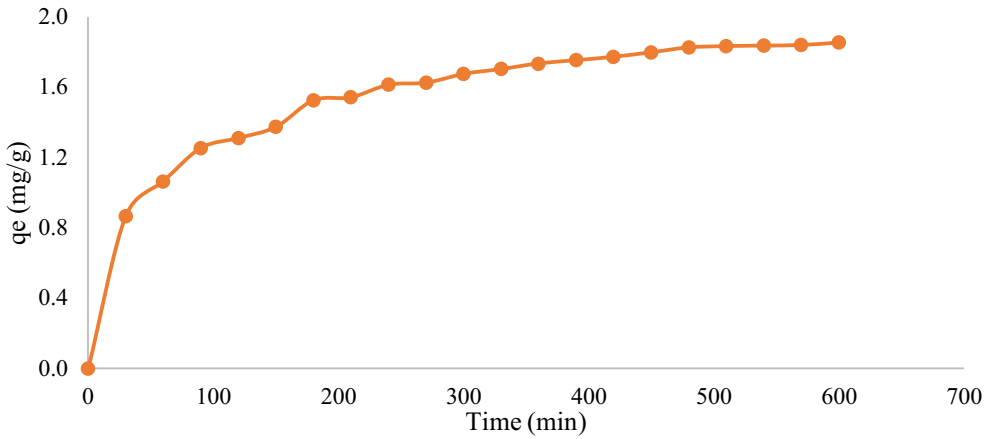

Fig. 2. Effect of time of contact on RB5 dye sorption onto MSH Conditions: (RB5 dye 91.2474 mg/L, MSH 2.5 g/50 mL, particle size; 300–600 µm, at 60 °C and pH 3).

of RB5 dye adsorbed at equilibrium onto MSH, Q_e (mg/g), was calculated using Eq. (1):

$$Q_e = \left(\frac{C_o - C_e}{w}\right) V \ . \tag{1}$$

Where C_o and C_e are the initial and the equilibrium dye concentrations (mg/L), V is the volume of solution (L) and W is the amount of biomass used (g).The percentage of RB5 dye removed from the original solution was calculated by the equation

$$\%\ \text{Dye Removal} = \left(\frac{C_o - C_e}{C_o}\right) \times 100. \tag{2}$$

Results and discussion

Effect of agitation time

Agitation time determines the effectiveness of the adsorbent in the removal of the pollutant molecules [22]. Fig. 2 presents a plot of the amount of RB5 dye adsorbed on MSH as a function of time. It is clear from the figure that the rate of adsorption was higher during the first 90 min before attaining equilibrium at around 510 min. Removal of RB5 dye by MSH was not spontaneous and hence the process required more time. Once equilibrium had been established, the amount of RB5 dye adsorbed over time was insignificant. High RB5 dye uptake at the start can be accredited to accessibility of numerous vacant active adsorption sites on the surface of MSH. The sorption capability of RB5 declined as time elapsed since the accessible adsorption sites had been occupied and the remaining sites became hard to occupy due to repulsive forces between RB5 adsorbate molecules on the MSH solid surface and the bulk phases. Similar findings have been recorded on the adsorption of recalcitrant dyes from aqueous solutions [12,13,17,21,23].

Effect of MSH particle sizes

The Surface area accessible for adsorption is considerably affected by the adsorbent particle size. Large surface area corresponds to a higher number of active adsorption sites hence greater adsorption capabilities. Effect of particle sizes on RB5 dye adsorption was examined and the results presented in Fig. 3. There was a steady decrease of RB5 dye removed as the size of the MSH particles increased. For instance, the percentage dye removal decreased from 98.9% to 33.2% as the particle sizes increased from 150–300 µm to 2360–4750 µm. The observation can be credited to the large surface area and therefore greater number of active adsorption sites of the smaller MSH particles as compared to larger MSH particles. These results are consistent with related research on the adsorption of synthetic dyes from solution [13,24,25].

Effect of adsorbent dose

The impact of MSH dosage on RB5 dye removal was examined at 25°C and at 60°C and the results illustrated in Fig. 4. The results revealed that the percentage dye removal increased as the MSH adsorbent dose increased for the two temperatures tested. Conversely, the percentage of dye adsorbed by the same amount of MSH dose was higher at 60°C as compared to room temperature. This points out that lesser doses could be employed at higher temperatures for sorption of RB5 dye. These trends can be attributed to an increase in the total surface area and active sorption sites as the amount of dosage increased. Similar results have been recorded in sorption of crystal violet on coffee husks [5].

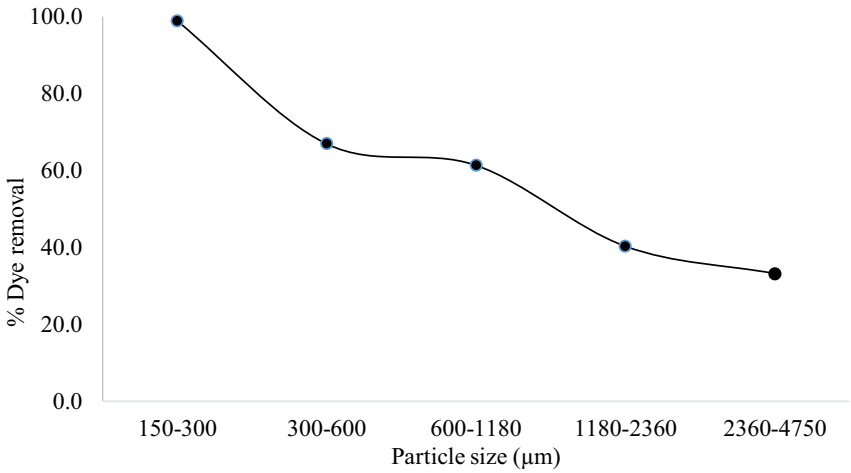

Fig. 3. Effect of MSH particle size on RB5 dye sorption Conditions: (RB5 dye 91.2474 mg/L, MSH 2.5 g/50 mL, particle size; at 60 °C and pH 3).

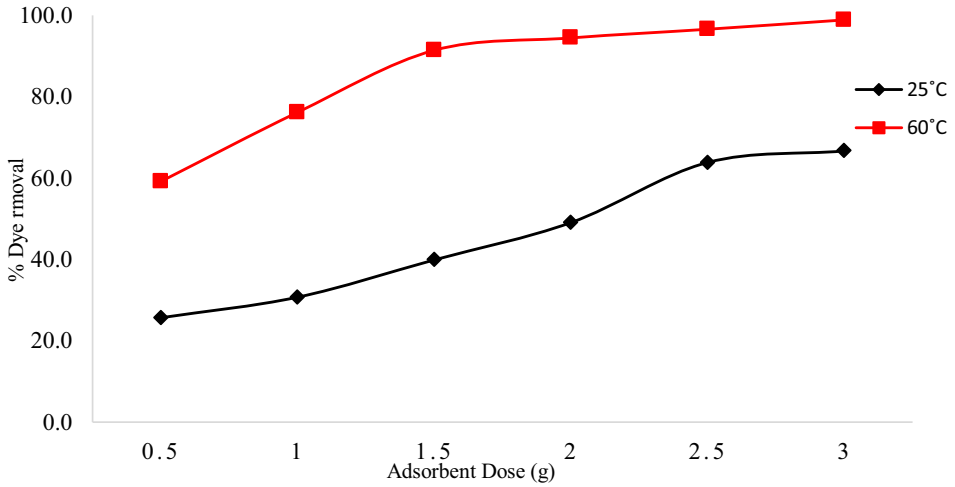

Fig. 4. Effect of MSH dose on RB5 dye sorption Conditions: (RB5 dye 91.2474 mg/L, MSH 2.5 g /50 mL, particle size; 300–600 µm at pH 3, 25 °C and 60 °C).

Effect of initial dye concentration on the sorption of RB5

Initial dye concentration effect on sorption of RB5 dyes by MSH was investigate and the results shown in Fig. 5. The graph demonstrates that the dosage of RB5 dye removed gradually improved with an increase in RB5 dye concentration. The capacity of dye adsorbed increased from 0.1543 mg/g to 1.1436 mg/g when the initial dye concentration was improved from 9.1247 mg/L to 91.2474 mg/L. The results can be ascribed to an increasing concentration gradient which acts as an increasing driving force overcoming all mass transfer resistances on the solute dye molecules between the aqueous solution and solid phase leading to an increasing equilibrium sorption until saturation is attained. The above outcome is in line with other results obtained by different researchers on the adsorption of synthetic dyes using bio- material [1].

Effect of temperature on the adsorption of RB5

Temperature changes can manipulate the viscosity of the solution as well as controlling dye diffusion rate over the external boundary layer and internal pores of the adsorbent [26,27]. The influence of solution temperature on the sorption of RB5 dye by MSH was studied and the outcomes displayed on Fig. 6. Percentage dye uptake increased from 63.8% at 25 °C to 100% at 80 °C. This designates that the adsorption of RB5 dye by MSH was endothermic process. The increase in the dye removal primarily with rise in temperature can be related to an increase in the mobility of the RB5 dye molecules/ reduced aqueous solution viscosity thus more adsorbate molecules could interact with the active adsorptive sites on MSH. Additionally, this can be credited to a rise in the strength of the intermolecular forces between the active adsorption sites of the MSH and the RB5 dye molecules than those between the solvent and dye molecules. Similar results have been previously

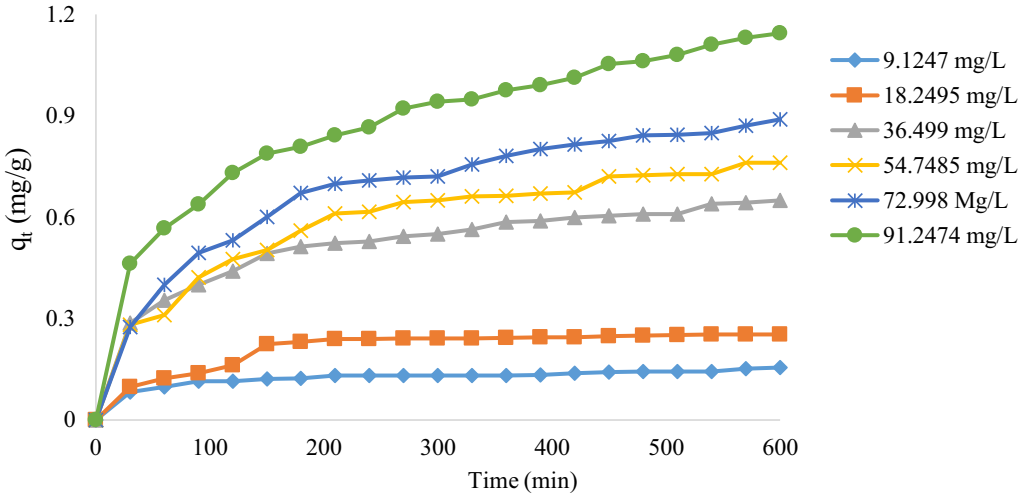

Fig. 5. Effect of initial RB5 dye concentration sorption Conditions: (MSH 2.5 g /50 mL of RB5 solution, particle size; 300–600 µm at 25 °C, pH 3).

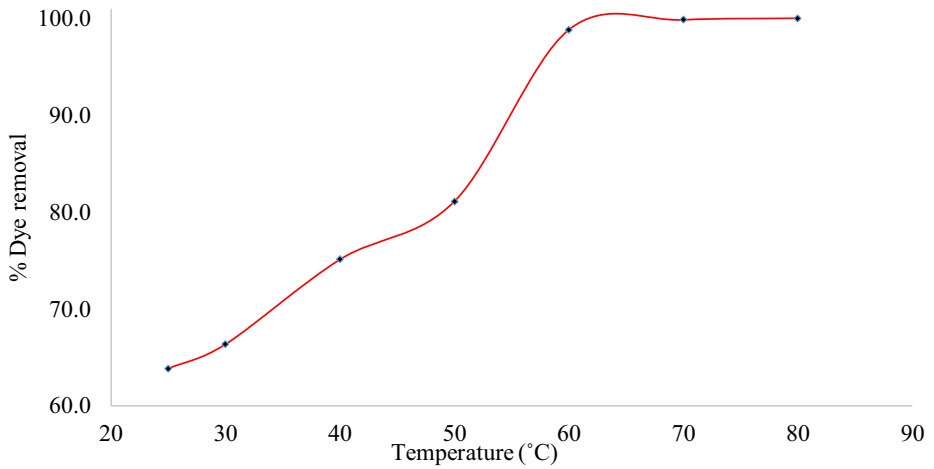

Fig. 6. Effect of temperature on the percentage RB5 removal. Conditions: (RB5 dye 91.2474 mg/L, MSH 2.5 g /50 mL of RB5 solution, particle size; 300 µm–600 µm, pH 3).

obtained in the adsorption of Burazol Blue ED dye onto dried *Neurospora crassa* biomass [26] and sorption of methylene blue dye onto poly (cyclotriphosphazene-co-4,4′-sulfonyldiphenol)nanotubes [28].

Effect of solution pH on adsorption of RB5

The pH of the aqueous dye solution transforms the surface charge of the sorbent material and controls the extent of ionization of the dye molecules. The impact of pH on the sorption of RB5 dyes by MSH was studied and the results presented in Fig 7. The results precisely show that adsorption took place in strong acidic conditions with the percentage dye removal gradually decreasing from 83.9% at pH 1 to 0% at pH 6. There were zero dye uptakes from pH 6 to pH 14. The higher rate of dye removal at lower pH could be attributed to higher electrostatic forces of attraction between the negative charged molecules and the positively charged adsorbent surfaces which were replaced by repulsive electrostatic forces as the solution pH was increased. A similar trend has been previously recorded [17,29]

Adsorption isotherms

Adsorption isotherms are significant in the designing an effective wastewater treatment system. Freundlich adsorption isotherm was employed to evaluate the practicability of adsorption as a technique of removing RB5 dye from aqueous solution using MSH powder. Freundlich isotherm model is commonly applied to describe heterogeneous adsorption process i.e. adsorption which takes place on a heterogeneous surface through a multilayer adsorption mechanism. Freundlich isotherm

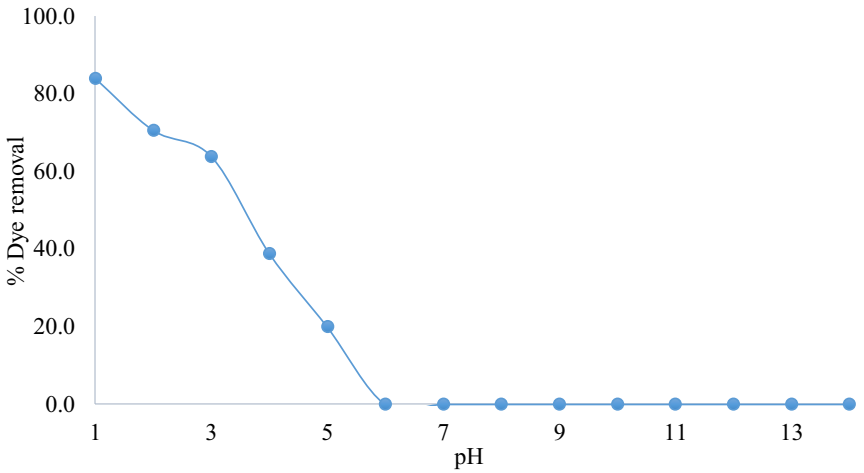

Fig. 7. Effect of solution pH on RB5 sorption. Conditions: (RB5 dye 91.2474 mg/L, MSH 2.5 g /50 mL of RB5 solution, particle size; 300 µm–600 µm, at 25 °C).

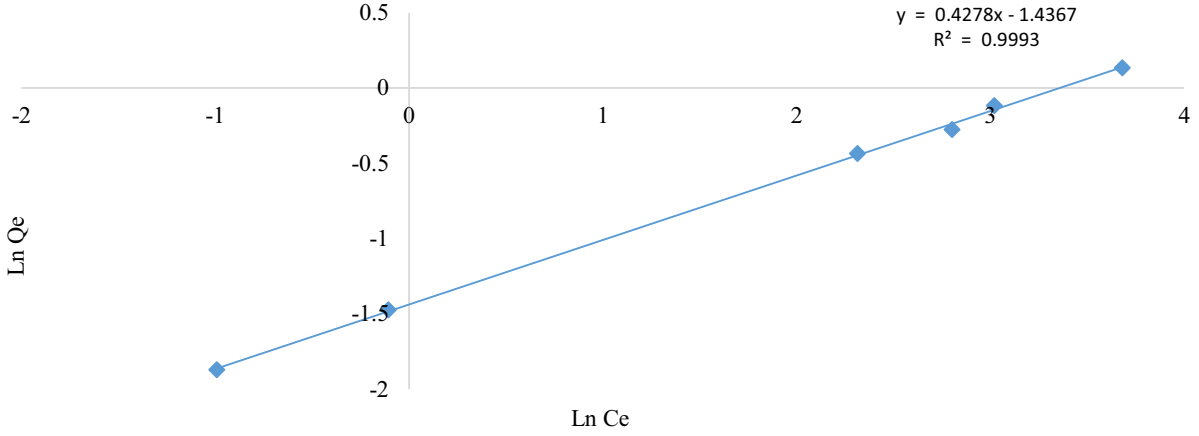

Fig. 8. Freundlich adsorption isotherm for RB5 dye sorption on MSH.

[30] is expressed as:

$$q_{e\ =\ K_f}.C_e^{1/n} \tag{3}$$

$$\text{In}\ q_e = InK_f + \left(\frac{1}{n}\right)InC_e \tag{4}$$

Where; q_e is the dye adsorbed on the adsorbent at equilibrium (mg/g), C_e is the concentration of dye solution at equilibrium (mg/L), K_f (mg/g) (L/mg)) is the empirical Freundlich constant, which indicate the adsorption capacity of the adsorbent, n is the Freundlich constant indicating the intensity of adsorption whose value should be ($n > 1$) for the conditions of the sorption process to be favourable. Values of k_f and n are obtained from the intercepts (Ink_f) and slopes (1/n) of the plot of lnq_e against lnC_e. A plot of lnq_e verses lnC_e for the adsorption of RB5 dye on MSH is shown in Fig. 8. The value K_f was found to be 4.20679. The value of $n = 2.3375$ perfectly fitted the condition for favourable multilayer sorption ($n > 1$). This indicated that there was physiosorption coupled with chemisorption during the process. The higher regression coefficient ($R^2 = 0.9993$) indicated the Freundlich adsorption isotherm model was best suitable for describing the equilibrium conditions for RB5 adsorption onto MSH. Previous results from adsorption of methylene blue dye by *Eichhornia crassipes* followed Freundlich model suggesting multilayer adsorption [24].

Adsorption kinetic studies

To understand the sorption mechanism of RB5 dye onto MSH in relation to time, the experimental data was analyzed using the pseudo second order kinetic model. The pseudo second-order model is expressed as:

$$\frac{dq_e}{dt} = k_2\ (q_e - q_t)^2 \tag{5}$$

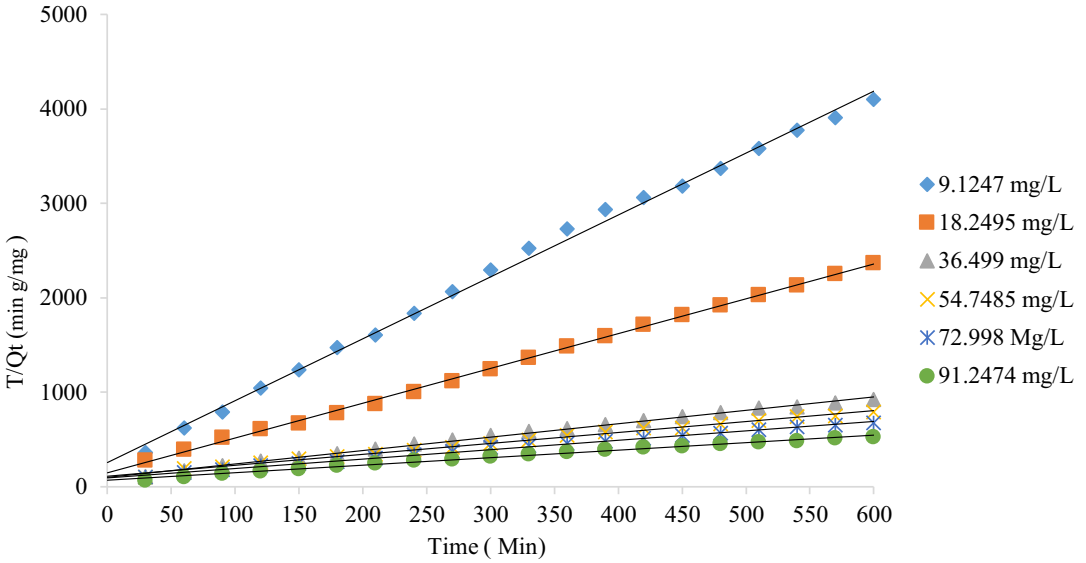

Fig. 9. Pseudo-second order kinetics plot for the adsorption of RB5 dye onto MSH.

Table 1
The pseudo second order kinetic model values for RB5 dye.

Pseudo second order kinetic models				
Concentration (mg/g)	$Q_{e\ cal}$(mg/g)	$Q_{e\ exp}$ (mg/g)	K_2(g/mg/min)	R^2
9.1747	0.1509	0.1543	0.2049	0.9956
18.2492	0.2668	0.2532	0.1152	0.9971
36.2495	0.6828	0.6489	0.0266	0.9916
54.7485	0.8276	0.7606	0.0161	0.9859
72.998	0.9640	0.8894	0.0136	0.9872
91.2474	1.2100	1.1436	0.0114	0.9844

When the initial condition is $q_t = 0$ at $t = 0$, integration leads to Eq. (6):

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \tag{6}$$

Where k_2 is the rate constant of the pseudo second order adsorption (gm/g min), q_e and q_t are the amounts of RB5 adsorbed (mg/g) at equilibrium and at time t (min) respectively. A plot of t/q_t vs t of Eq. (6) yield a linear relationship, from which, q_e and k_2 can be determined from the slope and intercept of the plot. Fig. 9 displays pseudo-second - order kinetics graph for the adsorption of RB5 dye by MSH at different dye concentrations. The correlation coefficients R^2, calculated $Q_{e.cal}$ and experimental $Q_{e.exp}$ equilibrium dye uptakes (mg/g) and the rate constants K_2 (g/mg/min) were tabulated in Table 1. It is clear from the results that the correlation coefficients (R^2) were close to a unit implying that the experimental values fitted well on the equation. The corresponding calculated $Q_{e.cal}$ (mg/g) values were comparable with the experimental $Q_{e.exp}$ (mg/g) values for all concentrations tested indicating that the sorption of RB5 dyes followed the Pseudo second order kinetic model perfectly. The small difference between the calculated and experimental $Q_{e.exp}$ values is most possibly due to the existence of boundary layer effects during the adsorption [20].The overall rate of RB5 sorption depended essentially on time of contact and concentration of the dye solution. It also suggested that chemisorption involving valence forces through exchange or sharing of electron between adsorbate and adsorbent dominated. These results are in line with recorded results for the removal of recalcitrant dye from solution using bio-waste [1,20,25,31,32].

Conclusion

The study demonstrates that MSH is certainly an effective adsorbent for removal of RB5 dye from polluted wastewater with almost 100% dye removal at optimum temperature. Experimental results analysis disclosed that the sorption process was highly controlled by time of contact, MSH particle size, initial RB5 dye concentration, MSH dosage solution pH and temperature. The amount of RB5 dye removed in percentage was found to increase with the increase of contact time, adsorbent dose, temperature and initial dye concentration. Conversely, the percentage dye uptake decreased with increase of solution pH and size of MSH particles. The experimental results fitted on Freundlich adsorption isotherm models showing

that phsiosorption process took place. Kinetic studies identified that the experimental data fitted well on the pseudo second order kinetic model implying that the rate of the process depended on time and concentration. The outcome of this study concludes that MSH can be used as an effective, highly potentiated, inexpensive, and locally available adsorbent for the removal RB5 dye from contaminated wastewaters.

Declaration of Competing Interests

None.

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