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Removal of Dyes by Using Rice Husk as Agricultural Solid Waste Adsorbent

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Abstract: Nano Activated Carbon (NAC) prepared from Rice Husk (RH) was used in batch adsorption experiments for removal of methylene blue (MB) and black B (BB) dyes from synthetic solutions. The effect of different operating parameters were investigated covering the pH (1-11), contact time (15-75 minutes) and temperatures (30-60^{O}C) for the two dyes of different concentrations (50-300 mg/l). The high adsorption capacity achieved at pH 3 for MB and at pH 11 for BB and at 30^{O}C. The adsorption equilibrium was attained after 60 minutes. Adsorption isotherms have been determined and the obtained data were analyzed using four isotherms models Langmuir, Freundlich, Temkin and Dubinin–Radushkevich. The two isotherm models Langmuir and Freundlich were favorable for MB dye but Langmuir was only favorable for BB. Two kinetic models have been studied namely: pseudo first order and pseudo second order kinetics. The results showed that the uptake rate of the NAC adsorbate obeyed pseudo second order kinetic model for both dyes.
Keywords: Nano activated carbon; Rice husk; Adsorption isotherm; Kinetic models.

1 Introduction

With the increasing of environmental awareness, stringent regulations are issued to control discharge of industrial wastewater in Egypt [1,2]. This concern eventually led to strict discharge limits for hazardous wastes such as dyes in industrial effluent which discharged to receiving water. Approximately 15% of the overall world production of dyes [3] are lost during the dying process and is released as liquid effluents. A very small amount of dyes in water is highly visible and can be toxic to creatures in water because it reduces the penetration of sunlight, causing a reduction in photosynthetic action that prevent the growth of the aquatic life [4-6]. They also pose a problem because they may be mutagenic and carcinogenic [7]. Hence, the removal of dyes from industrial processes or wastewater effluents becomes environmentally important and must be treated before disposal. Treatment of dye-based effluents is considered to be most challenging in the environmental fraternity, respecting to the large degree of organics present in these molecules, the stability and low biodegradability of modern dyes, conventional biological treatment methods are ineffective for their removal. Some existing technologies may have certain efficiency in the removal of dyes [8-11], but their initial and operational costs are very high, on the other hand low cost technologies do not allow a wishful color removal or have certain advantages. Hence, research has been directed to use cheap materials and simple procedures for efficient dye removal. Adsorption has become increasingly popular treatment technique compared to other techniques. It is a high quality treatment process for the removal of dissolved organic pollutants, such as dyes, from industrial wastewater. The adsorption efficiency depend on the nature of adsorbent used. The adsorption process has many advantages over several other conventional treatment methods for wastewater treatment. These include (i) less land area, (ii) not getting affected by toxic chemicals, (iii) greater flexibility in the design and operation and (iv) superior removal of organic contaminants. In the present study, attempts have been made to explore of the removal of cationic Methylene Blue(MB) dye and anionic Black B (BB) dye by using NAC prepared from low cost Rise Husk based on adsorption technique.

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2 Experimental Procedures

2.1 Raw Materials and Chemicals

Raw material used was Rice Husk (RH) for preparation of ACs, received from one of the rice mill in Egypt. The chemicals used were ortho-phosphoric acid (H_3PO_4), hydrochloric acid (HCl) and sodium hydroxide (NaOH) of analar grades from Merk Company, in addition of analytical grade of methylene blue and black B dyes (Purchased from Egyptian Chemical Company)

2.2 Preparation of NAC

Preparation of NAC was carried out in two steps activation and carbonization steps according to previous work carried out by the present authors [12] . The best operating parameters for the preparation were 90 micron particle size of RH and 30% H_3PO_4 as activating agent with solid/liquid ratio of 1:5 at 400^{o}C for 2 hours carbonization time.

2.3 Dye Solution Preparation

An accurately weigh of each dye (MB and BB) dissolved in distilled water was used to prepare stock solution (1000 ppm). Desired concentrations were obtained by successive dilutions. Final Dye concentration was determined by using absorbance values measured before and after treatment, at wavelength 665 nm for MB and at wavelength 596 nm for black BB using UV visible Spectrometer (model 559 UV-VIS) supplied from Perkin-Elemer.

3 Batch Adsorption Experiments

The prepared NAC was used as adsorbent material for color removal via batch experiments. MB and BB dyes were tested as adsorbates. The experiments were carried out by preparing different concentrations 100, 200 and 300 mg/l of synthetic solutions for both dyes with 0.05 mg/100 ml dose of NAC. The MB and the BB dyes concentrations were determined for each run after adsorption. The tested parameters were pH, contact time, initial concentrations of adsorbate and temperatures.

4 Equilibrium Isotherms

Sorption isotherms is a tool to describe how adsorbates have interaction with adsorbents to be able to optimize using adsorbents within the adsorption method to optimize the design of adsorption system. For isotherm identification, an amount of 0.05 gram NAC and 100 ml solution of an aqueous phase (MB or BB dye) were placed in a 250 ml glass stopper for 24 hours using a shaking water bath controlled at a specific temperature (30-60^{o}C) and at best pH for both dyes. Four isotherm equations (1 to 4) were used namely, Langmuir, Freundlich, Temkin and Dubinin–Radushkevich for testing both MB and BB dyes and presented as follows

Langmuir isotherm equation $qe = \frac{K_L C_e}{1+a_{lC_e}}$ …………………….………… (1)

Where: q_e and Ce are the equilibrium solid and liquid phases concentrations respectively, K_L and a_L are the Langmuir isotherm constants.

Freundlich isotherm equation $\log qe = logk_f + \frac{1}{n} logC_e$……………….…… (2)

Where: K_F and n are Freundlich constants

Temkin isotherm equation. $qe = B_T lnA_T + B_T lnC_e$………………………. (3)

Where: BT = RT/b, T is the absolute temperature(^{o}K); R is the universal gas constant (8.314 J/Kmol); A_T is the equilibrium binding constant that corresponds to the maximum binding energy; (L/mg), B_T is related to the heat of adsorption; qe is are the amount of adsorbate adsorbed per unit weight of adsorbent, C_e is equilibrium concentration of adsorbate remained in solution.

Dubinin–Radushkevich isotherm $lnqe = lnqs - (k_{ad} £^2)$…………………… (4)

Where: qe is the amount of adsorbate in the adsorbent at equilibrium (mg/g), K_{ad} (mol^2/kJ^2) and qs (mg/g) are Dubinin–Radushkevich isotherm constants, $£^2$ = potential energy, T is the absolute temperature ^oK and R is the universal gas constant (8.314 J/Kmol).

5 Adsorption kinetics Models

The mathematical models that may describe the behavior of a batch adsorption method operated under completely different experimental conditions are very helpful for scaling up studies or process optimization. Two kinetic models were tested for the simulation of the obtained experimental data ,to have a reasonable adsorption mechanisms for MB and BB dyes.

First model is pseudo first order model which presented in equation (5):

$$\log(q_e - q_t) = log q_e - \frac{k_1}{2.303} t \ldots\ldots\ldots\ldots \quad (5)$$

Where: qe is the amount of dyes that was adsorbed at the equilibrium (mg/g), q_t is the amount of dyes that was adsorbed at time t (min) (mg/g) and k_1 is the rate constant (1/min).

Second model is pseudo-second-order is presented in equation (6):

$$\frac{t}{q_t} = \frac{1}{k_2 qe^2} + \frac{1}{q_e} t \ldots\ldots\ldots\ldots \quad (6)$$

Where: qe is the amount of dyes that was adsorbed at the equilibrium (mg/g), qt is the amount of dyes that was adsorbed at time t (min)(mg/g), k_2 is the pseudo-second-order rate constant of adsorption (g/mg min).

6 Results and Discussion

6.1 Physical Characteristics of NAC

The physical characteristics of the used NAC are summarized at Table.1. It is remarkable that the NAC has surface area of 339 m^2/g and 61.83% carbon.

Table 1: Physiochemical Characteristics of NAC.

Characteristics	Surface area, (m^2/g)	Particle Size, (nm)	Moisture content, %	Density, (gm/l)	Pore Vol. (ml/g)	Carbon, %	Oxygen, %	Silica, %
value	339	5-7	5.24	23.7	0.31	61.83	27.67	6.6

6.2 Batch Adsorption Experiments

6.2.1. Effect of pH on Adsorption Capacity of NAC

Effect of pH is one of the maximum vital parameter on the adsorption capacity due to its effect on both the degree of ionization of dye and surface properties of the adsorbent. Effect of pH on the adsorption capacities of both MB and BB dyes was studied by varying the pH (1, 3, 7, 9 and 11). Figure 1 represents the adsorption capacity (qe) of MB and BB dyes of 300 mg/l concentration as function of pH by using 0.05 g/100 ml NAC at 30 ^oC. The obtained results indicated that the adsorption capacity of MB dye was favorable in acidic medium at pH 3, while in case of BB the adsorption process was favorable in the basic medium at pH 11. As the surface of the AC is negatively charged, the low pH region which considered acid region in which the concentration of H+ ion will increase so this cause neutralization to NAC surface which have negative ion, and therefore reduction in the absorption of the (+ charged) dye cation due to the reduction in the attraction force among the adsorbent and the adsorbate, while in high region of pH, the NAC might be (**-** charged) and the electric double layer formation changes the polarity of it, so the adsorption capacity decreases.

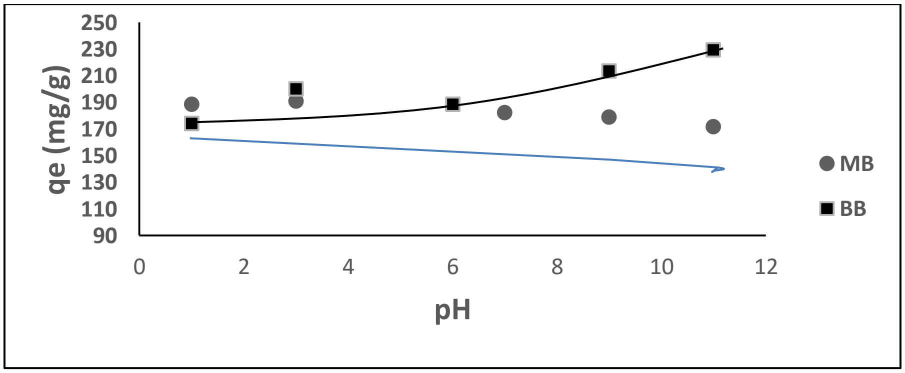

Fig.1: Effect of pH on adsorption capacity of 0.05 dose of NAC for 300 mg/l dyes concentrations at 30 ^{o}C, time 60 minutes.

6.2.2 Effect of Contact Time on Adsorption Capacity of NAC

The capacity of adsorption as a time function by using 0.05 g/100 ml of NAC for three different initial concentrations of MB and BB dyes (100, 200 and 300 mg/l) at 30 ^{o}C, was investigated and plotted in Figures 2 and 3, for determination the minimum contact time to attain equilibrium. From the two Figures, it is remarkable that fast adsorption is observed during the first 15 minutes for both dyes concentrations because of large active sites numbers on NAC surface which attracted the dye particles from the bulk solution, then the adsorption capacity becomes slower from 15 till 60 minutes as active sites decreased and nearly the adsorption becomes constant from 60- 75 minutes which indicated that all available sorption sites had been occupied. Fore that it is clear that equilibrium is attained after 60 minutes for both dyes.

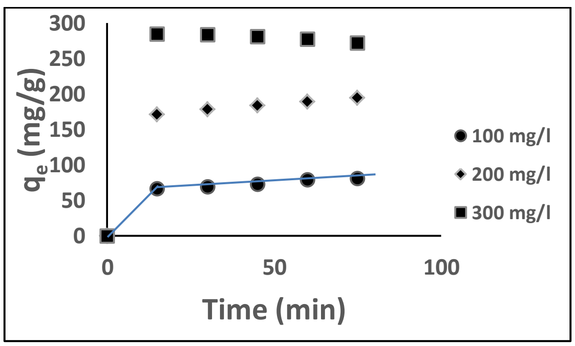

Fig. 2: Effect of contact time on the adsorption capacity of MB dye by using 0.05 mg/100 ml NAC.

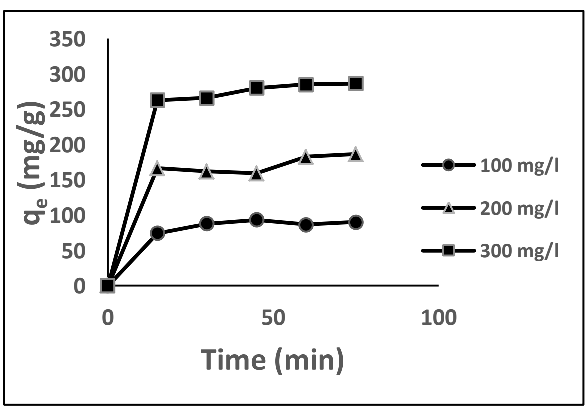

Fig. 3: Effect of contact time on the adsorption capacity of BB dye by using 0.05 mg/100ml NAC.

6.2.3 Effect of Initial Concentration on Adsorption Capacity of NAC

Effect of initial concentrations of MB and BB dyes (100, 200 and 300 mg/l) on adsorption capacity of NAC by using 0.05 g/100 ml dose at best pH are presented in Figure 4. It is observed that the adsorption capacities increase as concentrations increase, so it is recommended to study the use of the prepared NAC for higher dye concentrations in the future.

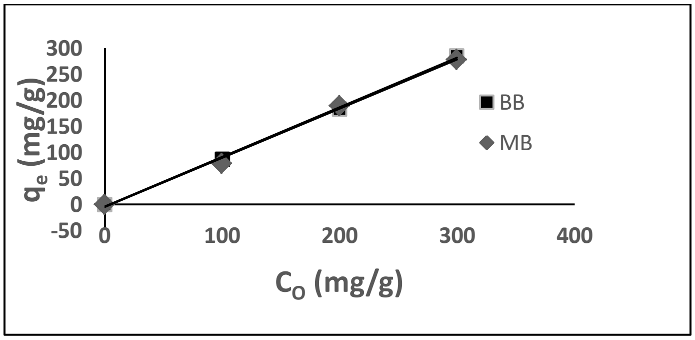

Fig. 4: Effect of different concentration on MB and BB dyes adsorption capacity onto 0.05 mg/100 ml NAC, 30 ^{o}C.

6.2.4. Effect of Temperature

Figures 5 and 6 demonstrate the effect of temperature change from 30 to 60 ^{o}C on the adsorption equilibrium of MB and BB dyes by using 0.05 g/100 ml dose of NAC at different initial concentrations (50-300mg/l.) and at pH 3 for MB and pH 11 for BB. It is observed from both figures that the adsorption process increased with decreasing in temperature. This phenomenon indicated that the equilibrium uptake decreased as the temperature increased. This may be due to the fact that the adsorption reaction is exothermic.

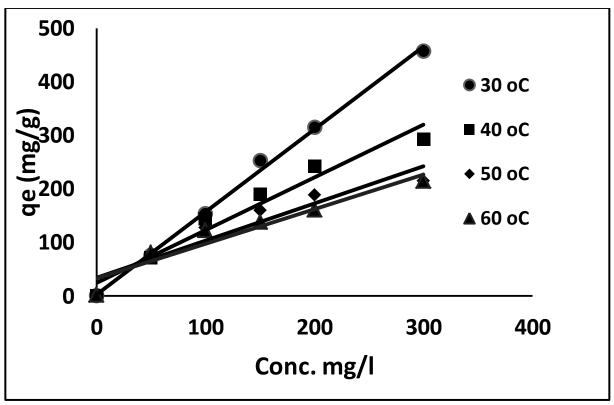

Fig.5 : Effect of temperature on MB dye adsorption Capacity by using 0.05gm/100ml NAC at different initial concentration.

6.3 Equilibrium Isotherms

The results of the four isotherms equations are presented as follows. The calculated parameters for the adsorption of MB and BB dyes have been calculated using the least square method.

6.3.1 Langmuir Isotherm

Using Equation 1, a series of straight lines have been obtained by plotting 1/qe versus 1/Ce for the MB and BB dyes at different temperatures (30, 40, 50 and 60 ^{o}C) and presented in Figures 7 and 8 respectively. The isotherms of MB and BB dyes were found to be linear over the whole concentrations ranges studied.

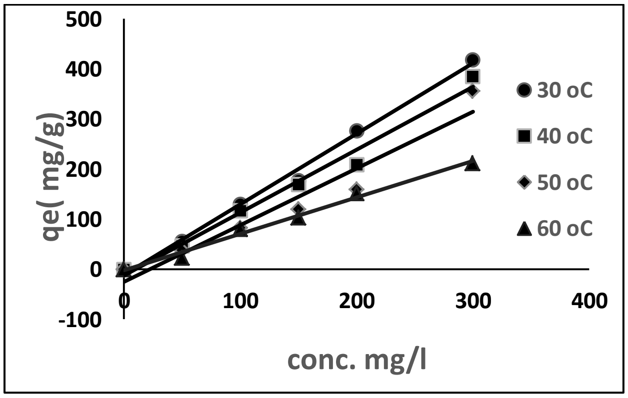

Fig.6: Effect of temperature on BB dye adsorption Capacity by using 0.05g/100 ml NAC at different Initial concentrations.

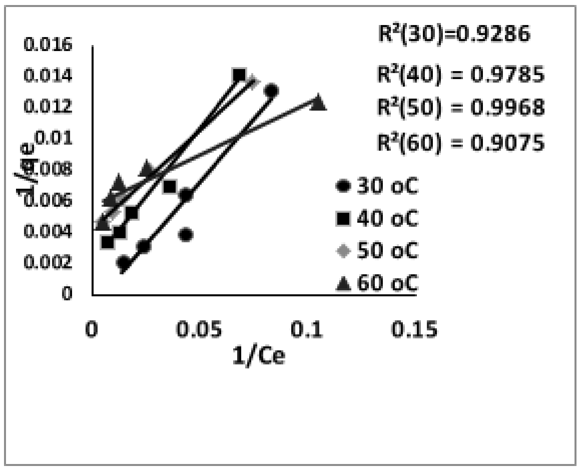

Fig. 7: Langmuir isotherm adsorption model for MB onto NAC at different temp.

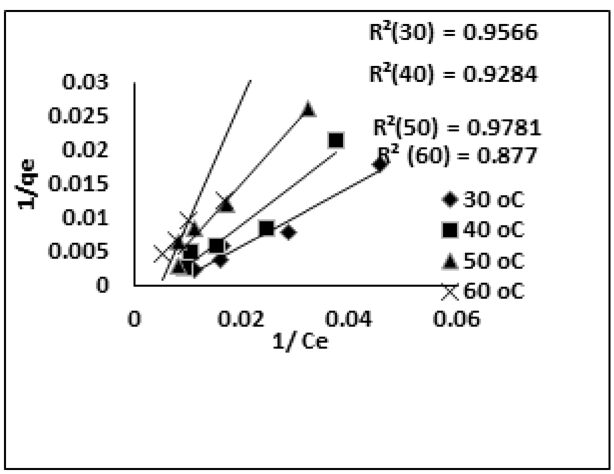

Fig. 8: Langmuir isotherm adsorption model for BB onto NAC at different temp.

Langmuir parameters are depicted in Table 2. The Langmuir $Q_{maximum}$ is decreased as temperature increases from 30 to 60°C for both MB and BB dyes meaning that adsorption capacity enhanced at low temperatures. The maximum adsorption capacities for MB and BB dyes are 1250 mg/g and 370.37 mg/g respectively.

Table 2: Parameters of Langmuir adsorption model for MB and BB dyes onto NAC.

Dyes	Temp, °C	K_{L}, (l/g)	a_{L}, (l/mg)	Q_{max}, (mg/g)	Correlation factor, (R^2)
MB	30	6.20732	4.9658x10^{-3}	1250	0.9286
	40	5.72409	0.010875	526.353	0.9785
	50	7.818608	0.032838	238.096	0.9968
	60	15.15151	0.086363	175.4398	0.9075
BB	30	2.32234	6.27x10^{-3}	370.37	0.9566
	40	1.63345	5.39x10^{-3}	303.0297	0.9284
	50	1.14168	2.854x10^{-3}	399.99	0.9781
	60	0.55108	4.739x10^{-3}	116.27	0.877

The equilibrium parameter (EP) of the Langmuir isotherm can be calculated from the following equation (7) for both MB and BB dyes

$$EP=1/ [1+K_L C_o]…………… ……..……….. (7)$$

Where: EP is the dimensionless equilibrium parameter and Co = initial MB dye concentration

The calculated EP for all temperature are presented in Figures 9 and 10**.** The EPs were <1 for both dyes at different temperatures , fore that Langmuir isotherm model is favorable for both dyes.

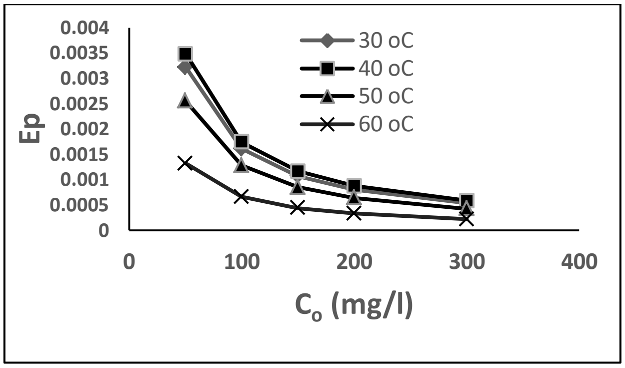

Fig. 9: The calculated dimensionless equilibrium parameter for MB dye at different temp.

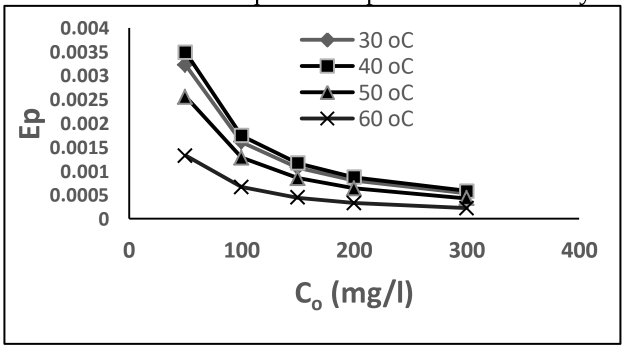

Fig. 10: The calculated dimensionless equilibrium parameter for BB dye at different temp.

6.3.2 Freundlich Isotherm

By using Equation 2, the experimental results of MB and BB dyes have been plotted as log qe versus log Ce, where straight lines have been obtained and presented in Figures 11 and 12 .

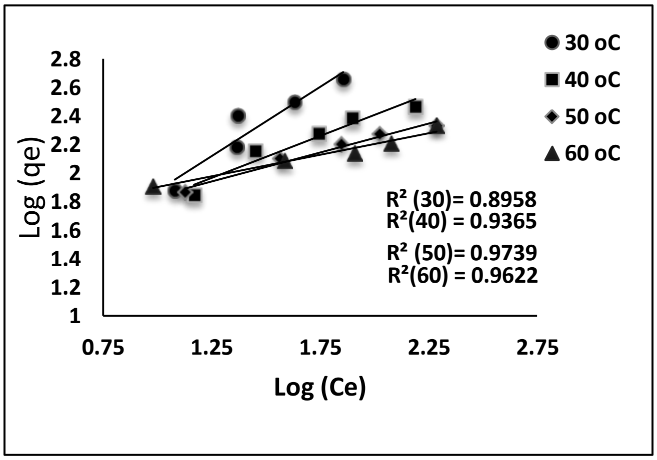

Fig. 11: Freundlich isotherm adsorption model for MB onto NAC at different temps.

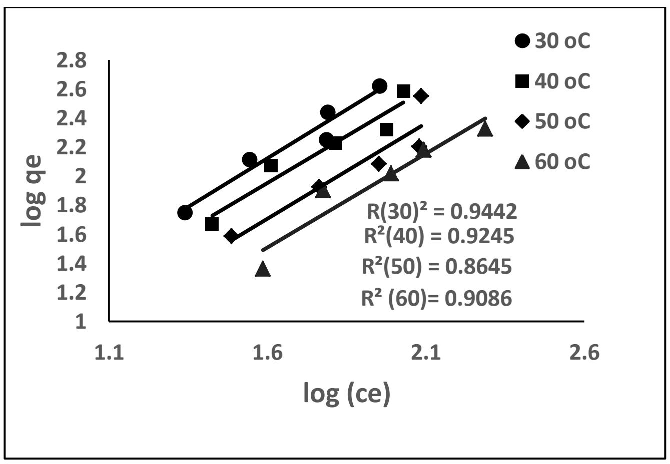

Fig.12: Freundlich isotherm adsorption model for BB onto NAC at different temps.

Freundlich parameters, K_f and n for the adsorption of MB and BB dyes have been calculated and tabulated in Table 3, which proved that the values of the Freundlich exponent, n, are greater than one in case of MB and less than 1 for BB meaning that the system is favorable for MB dye and non-favorable for BB dye.

Table 3: Parameters of Freundlich adsorption model for MB and BB onto NAC.

Dye	Temp, ^oC	K_f	n	Correlation factor (R^2)
MB	30	8.01124	1.0283	0.8958
	40	16.8035	1.6897	0.9365
	50	27.1831	2.4582	0.9739
	60	40.1143	3.3300	0.9622
BB	30	0.9598	0.7470	0.9442
	40	0.7787	0.7756	0.9245
	50	0.39976	0.7597	0.8645
	60	0.27422	0.77285	0.9086

6.3.3 Temkin Isotherm

The experimental results by using Equation 3 have been plotted as qe versus ln Ce, where straight lines have been obtained as shown in Figures 13 and 14 for MB and BB dyes respectively.

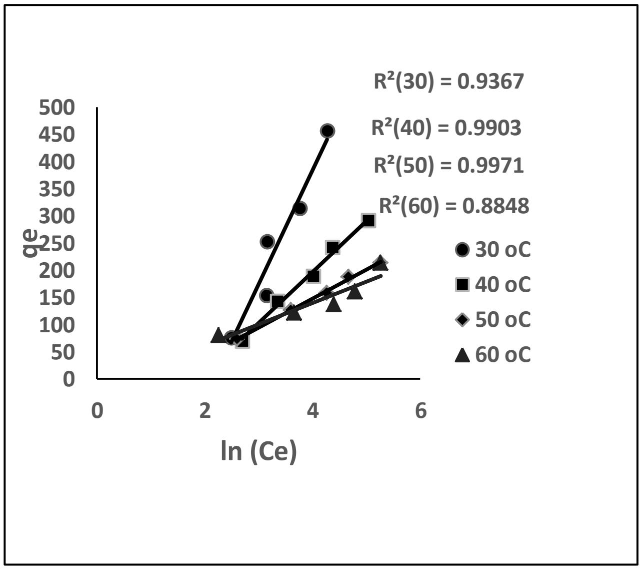

Fig.13: Temkin isotherm adsorption model for adsorption of MB onto NAC at different temp.

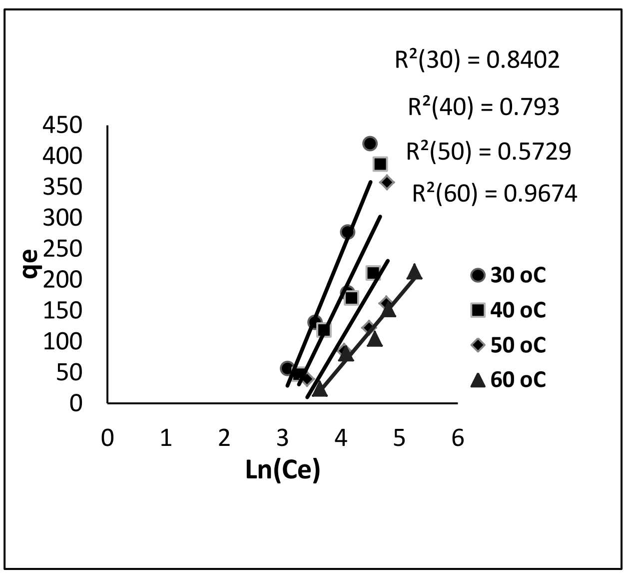

Fig.14: Temkin isotherm adsorption model for adsorption of BB onto NAC at different temp.

The calculated Temkin parameters, B_T and A_T for the adsorption of MB and BB dyes have been calculated and are illustrated in Table 4 . The positive values of adsorption energy (B_t) suggest that the process is exothermic, that's contrary to the results. In case of MB dye Temkin isotherm model is favorable for all ranges of temperatures (30-50 ^oC) except at 60 ^oC as proved by the correlation coefficient (0.8848), while in case of BB dye Temkin isotherm model is not favorable for all ranges of temperatures (30-50 ^oC) except at 60 ^oC where the correlation coefficient is 0.9674.

Table 4 : Parameters of Temkin adsorption model for MB and BB dyes onto NAC.

Dye	Temperature °C	A_t	B_t	Correlation factor (R^2)
MB	30	0.1138	210.12	0.9367
	40	0.14927	94.081	0.9903
	50	0.2913	53.515	0.9971
	60	0.6422	39.448	0.8848
BB	30	0.05169	232.06	0.8402
	40	0.04395	194.73	0.793
	50	0.0344	160.43	0.5729
	60	0.03119	112.37	0.9674

6.3.4. Dubinin–Radushkevich Isotherm Model

The experimental results by using Equation 4 have been plotted as **£²** versus lnqe, where straight lines have been obtained as shown in Figures 15 and 16 for both dyes .

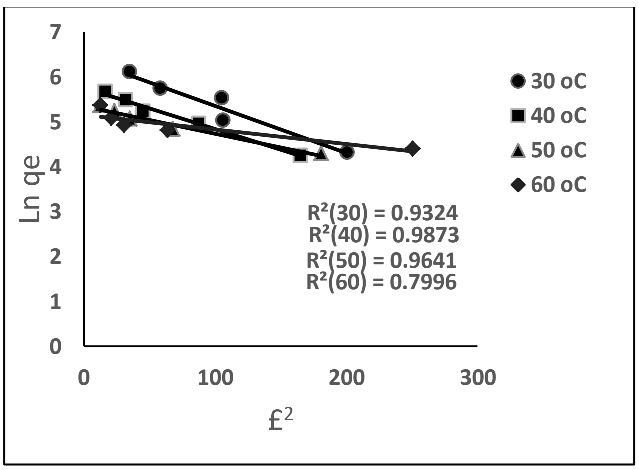

Fig. 15: Dubinin–Radushkevich adsorption model for adsorption of MB at different temp.

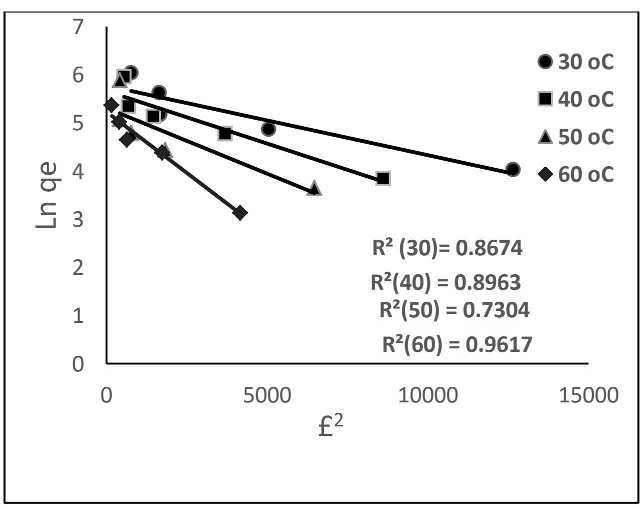

Fig. 16: Dubinin–Radushkevich adsorption model for adsorption of BB onto NAC at different temp.

The calculated Dubinin–Radushkevich parameters, q_s, k_d and E for the adsorption of MB and BB dyes have been calculated are illustrated in Table 5. It is remarkable from the values of energy that the adsorption type can be explained by physisorption. The adsorption isotherms parameters are presented in Table 5, where Dubinin Radushkevich coefficient correlation R^2 indicated that for MB dye the model is favorable for all ranges of temperatures except for 60 ^oC, the correlation coefficient was 0.7996. While in case of BB dye the model is favorable only at 60 ^oC.

Table 5: Parameters of Dubinin–Radushkevich adsorption model for MB and BB dyes onto NAC.

Dye	Temperature ^oC	q_s (mg/g)	k_d $(1/(J/mol)^2)$	Correlation factor (R^2)	E (J/mol)
MB	30	0.989	6.4196	0.9324	0.279
	40	0.9908	5.7624	0.9873	0.294
	50	0.9939	5.3497	0.9641	0.3057
	60	0.9968	5.1588	0.7996	0.3113
BB	30	0.9999	5.7794	0.8674	0.294
	40	0.9998	5.6677	0.8963	0.297
	50	0.9997	5.3067	0.7304	0.3069
	60	0.9995	5.2341	0.9617	0.3091

Table 6 represents the conclusion of the previous adsorption isotherm four models, where Langmuir and Freundlich isotherm models are favorable for both dyes in the temperature ranges of 30- 50^{0}C.

Table 6: Comparison between the adsorption isotherms models for MB and BB dyes onto NAC.

Model / Dye	Langmuir isotherm model	Freundlich isotherm model	Temkin isotherm model	Dubinin–Radushkevich isotherm model
MB	Favorable for all temperatures	Favorable for all temperatures	Favorable for all temperatures except at 60 ^oC	Favorable for all temperatures except at 60 ^oC
BB	Favorable for all temperatures except at 60 ^oC	Nonfavorable for all temperatures	Favorable only at 60 ^oC	Favorable only at 60 ^oC

6.4 Adsorption kinetics Models

6.4.1 Pseudo First Order Model

Figures 17 and 18 represent the relationship between log (q_e – q_t) and time (t) for MB and BB dyes onto NAC. It is observed that the correlation coefficient of MB and BB dyes are 0.2486 and 0.7958 respectively, which are small and indicates that the pseudo first order equation is not fit the behavior of MB and BB adsorption.

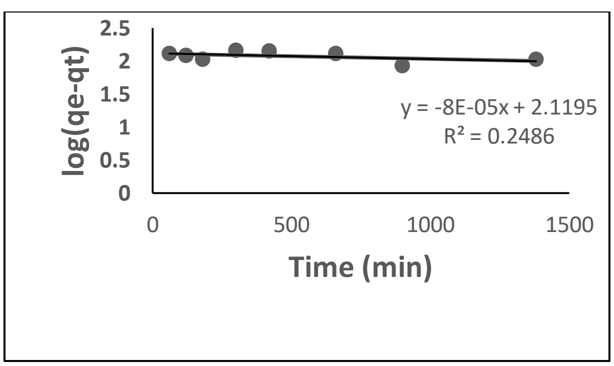

Fig. 17: First order kinetic model for MB dye adsorption onto NAC.

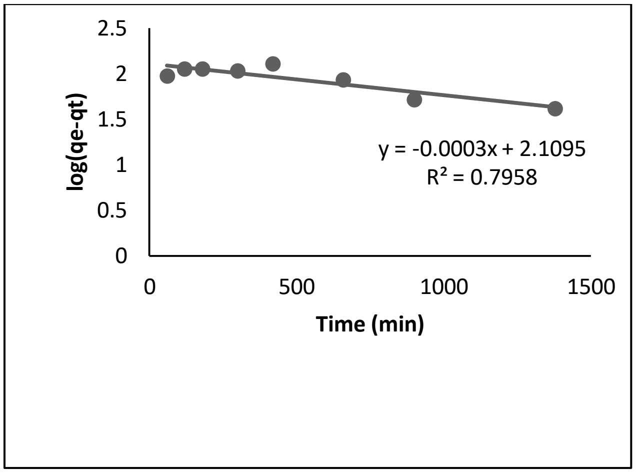

Fig. 18: First order kinetic model for BB dye adsorption onto NAC.

6.4.2 Pseudo Second Order Model

By plotting t/q_t verses t as presented in figure 19 and 20 for MB and BB dyes where the correlation coefficients were 0.9951and 0.9914 respectively which are high, depicted that the pseudo second order equation fit well to the whole range of contact time for both dyes.

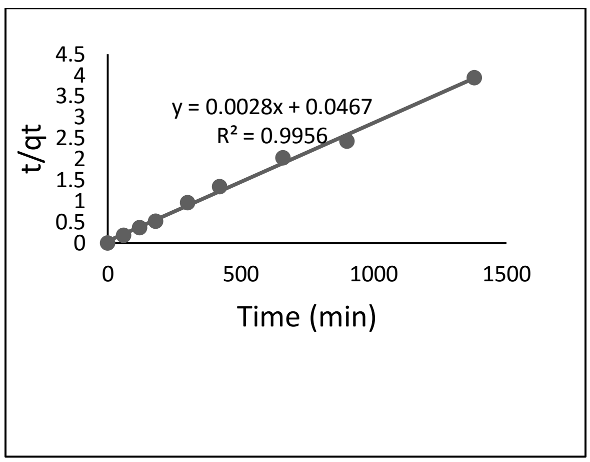

Fig.19: Second order kinetic model for MB dye adsorption onto NAC.

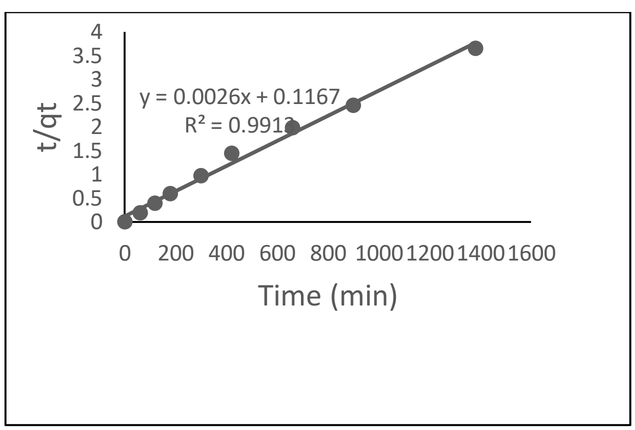

Fig.20: Second order kinetic model for BB adsorption onto NAC.

Table 7 summarizes the kinetic parameters of both models for MB and BB dyes. It can be concluded that pseudo second order mechanism is fitted for both MB and BB dyes and applicable for this system.

Table 7: Kinetic parameters for adsorption of dyes onto NAC at 30 ^oC.

Dye	Pseudo First order		Pseudo Second Order	
	K, l/min	R^2	K_2, l/min	R^2
MB	$-8x10^{-5}$	0.248	0.00467	0.9956
BB	-0.0003	0.795	0.0046	0.9913

Conclusions

Batch adsorption experiments were carried out to remove MB and BB dyes from synthetic solutions. The high adsorption capacity achieved at pH 3 for MB and at pH 11 for BB at 30^{O}C after 60 minutes. Equilibrium isotherms have been studied for the prepared NAC adsorbate. Four models were investigated: Langmuir, Freundlish, Temkin and Dubinin–Radushkevich. Freundlich isotherm models was favorable for both dyes in the temperature ranges of 30- 50^{0}C. The results showed that the uptake rate of the NAC adsorbate obeyed pseudo second order kinetic model for both dyes.

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