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Biochar from waste verbena foumanat as an adsorbent to remove phenol from aqueous solution

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ABSTRACT

This paper studies the efficiency of biochar prepared from waste verbena foumanat (BWV) to adsorb phenol from aqueous solution. The waste verbena foumanat were collected from cafeterias, after then it was washed and dried at 70 °C. The dried verbena carbonized in muffle furnace at 800 °C. The characterizations of BWV before and after sorption were determined by using scanning electron microscopy (SEM) and Fourier transform infrared (FTIR). Batch adsorption process were conducted at contact time (0 – 120 min), BWV amount (0.2 to 1 gl^{-1}), phenol initial concentration (25 - 150 mgl^{-1}), pH solutions (2 – 12) and temperature (20 – 50°C). The adsorption isotherm and the kinetic models were used in these investigations to fit data analysis BWV equilibrium data was fitted by Freundlich isotherm model. Although, a pseudo-second order was the better model to fit the adsorption kinetic. The maximum adsorption capacity calculated was 43.29 mgg^{-1}. The following investigation indicates that biochar from waste verbena (BWV) is an effective adsorbent with an important adsorption capacity. It could be used as bio sorbent to eliminate phenol from industrial wastewater.

Key words: *Phenol, Adsorption, Aqueous solution, Waste verbena, Biochar, Pollutant.*

Introduction

The chemical compound phenol is an aromatic organic hydrocarbon. It is considered as a most important organic pollutants found in refining, petrochemical and gas treatment industries (Jonidi *et al.*, 2019). However the phenol molecules were synthesised with waste water industrials, after then it was rejected on the sea. Unfortunately some amounts of these chemicals products (phenol) containing in water are dangerous for environment and effect in the life cycle of our planet. Whereas, its can effect on human health by causing carcinogenic and nervous system illness (Nirmala *et al.,* 2021). In the last years the environmental researches study to eliminate organic pollutants from waste water with an efficacy and green methods, such as adsorption, photocatalytic degradation, extraction and membrane (El-Naas *et al*., 2010).

The adsorption by waste agriculture was a popular method used by researches to treat waste water from aromatic hydrocarbon. It was considered as a safety method for environment. Many agriculture waste were used as an adsorbent such as; waste coffee (Jeon, 2017), bamboochips (Li*et al*., 2016), Banana Peel (Maia *et al*., 2021), citrus limetta peel (Mondal *et al*., 2019), Moringa seeds (Nonfodji *et al*., 2019), *Enteromorpha prolifera* (Li *et al*., 2020), *Carica papaya*

wood (Rangabhashiyam *et al.*, 2018). The pyrolysis of waste agriculture at high temperature is method to fabricate an effective biomaterial (biochar) to use it as a bio-adsorbent. This biochar have high surface area, high carbon contain, important porosity and high selectivity (Devi and Saroha, 2015). Whereas, it can hence the adsorption efficiently of industrial organic pollutants from waste water.

The phenol is the most organic pollutant rejected in the wastewater by different industrials petroleum. Many paper studies the sorption of phenol form aqueous solution. Lütke *et al.* (2019) and Mandal *et al.* (2020) studied the elimination of Phenol by black wattle barkwasteandneem leaves, respectively. Although, Ingole *et al.* (2017) used banana peels activated carbon to adsorb phenol. Rana *et al*. (2017) used orange peel and tea waste to remove phenol.

Verbena plant is popular and healthy drink for Algerians people, can be used as a traditional medicines for stomach illness and thermal stability (Abderrahim *et al.*, 2011). It sells in the supermarkets, pharmacies and cafeterias as a foumanat. The waste of verbena foumanat can be used to prepare a biochar by heating this waste biomass at high temperature. This biochar was considered as a green adsorbent to eliminate organic pollutant from waste water.

This paper studies the removal of organic pollutants (phenol) form solution aqueous. The absorbent were prepared by pyrolysis the wast everbena foumanat (WV) at high temperature 800 °C to obtain an effective biochar withan important surface area. The effects of initial concentration, absorbent dose, time of contact and pH solution were carried out to study the sorption of phenol from waste water by biochar from waste verbena (BWV). The isotherm models (Langmuir and Freundlich) at equilibrium and kinetic study were carried out to examine the capacity of BWV to remove phenol from wastewater industrials.

Materials and Methods

Solution preparation

The organic chemical phenol was used as pollutant in this work, molar masse; 94.11 $gmol^{-1}$. The molecular structure was C_6H_6O. It was purchased from Biochem laboratory, tortola-bri- United Kingdom. The phenol solutions were prepared by dissolving 1 g of phenol in 1 liter of demineralised water. The diluted solution (1000 mgl^{-1}) prepared was stored in glass container. Hydrochloride acid (HCl) and sodium hydroxide (NaOH) purchased from sigma-aldrichlaboratory, India. It was used to prepare different pH solutions (0.1M).

Biochar preparation

Waste verbena foumanat (WV) was collected from cafeterias to prepare abiochar. WV was washed with distilled water to eliminate dust. Then it was boiled with demineralised water for 30 min several times until the green colour of verbena was removed. The boiled verbena was dried in an oven at 70 °C for 24 h to eliminate humidity. After then the dried verbena was introduced in crucible ceramic to heat in muffle furnace at different temperature. The sample was pyrolysis at 400, 600 and 800 °C for 1h in each temperature with 5 °C/min raw rat. The biochar from waste verbena foumanat (BWV) was ground using mortar (MICROTRON SYSTEM MB 800) and then sieve using 0.08 mm sieve mesh. The final material was stored in glass airtight container for following use.

Batch absorption

The removals of phenol from aqueous solution by BWV were studied by batch method. The batch absorption were carried out by varying the contact time (0 – 120 min), BWV amount (0.2 to 1 gl^{-1}), phenol initial concentration (25 - 150 mgl^{-1}), pH solutions (2 – 12), the temperature (20 – 50 °C) and the speed shaker was fixed at 300 tr min^{-1} in all experiment. All samples were conducted by mixing the phenol compounds with BWV adsorbent in an Erlenmeyer flask (100 ml). Each sample was filtered to separate saturated BWV adsorbent from aqueous solutions. The filtration was carried out by using sieve paper mesh 3. The equilibrium concentrations of phenol solutions were determined by using UV-vis spectrometer at 270 nm wave lengths. All experiments were repeated twice, to find a precise value of equilibrium concentrations. These experiments values were used in the following paper to study the efficiently of BWV adsorbent to eliminate phenol from aqueous solutions. The phenolamount (q_m) and percentage (R) removal by BWV adsorbent at equilibrium were determined by equation (1) and (2), respectively.

$$q_m = (C_0 - C_{eq})\frac{V}{m}(1) R = \frac{C_0 - C_{eq}}{C_0} 100 \quad .. (2)$$

Where

C_0: the initial concentration of phenol (mgl^{-1})

C_e: the concentration of phenol at equilibrium (mgl^{-1})

m: BEDB adsorbent weight (g)

Analysis methods

The concentrations of phenol solutions at equilibrium were measured using UV-vis spectrophotometer (model: Optizen 3220 UV), scanning electron microscopy SEM (model: Hitachi TM 100) was used to capture a morphology image of biochar from waste verbena before and after phenol sorption. BWV functional groups before and after adsorption process were detected using Fourier transform infrared FTIR spectrometer (model: Agilent technologies cary 600).

Results and Discussion

Surface morphology of BWV

The Figure 1 illustrates the image morphology of biochar from waste verbena before and after phenol sorption. The magnification is 4000-time. Before adsorption (Figure 1a), the structure of biochar shown as porous form whereas high number of active sites were free, this structure allows organic pollutant to penetrate inside these active pores and enhance the adsorption of phenol molecules (Girish *et al.*, 2017). After phenol sorption (Figure 1b) the structure of BWV was shown impermeable whereas the pores were covered by phenol molecules, this due to the adsorption process, where the free pores of BWV adsorb phenol molecules.

FTIR spectrum of BWV

The Figure 2 presents FTIR/kbr spectrum of BWV before and after phenol sorption. From the trend many peaks transmittance of BWV were detected at different wavelength, which are 3392, 1577, 1418, 1350, 1194 and 1040 cm^{-1}. Although, another transmittance are shows between 900 – 700 cm^{-1}range. The peaks at 3392, 1350, 1194 and 1040 cm^{-1} are assigned to hydroxyl groups (OH stretching). Furthermore, the peaks at 1577 and 1418 cm^{-1} are attributes to C=C and N=N bending, respectively. The last absorbance peaks between 900 – 700 cm^{-1}, due to aromatic stretching. After adsorption process, a new

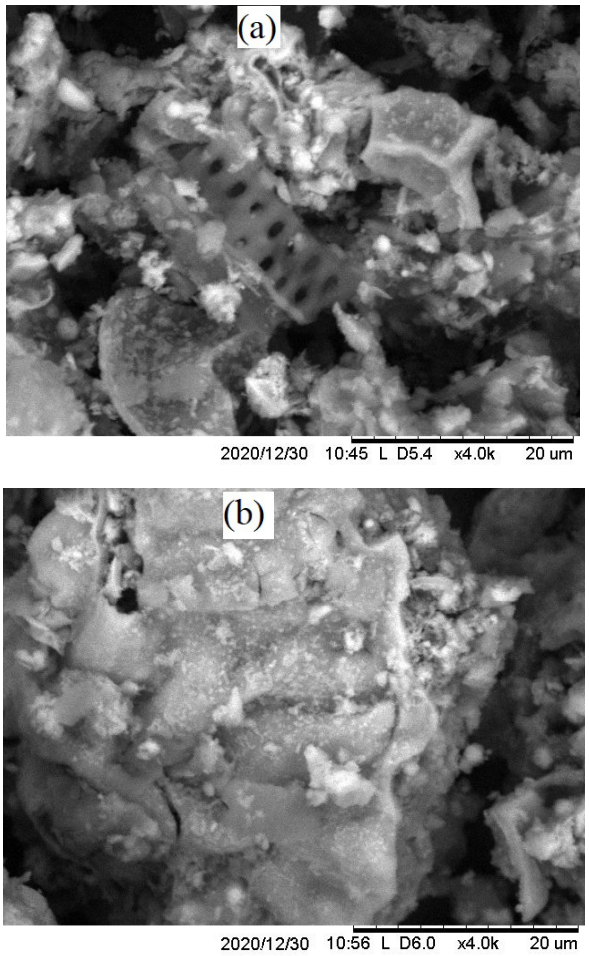

Fig. 1. Surface morphology of BVW with 4000 time magnification, (a) before absorption, (b) after phenol absorption

transmittance peaks were appeared, which are 2573 and 1680 cm^{-1} wavelength, are due to carboxyl groups and aromatic bending, respectively. However the transmittances at 1194 and 1350 cm^{-1} were disappeared after phenol sorption, this because to the attachment of BWV functional groups with phenol molecules and shifting new peaks absorbance. Whereas the intensity peaks at 2513 and 1680 cm^{-1}were decreased, this can be explained by the reaction between BWV groups and phenol.

Effect of contact time on phenol adsorption

The effect of ccontact time on phenol sorption by BWV adsorbent were shown in Figure 3, the experiments were carried out at different contact time (10 – 120 min) and by varying the initial concentration (75, 50, 150 mgl^{-1}). The adsorption capacity increase

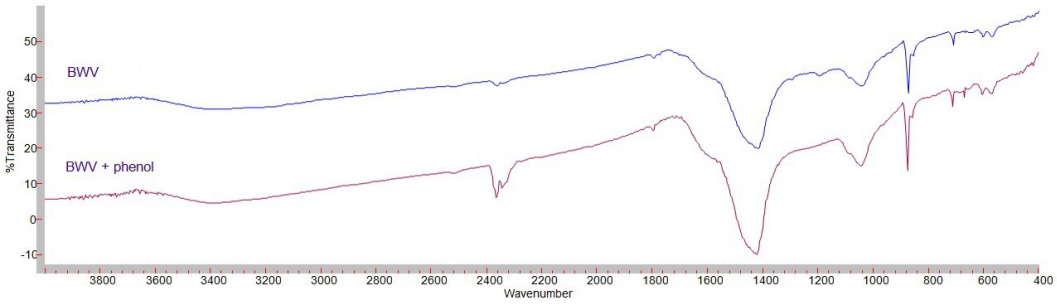

Fig. 2. FTIR/kbr spectra of BWV before and after adsorption.

rapidly from 0 min to 15 min for each initial concentrations, this because as the availability of BWV surface active (Li *et al*., 2018). Then the phenol removal was increase slowly from 15 to 75 min, this due to the few free active sites of BWV (Rawat and Singh, 2019). After then the adsorption process reach the equilibrium for all initial concentration, this can be explain by the all free active surface of BWV were occupied by phenol molecules.

Effect of BWV amount on phenol sorption

The graphs in the Figure 4 present the effect of adsorbent amount on phenol sorption by BWV. The parameters which; pH 6, T=25°C and contact time 60 min were used in these experiments. According to the phenol removal Trend, the percentage eliminate increase from 0 to 45% with increasing in BWV dose (0 to 1 gl^{-1}), these results can be explain by more free sites active were available, the later was the main raison to increase the percentage of phenol eliminate from aqueous solution (Gautam *et al*., 2018). However, from the second trend, the adsorption capacity increases quickly from 0 to 22.8 mgg^{-1} with increasing in the BWV quantities. Then the phenol amount eliminate was decreased slowly with increasing in adsorbent dose, this due to the desorption phenomena between BWV adsorbent and phenol molecules.

Effect of initial concentration on phenol elimination

The graph 5 illustrates the effect of initial concentration of phenol removal by BWV adsorbent. The experiments were carried out at pH 6, T= 20 °C and 60 min of contact time. From the trend the percentage of phenol removal was increase from 23 to 28 % with increasing in initial concentration. After then the yield of phenol was decrease from 28 to 14% with increasing of initial concentration from 75 to 150 mgl^{-1}, this due to all surface active of biochar from waste verbena were occupied by phenol molecules (Mandal *et al.*, 2019). The highest percentage removal is at 75 mgl^{-1} which is 29%, this is because the transfer mass process between the surface active of BWV and phenol molecules (Khan *et al*., 2019).

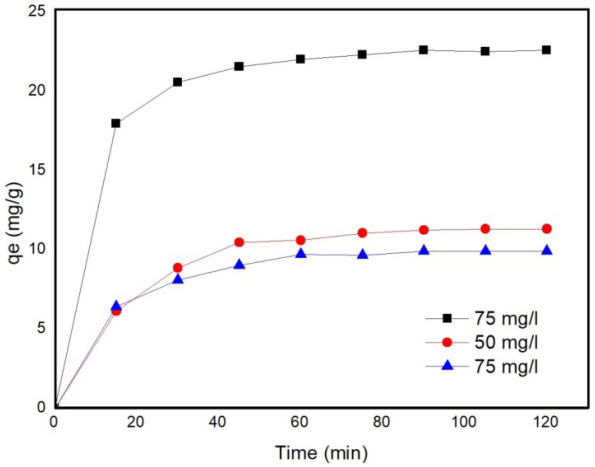

Fig. 3. Effect of contact time on phenol removal onto BWV

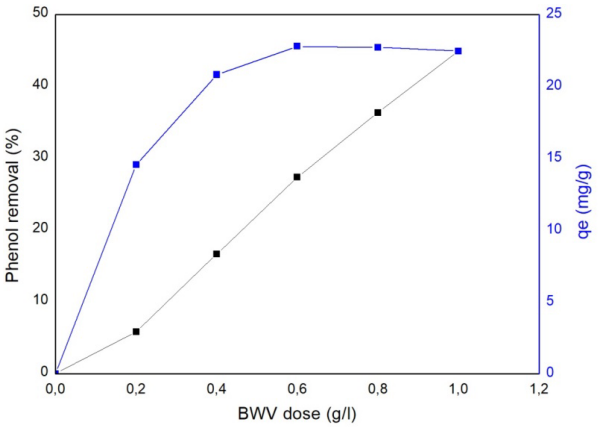

Fig. 4. Effect of BWV dose on phenol removal

Effect of pH solutions on phenol removal

The data in the Figure 5 illustrates the effect of pH solutions to remove phenol by biochar from waste verbena (WBV). The adsorption process was carried out with 1gl^{-1} of biochar, the contact time was 60 min and T = 20 °C. It's clearly shows at pH 2 the percentage of phenol adsorbed by BWV was high which is 39%, this because in the acidic solutions the surface of bio adsorbent has a negative charge (OH^-) which allowed the interaction between phenol ions and adsorbent surface (Qiao *et al.*, 2018). However the increasing in the pH solution from 4 to 12 decrease the efficiently to remove phenol molecules from aqueous solutions, this due to the surface charge of biochar become positive (H^+) in the basic solutions, this decrease the interaction between phenol molecule and BWV. Whereas the efficiently of adsorption was decrease.

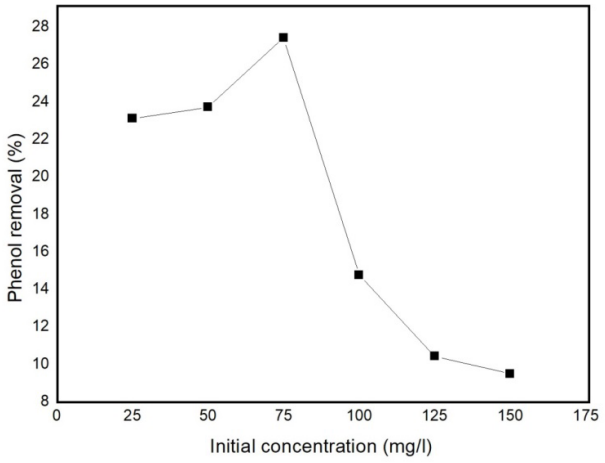

Fig. 5. Effect of initial concentration on phenol removal onto BWV

Effect of temperature on phenol removal

The effect of temperature on phenol adsorption was illustrated in the Figure 7. The experiments were carried out by varying the temperature from 20 to 50 °C, pH 6, C_0=75 mgl^{-1}, and BWV dose 0.1 gl^{-1} From the trend the adsorption of phenol by biochar from waste verbena was decrease with increasing in the temperature from 11.5 to 10.5 mgg^{-1}; this can be explain by the kind of sorption process which is exothermic (Nirmala *et al.*, 2021).

Adsorption isotherm

The analysis data of BWV adsorbent on phenol removal from waste water were studied by using two isotherm models, which are Langmuir and Freundlich. These isotherms evaluate the efficiently of present biochar to adsorb phenol molecule from aqueous solutions. Langmuir model assume a monolayer sorption and the surface of adsorbent was homogeny (Mohammed *et al.*, 2015). The equation was given by

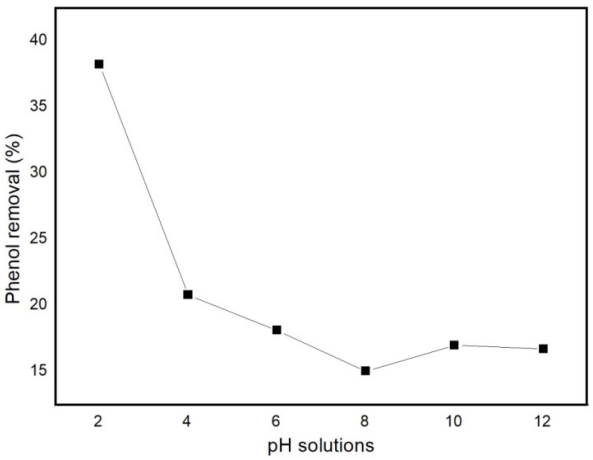

Fig. 6. Effect of pH solutions on phenol removal onto BEDB

$$\frac{Ce}{qe} = \frac{1}{q_m K_l} + \frac{Ce}{qm} \quad .. (3)$$

Where,

q_e and q_t are the Adsorption capacity of BWV adsorbent at equilibrium and t (min), Ce is the concentration of phenol solution at equilibrium (mgl^{-1}), K_L is Langmuir constant (Lg^{-1}) and Qm is the maximum adsorption capacity (mgg^{-1}).

However, Freundlich model assume a multilayer

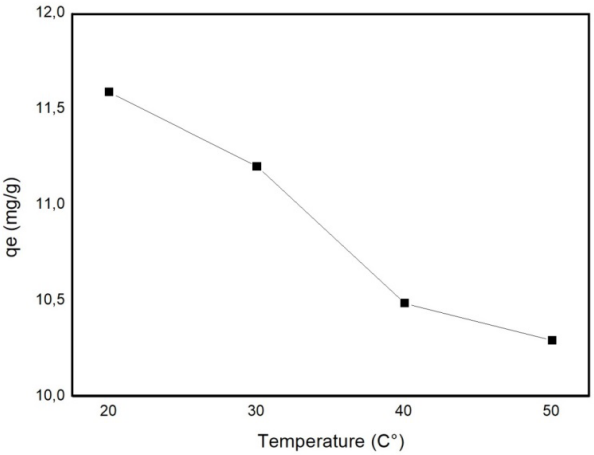

Fig. 7. Effect of temperature on phenol removal onto BEDB

adsorption with heterogeneous surface of adsorbent (Šoštaric *et al.*, 2015). The isotherm was presented by the following equation (Eq 04)

$$q_e = K_F\, C_e^{1/n} \quad .. (4)$$

Where K_F is the Freundlich constant, C_e is the concentration of solutions at equilibrium and 1/n is the intensity of adsorption process.

Figure 8.a present Langmuir Isotherm plot Ce/qe against Ce, the plot is clearly shows as straight line for each initial concentrations (75 and 150mgl^{-1}). The adsorption capacity maximal (Qm) and Langmuir coefficient (K_L) were calculated using the slop and the interception of these plots. The figure 8.b illustrate Freundlich isotherm trend ln (qe) versus ln (ce), the straight line as shows in the plot, can be used to determine Freundlich constant (K_F) and the adsorption intensity (1/n). The regression coefficient R^2 of each isotherms, were calculated from the plots. This coefficient evaluates the model which is better than other to fitted adsorption process. The calculate values of Langmuir and Freundlich isotherms were presented in the Table 2. According to R^2 values which shows in Table 2, the Freundlich isotherm model was evaluated the adsorption of phenol by BWV better than Langmuir model, this can be explain by the heterogeneous surface of BWV adsorbent (Šoštaric *et al.*, 2015).

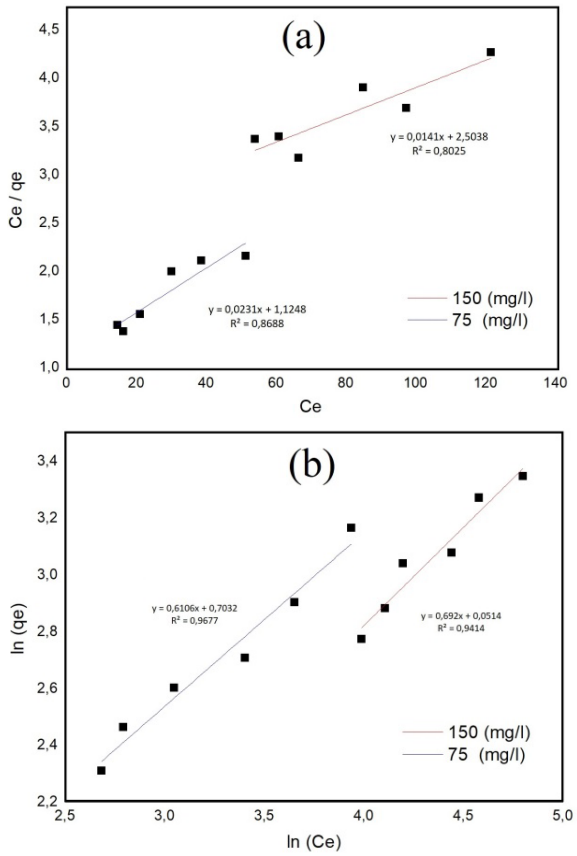

Fig. 8. (a)/(b) Langmuir/ Freunlich isotherms for phenol adsorption onto BWV

kinetic study

The kinetic study was carried out to evaluate the performance of BWV adsorbent to eliminate phenol from aqueous solutions. The analysis was used two kinetic models, which are pseudo first and pseudo second order and were given in the following expressions (Eq 05) and (Eq06), respectively.

$$\ln\,(q_e - q_t) = -\frac{K_1}{2{,}303}\, t + \ln q_e \quad .. (5)$$

$$\frac{t}{q_t} = \frac{1}{q_e}\, t + \frac{1}{K_2 q_e^2} \quad .. (6)$$

Where, qe and qt are the phenol quantities

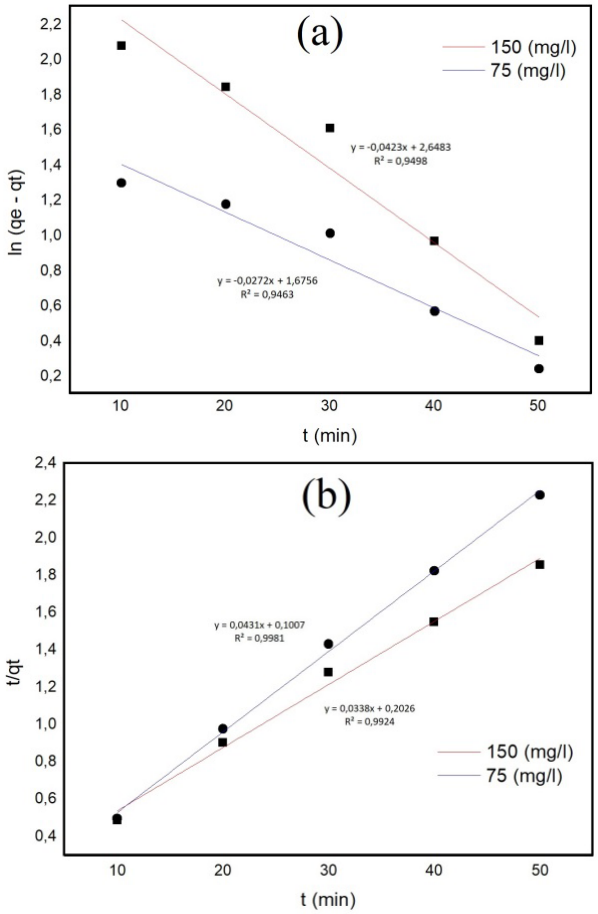

Fig. 9. (a) /(b) Pseudo-first/second order kinetic for phenol adsorption onto BWV

Table 1. Langmuir and Freundlich parameters for phenol removal onto BWV

	Langmuir			Freundlich		
	q_m	K_L	R^2	K_F	n	R^2
75 mgl^{-1}	43.29	0.020	0.8688	2.02	1.36	0.9677
150 mgl^{-1}	70.92	0.005	0.8025	1.05	1.44	0.9414

Table 2. Pseudo-first / second order kinetic parameters for phenol removal onto BWV

	Pseudo-first-order				Pseudo-Second-order		
	q_{exp}	q_{cal}	K_1	R^2	q_{cal}	K_2	R^2
75 mgl^{-1}	23.30	5.34	0.063	0.9463	23.20	0.0184	0.9981
150 mgl^{-1}	27.69	14.13	0.097	0.9498	29.58	0.0056	0.9924

adsorbed at equilibrium and t, respectively. K_1 and K_2 are the pseudo first and pseudo second order constants ($min^{-1}.g.mg^{-1}min^{-1}$), respectively.

The graph in the Figures 8a and 8b analysed pseudo first and pseudo second order experiment, respectively. It's clearly Shows the plot of kinetic models ln (qe-qt) versus t and t/qt against t were straight line. According to the slop and interception of each kinetic isotherms, different experimental parameters were obtained, which are qe, K_1, K_2 and R. The results of these investigations were presented in the Table 2.

The regression coefficients R^2 of pseudo first order are 0.90 and 0.94 for 75 and 150 mgl^{-1}, respectively, these results conducted the first order is not well to fit this adsorption. However the R^2 of pseudo second order are 0.99 for each initial concentrations, this mean the pseudo second order is the good isotherm to fitted phenol removal by BWV adsorbent from aqueous solutions. Furthermore the experimental and the calculated values of phenol amount adsorbed for pseudo second order are convergent, compared with pseudo first order are divergent (Nirmala *et al.*, 2021).

Conclusion

In this investigation, the biochar from waste verbena foumanat (BWV) was prepared as an adsorbent to eliminate phenol molecules from aqueous solution. The surface morphology and the functional groups were detected by SEM and FTIR, respectively. Batch adsorption experiments were carried out at different parameters, which including; time, BWV dose, initial concentration, pH solution and temperature. The maximum percentage of phenol removal is 45% at m = 0.6 gl^{-1}, T = 30 C° and C_0 = 50 mg/l. Freundlich isotherm was the best model to present the adsorption data of phenol by BWV sorbent. Pseudo second order was the better isotherm to describe the kinetic study than pseudo first order model. The monolayer adsorption capacity for BWV sorbent was 43.29 mgg^{-1}. According to the results, the proposed BWV can be used as an effective adsorbent to eliminate phenol from industrial wastewater. Whereas, it was considered as green and friendly for environment.

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