



Comparison of the Efficiency of Local Bentonites and Activated Carbon for Removing of Phenol from Waste Water

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Article information

Article history:

Received: November, 17, 2025

Accepted: February, 14, 2026

Available online: June, 14, 2026

Keywords:

Bentonite clay,

Activated carbon,

Phenol adsorption

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DOI:

<https://doi.org/10.53523/ijoirVol13I1ID628>

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Abstract

The aim of this study is the assessment of Iraqi local bentonite clay (Be) as an adsorbent for the removal of phenol from laboratory wastewater, where activated carbon (AC) was used for the same target as a conventional adsorbent under different experimental variables. Moisture content, loss of ignition (%), and FTIR tests were done to characterize bentonite clay. The batch adsorption process was carried out using AC and Be under experimental conditions with adsorbent dosage (0.1–1) g at an initial phenol concentration of 100 mg/L to study the dosage effect, while the initial concentration effect at constant adsorbent dosage was (20–100) mg/L with a dosage of 1 g of Be and (100–1000) mg/L with a dosage of 0.1 g of AC and 240 min contact time. A kinetic study of the contact time effect (15–240) min was conducted at an initial phenol concentration of 50 mg/L for Be and 250 mg/L for AC, for a constant volume of 500 mL. The results show removal percentages of 45.58% and 100% of phenol at 1 g Be and 0.4 g AC, and adsorption capacities at variable initial phenol concentrations were 1.282 mg/g and 138.31 mg/g using Be and AC, respectively. Adsorption isotherm models show good fitting with the Langmuir model for the AC/phenol system, with an R^2 value of 0.987 and a theoretical maximum adsorption capacity of 142.857 mg/g, while the Be/phenol system shows slightly better fitting with the Freundlich model, with an R^2 value of 0.981. The kinetic study shows good fitting with the pseudo-second-order model, with R^2 values of 0.994 for both Be and AC, which refers to chemical adsorption.

1. Introduction

One of the main problems of surface and groundwater pollution is domestic and industrial wastewater that is discharged directly into rivers, as it is often untreated or partially treated due to the lack of treatment plants or their inefficiency [1]. This problem was reported by the United Nations Educational, 2015, in many countries, especially Iraq. Liquid wastes that are discharged directly or indirectly into water bodies often cause serious problems for living organisms [2] because they are untreated or insufficiently treated. There are many pollutants in wastewater [3], most of which are minerals, nutrients, hydrocarbons, heavy elements, pesticides, and others [4]. Hydrocarbons are among the most important sources of energy in the current era, and because of increasing industrial activities and the world's needs, contamination by hydrocarbons occurs [5] due to toxic organic substances, petroleum [6], and pesticides, which is a serious concern for the environment. Numerous sources,

such as pesticides, petroleum, or different harmful organic substances discharged into water streams as effluents, are responsible for hydrocarbon pollution in wastewater [7]. Water contaminated with hydrocarbons is known to be carcinogenic, neurotoxic, and mutagenic to the ecosystem. Phenol is a mono-substituted aromatic hydrocarbon. In its pure state, it exists as a colorless or white solid. This pure compound is mixed with water and commercially sold as a liquid product. Phenol gives off a sweet, acrid smell detectable to most people at 40 ppb in air and at about 1–8 mg/L in water, where phenol is also combustible [8]. The Iraqi recommended level of phenol is 0.005 ppm in surface and groundwater, while 0.01–0.05 mg/L is the acceptable concentration for discharging into rivers according to Iraqi Law No. 25/1967. The World Health Organization (WHO) has established a maximum permissible concentration in drinking water of 0.001 mg/L. Therefore, it is necessary to remove phenol from domestic, industrial, and agricultural wastewaters in order to reduce its concentration to below the acceptable level [9].

Phenol is produced through both natural and anthropogenic processes [10, 8]. It occurs naturally in some foods, in human and animal wastes, and in decomposing organic material, and it is also produced endogenously in the gut from the metabolism of aromatic amino acids. Phenol has been isolated from coal tar, but it is now synthetically manufactured. Currently, the largest use of phenol is as an intermediate in the production of phenolic resins, which are used in plywood and adhesives [8]. Phenol is highly irritating to the skin, eyes, and mucous membranes in humans after acute (short-term) inhalation or dermal exposure. It is considered to be quite toxic to humans via oral exposure. Anorexia, progressive weight loss, diarrhea, vertigo, salivation, dark coloration of the urine and blood, and liver effects have been reported in chronically (long-term) exposed humans, according to the Agency for Toxic Substances and Disease Registry's (ATSDR's) Toxicological Profile for Phenol [8].

Treatment methods for wastewater contaminated with phenol incorporate physical, chemical, and biological technologies, which are essential for complete, high-efficiency commercial use to eliminate phenol from wastewaters. For instance, biological treatment [11] or chemical oxidation methods eliminate organic contaminants from petroleum industry wastewater [12]. Additional special treatment protocols are also required when other pollutants are present in phenol waste. The selection of efficient and acceptable treatment methods relies on specific wastewater characteristics and economic factors [13].

Different types of adsorbents can be applied for phenol removal from wastewater, according to many factors to consider when selecting the most suitable adsorbents, including surface area, pore size, structural features, adsorption performance, ease of regeneration, and a wide range of applications [9], such as activated carbon, natural clay [15], zeolite [16], rice straw [17], and bentonite and modified bentonite with graphene oxide–bentonite [18] or with zinc oxide [19].

The aim of the study involves the characterization of local bentonite through a group of physical and chemical analyses as an adsorbent, and its application in the batch adsorption process of phenol in a laboratory solution to assess adsorption capacity and removal efficiency, comparing it with commercial activated carbon under different conditions such as adsorbent dosage, initial phenol concentration, and contact time. Equilibrium isotherm models, Langmuir and Freundlich, were applied to fit the results under isotherm conditions, and pseudo-first-order and pseudo-second-order models were used as kinetic models to test the mass transfer mechanism. The novelty of the study is the evaluation of raw bentonite as a local adsorbent for pollution treatment.

2. Method/ Experimental Work

2.1. Materials

2.1.1. Phenol

C_6H_5OH is a 94.11 g/mol aromatic chemical. The 1000 mg/L phenol stock solution was prepared by dissolving 1000 mg of crystalline phenol in 1 liter of distilled water and stirring the mixture until the crystals were completely dissolved, where experimental phenol concentrations were prepared by dilution to obtain the required concentration.

2.1.2. Bentonite Be

Bentonite Be is obtained from the Iraqi Geological Survey/ Iraqi Ministry of Industry, with characteristic content according to source analyses: Al₂O₃ 15.62%, SiO₂ 54.22%, Fe₂O₃ 5.26%, and CaO 5.91%. After being washed with distilled water to remove dissolved material by checking the total dissolved solids (TDS) value, the bentonite was dried in an oven at 100 °C for 6 hr to determine moisture content. The dried bentonite was then ground to prepare it in powder form.

2.1.3. Activated Carbon AC

Granular activated carbon was produced by Unicarbo Company, Italy, and supplied from local markets. It is of coconut shell origin, with bulk and real densities of 711.8 and 1529 kg/m³, respectively, a surface area of 911.581 m²/g, and a particle size of 0.5–0.75 mm.

2.2. Adsorption Batch Experiment

Using a batch process method for the application of phenol removal by adsorption, three experimental sets were conducted that involved adsorbent dosage, initial phenol concentration, and contact time at constant pH and temperature. The volume of the solution was 50 mL for Be and AC dosage effect (0.1–1) g at constant other experimental conditions, with an initial phenol concentration of 50 mg/L, temperature of 30 °C, normal pH, and shaking speed of 200 rpm. The effect of initial phenol concentration was investigated at 20, 40, 60, 80, and 100 mg/L with a Be dosage mass of 1 g, while for 0.1 g AC, phenol initial concentrations of 100, 250, 500, 750, and 1000 mg/L were used. The effect of contact time was investigated individually under the best conditions for a 500 mL solution of selected phenol concentrations of 50 mg/L and 250 mg/L using Be and AC, respectively. The removal of phenol and adsorption uptake (q_e or q_t) were computed using the following Eqs (1 & 2) [20]:

$$\text{Phenol Removal \%Re} = \frac{C_0 - C_e}{C_0} \times 100 \dots\dots\dots (1)$$

$$\text{Adsorption uptake (q}_e \text{ or q}_t) = \frac{(C_0 - C_e) \times V}{M} \dots\dots\dots (2)$$

Where C₀ is the initial concentration (mg/L, ppm), C_e is the concentration at equilibrium (mg/L, ppm), q_e is the adsorption uptake at equilibrium or at a time interval (mg phenol/g of Be or AC), V is the sample volume (litre), and M is the mass of the adsorbent (Be or AC) (g).

2.3. Adsorbent Be and Adsorbate Analyses

2.3.1. Moisture Content of Be

It is the weight percentage of water contained in the pore spaces of soil relative to the solid mass of particles in that material, expressed as a percentage. A standard temperature of 100 °C is used to determine the mass of the Be sample according to Equation (3):

$$\text{Moisture Content} = \frac{M_1 - M_2}{M_1} \times 100 \dots\dots\dots (3)$$

Where M₁ and M₂ mass of Be before and after drying g.

2.3.2. Lose of Ignition (LOI)

It refers to the process of measuring the weight loss of a material after it has been heated to a high temperature (550–600 °C), typically in a furnace or oven. This loss in weight is mainly due to the burning or volatilization of volatile substances within the sample, such as organic matter and other volatile compounds, according to Equation (4):

$$\text{LOI} = \frac{W_1 - W_2}{W_1} \times 100 \dots\dots\dots (4)$$

Where W₁ and W₂ are the Be mass (g) before and after burning, respectively.

2.3.3. pH, Total dissolved Solid TDS and Electrical Conductivity EC

pH is a measure of how basic or acidic the Be and distilled water mixture is, with a ratio of 1:5. TDS stands for total dissolved solids and is a way to measure the total dissolved solids in water, expressed in mg/L, using a pH meter, while EC is a measure of the electrical conductivity of soluble salts in solution, expressed in $\mu\text{S}/\text{cm}$, using a multi-test TDS/EC meter.

2.3.4. Fourier Transform Infrared Spectroscopy (FTIR) Analysis

FTIR spectroscopy is a technique used to identify chemical bonds in a molecule by producing an infrared absorption spectrum. It provides a "fingerprint" of the sample, allowing for the identification of chemical functional groups and the characterization of the molecular structure, where FTIR type (Thermo Fisher, USA) was applied for Be testing, where an amount of Be was mixed with potassium bromide (KBr) at a ratio of 1:5, ground manually, and shaped into a tablet form as a thin circular shape using a manual press.

2.4. Phenol Measurement

High-performance liquid chromatography (HPLC), type (CECIL CE4600, JAP), is a widely used technique for identifying and quantifying phenol compounds. It separates these compounds based on their different interactions with the stationary phase. A C18 column (octadecyl silane) is the standard, and a mobile phase consisting of 10% acetonitrile (ACN) and water (H_2O) (10:90) was used, with a Phenomenex C18 column ($250 \times 4.6 \text{ mm}$, $5 \mu\text{m}$). The separation was monitored for 15 min at 270 nm using a UV-visible detector, with a flow rate of 1.5 mL/min and λ_{max} of 272.6 nm for UV measurements of different phenol compounds, allowing for the analysis of even complex mixtures.

3. Results and Discussion

3.1. pH, Moisture Content%, LOI % and TDS (EC) of Bentonite Analyses

Properties of Be after three washing times [21, 15] showed that the acidity ranged from 8.2–8.3. The total dissolved solids (TDS) decreased from 2020 to 405 mg/L, and the electrical conductivity (EC) was reduced from 3370 to 801 $\mu\text{S}/\text{cm}$, which refers to soluble electrolyte salts that can be reduced by leaching with water, while the moisture content is 5.934% and the loss of ignition (LOI) is 9.2%.

3.2. FTIR Analyses of Washed Dried Bentonite

Figure (1) shows the FTIR test of washed, dried Be, where identifying the functional groups present on Be is the objective of the FTIR analysis. The peak value at 3448.35 cm^{-1} is attributed to the hydroxyl ($-\text{OH}$) stretching vibrations of the Si-OH (silanol) group on the surface of Be [20, 22]. The observed bands at $1100\text{--}800 \text{ cm}^{-1}$ can be assigned to the Si-O-Si stretching vibrations of the clay mineral or quartz, while the band at 600 cm^{-1} can be assigned to the $-\text{OH}$ deformation linked to Al. The vibrations of SiO_4 tetrahedral stretching and bending are represented by the bands at 522.49 cm^{-1} and 469.42 cm^{-1} [22]. The stretching and bending vibrations of Al-Al-OH cause the bands at 3630 and 910 cm^{-1} . Additionally, a band of medium intensity at 1038.16 cm^{-1} was detected, which is attributed to the stretching and bending vibration of the Si-O-Si group. At 586 cm^{-1} , the Si-O-Al family (in an octahedral sheet) appears. Furthermore, the presence of quartz in the bentonite composition is demonstrated by the bands at 873.42 and 797.35 cm^{-1} . Calcium, magnesium, and other carbonate minerals may be responsible for the band at 1456.91 cm^{-1} . The stretching and bending vibrations of water account for the bands at 3448.35 and 1696.91 cm^{-1} [24–26].

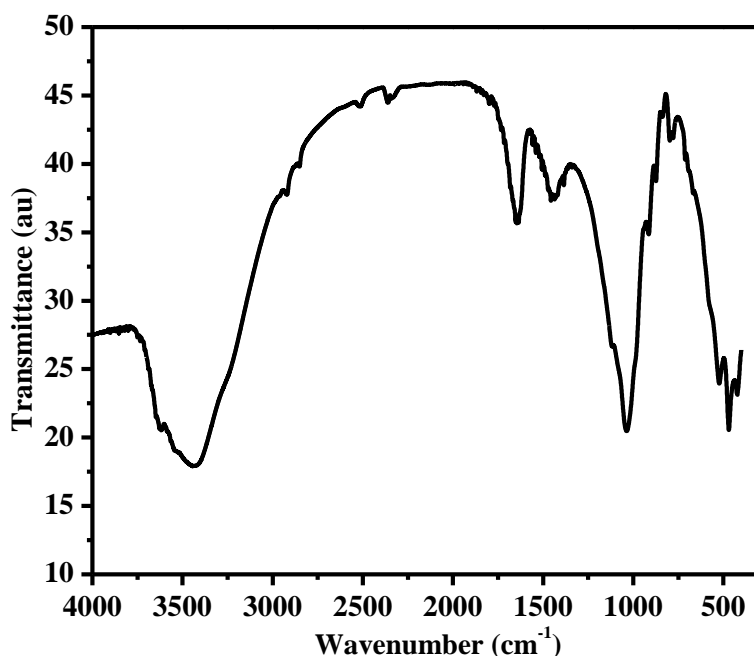


Figure (1): FTIR analyses of washed dried Be.

3.3. Equilibrium Isotherm Results

3.3.1. AC and Be Mass Dose Effect

Variable mass doses of adsorbents, AC and Be (0.1–1 g), were studied at a constant initial phenol concentration of 100 mg/L, a volume of 50 mL, and a contact time of 240 min, where the obtained results are shown in Table (1). The results show a decrease in phenol equilibrium concentration, with maximum phenol removal in the range of 97–100% at doses of 0.1–0.4 g of AC, while phenol removal was 45.58% at a dosage of 1 g of Be, as shown in Figure (2). The increase in removal percentage is due to the greater availability of surface area at higher adsorbent concentrations and the complete utilization of all active sites in the adsorbent [23].

Table (1): dose effect at removal efficiency and adsorption uptake.

Ac and Be mass dose gm	Be			AC		
	C_e (mg/L)	Phenol removal Re%	Adsorption uptake mg/g	C_e (mg/L)	Phenol removal Re%	Adsorption uptake mg/g
0.1	45.05	9.9	2.475	1.5	97	24.25
0.2	41.24	17.52	2.19	0.5	99	12.375
0.4	38.75	22.2	1.406	0	100	6.25
0.8	28.4	43.2	1.35	0	100	3.125
1	27.21	45.58	1.1395	0	100	2.5

The adsorption uptake results show a clear difference between AC and Be due to the nature of surface area availability in AC layers, which provide more active sites to improve removal efficiency due to the equilibrium state between the adsorbent (solid) and adsorbate (liquid), while Be has a limited number of active binding sites, which become harder to occupy with phenol [9].

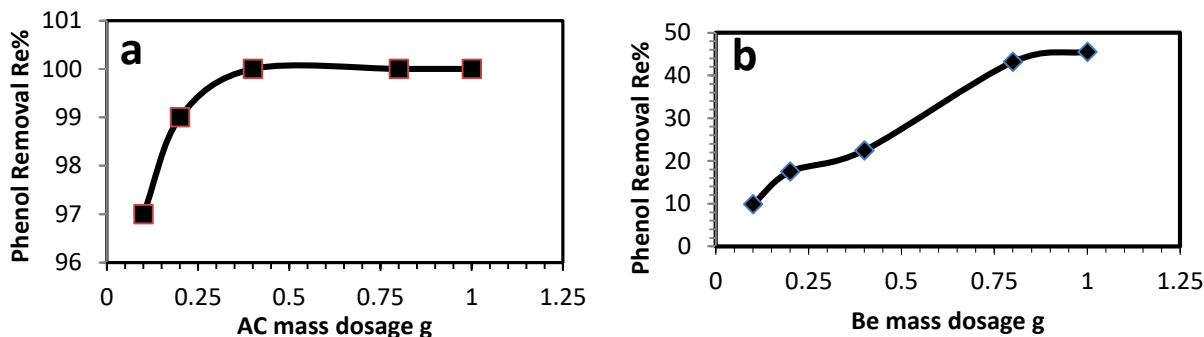


Figure (2): Adsorbent mass effect (a) AC (b) Be for phenol C_0 50 (mg/L) and contact time 240 min.

3.3.2. Phenol Initial Concentration Effect

The initial phenol concentration was studied in the range of 100–1000 mg/L at a constant AC dose of 0.1 g, and 20–100 mg/L at a constant Be dose of 1 g, with a contact time of 240 minutes. Table (2) shows that phenol removal efficiency decreases as the initial phenol concentration increases, reaching a maximum removal of 94.5% by AC at an initial phenol concentration of 100 mg/L, while it was 53.35% by Be at an initial phenol concentration of 20 mg/L, as shown in Table (2) and Figure (3a & b). Meanwhile, the adsorption uptake shows an increase with increasing initial phenol concentration, reaching maximum adsorption capacities of 138.31 mg/g and 1.232 mg/g by AC and Be, respectively. This is due to the increasing pollutant concentration acting as a driving force between the two phases, adsorbent (solid phase) and adsorbate (liquid phase) [9], which enhances mass transfer from the higher concentration phase to the lower one until equilibrium is reached. The decrease in efficiency at high concentrations is due to the accumulation of phenol particles on the adsorption surface, reaching saturation at a specific phenol concentration [23]. At the same time, adsorption uptake increases for a constant adsorbent dosage [24], and these results are in agreement with the principles of the adsorption mechanism reported in previous works [18, 15].

Table (2): Phenol initial concentration effect at removal efficiency and adsorption uptake.

Be			AC		
C_0 (C_e) (mg/L)	Phenol removal Re%	Adsorption uptake (mg/g)	C_0 (C_e) (mg/L)	Phenol removal Re%	Adsorption uptake (mg/g)
20 (9.9)	50.5	0.5335	100 (5.5)	94.5	47.25
40 (23.6)	41	0.82	250 (45.2)	81.92	102.4
60 (38.2)	36.3	1.09	500 (276.2)	44.76	111.9
80 (57.24)	28.45	1.138	750 (509.54)	32.06	120.23
100 (74.36)	25.64	1.282	1000 (723.38)	27.662	138.31

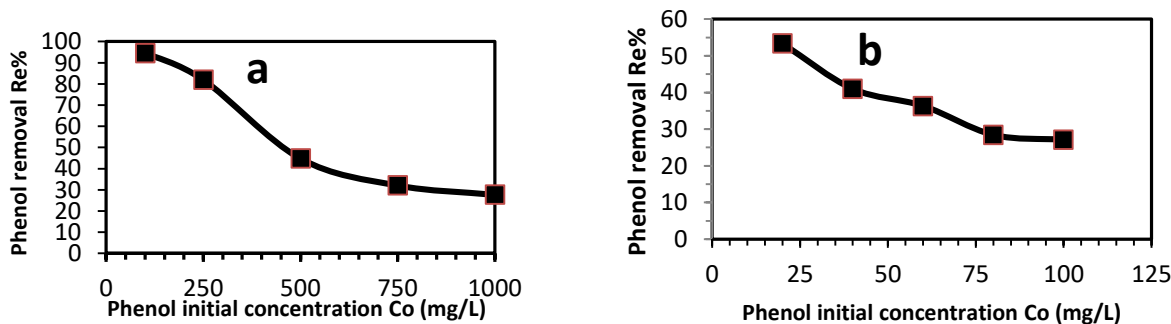


Figure (3): Effect of initial phenol concentration (a) AC, (b) Be, at phenol C_0 = 50 mg/L and a contact time of 240 min.

3.3.3. Equilibrium Isotherm Models

Well-known equilibrium isotherms are the Langmuir and Freundlich adsorption isotherm models, Equations (5 & 6). The Langmuir adsorption isotherm equation is a widely used model describing the relationship between the amount of adsorbate on a surface and the adsorbent's pressure or concentration at constant temperature, assuming a monolayer of adsorption on a homogeneous surface without interactions between adsorbed species, while the Freundlich isotherm model is an empirical approach that extends the limitation of monolayer coverage and broadens its use to multilayer adsorption processes [18, 23].

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \dots\dots\dots (5)$$

$$q_e = K_F C_e^{1/n} \dots\dots\dots (6)$$

Where C_e is the phenol concentration at equilibrium (mg/L), K_L and K_f are the Langmuir and Freundlich constants, and q_m is the theoretical maximum adsorption capacity (mg/g). According to the results listed in Table (2), the effect of initial phenol concentration is shown.

The linear forms of Eqs. (5 & 6) were obtained by plotting C_e/q_e vs. C_e for the Langmuir model and $\ln q_e$ vs. $\ln C_e$, as shown in Figure (4), and the isotherm model parameters were obtained.

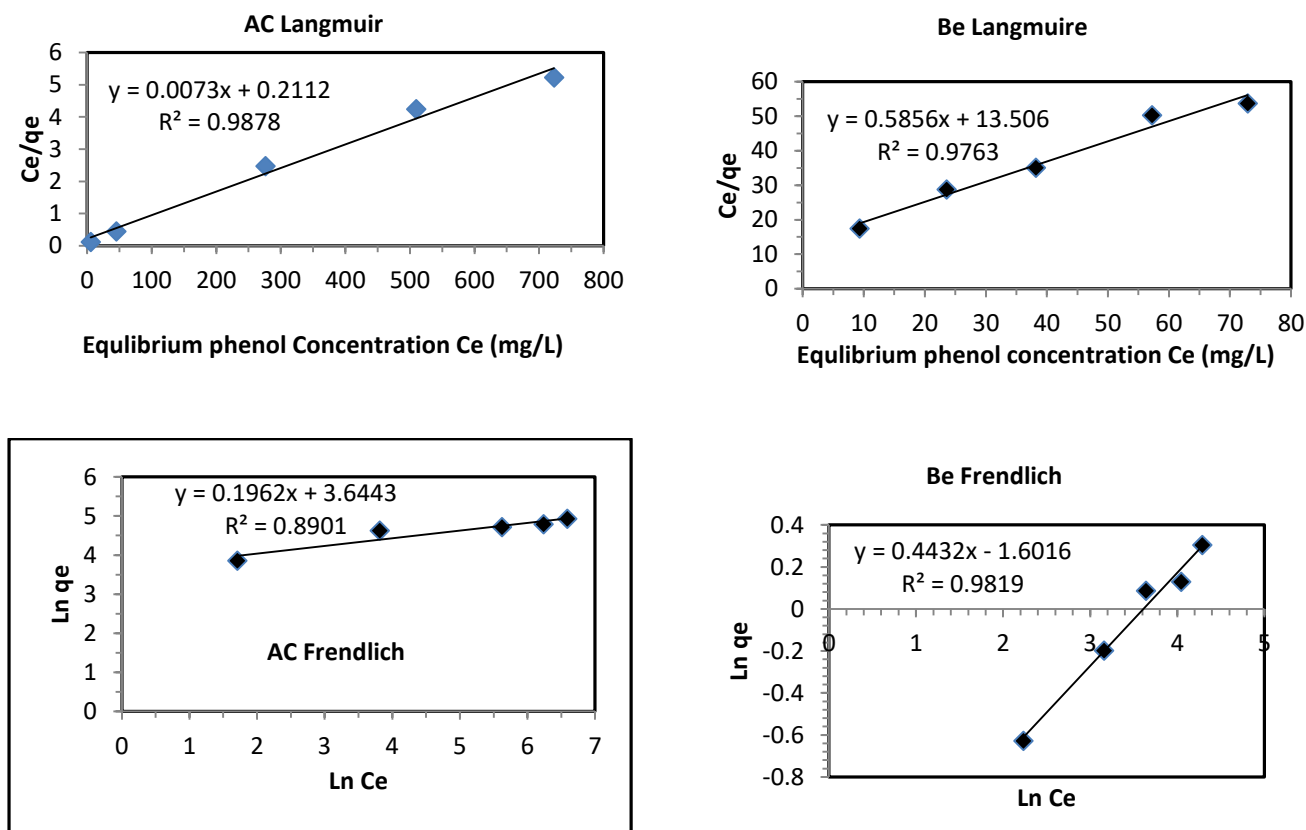


Figure (4): Linear form of Langmuir and Freundlich isotherm model.

Table (3) shows the calculated isotherm model parameters of Langmuir and Freundlich. The results show that AC/phenol adsorption fits the Langmuir model with an R^2 value of 0.987 and a theoretical maximum adsorption capacity of 142.857 mg/g, compared to the actual value of 138.31 mg/g, which refers to monolayer adsorption of phenol on the AC surface and a homogeneous surface nature [18]. While Be/phenol adsorption was fitted with the Freundlich model with an R^2 value of 0.981 and an n value of 2.257, as recommended according to the limit

values ($1 < n < 10$), which refers to multilayer adsorption with a heterogeneous surface and non-equivalent or independent active sites [23, 24].

Table (3): Langmuir Freundlich isotherm models parameters.

Adsorbent	Langmuir isotherm			Freundlich isotherm		
	q_e (mg/g)	Kl	R2	n	Kf	R ²
AC	142.857	0.0331	0.987	5.1	38.24	0.89
Be	1.71	0.0436	0.976	2.257	0.2017	0.981

3.4. Kinetic Results of Time Effect

Table (4) shows the results of phenol equilibrium concentration, removal percentage, and adsorption uptake by AC and Be at different contact time intervals, with other parameters fixed: phenol concentration of 250 mg/L, AC dosage of 1 g, and sample volume, and 50 mg/L phenol concentration, Be dosage of 10 g, and a sample volume of 500 mL.

The tabulated results show a decrease in equilibrium concentration and an increase in removal percentage with increasing time, as shown by the reduction of phenol concentration from 250 mg/L to 16.8 mg/L by AC and from 50 mg/L to 22.2 mg/L by Be, with removal percentages of 93.28% and 55.6%, respectively. Figure (5) shows the removal of phenol by AC and Be over time, where the results show an increase in adsorption uptake with increasing time, which refers to increased removal of phenol. The best contact time for phenol removal by AC is 60 min, with an adsorption capacity of 115.9 mg/g, while the best contact time for phenol uptake by Be is in the range of 240 min, with a maximum adsorption capacity of 1.39 mg/g. The effect of time shows a positive influence on removal and adsorption uptake due to increased contact time providing more ability for mass transfer of phenol accumulated on the solid surface until equilibrium, which is in agreement with previous works [9, 23].

Table (4): Time effect result of phenol removal by AC and Be.

Time (minutes)	AC+ Phenol initial Conc., Co 250 (mg/L)			Be+ Phenol initial Conc., Co 50 (mg/L)		
	C_e mg/l	Re%	q_e (mg/g)	C_e mg/l	Re%	q_e (mg/g)
15	58.7	76.52	95.65	38.5	23	0.575
30	22.78	90.88	113.61	34.24	31.52	0.788
60	18.2	92.72	115.9	31.1	37.8	0.945
120	17.5	93	116.25	25.6	48.8	1.22
180	17	93.2	116.5	23.1	53.8	1.345
240	16.8	93.28	116.6	22.2	55.6	1.39

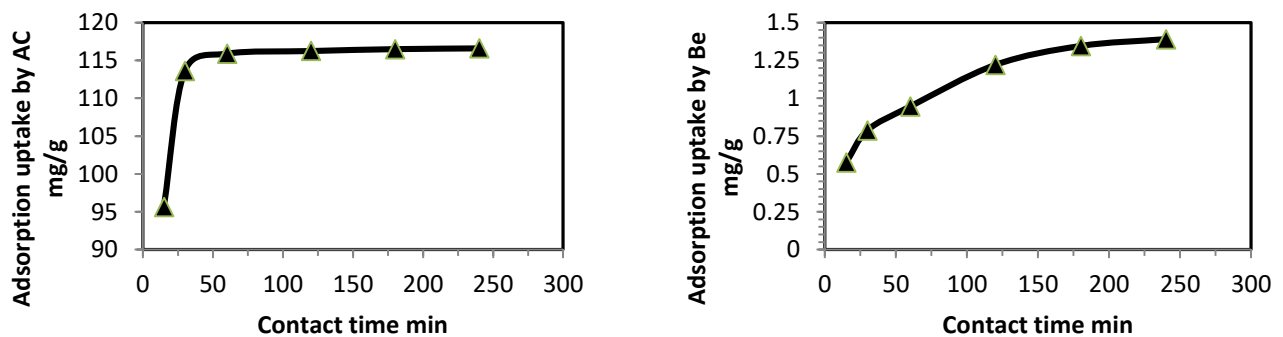


Figure (5): Phenol adsorption uptake at time effect: (a) AC/phenol 250 mg/L, (b) Be/phenol 50 mg/L.

3.4.1. Kinetic Adsorption Models

To analyze the adsorption rate and pinpoint the rate-limiting phase, the pseudo-first-order (1st PS) equation (7) and pseudo-second-order (2nd PS) equation (8) were used. Together, these kinetic models offer essential insights into the kinetics of the adsorption process, making it possible to identify the rate-limiting steps and provide a thorough understanding of the adsorption mechanisms within the context of the investigated materials [18, 24].

$$\ln(q_e - q_t) = \ln(q_e - K_1 t) \dots\dots\dots (7)$$

$$q_t = \frac{k_2 q_e^2 t}{1 + k_2 q_e t} \dots\dots\dots (8)$$

Where q_t and q_e are the adsorption uptake at a specific time and at equilibrium, respectively (mg/g), K_1 and K_2 are the rate constants of the pseudo-first-order and pseudo-second-order models (min^{-1} and $\text{g/mg}\cdot\text{min}$), respectively, and t is the time of sample analysis (min).

Plotting of $\ln(q_e - q_t)$ vs. t and t/q_t vs. t was carried out for the linearization of the pseudo-first-order and pseudo-second-order models of the obtained results in Table (4), as shown in Figure (6), to estimate the kinetic model parameters, which are listed in Table (5).

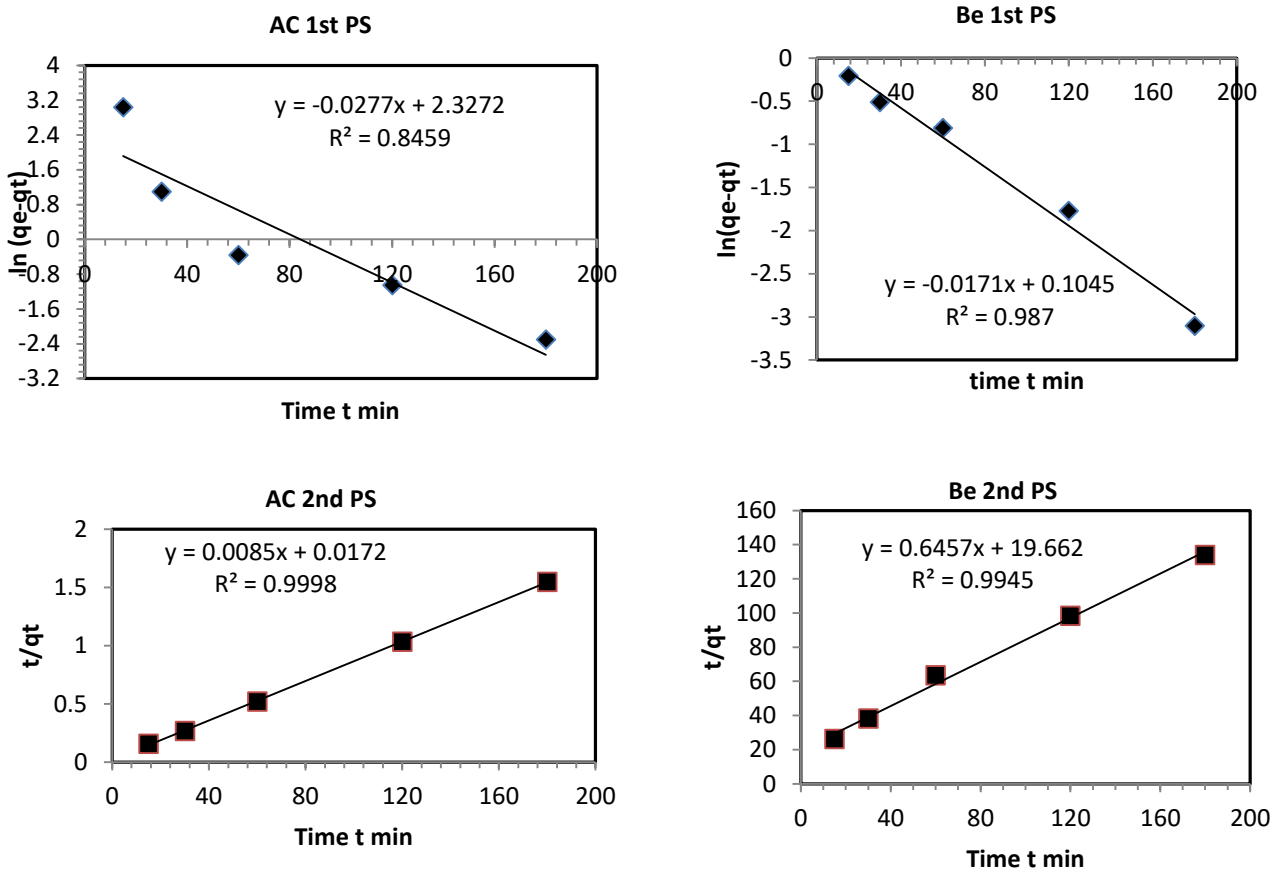


Figure (6): Linearization form of 1st and 2nd PS kinetic models of phenol adsorption by AC and Be.

Table (5): Estimated 1st and 2nd PS kinetic models of effect time at phenol adsorption.

Adsorbent	1 st PS model			2 nd PS model		
	q _e (mg/g)	K ₁ min	R ²	q _e (mg/ g)	K ₂ (g/mg.min)	R ²
AC	10.247	0.027	0.845	125	0.0039	0.999
Be	1.11	0.017	0.987	1.55	0.0211	0.994

The tabulated results in Table (5) show better fitting with the pseudo-second-order model than the pseudo-first-order model, with R² values of 0.999 and 0.994 for AC and Be, respectively, even though there are close R² values for both models. This refers to chemical adsorption [24] of phenol by AC and Be. The calculated q_e values were 125 and 1.55 mg/g, which are close to the experimental values (116.6 and 1.39 mg/g). These conclusions are in agreement with previous works [19, 23].

4. Conclusions

The study results show the ability of phenol removal by AC and Be, with the high capability of AC due to the nature of its surface, which is characterized by available surface area. The maximum adsorption capacities were 142.587 mg/g and 1.71 mg/g for AC and Be, respectively. Experimental conditions of the batch process show an increase in removal percentage with increasing mass dosage of AC, where 0.4 g achieved 100% phenol removal, while 1 g of Be achieved 45.58% phenol removal. The effect of initial phenol concentration shows 50.5% phenol removal by 1 g of Be at a phenol C_o of 20 mg/L and 94.5% removal by 0.1 g of AC at a phenol C_o of 100 mg/L. Equilibrium isotherm models show good fitting with the Langmuir isotherm model for AC, with an R² value of 0.987, while for Be the model matches the Freundlich isotherm model with an R² value of 0.981 and an n value of 2.257, as recommended within the limit values (1 < n < 10), due to the homogeneity and heterogeneity of the surface nature for mono- or multilayer adsorption. The kinetic study shows good fitting for AC and Be, with better fitting to the pseudo-second-order model than the pseudo-first-order model, with R² values of 0.999 and 0.994 for AC and Be, respectively, indicating a chemisorption mechanism of phenol accumulation on the adsorbent surface.

Conflict of Interest: The authors declare that there are no conflicts of interest associated with this research project. We have no financial or personal relationships that could potentially bias our work or influence the interpretation of the results.

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