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Phenol Adsorption from Simulated Wastewater Using Activated Spent Tea Leaves

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Abstract

The removal of phenol from wastewater has become a significant environmental concern because of its toxicity, even at low concentrations. This research focuses on the removal of phenol using spent tea leaves (STLs) as an adsorbent. SEM and FTIR tests were used to evaluate the surface characterization of the STLs. The effect of the parameters such as initial phenol concentration (20 - 80 mg. L^{-1}), pH (3 - 12), temperature (25 - 55 °C), contact period (30-330 min), and dose (0.25 - 2.5) on the removal of phenol was studied. The highest removal of phenol of 82% was obtained at 20 mg. L^{-1} of initial phenol concentration, 4 h of the contact period, pH of 3, adsorbent dose of 2 g, and temperature of 55 °C. The results of the kinetic study fitted the pseudo-second-order model. The obtained data for phenol adsorption isotherm fitted to the Langmuir, Freundlich, and Temkin models. The Temkin isotherm model presented the best fitting with phenol adsorption isotherm experimental data based on the correlation coefficients. According to the findings, the STLs can effectively remove phenol from wastewater.

Keywords: Phenol; Spent Tea Leave; Adsorption; isotherm; Kinetic.

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1- Introduction

Water pollution is one of the dangers which humans face. Human activities frequently lead to polluted water sources [1]. Phenol is released from textile, rubber, pulp and paper, plastics, pharmaceutical, coke manufacture, petroleum refineries, and ferrous sectors exceed safe levels [2]. Phenol is readily absorbed via skin contact, inhalation, or ingestion [3]. Due to its dangerous effects in tiny doses, the Environmental Protection Agency (USEPA) has categorized phenol as a priority pollutant, requiring treatment even if a phenol concentration is less than 1 ppm before release [4].

It is possible to remove phenol from wastewater via different processes, including chemical oxidation [5], filtration[6], biodegradation[7], electrocoagulation[8], photodegradation [9], solvent extraction [10], and adsorption [11, 12]. Adsorption is the most favorable among these techniques because of its simplicity and low cost. Researchers have studied the efficacy of phenol removal using a wide range of adsorbents, including zeolites [13, 14], clay [15], coal fly ash [16], activated carbon [17], and agriculture waste (including peat, rice husk, sawdust, peels of various fruits, modified corncob, and peanut hull [18].

Since spent tea leaves (STLs) are inexpensive and abundant, they can be used as an adsorbent in wastewater treatment. The yearly global consumption of tea is an astounding 3.5 million tons [19], making it one of the most widely consumed drinks. The potential to use STLs in wastewater treatment is a major factor in the technology's due to growing popularity [20]. In this paper, the activation of STLs using thermal and chemical methods will be considered for the removal of phenol from wastewater. The main objectives are to determine the ability of thermal and chemically activated STLs to remove phenol, study the effect of different operating parameters on the adsorption process, such as the initial concentration of material being adsorbed, temperature, contact period, adsorbent dose, and the pH value. Kinetic models will be used to identify the possible mechanisms of such an adsorption process. The Freundlich, Langmuir, and Temkin models will be used to analyze the adsorption isotherm.

2- Experimental Work

2.1. Materials

STLs were collected from daily use. Phenol of 97% purity, hydrochloric acid (HCl, 35-38% concentration), and ortho-phosphoric acid (H_3PO_4, 98% concentration) were obtained from BHD Laboratory, Germany), Sodium hydroxide (NaOH, 98% concentration) was obtained from SDFCL, India), and distilled water.

2.2. Preparation of the Adsorbent

The STLs were washed with water and then dried in the sun. After that, the sample was dried in an oven at 100 ^{0}C

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for an hour to remove any remaining moisture. The sample was immersed for about 20 min in a solution with a 1:4 ratio of water and 2 M ortho-phosphoric acid. To further improve the adsorbent's properties, the slurry shown in Fig. 1 was heated at 450 °C for 1.5 h inside a muffle furnace. The sample was then rinsed with water to remove any residual acid until the pH reached 7. Next, it was filtered and dried for being used in this work.

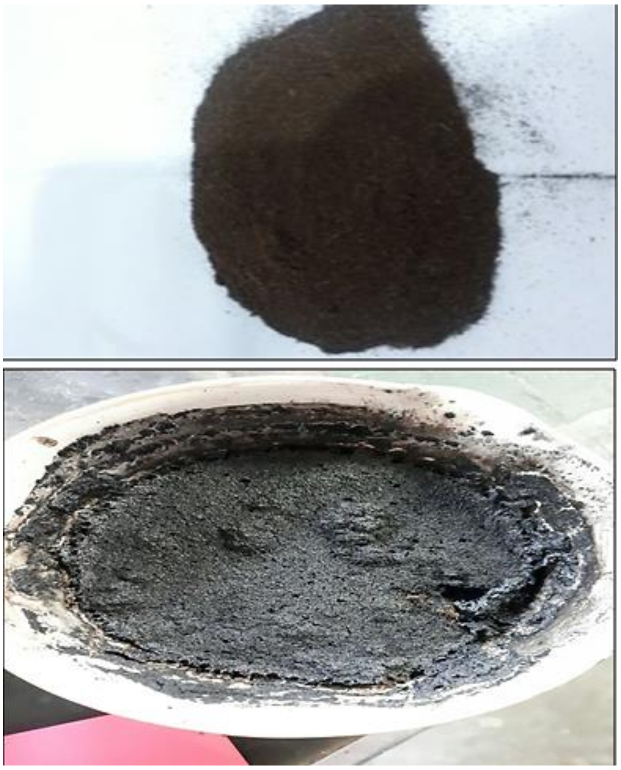

Fig. 1. STL after Washing, Grinding, and Activation

2.3. Characterization techniques

The characterization techniques used in this work were FTIR spectroscope (FTIR - 8400 S, Shimadzu, Japan), and SEM (Mirra 3-XMU, Czech Republic, TESCAN). A UV-visible spectrophotometer (model 9200, Shimadzu, China) was used to read the concentration of phenol in the solutions.

2.4. Phenol Solution Preparation

To prepare a stock phenol solution, 1 g of phenol crystals was dissolved in 1 L of water. To obtain a calibration curve, standard phenol solutions of 20, 40, 60, 80, and 100 mg. L^{-1} were prepared from the stock solution. The wavelength of phenol was 265 nm was used.

2.5. Phenol Adsorption Study

The adsorption experiment was conducted using constant doses for all trials. The quantity of activated STLs of 2 g was added to the phenol solution in a 250 mL flask. The mixture was stirred for a predetermined time at a speed of 125 rpm. Following the end of the agitating procedure, filtration paper was used to separate the phenol solution. The amount of phenol present in the supernatant was measured using a UV-Vis spectrophotometer. The impact of initial phenol concentrations was studied at 20, 40, 60, and 80 mg. L^{-1}. The impact of adsorption pH was studied at 3, 6, 9, and 12. The impact of the adsorption temperature was studied at 25, 35, 45, and 55 °C. The removal of the phenol and adsorption capacity were determined by Equation 1 and Equation 2:

$$\%R = \frac{(C_o - C_f)}{C_o} * 100 \qquad (1)$$

$$q_e = \frac{V}{m} \times (C_o - C_f) \qquad (2)$$

Where: %R is the phenol removal efficiency, C_0 (mg. L^{-1}) is the initial concentration of phenol, C_f (mg. L^{-1}) is the phenol concentration at any time, q_e (mg. g^{-1}) is the adsorption capacity, m (g) is the adsorbent mass, and V (mL) is the solution volume.

3- Result and Discussion

3.1. Characterization of STLs

The FTIR spectrometer was used to analyze the morphology of STL. Each chemical bond often has its distinct energy absorption band. It has been used to determine the existence of certain biosorbent functional groups. FTIR spectra are presented in Fig. 2. The O-H group is represented in the spectrum by a broad, U-shape peak at around 3606.89-3340.71 cm^{-1}. The aliphatic C-H stretching mode is indicated by peaks at 2920 cm^{-1} and 2858.51 cm^{-1}. This coincides with the frequency of 1612 cm^{-1}, which is the aromatic C=C peak. Alcohol's C–O bonds could be responsible for the band's formation at 1157.29–1029 cm^{-1}. STLs have a large capacity to remove phenol from wastewater because of the functional groups that they contain [21]. Fig. 3 shows the SEM image of the STLs. The image shows the adsorbent as small particles with a coarse porous surface. The adsorbent was able to offer the best surface area for the absorption of phenol because of the varying pore sizes [22].

3.2. Adsorption Experiment

a. Influence of Contact Time

The equilibrium contact period was studied at an adsorbent dose of 2 g, pH of 7, the initial concentration of phenol of 20 mgL^{-1}, and a temperature of 25 °C. It can be observed from Fig. 4 that the removal percentage rose with increasing time due to the ability of phenol to reach the adsorbent sites. Also, the figure shows that phenol removal was nearly constant after 4 h because the adsorbent had reached equilibrium [23].

b. Influence of the Adsorbent Dose

This study was conducted with different doses from 0.25 to 2.5 g at a phenol concentration of 20 mg. L^{-1}, pH of 7, temperature of 25 °C, and 4 h contact time. Fig. 5 shows that the adsorption increased initially, but the percentage remained relatively constant when the adsorbent dosage was 2 g. As the adsorbent dose increased, more surface area was accessible for adsorption due to increasing active sites. However, using more amount of adsorbent makes the removal decline due to the buildup of adsorbent particles, leading to a reduction in the surface area and a saturation of the active adsorbent situates [24].

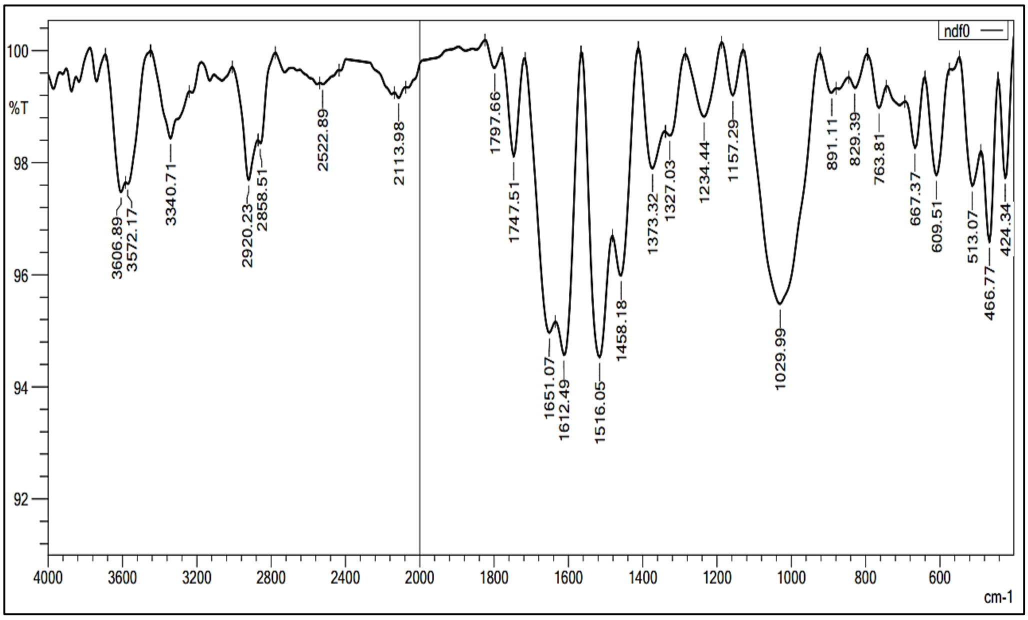

Fig. 2. FTIR of the Adsorbent

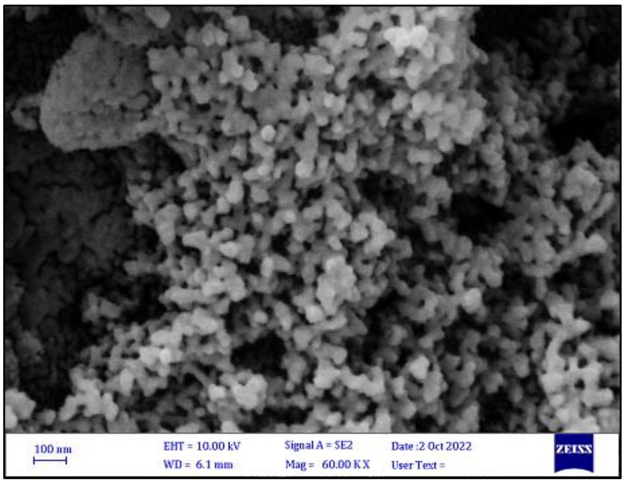

Fig. 3. SEM of the Adsorbent

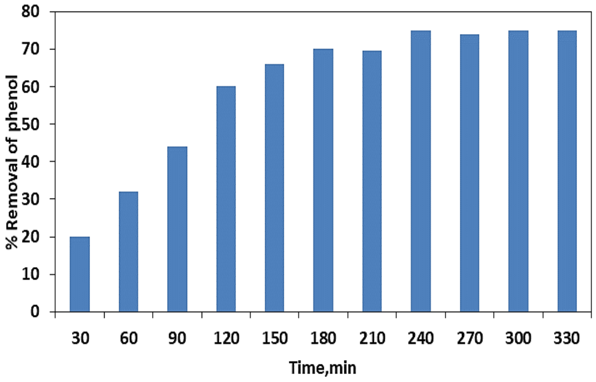

Fig. 4. Influence of Contact Period on Phenol Removal Efficiency

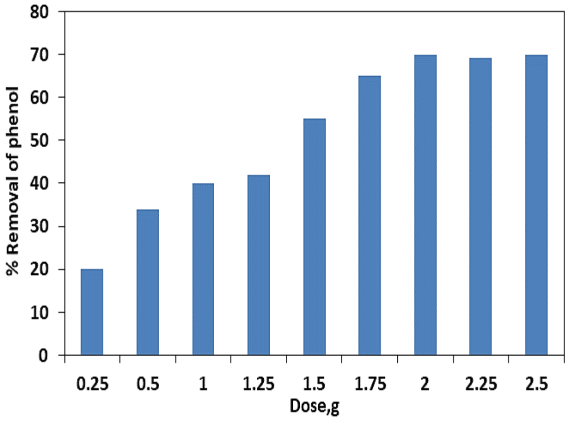

Fig. 5. Influence of the Adsorbent Dose on Phenol Removal Efficiency

c. Influence of pH

The effects of the pH of the solution on the removal of phenol from wastewater were studied from (3-12) using the dose of 2 g, contact time of 4 h, temperature of 55 ^{0}C, and an initial concentration of 20 mg. L^{-1}. Fig. 6 depicts the relationship between removal efficiency and pH. The figure shows that the removal increased when the medium is acidic. A more significant concentration of H$^+$ ions in the acidic medium favors phenol sorption by STLs, where the surface of adsorbents becomes more positively charged in the existence of high concentrations of H$^+$ ions to boost the removal of phenol. It can be seen that the highest removal occurred at a pH of 3 [25].

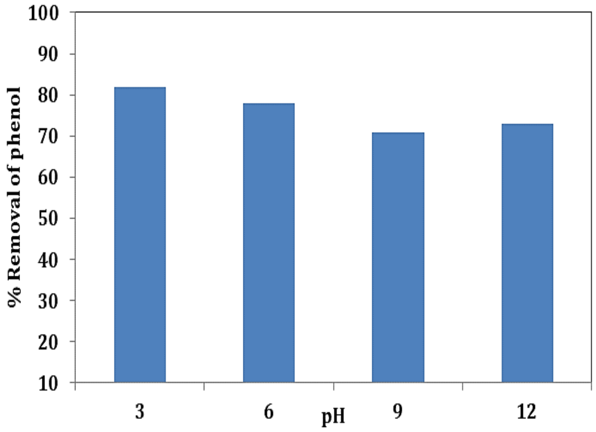

Fig. 6. Influence of pH on the Removal of Phenol

d. Influence of Temperature

The study was performed by varying the adsorption temperature from 25 to 55 °C at an adsorbent dose of 2 g, contact period of 4 h, pH of 3, and initial phenol concentration. Fig. 7 shows the influence of adsorption temperature on phenol removal. According to the graph, the phenol removal increased when temperature increased because the phenol and the STLs could interact chemically with each other at higher temperatures. According to the findings, the adsorption of phenol by the STLs depends on temperature, and the approach would be endothermic if used. This finding accords quite well with [26].

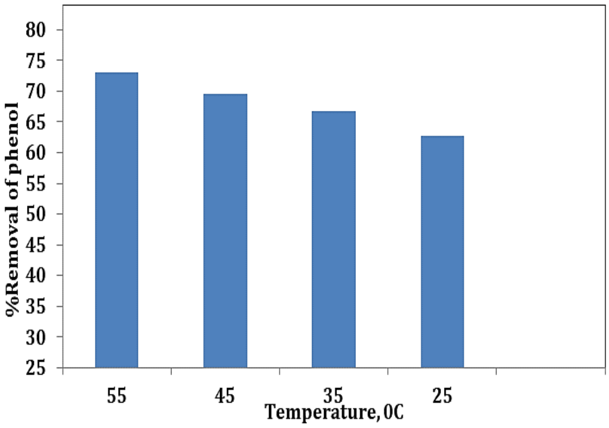

Fig. 7. Influence Temperature on Phenol Removal Efficiency

e. Effect of the Initial Phenol Concentration

The effect of the concentration on the removal of phenol by STLs was studied in the range of (20 - 80 mg. L^{-1}) at an adsorbent dose of 2 g, time of 4 h, temperatures of 55 ºC, and pH of 3. The results are shown in Fig. 8. The %Removed decreased as the initial adsorbate concentration increased because there are no binding groups on the surface of STLs at high concentrations and adsorption reaches saturation at high initial concentrations [27].

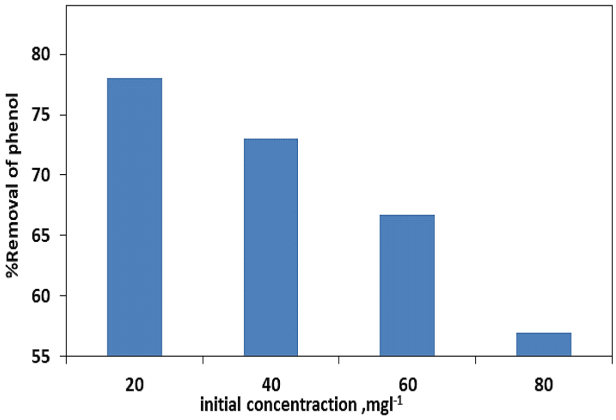

Fig. 8. Effect of Initial Phenol Concentration on the Removal

3.3. Equilibrium Isotherm

The equilibrium features of the adsorption of phenol on STL were described using the Freundlich [28], Langmuir [29], and Temkin [30] isotherm models. The Langmuir isotherm model assumes that sorption is constant at all the binding sites, while the Freundlich isotherm model suggests that the heat of adsorption varies throughout the surface heterogeneously. The Temkin isotherm involves multilayers and is relevant to heterogeneous systems.

Eq. 3 shows the Freundlich isotherm model:

$$q_e = K_F C_e^{\frac{1}{n}} \qquad (3)$$

Where, K_F is the Freundlich adsorption model constant ($L.mg^{-1}$) related to adsorption capacity, and n is the Freundlich adsorption intensity parameter. The Freundlich constant (K_f) and n can be calculated from the slope and the intercept of the straight line obtained by plotting LnC_e versus Lnq_e.

Eq. 4 shows the Langmuir isotherm model:

$$\frac{1}{q_e} = \frac{1}{q_{max}K_L}\frac{1}{C_e} + \frac{1}{q_{max}} \qquad (4)$$

Where, C_e is the equilibrium concentration of adsorbate (mg. L^{-1}), q_e is the amount of the adsorbate adsorbed at equilibrium (mg. g^{-1}), q_{max} is the maximum adsorption capacity (mg. g^{-1}), and K_L is the Langmuir equilibrium constant ($L.mg^{-1}$). The Langmuir constants q_e and k_L can be calculated from the slope and the intercept of the straight line obtained by plotting $1/q_e$ versus $1/C_e$.

Eq. 5 shows the model of the Temkin isotherm model.

$$q_e = BlnA + BlnCe \qquad (5)$$

Where A is the Temkin isotherm model equilibrium constant ($L.mg^{-1}$), B is the heat of adsorption constant ($J.mol^{-1}$), R is the universal gas constant (8.314 $J.mol^{-1}. K^{-1}$), T is absolute temperature (K), b is the Temkin isotherm constant, which shows the adsorption potential of the adsorbent. The Temkin model constants can be obtained from the straight-line obtained from plotting q_e versus LnC_e.

The comparison of the values of the correlation coefficient can be seen in Fig. 9, Fig. 10, and Fig. 11. R^2 of 0.7805 was for the Freundlich isotherm model, R^2 of 0.4042 was for the Langmuir isotherm model, and R^2 of 0.9728 was for the Temkin isotherm model. The Temkin model is the most accurate description of phenol adsorption by STLs.

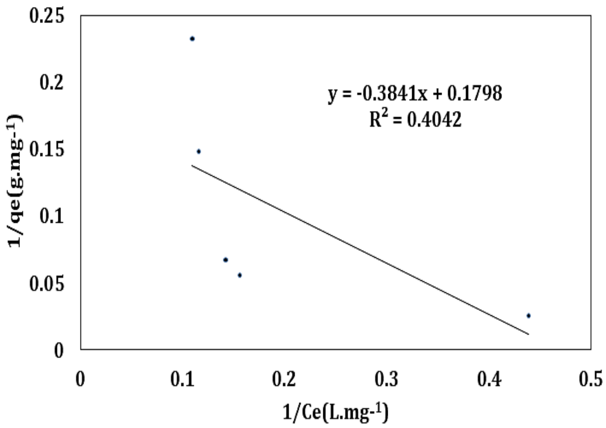

Fig. 9. Langmuir Isotherm Model

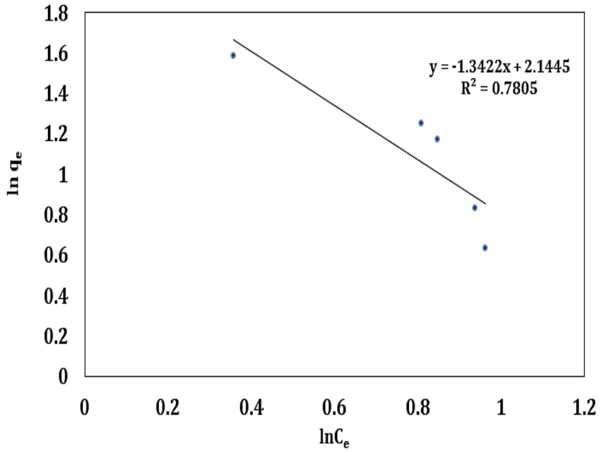

Fig. 10. Freundlich Isotherm Model

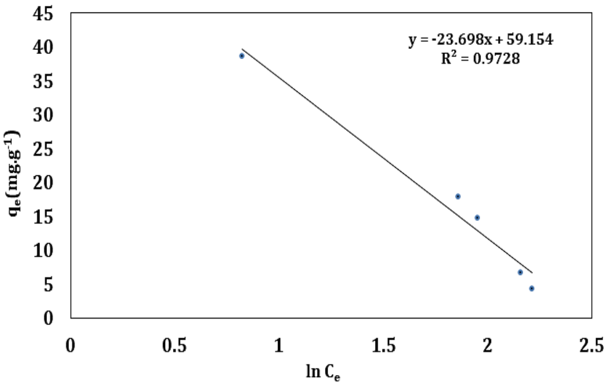

Fig. 11. Temkin Isotherm Model

3.4. Kinetic Studies

The kinetics isotherm data was also studied using the pseudo-first-order and pseudo-second-order models, respectively. Eq. 6 shows the pseudo-first-order model and Eq. 7 shows the pseudo-second-order model [31].

$$\log(qe - qt) = \log qe - \left(\frac{K1t}{2.303}\right) \qquad (6)$$

Where q_t is the amount of phenol adsorbed (mg/g) at time t (min), qe is the amount of phenol adsorbed (mg/g) at the equilibrium, and k_1 is the adsorption rate constant for the pseudo-first-order kinetic model ($\min^{-1}$) .The value of the adsorption rate constant (k_1) is calculated from the slope obtained from plotting $\log(q_e - q_t)$ vs t. Fig. 12.

$$\frac{t}{qt} = \left(\frac{1}{K2qe2} + \frac{t}{qt}\right) \qquad (7)$$

Where the k_2 is the pseudo-second-order kinetic model (g mg^{-1}. $\min^{-1}$). The value of k_2 was calculated from the intercept of the plot of t/q_t vs t (Fig. 13). The correlation coefficient is closer to unity for the pseudo-second-order than for the pseudo-first-order model.

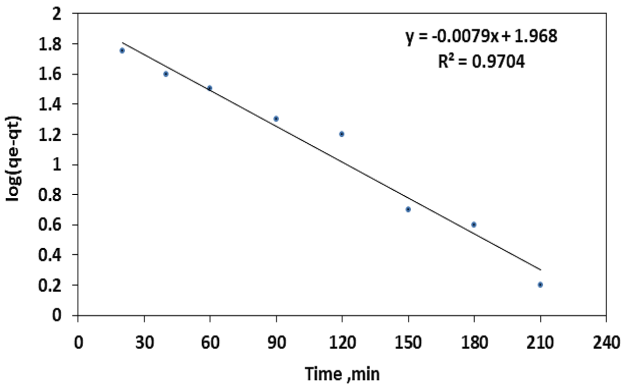

Fig. 12. Pseudo-First-order Kinetic Model

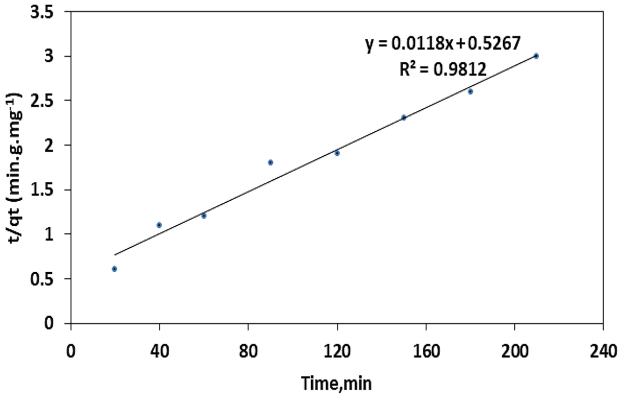

Fig. 13. Pseudo-Second-order Kinetic Model

4- Conclusion

STLs were activated by acid and thermal methods to increase their adsorption capacity. It was used to remove phenol from simulated wastewater. The efficiency of phenol removed in the experiment was high at the initial phenol concentration of 20 ppm, pH of 3, dose of 2 g, temperature of 55 ^{0}C, and contact time of 4 h. The maximum percentage of removal was 88 %. When analyzing the adsorption data, the Temkin isotherm fitted the most accurate results. The results of the kinetic analysis fitted the pseudo-second-order model more than the pseudo-first-order model.

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امتزاز الفينول من مياه الصرف الصحي المحاكاة باستخدام أوراق الشاي المنشط المستهلك

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الخلاصة

أصبحت إزالة الفينول من المياه الصرف الصحي من العوامل البيئية الهامة بسبب سميته حتى عند التركيز المنخفض. يركز هذا البحث على إزالة الفينول باستخدام أوراق الشاي المستهلكة كمادة ماصة. تم استخدام اختبارات SEM وFTIR لتقييم توصيف سطح STLs. تأثير بعض العوامل مثل تركيز الفينول الأولي (٢٠–٨٠ مجم لتر $^{-١}$)، درجة الحموضة (٣–١٢)، درجة الحرارة (٢٥ –٥٥ درجة مئوية)، فترة التلامس (٣٠–٣٣٠ دقيقة)، وزن اوراق الشاي المستهلكة (٠,٢٥–٢,٥) غم تمت دراسة إزالة الفينول. عند تركيز الفينول الأولي ٢٠ مجم لتر $^{-١}$، ٤ ساعات من فترة التلامس، درجة الحموضة ٣، ٢ جرام من اوراق الشاي المستهلكة، ودرجة حرارة ٥٥ درجة مئوية، تم الحصول على اعلى ازاله للفينول بمقدار ٨٢%. نتائج الدراسة الحركية تناسب نموذج الدرجة الثانية الزائفة. البيانات التي تم الحصول عليها من نماذج متساوي الحرارة لامتصاص الفينول المجهزة لنماذج لانجمير، فروندليتش، و تيمكين. قدم نموذج تيمكين أفضل ملاءمة مع البيانات التجريبية لأيزوثرم امتزاز الفينول بناءً على معاملات الارتباط. ووفقا للنتائج، يمكن لـ STLs إزالة الفينول بشكل فعال من مياه الصرف الصحي.

الكلمات الدالة: الفينول، أوراق الشاي المستهلكة، الامتزاز، متساوي الحرارة، الحركية.