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RESEARCH ARTICLE

Comparing the efficiency of unmodified dried sludge adsorbents and those modified via chemical and microwave methods in removing 2,4-dinitrophenol from aqueous solutions

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Abstract
2,4-dinitrophenol (DNP) is found in small amounts in the effluent of many wastewater treatment plants. The contamination of drinking water with this pollutant, even in trace amounts, causes toxicity, health problems, and unfavorable taste and odor. This study aims to compare the efficiency of non-modified and modified dried sludge adsorbents in removing 2,4 DNP from aqueous solutions. The results of 2,4DNP removal by high-performance liquid chromatography method at the wavelength of 360 nm in a batch mode were obtained by changing the influential factors including contact time, pH, initial concentration of the contaminant, and adsorbent dosage. Eventually, the results were analyzed by kinetic and isotherm models. In this research, the optimal time was obtained as 60 min and pH as seven for all three adsorbents. The results showed that the removal percentage increases by rising adsorbent dosage and reducing contaminant concentration. The correlation coefficient value of linear and non-linear led that in kinetic studies, it follows the pseudo-second order model. In contrast, in isotherm studies, examining linear and non-linear models of isotherms showed that the data for every three types of adsorbents follow the Freundlich model well. The adsorption process is highly dependent on pH and affects the adsorbent surface properties, ionization degree, and removal percentage. At high pH, hydroxide ions (OH) compete with 2,4 DNP molecules for the adsorption sites. The adsorption occurs quickly and gradually reaches a constant value because, over time, the adsorption sites are occupied until reaching a saturated limit. By increasing the adsorbent dosage, the adsorption percentage increased significantly, which is due to the fact that higher amounts of adsorbent cause more adsorption sites.

Keywords Dried sludge · 2,4 DNP · Adsorption · Isotherm · Kinetics

Introduction

Nitrophenol compounds are one of the highly used organic materials in industries. These materials are used as intermediate compounds in the production of explosives, pharmaceuticals, pesticides, paint, wood protector, and detergents [1, 2]. Nitrophenol materials are considered as important environmental contaminants because of their toxicity and resistance to microbial degradation. Thus, these compounds have been regarded as high priority pollutants according to the classification by the American Environmental Protection Agency. The standard of their concentration in natural waters should be less than ten ng/L [3–6]. Dinitrophenol (generally isomer 2,4) is the most critical nitrophenol compound, which is extensively used in chemical industries for producing nitroso dyes and their derivatives, picric acid, diamino phenol dihydrochloride, solvents, and pesticides. 2,4 DNP is a highly toxic material whose long-term contact in humans and animals through inhalation or skin contact affects the bone marrow, central nervous system, and cardiovascular system. In this way, it causes the development of cataracts, inflammation of lymph nodes, eczema, loss of nails, increased rate of metabolism, the elevation of body temperature, headache, heavy perspiration, thirst, and fatigue [4]. Due to the toxic and

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environmental effects of 2,4 dinitrophenol pollutants and the need to remove or reduce their concentration in industrial effluents, various treatment methods have been investigated to remove this pollutant. Currently, multiple methods are employed for treating the wastewater containing compounds, including adsorption, chemical oxidation, precipitation, concentration, evaporation, and incineration [7, 8]. The adsorption process is one of the standard methods for the removal of organic pollutants from aqueous environments. Its advantages include inexpensive process (compared to other treatment processes), simple operation, quick adsorption and high-efficiency, excellent adsorption capacity, selectivity, adsorbent recoverability, reusability, and recyclability of the absorbed pollutant, making it desirable for removal of organic contaminants. Adsorption is a mass transfer process whereby a material transfers from the liquid phase to the surface of a solid and binds through physical or chemical forces [9, 10]. Various adsorbents have been used for the removal of phenolic compounds from wastewater, one of which is active carbon. However, because of its high cost of preparation and the need to spend high prices for its recovery [11], attempts have been made to employ inexpensive and natural adsorbents, including natural rubber core shell [12], rice husk ash [10], etc. Thus, the usage of dried sludge discharged from the wastewater treatment plant for the adsorption of organic contaminants is economical thanks to access to inexpensive and local materials [13]. This activating agent removes water from the structure of raw material and decreases the required temperature for carbonization and prevents tar during the process, creating a porous structure in the adsorbent [14]. The use of microwaves in drying sludge Causes solids to coagulate breaks gelatinous forms, and reduces the water affinity of the sludge solids. As a result, the sludge was almost sterile and easily dehydrated [15]. Accordingly, the present study aims to compare the efficiency of unmodified dried sludge adsorbents and those modified via chemical and microwave method in the removal of 2,4 dinitrophenol from aqueous solutions.

Materials and methods

This study has been of experimental type in which the efficiency of unmodified and modified dried sludge has been investigated to remove a synthetic solution of 2,4 DNP. The discharged sludge required was prepared from the treatment plant in the south of Tehran. After collection, the sludge sample was washed several times with distilled water so that the possible extra contaminations and particles would be removed from its surface. It was then dried in an oven at 60 °C for 48 h. The drive adsorbent was screened using standard sleeves within the size of 0.2–0.3 mm with mesh 50–70 [13]. The preparation of modified sludge with calcium chloride, 20 g of the sludge dried in the previous stage, underwent chemical preprocessing and protonation with 0.1 M solution of calcium chloride (400 mm) for 15 min. In this process, the pH of the solution was kept constant at five because it is the optimal pH value for the activation of adsorbent with calcium chloride. Subsequently, the adsorbent process with calcium chloride was washed with distilled water several times so that the extra calcium would be removed. Next, it was placed on a paper filter so that its water content would diminish. This non-live adsorbent was heated in an oven at 60 °C for 48 h. After that, it was screened using standard sieves within the size of 0.2–0.3 mm with mesh 50–70 [14]. The preparation of microwaved sludge, the dried sludge in the previous stage, underwent chemical preprocessing with a solution of H_3PO_4 3 M, KOH 2 M, and $ZnCl_2$ 2 M. These activating factors remove water from the structure of the raw material, which helps to create porous forms in the adsorbent. In this process, the pH of the solution was kept constant at 6.5. Subsequently, the processed biomass was washed several times with distilled water and then transferred to a filter paper so that its water content would shrink. This biomass was heated in a microwave with an input power of 700 W and a frequency of 2450 MHz for 7 min. Next, it was screened using standard sleeves within the size of 0.2–0.3 mm with mesh 50–70 [15, 16]. The properties of the adsorbent, including granulation, pH_{zpc}, and for determining the morphological and structural properties, SEM and BET tests were used. In order to specify the pH_{zpc}, 30 ml of sodium chloride solution (0.01 M) was poured into six 50-ml Erlene flasks, and the pH of the solution was adjusted within the range of 2–12. Next, 1 g of the dried sludge, sludge modified with calcium chloride, and microwaved sludge adsorbents were added to each of the Erlene flasks, and placed on a shaker with 200 rpm for 48 h. Thereafter, the samples were filtered, and the final pH of each sample was measured. After drawing the curve of pH variations against the initial pH, the pH_{zpc} value was calculated [17]. The experiments were performed as a batch in all stages on a rotary shaker at laboratory temperature (25 ± 1 °C). Measurements of 2,4 DNP were performed by High-performance liquid chromatography (*HPLC*) at 360 nm. Measurement of 2,4 DNP was performed at different pH's (3, 5, 7, 9, and 11), different concentrations (10, 30, 50, 70, and 100 mg/L), and at different contact times (10, 30, 45, 60, 90, 120, 150 min) by performing kinetic

Table 1 The physical and chemical properties of the three adsorbents used

Characteristics	Dried sewage sludge	chemically modified waste sludge	Microwaved sludge
Colour	Brown	Brown	Brown
BET surface area ($m^2 g^{-1}$)	68/14	83/51	98/76
pHzpc	7/4	7/3	7/2
Particle size (mm)	0/2–0/3	0/2–0/3	0/2–0/3
Particle porosity	0/58	0/65	0/79

experiments. For determining the optimal values of the mentioned parameter, the other ones were kept constant. The adsorption equilibrium time of compounds on adsorbents, optimal pH of adsorptions, optimal concentration, and eventually of adsorption capacity and adsorption kinetic model was obtained for each adsorbent. And then, the efficiency of the three adsorbents in absorbing the selected compound was compared with each other. Next, equilibrium (isotherm) experiments were performed with different amounts of adsorbents (0.1, 0.5, 1, and 1.5) and optimal values of time, pH, and concentration. After that, Langmuir and Freundlich adsorption models regarding the adsorption of 2,4 DNP on each of the adsorbents were investigated. The data obtained from different stages of the experiment, as well as the results gained from analysis of the collected samples as well as other measured parameters, were analyzed by Excel software.

Adsorption kinetics: in this research, interparticle diffusion, pseudo-first-order, and pseudo-second order models were used for describing the adsorption process, whose mathematical expression is as follows Eq.(1 to 3):

$$q_t = K_{dif} t^{0.5} + C \quad (1)$$

[18–20] Intraparticle diffusion

$$\log(q_e - q_t) = \log(q_e) - \frac{k_1}{2.303} t \quad (2)$$

[18, 21]Pseudo-first order

$$\frac{t}{q_t} = \left(\frac{1}{k_2 q_e^2}\right) + \left(\frac{1}{q_e}\right) t \quad (3)$$

[9, 22]Pseudo-second order Where K_d represents the intra-particular diffusion rate constant, and C is the intra-particular diffusion constant. q and q_e show the amounts of 2,4 dinitrophenol absorbed per every gram of the adsorbent at the time of t at equilibrium (mg/g). K_1 represents the first-order kinetic constant(1/h), and K_2 is the second-order kinetic constant (gmg^{-1} h^{-1}). Also, non-linear forms of pseudo -first-order and pseudo-second order kinetics models (Eqs. 4 and 5) were used. Its aquations as follow (aquations of 4 and 5):

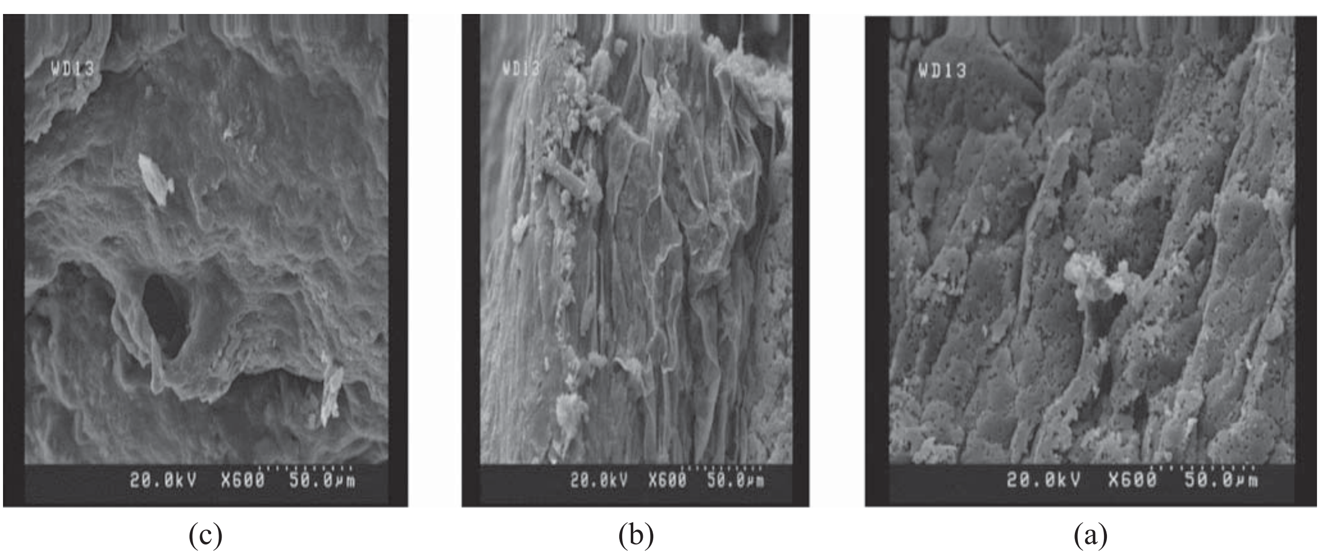

Fig. 1 Scanning electron microscopy (SEM) images of dried sludge (a), modified sludge (b), and microwaved sludge (c)

$$q = q_e\left(1 - q^{kt}\right) \quad (4)$$

$$q = \frac{q^2_{g+K_t}}{1 + q_{e\,K_t}} \quad (5)$$

where q is the amount of adsorbate adsorbed per unit mass of solid at the time of t (mg/g), and K_t is the pseudo-first-order and the pseudo-second-order rate constants (g/mg min) [23–25].

Adsorption isotherm: there are various isotherm models for analyzing experimental data and describing equilibrium in adsorption, including Langmuir and Freundlich. Freundlich isotherm is obtained by assuming a heterogeneous surface with a non-uniform distribution of adsorption heat across the surface. Langmuir isotherm indicates monolayer, uniform adsorption by removing the interaction effects of the absorbed molecules. According to the aquations of 6 and 7.

$$\text{lnqe} = \frac{1}{n}\ln c_e + \ln K_F \quad (6)$$

[26–28] Isotherm Freundlich

$$\frac{c_e}{q_e} = \frac{c_e}{q_m} + \frac{1}{q_m b} \quad (7)$$

[29] Isotherm Langmuir

In the Freundlich model, K_F indicates the adsorption capacity per unit concentration, 1/n shows the degree of adsorption, and C_e indicates the equilibrium concentration in terms of mg/L. 1/n represents the manner of the process; if $1/n = 0$, then the adsorption process is irreversible; $0 < 1/n < 1$, it is desirable, and if $1/n > 1$, it is undesirable [30]. In the Langmuir model, q_m represents the maximum adsorption capacity (mg/g), and b shows the adsorption correlation energy [31–33]. Non-linear models of the Langmuir and Freundlich isotherms (Eqs. 8 and 9, respectively) were used to evaluate the adsorption equilibrium data.

$$q_e = \frac{q_{m\,K_L\,C_e}}{1 + K_{L\,C_e}} \quad (8)$$

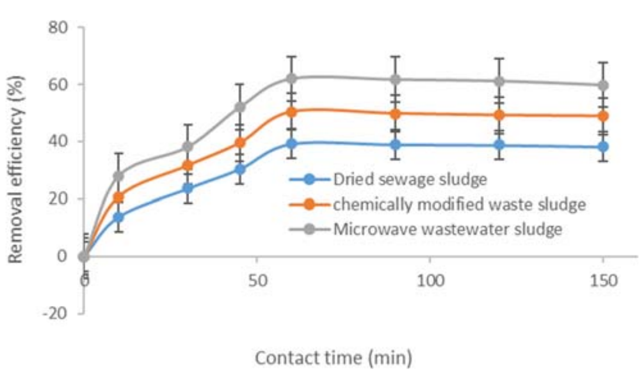

Fig. 2 The effect of different contact times on the adsorption of 2,4 DNP on dried sludge, modified sludge, and microwaved sludge adsorbents (initial pH = 7, c_0 = 50 mg/L, adsorbent dose = 0.5 g/40 mL)

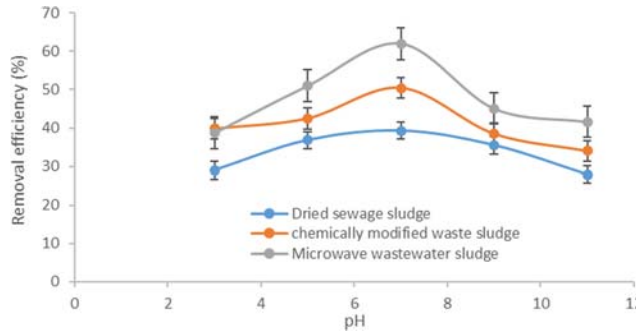

Fig. 3 The effect of different solution pH's on the extent of adsorption of 2,4 DNP on the dried sludge, modified sludge, and microwaved sludge adsorbents (contact time = 60 min, c_0 = 50 mg/L, adsorbent dose = 0.5 g/40 mL)

$$q_e = K_F c_e^{1/n} \quad (9)$$

In these equations, C_e is the equilibrium solution concentration of the adsorbate (mg/L), q_e the amount of adsorbate adsorbed per unit mass of solid at the time of t (mg/g), q_m the maximum adsorption capacity (mg/g), K_L is the Constant absorption equilibrium(L/mg), K_F and n are Freundlich constants in which the K_F and n indicates constants associated with the sorption capacity (mgg^{-1}) and intensity [34–36].

Results

The properties of the three utilized adsorbents are provided n Table 1. Also, SEM images for the three adsorbents are shown in Fig. 1.

The results of contact time on the adsorption of 2,4 DNP on the dried sludge, modified sludge, and microwaved sludge are shown in Fig. 2; the removal percentage by these adsorbents has been 39.24, 50.4, and 62%, respectively, with the optimal time obtained as 60 min.

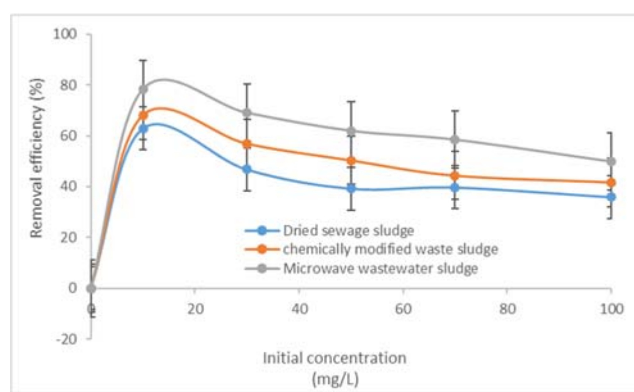

Fig. 4 The effect of different concentrations of 2,4 DNP on the adsorbents of dried sludge, modified sludge, and microwaved sludge (optimal time, optimal pH, and adsorbent dose 0.5 g/40 mL)

Table 2 Adaptation of equilibrium data with Langmuir and Freundlich linear models

Isotherm Freundlich			Isotherm Langmuir			adsorbent
1/n	k_f	R^2	b	q_{max}	R^2	Dried sewage sludge
0/604	0/218	0/9896	0/0248	4/355	0/8401	
0/613	0/273	0/9977	0/033	4/752	0/9477	chemically modified waste sluge
0/60	0/411	0/9933	0/050	5/494	0/9835	Microwave wastewater sludge

The effect of pH on the adsorption of 2,4 DNP on the dried sludge, modified sludge, and microwaved sludge adsorbents was tested at pH's 3–11, with its results shown in Fig. 3. The maximum removal of pollutants by the three adsorbents occurred at pH 7. Larger or smaller values lead to diminished adsorption of 2,4 DNP in all of the three adsorbents.

The effect of the initial concentration of 2,4 DNP on the removal efficiency by the three mentioned adsorbents was tested within the range of 10–100 mg/L (Fig. 4). The results indicate that adsorption is heavily dependent on the initial pH concentration of the solution; as the pollutant concentration rose from 10 to 100 mg/L, the extent of removal of this pollutant by dried sludge dropped from 63 to 35.9%. And In the modified sludge adsorbent, the efficiency of removal change decreased by increasing the concentration of 2,4 DNP from 10 mg/l to 100 mg/l to 68.2% to 41.7%. Finally, in the microwaved sludge adsorbent, the extent of changes in the removal efficiency with an elevation of 2,4 DNP concentration from 10 to 100 mg/L diminished from 78.3 to 49.9%. Nevertheless, in all of the three adsorbents, the trend of adsorption capacity has been different, and with concentration elevation, the adsorption capacity increased.

The results of investigating different amounts of adsorbent and its efficiency in the removal of 2,4 DNP are shown in Fig. 5. The increase in the content of adsorbent led to enhance the removal of contaminants by all of the three adsorbents. Increased adsorption by elevating the adsorbent content is associated with more adsorption sites and greater surface area at higher doses. The Langmuir and Freundlich equations are among the standard models used to fit data with adsorbents, and the results of non-linear fitting for all three adsorbents followed Freundlich's model according to fig. 6. The information related to the correlation coefficients of each of the Langmuir and Freundlich models is shown in Table 2. The correlation coefficients of Langmuir and Freundlich isotherm models for the adsorption of 2,4 DNP on dried sludge, modified sludge, and microwaved sludge indicated that The highest correlation coefficient was related to the Freundlich model. The results of kinetic studies are listed in Table 3. The results showed that the pseudo-second order model provides a better correlation for adsorption of 2,4 DNP on dried sludge, modified sludge, and microwaved sludge compared to pseudo-first order model and interparticle diffusion. Moreover, the graphs of the non-linear fitting results for all three adsorbents confirm the adherence to the pseudo - quadratic model, as shown in the Figures of 7. Table 4 compares the adsorption capacity of the three adsorbents utilized in this research and other studies.

Discussion

In this study, it was found that by an increase in time and reduction of the initial concentration of phenolic compounds, the extent of removal increases. At the beginning of the adsorption process, due to many adsorption sites, there is a significant difference between the adsorbent concentration in the solution and its amount on the adsorbent surface. This increase in concentration difference leads to enhanced adsorption in the primary stages of adsorption [42]. Based on results, the removal efficiency of the pollutant in all of the three adsorbents was maximum within

Table 3 Correspondence between the obtained data and adsorption kinetics linear models

Intraparticle diffusion			Pseudo-second order			Pseudo-first order			
K_d	C	R^2	k_2	qe	R^2	k_1	Qe	R^2	adsorbent
0/034	0/1949	0/894	1/684	23/774	0/997	0/0038	0/629	0/835	Dried sewage sludge
0/038	0/1936	0/932	1/46	29/603	0/996	0/0036	0/463	0/868	chemically modified waste sluge
0/046	0/198	0/917	1/23	29/54	0/993	0/0019	0/03	0/529	Microwave wastewater sludge

60 min, after which it diminished and reached a constant value at 120 min. In a study by Darvishi et al. in 2009 entitled "cadmium adsorption by excessive sludge of urban wastewater active sludge process," it was observed that the optimal type of adsorption was 60 min [13]. pH affects the electric charge of the adsorbent surface. Different charges at different pH's influence on the adsorption process [26]. The maximum 2,4 DNP removal occurred at pH = 7 in all of the three adsorbents. At low pH, the adsorbent surface is protonated, leading to further adsorption of phenolic ions. These ions are negatively charged and are directly absorbed on the adsorbent surface via electrostatic force. At high pH, OH^- ions compete with the molecules of the phenolic compounds to acquire the adsorption sites [42], where pH variations through the impact of pH_{zpc} of the adsorbent surface have affected the pollutant adsorption efficiency [43]. At pH above pH_{zpc}, the adsorbent has a negative charge, while at pH below it, it has a positive charge; since the superficial charge of all three adsorbents at pH is less than $pH_{zpc,}$ all of the three adsorbents have a positive charge. Considering the anionic nature of the phenolic compound, in these conditions, electrostatic attraction between the adsorbent and pollutant increases, and so does the removal efficiency [44, 45]. Norton et al. (2004) examined zinc adsorption by discharged active sludge. In that study, the optimal pH was obtained as 4 [46]. With the elevation of the initial concentration of compounds, their removal efficiency diminishes, but their adsorption capacity increases. This is because, by elevating the superficial charge of absorbates on the adsorbent, the adsorption sites of the top levels are rapidly saturated on the adsorbent, whereby the removal efficiency diminishes [10]. Different doses of these three adsorbents (0.1–1.5 g) were tested in this study. The results showed that by reducing the adsorbent content, the extent of pollutant removal decreased, where the maximum extent of removal for this phenolic compound was related to 1.5 g. This is because of the fact that higher contents of adsorbent lead to more adsorption sites for phenolic compounds. Gulay Bayramoglu et al. indicated that with increase in the adsorbent dose, the adsorption percentage grows dramatically [9]. The results obtained from the adsorption data showed that adsorption of 2,4 DNP in both linear and non-linear methods follows Freundlich isotherm, suggesting their multilayer adsorption on all of the three adsorbents. Freundlich coefficient value K_F and 1/n from the linear diagram $\log q_e$ versus $\log c_e$ alongside their correlation coefficient have been shown. Freundlich constant 1/n for this pollutant is close to the unit value, suggesting that the adsorption process is under suitable experimental conditions [31]. In adsorption kinetic studies, it was observed that the kinetics of this phenolic compound follows pseudo-second order equation. This equation is based on the assumption that the determinant step for the reaction rate may be related to chemical adsorption, which involves valence force through sharing or exchanging electrons between the adsorbent and absorbate [9, 47]. In relation to kinetic equations, both linear and non-linear methods were used. In the linear method, the rate constant of the pseudo-quadratic equation k_1 and the value of qe were calculated by drawing ln (qe-qt) versus t, and the results are shown in Table 3 are given. Regarding kinetic equations, pseudo-first order equation rate constant k_1 and q_e value was calculated from plotting ln (q_e-q_t) versus t, with its results presented in Table 2. In this model, the correlation coefficient (R^2) has been low, suggesting its low correlation. Thus, adsorption of 2,4 DNP by these three adsorbents does not match pseudo-first order model. The C resulting from the intraparticle diffusion equation was nonzero, and its correlation coefficient was also low. Accordingly, the intraparticle diffusion model is not a controlling factor in determining the process kinetics. Pseudo-second order kinetic coefficient k_2 and q_e value were selected from the model, with its results presented in Table 3. The correlation coefficient in this model is high for all of the three adsorbents. And the highest correlation coefficient obtained from the non-linear method was in the quasi-quadratic

Table 4 Comparing different adsorbents in the removal of 2,4DNP

Adsorbent	q_{max} (mg/g)	Reference
Charcoal activated powder	1.01	[37]
Activated carbon fibers	2.27	[38]
cross-linked starch-based polymers	5.43	[39]
Molecularly Imprinted Polymer	2.87	[40]
activated carbons	2.96	[41]
Dried sewage sludge	4.35	This study
chemically modified waste sludge	4.75	This study
Microwaved wastewater sludge	5.49	This study

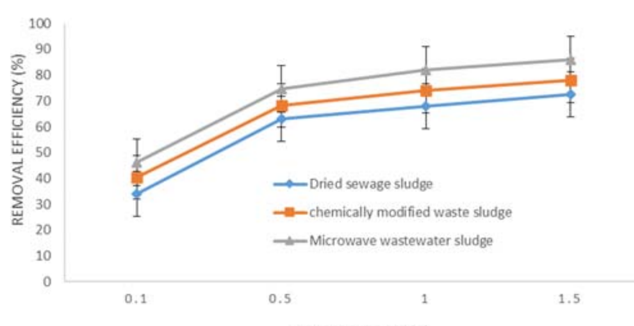

Fig. 5 The effect of different doses of adsorbents of dried sludge, modified sludge, and microwaved sludge (optimal time, optimal concentration, optimal pH)

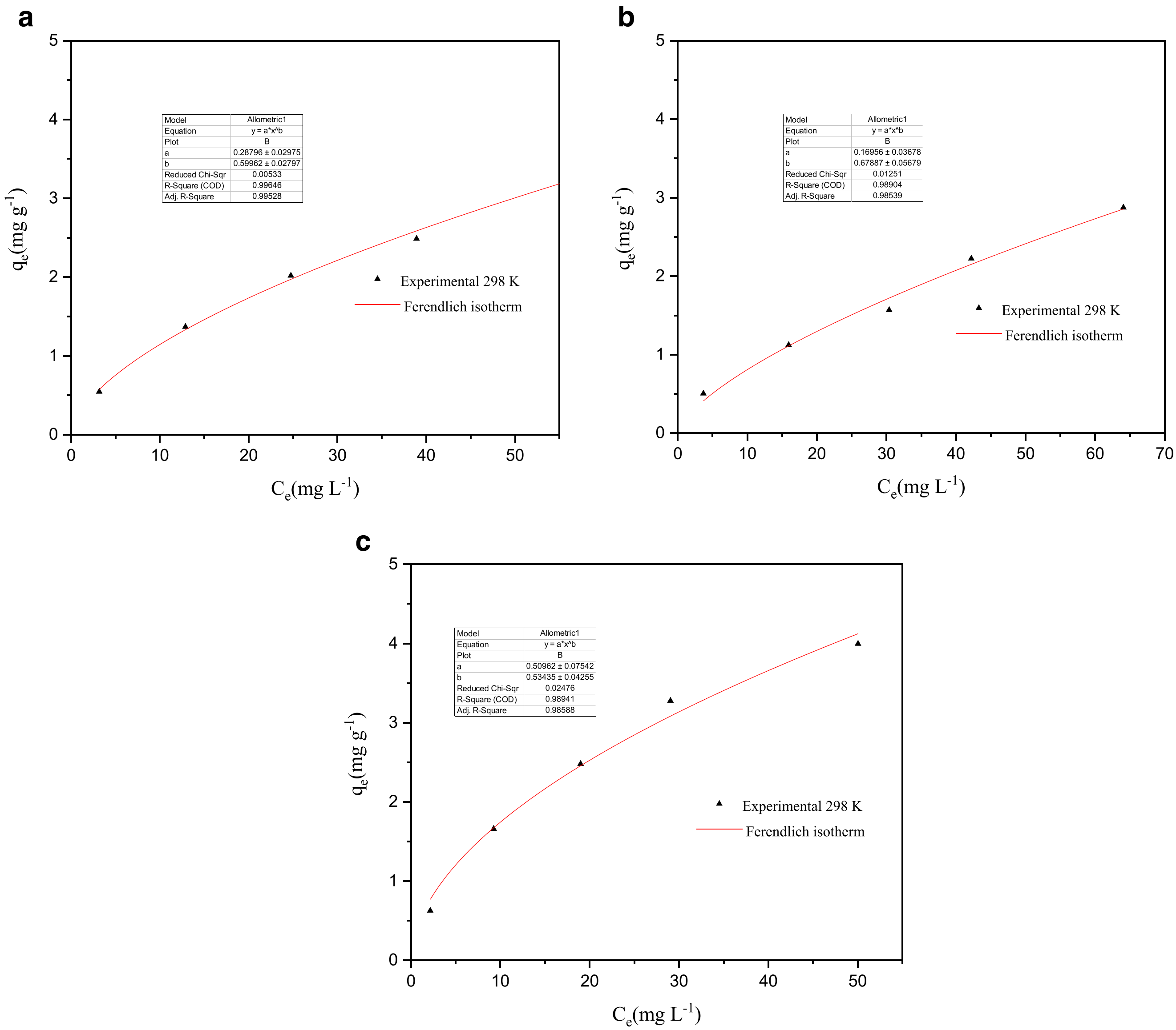

Fig. 6 Non-linear diagram of the sludge's Friendlich isotherm for A) the chemically modified sludge, B) the dried sewage sludge and, C) the microwave wastewater sludge

kinetics model. The fact that the adsorption process follows the pseudo-second order model indicates that in the adsorption process, two reactions are useful in parallel for the adsorption of the absorbate on the adsorbent; the first is fast and reaches equilibrium quickly, while the second is slow and continues for a long time [22]. Table 4 compares the adsorbents studied in this paper with other adsorbents utilized for the removal of 2,4 DNP from aqueous solutions. It indicates that the adsorbents used in this study, especially the use of microwaves in drying sludge due to chemical processing and then the use of microwave radiation, which penetrates into the deeper parts of the adsorbent and removes interstitial fluid, causing porosity and greater adsorption capacity of that adsorbent than the other two ones in this study. The utilized adsorbents have better adsorption capacity in this study and lower adsorption capacity than the adsorbents reported in this table.

Conclusion

The process of adsorption of 2,4 DNP on the three adsorbents occurred quickly, where the maximum extent of adsorption was at 60 min, after which it decreased and reached a constant value at 120 min, and later desorption occurred. The removal efficiency of the pollutant on all

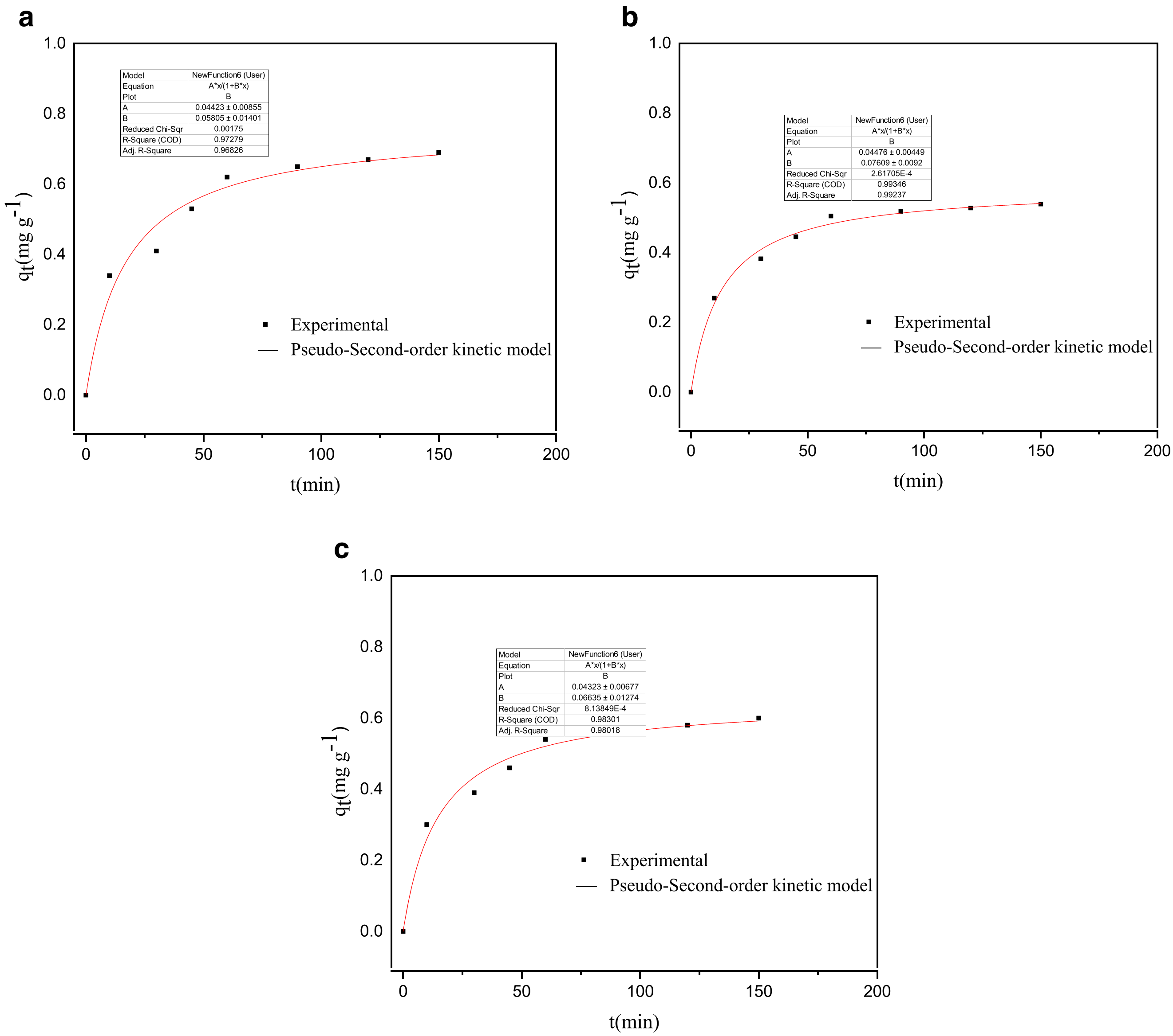

Fig. 7 Non-linear pseudo-second-order diagrams of A) the chemically modified waste sludge, B) the dried sewage sludge and, C) the microwave wastewater sludge

three adsorbents was maximum at pH = 7, while for pH's above and below that, removal efficiency decreased. The results of changes of concentration on the removal efficiency showed that by increasing 2,4 DNP concentration, its removal level diminishes, but the adsorption capacity increased. Concerning the impact of the adsorbent dose, with a reduction of the adsorbent content, the extent of adsorption decreased. In kinetic studies, the results of the experiment for all of the three adsorbents followed pseudo-second order model. In equilibrium studies, 2,4 DNP adsorption on all three adsorbents followed Freundlich isotherm, suggesting uniform and multilayer distribution of active sites on the adsorbent. Eventually, it can be stated that since the adsorption process is relatively common in the treatment of aqueous media, alternative and inexpensive adsorbents available in our countries such as dried sludge and process sludge can be suggested which have good efficiency in the removal of pollutants including 2,4 DNP from aqueous solutions.

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Compliance with ethical standards

Conflict of interest The authors of this article declare that they have no conflict of interests.

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