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Adsorption capacity of NH_4Cl-induced activated carbon for removing sodium dodecyl sulfate from water

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ABSTRACT

Surfactants are a very important group of compounds that are used extensively in modern life and have adverse effects on human health and aquatic life. The aim of this study was to evaluate the adsorption characteristics of sodium dodecyl sulfate (SDS) on standard activated carbon (SAC) and NH_4Cl-induced activated carbon (NAC). It was found that, in the batch experiment conducted at 50 mg/L initial concentration of SDS, pH of neutral, and adsorbent dose of 1 g/L, SAC and NAC could remove up to 81 and 99% of SDS from the solution. The removal of SDS by both adsorbent followed the pseudo-second-order reaction kinetics. Results of the equilibrium experiments revealed that the adsorption of SDS onto SAC and NAC was better fitted by the Langmuir model. The maximum adsorption capacity of SDS onto SAC and NAC was 117.2 and 178.6 mg/g, respectively. The results suggested that NAC is suitable as a sorbent for the adsorption of SDS from aqueous solutions in view of its effectiveness and high capacity.

Keywords: Activated carbon; Adsorption capacity; Anionic surfactant; Kinetic coefficients

1. Introduction

Surfactants are the substances that decrease the surface tension of a solvent. They have cleaning and solubilization features [1]. These compounds contain both hydrophobic and hydrophilic groups. The hydrophobic group is usually a hydrocarbon radical with 10–20 carbon atoms. The hydrophilic part based on the type of surfactant may or may not be ionized [2]. Surfactants based on the charge of molecular chain are classified into four groups: anionic, cationic, zwitterionic, and non-ionic [3].

Due to simple synthesis and low production costs, anionic surfactants are the main class of surfactants that are used in detergent formulations [4]. Alkylbenzene sulfonate, linear alkylbenzene sulfonate, and linear alkyl sulfate are the major classes of anionic surfactants. One of the examples of linear alkyl sulfate is sodium dodecyl sulfate (SDS). Today, synthetic surfactants are widely used in domestic washing detergents, personal care products, textiles, paints, polymers, pesticide preparations, medicines, mining, oil recovery, and paper industries [1].

It has been estimated that the amount of surfactant which is produced worldwide is about 10 million ton per year, and anionic surfactants account for about 60% of them. Textile industries consume 10% of the

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surfactant produced in the world, and the wastewater of these industries contains high concentration of surfactant [4]. Surfactants are harmful for both terrestrial and aquatic life and cause health threats such as dermatitis and eye irritation. Furthermore, surfactants persist in biota for a long time [5], and the presence of these compounds in aqueous media diminishes the quality of water, causes unpleasant taste and odor, produces foams in rivers and wastewater treatment plants, and causes both short- and long-term disorders in the receiving ecosystems. Surfactants reduce the surface tension of water, which contributes to their easy penetration into *in vivo* of aqueous media and thus adversely affects aquatic life. Also, they have been found to interact synergistically with other toxic chemicals [6]. Some of them are biodegradable, but many of them are not [7]. Due to their toxic nature, surfactant concentrations in water and wastewater should be reduced to acceptable levels [7]. Many environmental organizations have set 0.5 mg/L as the standard level for anionic detergents in drinking water and up to 1.0 mg/L for other purposes [2].

The conventional methods that are used for surfactant removal from water include physical, chemical, and biological processes. They are chemical or electrochemical oxidation [8], membrane technology [9], liquid–liquid extraction [10], chemical precipitation [11], photocatalytic degradation [12], biological degradation [13], and adsorption or electrosorption [3]. Some of the surfactants are biodegraded partly under aerobic conditions; but, most of them are not biodegradable [3]. Chemical processes are cost-intensive; thus, they are not appropriate for scale application or are incapable of meeting the permissible limits of surfactants [3,6]. Accordingly, the physical techniques might be the method of choice for removing water contaminants including surfactants. Adsorption is one of the most conventional physical methods used for removing pollutants, especially organic compounds, from water and wastewater [7]. Adsorption is efficient, simple for design and operation, unaffected by toxicity, and inexpensive for the accumulation of organic compounds from contaminated streams into a solid material. Saturated adsorbent is separated and regenerated/disposed using an appropriate method [14]. Due to the main features of adsorption, this method is frequently used in full scale for the removal of organic and refractory compounds from water and wastewater [15].

The characteristics of adsorbents have an important role in the performance of an adsorption process [14]. Many adsorbents such as alumina [2], zeolites [16], sediment [17], bentonite [18], sand [19], silica gel [20], resins [21], rubber granules [22], and activated carbons [23] have been investigated for the removal of surfactants [4]. Due to porosity and very high specific surface area and capacity, activated carbon is the most widely used adsorbent for removing various compounds from contaminated streams. However, the main defects of activated carbon are high cost of production and regeneration [14]. Therefore, new carbons must be investigated for introducing a more efficient and thus cost-effective adsorption process.

In this study, the capability of a newly prepared NH_4Cl-induced activated carbon (NAC) is compared with the standard activated carbon (SAC) in the adsorption of SDS from contaminated water. Effects of the most important variables including solution pH, adsorption concentration, SDS concentration, and reaction time are evaluated. Also, the equilibrium, kinetics, and mechanism of SDS adsorption onto SAC and NAC are determined.

2. Material and methods

2.1. Reagents and adsorbents

SDS ($CH_3\ (CH_2)_{11}SO_4^-\ Na^+$, 288 g/mol), Acridine orange (ACO) chemically known as 3, 6-bis (dimethylamino) acridine, glacial acetic acid, toluene were purchased from Merck Company. All the chemicals that used in this study had high purity and analytical grade.

Aqueous solutions of the surfactant were prepared with deionized water. A stock solution of SDS was made by dissolving 1 g of analytical grade SDS in distilled water. In this study, two types of activated carbon were used: NAC and SAC. The NAC was prepared from pomegranate wood waste. The specific surface area determined by the Brunauer–Emmett–Teller approach in SAC and NAC were 1,024 and 1,029 m^2/g, respectively. Other properties and more information about SAC and NAC were reported in the previous works [14]. Activated carbon from Merck Company was used as the SAC to make a comparison and highlight the adsorption potential of the NAC prepared in this study.

2.2. Instrumentations

An electrical balance (One I 160) was used for weighting. A digital pH meter (Jenway, 3505) was used for pH measurements. A spectrophotometer (UV–vis, Unico, 4802) was used for measuring absorbance.

2.3. Analytical method

A quick and reliable solvent extraction spectrophotometric method was used for determining anionic surfactants. ACO has the capability to make an ion-pairing agent with anionic surfactants. This ion-pair component is extractable in toluene. 10 mL of the sample solution containing SDS in the range of 0.1–6.0 ppm was transferred to a 25 mL separating funnel. ACO (0.005 M) and glacial acetic acid, 100 µL each, were then added and 5 mL of toluene was added to it and shaken for 1 min. After 5 min, the aqueous layer was discarded and the toluene layer was used for absorbance measurement at 467 nm [2,24].

2.4. Batch adsorption studies

All the batch tests were carried out using synthetic SDS solutions at room temperature. Solutions of 1 M HCl or NaOH were used to adjust the solution pH prior to the reaction to achieve an optimum pH. Parameters of this test were as follows: solution pH (2.0–12.0), initial SDS concentration (50–500 mg/L), and reaction time of 1–60 min in kinetic experiment and 2 h in equilibrium tests. All the tests were conducted in duplicate, and mean of these two measurements was taken to represent each evaluation. The experimental runs and conditions are given in Table 1.

It was performed by introducing a mass of SAC and NAC into a glass conical flask containing 100 mL of an aqueous SDS solution with 50, 100, and 200 mg/L concentrations. The solution was agitated by a magnetic stirrer at 100 rpm for the desired period. Then, the separation was filtered with 0.2-µm pore size, and the filtrate was analyzed by UV–vis spectroscopy at the wavelength of 467 nm to determine the residual concentration of SDS. The adsorbed amount of SDS was obtained by measuring the SDS concentration solution before and after exposing using SAC and NAC. The amount of SDS adsorbed by the SAC and NAC q_e (mg/g) is computed as (Eq. (1)):

$$q_e = \frac{V(C_0 - C_e)}{M} \tag{1}$$

The removal efficiency is expressed by (Eq. (2)):

$$R = \frac{(C_0 - C_e)}{C_0} \tag{2}$$

where V is the volume of the solution in L, C_0, and C_e are initial and equilibrium concentrations in mmol L^{-1}, respectively, M is the mass of NAC in g, and R is removal efficiency of SDS.

2.5. Adsorption isotherms

The adsorption isotherm determines the equilibrium condition of the process [20]. The adsorption isotherm experiment was done at room temperature with 100-mL solutions of SDS at varying initial concentrations (50–500 mg/L) while keeping SAC and NAC doses fixed at 0.5 g/L. The flasks were shaken at 100 rpm for 2 h to attain equilibrium, after which the suspension was analyzed as described in Section 2.3. The equilibrium adsorption of SDS onto the prepared SAC and NAC was modeled using isotherm models (Langmuir and Freundlich) by fitting results from the experiment onto the models.

2.6. Adsorption kinetics

Kinetic study is very significant in batch experiments to determine the optimum contact time of the adsorbent with adsorbate and to assess reaction rate coefficients. For designing adsorption system, kinetic is a key parameter which is needed to choose optimum operating situations for batch studies [6]. The kinetic study was done on the SDS solution (initial concentration: 100 mg/L) with SAC and NAC as the adsorbent (dose 0.4 and 0.6 g/L) and the shaking time was varied from 1 to 60 min. The amount of adsorbed

Table 1
The experimental runs and conditions

No.	Run experiment	SDS concentration (mg/L)	pH	Adsorbent dose (g/L)		Contact time (min)
				SAC	NAC	
1	Effect of solution pH	50	2–12	1	1	30
2	Effect of SDS concentration	50–200	Natural	0.1–1	0.1–1	30
3	Equilibrium adsorption	50–500	Natural	0.5	0.5	120
4	Adsorption kinetic	100	Natural	0.4, 0.6	0.4, 0.6	1–60

surfactant was determined by measuring the solution concentration after the equilibrium with the adsorbent.

3. Results and discussion

3.1. Influence of process variables

3.1.1. Effect of adsorbent dose on adsorption

The amount of SDS that can be removed from a solution by adsorption using SAC and NAC depends on the process variables used in batch systems such as adsorbent dosage, initial SDS concentration, and contact time [6]. In any adsorption process, the amount of adsorbent plays an important role. It is very necessary to find optimum adsorbent dose and contact time for the maximum possible removal of the adsorbate. The effect of SAC and NAC doses was examined at various SDS concentrations of 50, 100, and 200 mg/L with a varying dose (0.1–1 g/L) of adsorbent. Percentage of SDS removal against adsorbent dose, after 30 min, is shown in Fig. 1. It can be observed that the removal of SDS was increased with the increase of SAC and NAC dose. At the SDS concentration of 50, 100 and 200 mg/L with 0.1 g NAC, removal efficiency was 99, 92.8, and 62.6%, respectively, while in SAC and similar situation, removal efficiency was 81, 42.5, and 29.1%, respectively.

The increase of removal efficiency should be due to the increase of adsorption sites available when more adsorbent is used. It must be noted that the removal extent of SDS could not be further increased when the adsorbent increases excessively, which might be due to the particle interaction, such as accumulation, resulting from high adsorbent concentration. Such accumulation causes a decrease in the total surface area of adsorbent and an increase in diffused path length [25]. Furthermore, surface chemistry of activated carbon plays a more important role in controlling the adsorption of a relatively hydrophobic molecule from dilute aqueous conditions than pore size distribution [26]. According to the previous work, FTIR spectrum showed greater density of functional groups observed on the surface of NAC than SAC. Also, the surface area of NAC was more than that of SAC. The surface morphology of NAC and SAC was different: NAC surface had a fibrous structure consisting of a series of long parallel, similarly shaped channels with a smooth surface, but SAC had a smooth surface with scattered hollows of various sizes [14]. Also, the total volume and size of pores in NAC were around 10% greater than those in SAC. The role of NH_4Cl can be attributed to the destruction of the carbon texture likely due to the generation and release of the excessive gas related to the evaporation and volatilization of ammonium impregnated onto carbon at high activation temperature [14]. For these reasons, adsorption capacity of NAC was greater than SAC's.

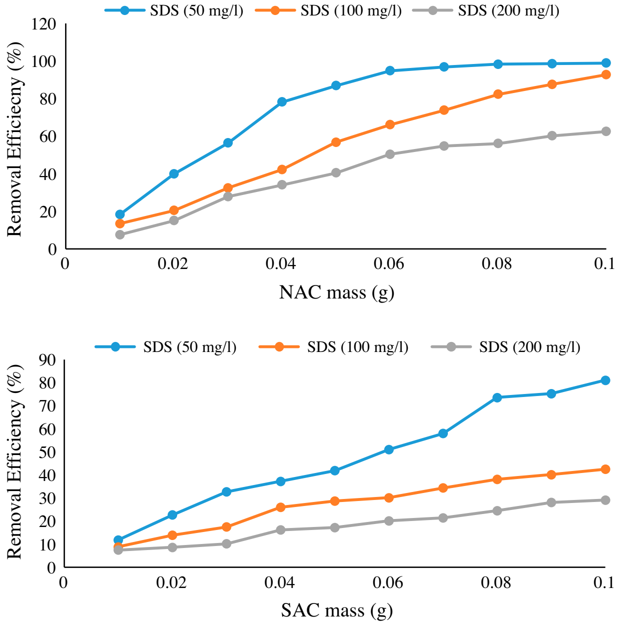

Fig. 1. The adsorption of various concentrations of SDS onto NAC and SAC as a function of adsorbent mass.

3.1.2. Effect of SDS concentration on adsorption

The adsorption of SDS depends on the initial SDS concentration (C_i), because it provides the driving force to overcome mass transfer limitations onto SAC and NAC surfaces [6]. The effect of initial SDS concentration in the range 50–500 mg/L, while keeping the SAC and NAC dose fixed at 0.5 g/L, was examined. The residual SDS concentrations were determined after reaching the equilibrium. It has been observed that the removal of SDS decreased gradually with the increase of initial SDS concentration. But, the amount of the SDS adsorbed onto SAC and NAC was increased with an increase in the initial SDS concentration, because with increasing initial SDS concentration, the driving force of the concentration gradient was also increased [6]. At low SDS concentrations, the active binding sites were occupied fast or bound to the surface mainly by electrostatic attraction, while at higher SDS concentrations, the active binding sites got saturated and formed monolayer or bilayer patches on the surface that contributed to the increased adsorption and mass transfer from the liquid to solid phase [27,28].

3.1.3. Effect of solution pH and adsorption mechanism

The pH of the aqueous solution is an important controlling parameter in adsorption by various adsorbents [19], because it affects the surface charge of the adsorbent, degree of ionization, and species of adsorbate. In many works that have been carried out in order to clarify the mechanism of organic molecules from aqueous solutions on carbon-based materials, a complex interaction between electrostatic and non-electrostatic forces has been reported. Also, these interactions depend on the characteristics of the adsorbent and adsorbate as well as the chemical properties of the solution. It has been observed that adsorption capacity is negatively influenced by the presence of similar groups on adsorbent surface and adsorbate molecule [29]. In the present study, the effect of pH on the adsorption of SDS (initial concentration: (50 mg/L) by SAC and NAC (dose: 1 g/L) was studied within the pH range of 2.0–12.0. The residual SDS concentrations were measured after 30 min of adsorption. The results of the adsorption of SDS on SAC and NAC at different pH levels are plotted in Fig. 2. In our study, no significant influence of pH on SDS adsorption by NAC was observed and removal efficiency at all pH levels was 98–99%. In contrast, in SAC, with increasing pH, the removal efficiency was decreased. The maximum removal was observed at pH 2 with 81.8% and the minimum removal was obtained at pH 12 as 56%, because pH change has no effect on SDS adsorption proposes that, along with the ionic interaction, there must be some hydrophobic interactions between the hydrophobic functions of the adsorbate and adsorbent molecules [6]. Raval and Desai [24] reported the maximum adsorption of SDS on alumina at pH 4.

The relationship between effects of SAC and NAC in SDS adsorption and solution pH can be described by considering both the surface charge of the adsorbent and the dissociation constant (pK_a) of SDS The pHpzc of SAC and NAC were 7.4 and 6.6, respectively. SDS has pK_a 1.9. At this pH, the adsorption capacity is almost zero. At low pH, the SAC and NAC particles become more positive and hence there is an increase in adsorption at lower pH.

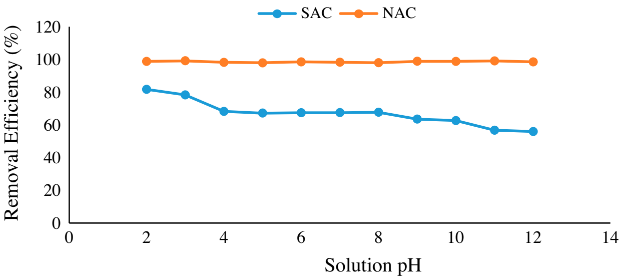

Fig. 2. Equilibrium uptake of SDS at different solution pH values in 50 mg/L initial concentration.

3.2. Adsorption behaviors and kinetics of SDS

The kinetic adsorption was determined by contacting SDS concentration of 100 mg/L and SAC and NAC with dose in the range 0.4 and 0.6 g/L for 1–60 min. The results in Table 2 indicate that the uptake of SDS was increased with an increase in contact time. Afterward, the SDS concentration stayed almost unchanged and equilibrium was reached. Adsorption kinetics describes reaction pathways and rate of adsorbate uptake on the adsorbent to reach the equilibrium [30]. In order to predict the adsorption kinetic model for SDS onto SAC and NAC, pseudo-first-order, pseudo-second-order, and intraparticle diffusion kinetic models were applied to the data at different doses of adsorbents.

The pseudo-first-order model equation:

$$(\ln q_e - q_t) = \ln q_e - k_1 t \tag{3}$$

The pseudo-second-order model equation:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \tag{4}$$

where k_1 and k_2 are adsorption rate constants, q_t is adsorption capacity at time t, and q_e is adsorption capacity in equilibrium conditions. In order to test these models, linear regression analysis was applied to plot ln ($q_e - q_t$) and t/q_t vs. t data for the pseudo-first-order and pseudo-second-order, respectively. The parameters and regression coefficients of the respective models are given in Table 2. It can be concluded that the kinetic of SDS adsorption onto the SAC and NAC from the solution obeys the pseudo-second-order model with the R^2 value of 0.99 for both adsorbents. The q_e values calculated from the model were also in good agreement with the experimental values. The fitting experimental data from this study with the pseudo-second-order model implies that the availability of adsorption sites on the SAC and NAC plays an important role in adsorption rather than SDS concentration in bulk solution [31]. The magnitudes of SDS removal at the initial concentration of 100 mg/L by SAC and NAC at different adsorbent doses (0.2, 0.4, and 0.6 g/L) are presented in Fig. 3. For the first 30 min, the removal rate by NAC was very fast and then it was gradually slowed down.

Table 2
The kinetics information of SDS adsorption onto NAC and SAC

			Pseudo-second-order kinetic		
Adsorbent	Adsorbent dose (g/L)	$q_{\text{e-test}}$	R^2	q_e (mg/g)	$K_2 \times 10^3$ (g/mg min)
SAC	0.4	82.6	0.994	87.3	0.35
	0.6	62.4	0.992	72.5	0.42
NAC	0.4	125.7	0.996	126.6	2.2
	0.6	122.4	0.999	122	4.3

The two main steps involved in mass transfer in the adsorption of organic molecules onto adsorbents are the liquid phase mass transport (film diffusion) and the intraparticle mass transport (pore diffusion or intraparticle diffusion) phases [14]. Prediction of the rate-limiting step is an important factor to be considered in adsorption process. The adsorption process of the adsorbate molecules from the bulk liquid phase onto the adsorbent surface is presumed to be composed of three stages: (1) Mass transfer of the adsorbate molecules across the external boundary layer; (2) intraparticle diffusion within the pores of the adsorbent; and (3) adsorption at a site on the surface. The intraparticle diffusion model provided the rate for intraparticle diffusion by a relationship between q_t and square root of time, $t^{1/2}$, as shown in (Eq. (5)) [32].

$$q_t = k_{\text{id}} t^{0.5} + C \tag{5}$$

In these equations, k_{id} is the constant of intraparticle diffusion. By plotting q_t vs. $t^{0.5}$ for intraparticle diffusion model, a very poor linear fit was found. The results showed that the model was not fit and it was concluded that the intraparticle diffusion model is not the rate controlling step.

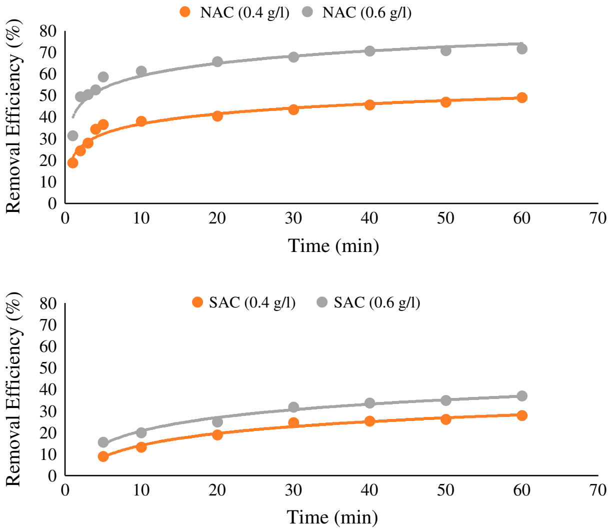

Fig. 3. Adsorption kinetic profiles for SDS (C_i = 100 mg/L) removal on NAC and SAC at different adsorbent dose.

3.3. Equilibrium of SDS and adsorption isotherms

For understanding the adsorption process, the equilibrium isotherms are very important. Adsorption isotherm expresses the relationship between the amount of a solute adsorbed at constant temperature on adsorbent and its concentration in the equilibrium solution. It gives important physiochemical data for evaluating the applicability of the adsorption process as a complete unit operation [32]. Many theoretical and empirical models have been published to describe the various types of adsorption isotherms. The Langmuir and Freundlich isotherm have been widely used to explain the relationship between the amounts of adsorbate that is adsorbed and its equilibrium concentration in the solution [30]. The equilibrium adsorption results were evaluated using Langmuir and Freundlich isotherm as given in Eqs. (6) and (7):

Langmuir isotherm:

$$\frac{C_e}{q_e} = \frac{1}{bq_{\max}} + \frac{C_e}{q_{\max}} \tag{6}$$

where q_e (mg/g) is the equilibrium amount of SDS adsorbed per gram of SAC and NAC, C_e (mg/L) is the equilibrium concentration of SDS, $q_{\max}$ (mg/g) and b (L/mg) are maximum adsorption capacity for adsorbent and the energy constant related to the heat of adsorption, respectively. By plotting linear regression C_e/q_e vs. C_e, values of $q_{\max}$ and b are determined. The Langmuir parameters can be used for predicting affinity between adsorbate and adsorbent by the dimensionless separation factor (R_L) expressed below:

$$R_L = \frac{1}{1 + bC_i} \tag{7}$$

where R_L is the dimensionless separation factor and C_i is the initial concentration of the adsorbate (mg/L). The adsorption system can be favorable if ($0 > R_L > 1$), unfavorable ($R_L > 1$), linear ($R_L = 1$), and irreversible ($R_L = 0$).

Freundlich isotherm:

$$\ln q_e = \ln k_f + \frac{1}{n} \ln C_e \tag{8}$$

where K_f is a characteristic parameter of the adsorption capacity (mg/g) and n refers to the adsorption intensity. These two constants were obtained by plotting ln q_e vs. ln C_e. The lower fractional values of $1/n$ [$0 < (1/n) < 1$] show that weak adsorptive forces were effective for the surface of activated carbon. Values of $n > 1$ represent a favorable adsorption condition, suitable for highly heterogeneous surfaces [33].

The equilibrium adsorption isotherms of SDS were determined at room temperature with 0.05 g adsorbent and SDS concentration in range of 50–500 mg/L for 2 h. The parameters and coefficients obtained from the experimental data are shown in Table 3. The results obtained from the Langmuir equation demonstrated better correlation to the experimental data than the Freundlich equation. The correlation coefficients (R^2) for the linear equation were greater than 0.99. The maximum adsorption capacity (q_{max}) at room temperature for SAC and NAC was 117.2 and 178.6 mg/g, respectively. The validity on the Langmuir isotherm depicted the monolayer adsorption onto the surface containing a finite number of identical sorption. A similar behavior has been obtained in a number of studies [22,34]. In two adsorbents, R_L expresses that the isotherm has a favorable behavior.

Table 3
The isotherm information of SDS adsorption onto NAC and SAC

	Adsorbent	
Isotherm	SAC	NAC
Langmuir		
R^2	0.991	0.9934
q_{max}	117.2	178.6
b	0.068	0.014
R_L	0.03–0.23	0.13–0.59
Freundlich		
R^2	0.986	0.8848
K_F	13.1	68.6
n	2.9	6.3

Table 4
SDS adsorption capacities by different adsorbents

Adsorbent	q_{max} (mg/g)	References
Granite sand	1.3	[19]
Montmorillonite	48.3	[35]
Tire rubber granules	4.164	[36]
Alumina	197.7	[24]
Chitosan	76.9	[6]
Silica gel	5.181	[37]
NAC	178.6	Present
SAC	117.2	Present

A comparison of the maximum SDS uptake in this study (q_{max}) with those reported in the literature for other adsorbents is presented in Table 4. It shows that the uptake capacity of NAC was comparable to other adsorbents and it can be considered as a low-cost alternative to commercially activated carbons for the removal of SDS from water. Also, NAC had higher adsorption capacity of other adsorbents.

4. Conclusions

In this study, the effectiveness of activated carbon prepared from pomegranate and induced by NH_4Cl for the uptake of SDS from water solution was evaluated. It was found that NAC, due to mesoporous particle with a higher volume of pores, greater surface area of functional groups, and greater interaction with SDS, can remove this compound efficiently from SAC in water solutions. The adsorption was not affected by pH solution, and the highest removal was observed in neutral conditions. Kinetics of SDS adsorption onto the NAC and SAC was found to follow pseudo-second-order model. Isotherm data were fitted to Langmuir model better than the Freundlich one.

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