

Sand sorption process for the removal of sodium dodecyl sulfate (anionic surfactant) from water

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Abstract

Granite sand was used to adsorb anionic surfactant, sodium dodecyl sulfate (SDS) from water at natural pH 6.25. The effect of adsorbent size, pH, temperature and amount of adsorbent has been examined. The results indicate that the Langmuir model provides the best correlation of experimental data. Thermodynamic parameters like entropy, enthalpy and free energy of adsorption were evaluated. Decreasing the temperature accelerates the adsorption of SDS onto sand surface. The kinetic data were analyzed by using pseudo-first order Lagergren equation. Adsorption of SDS was exothermic and dominated by physisorption with activation energy (E_a) 33.65 kJ mol⁻¹. In addition, regeneration of granite sand by washing with Fenton like reagent was examined. The results suggested that granite sand is suitable as a sorbent material for recovery and adsorption of SDS from aqueous solutions in view of its effectiveness and cheaper cost.

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Keywords: Granite sand; Sodium dodecyl sulfate; Adsorption; Kinetics; Activation energy

1. Introduction

Surfactants are one of the major ingredients in the formulation of various industrial and household products. Besides its routine working as a washing agent, surfactant based detergents are widely used in hospitals to eliminate pathogenic organisms, as a major component in pesticides, to disperse oil spoilage at sea and as extractant for removal of organic pollutants [1,2]. These applications of the surfactant, increasing its discharge in the wastewater, produce foam and enter into the underground water resources and constituting an ecological risk for aquatic organisms. They also create many health hazards like dermatitis and harmful for the aquatic flora and fauna [3–5].

So, the water treatment process is necessary in order to remove surfactant from industrial and domestic wastes to reduce its concentration. Some processes have already been employed for this purpose, such as aerobic and anaerobic degradation, biodegradation and sorption technique [6–14]. Adsorption of anionic surfactant has been investigated onto numerous adsorbents like activated carbon, silica gel, clay, soil, kaolinite, sand stone and rubber granules surface [15–25] etc.

In this work, we have chosen a low cost, environmental friendly, regeneration ability and widely abundant granite sand as an adsorbent. Several experiments have been carried out to optimize the adsorption process.

Granite sand is primarily composed of feldspar, quartz, microcline and mica along with various other minerals in varying percentage (e.g. 70–77% SiO₂, 11–14% Al₂O₃, 04–07% iron, 03–05% lime) and 0.264% organic content.

2. Experimental

2.1. Preparation of adsorbent

Granite sand was purchased from the local market in a very cheap rate (60 rupees per 10 kg). The sand was first sieved to remove biggest particles and washed several times with double distilled water, followed by settling and decanting. The residual solid was dried at 50 °C for 24 h.

2.2. Grain size analysis

Grain size analysis was made by sieve technique. A series of ASTM sieves was selected for this purpose. Approximately, 200 g of grinded, dried granite sand was passed

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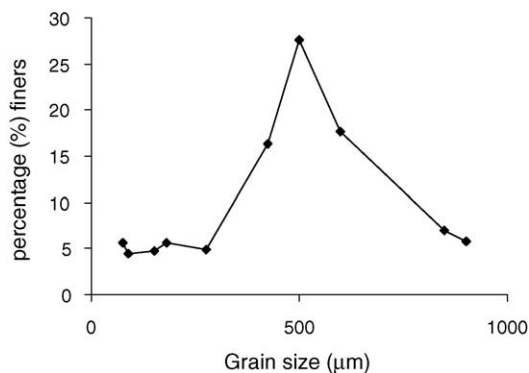


Fig. 1. Grain size analysis by the sieve technique. The plot of the grain size (in μm) versus percentage finer.

through these sieves. The Collected various fractions of particle sizes were weighted using electronic balance (Model: Mettler Toledo B204-S). Computed the cumulative weights and calculated the percent finer for each sieve size and plotted against grain size (Fig. 1). This plot showed that the particle size $500\ \mu\text{m}$ has the maximum percentage finer. Thus, size range ($475\text{--}700\ \mu\text{m}$) was used in batch sorption process.

2.3. Surfactant

The anionic surfactant sodium dodecyl sulfate (SDS) of AR grade was purchased from BDH laboratory of 100% pure. The stock solution of SDS was prepared by dissolving $0.7209\ \text{g}$ in double distilled water.

2.4. Batch sorption methods

In this study, characteristics of surfactant sorption onto the granite sand were investigated. The batch work was carried out in 100 ml Erlenmeyer flasks by transferring 10 ml of $3 \times 10^{-3}\ \text{M}$ SDS solution into the weighed sand ($7.0\ \text{g}$) and the pH of the solution was 6.25. The flasks were tightly stoppered and shaken at the rate of 125 rpm with the mechanical orbital shaker (VRN-360) for 15 min at $35 \pm 0.5\ ^\circ\text{C}$ and filtered. The adsorption studies were performed in triplicate. Adsorbed amount of surfactant was obtained by measuring surface tension of the solution using the stalagmometer (TRAUB'S Model: 4855) before and after exposing with the adsorbent [26,27]. The average size of the adsorbent varied from $300\text{--}1450\ \mu\text{m}$, examined by compound microscope (Cole Parmer Model: 48923-44). The effect of pH on the adsorption phenomenon was studied by adding either 0.1N HCl or NaOH.

The surfactant concentration in the sorbent phase $q\ (\text{mg g}^{-1})$ was calculated from the following expression,

$$q = (C_0 - C_f) \frac{V}{m} \quad (1)$$

where C_0 and C_f are the initial and final concentration (mg l^{-1}) of the adsorbate in solution, V is the volume (ml) of SDS solution and m is the mass (g) of the granite sand.

2.5. Column experiment

Recovery of the adsorbed material as well as the regeneration of column has also an important aspect for the practical application of any adsorption process. For this purpose, dry granite sand was packed in a 20 cm long column having 0.9 cm internal diameter, yielding the packing density $2.36\ \text{g cm}^{-3}$. The column was slowly flooded with water then 25 ml SDS solution of $4 \times 10^{-3}\ \text{M}$ was fed into the column from the top. The flow rate was 0.5 ml/min. The regeneration of the column was carried out by two ways, washing with the distilled water and with H_2O_2 solution.

Similar procedure was followed for the decisive effect of interference ions on the adsorption of SDS solution. The concentration of each trace metal ions (i.e. Cd^{2+} , Fe^{2+} , Pb^{2+} and Ni^{2+}) and the minor metal ions (i.e. Na^+ , Li^+ , K^+ and Ca^{2+}) were 10 and 100 mg/l, respectively.

3. Results and discussion

Particle size distributions of the samples are illustrated in Table 1. Adsorption studies were carried out to observe the effect of these particles size on the attainment of equilibrium time. Results showed that S1 (size range $300\text{--}450\ \mu\text{m}$) adsorbed greater amount of surfactant molecules due to the large surface area.

The dependence of the adsorption of SDS with mass of sand is presented in Fig. 2. 10 ml of $3 \times 10^{-3}\ \text{M}$ SDS solution at the temperature of $34 \pm 01\ ^\circ\text{C}$ and pH 6.25, shaking for 15 min at 125 rpm. The amount of sand varied from 1.0 to 9.0 g. The data

Table 1
Effect of adsorbent size on the adsorption of SDS

| No. of samples | Adsorbent size (μm) | $q\ (\text{mg g}^{-1})$ |
|----------------|----------------------------------|-------------------------|
| S1 | 300–450 | 1.205 |
| S2 | 475–700 | 0.980 |
| S3 | 700–1000 | 0.764 |
| S4 | 1000–1450 | 0.365 |

Conditions: initial concentration of SDS, $3.28 \times 10^{-3}\ \text{M}$, pH 6.25, temperature, $35 \pm 0.5\ ^\circ\text{C}$, orbital shaking 15 min at 125 rpm.

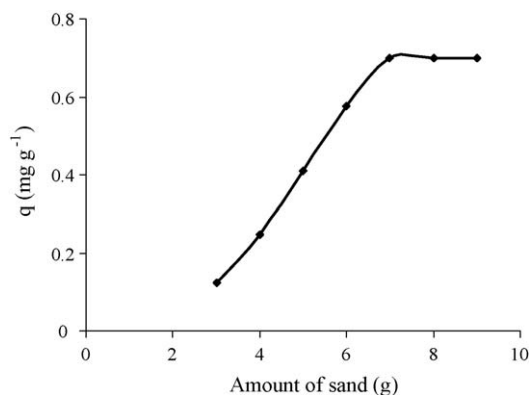


Fig. 2. Adsorption of SDS from solution of different pH by 7.0 g of granite sand mixed in $8 \times 10^{-3}\ \text{M}$ SDS solution during orbital shaking of 15 min at 125 rpm incubated at $34 \pm 01\ ^\circ\text{C}$.

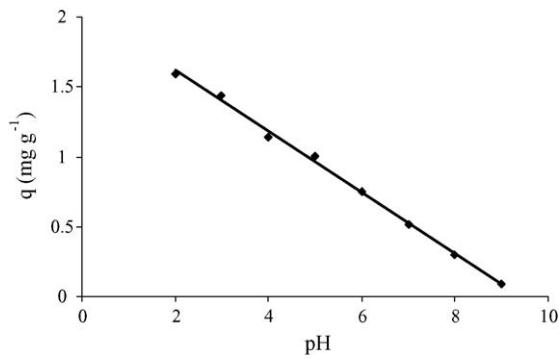


Fig. 3. The effect of the amount of sand on the adsorption process of SDS. The initial concentration of SDS was 3×10^{-3} M at the temperature of 34 ± 01 °C and pH 6.25 for 15 min shaking at 125 rpm.

indicates the dependency of amount adsorbed on the dose of the sand. The maximum uptake was exhibited at 7.0 g of adsorbent. Thus, this amount of sand was taken for further adsorption kinetic study to achieved pseudo-order condition with respect to adsorbent.

The pH of the aqueous solution is an important controlling parameter in the adsorption process. A number of works has been reported a non-specific ion adsorption, which measure changes in the electrostatics adsorption of the cations and anion with changes in the activities of H^+ and OH^- , to find the point of zero net charge (PZNC) [28,29]. A granite sand was comprised 70–77% SiO_2 whose reported PZNC is 2.0 [30]. In the present study, adsorption of SDS on granite sand at different pH was observed and the results are plotted in Fig. 3. The maximum adsorption took place at pH 2.0 and gradually decreased to minimum at pH 9.0, confirmed that PZNC of the granite sand is 2.0.

The observed effects of Ca^{2+} and Na^+ ions in the range of (0.10–0.60) M on the adsorption of SDS were remarkable. It was noticed that the presence of Na^+ ions in the range of (0–0.30) M slightly decreased the adsorption of SDS because of the common ion effect, which reduced the availability of negatively charged dodecyl molecule (Fig. 4). While on the other hand, the adsorbed amount sharply increased with increasing Ca^{2+} ion concentra-

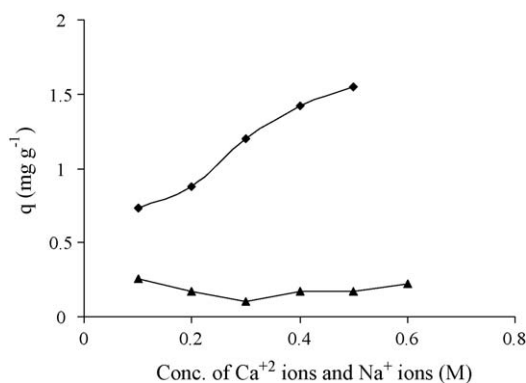


Fig. 4. Effect of Na^+ (\blacktriangle) and Ca^{2+} (\blacklozenge) ions on the adsorption process. The initial concentration of SDS was 4.23×10^{-3} M at 32 ± 0.5 °C and amount of sand was 7.0 g.

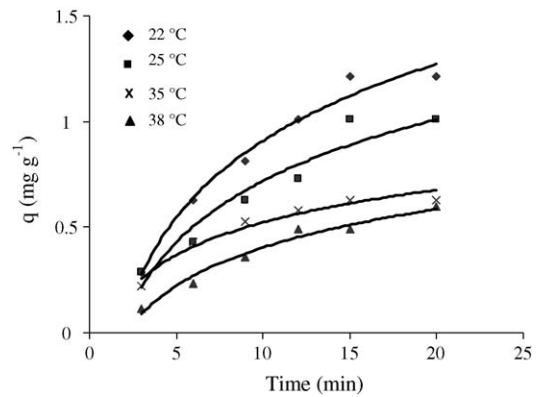


Fig. 5. Adsorption isotherms for SDS onto the granite sand. Initial concentration of SDS was 4.23×10^{-3} M at the natural pH 6.25 during 15 min orbital shaking at 125 rpm and the amount of sand was 7.0 g.

tion. It is because of the formation of scum, which is sufficiently adsorbed onto the sand surface.

A series of experiments were undertaken to study the temperature effect by performing experiments at 22, 25, 35 and 38 °C. In all the experiments, the particle size range of the sand was 475–700 μm . The initial concentration of SDS was 3×10^{-3} M, agitation speed 125 rpm and natural pH 6.25 was used. The influenced of temperature on the sorption of SDS is illustrated in Fig. 5. The increasing trend of adsorption at low temperature attributed to the fact that the adsorption interactions are exothermic in nature.

3.1. Adsorption kinetics

The adsorption kinetics described a pseudo-first order trend (Lagergren 1898) [31]:

$$\ln(q_e - q_t) = \ln q_e - k_{ads}t \quad (2)$$

where q_t and q_e ($mg g^{-1}$) are the amounts adsorbed at time t (min) and at equilibrium, respectively. Eq. (2) clearly justified that the sand sorption process followed the pseudo-first order kinetics (Fig. 6). The values of adsorption rate constant k_{ads} , at different temperature were calculated and used further in the evo-

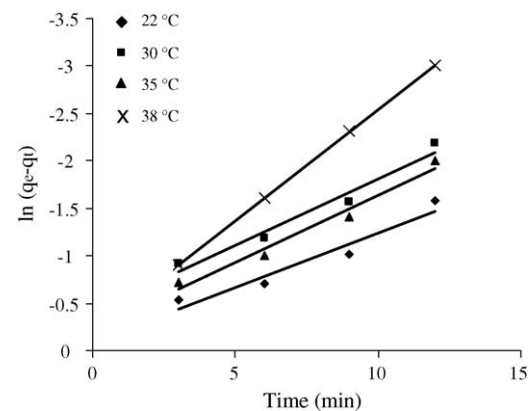


Fig. 6. Kinetics study of the adsorption process at different temperature. Conditions: concentration of SDS = 3×10^{-3} M, pH 6.25, amount of sand = 7.0 g, orbital shaking at 125 rpm.

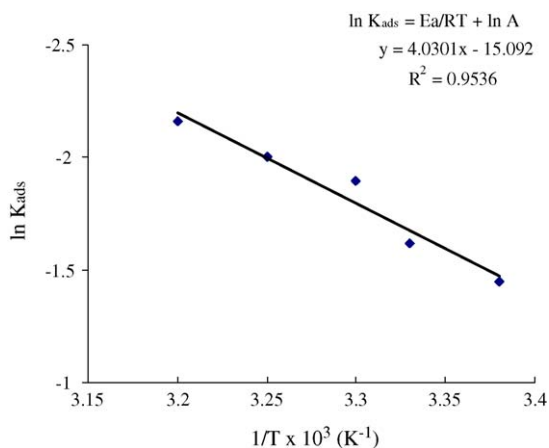


Fig. 7. Plot of $\ln K_{\text{ads}}$ against reciprocal temperature for SDS onto granite sand. Conditions: concentration of SDS = 3×10^{-3} M, pH 6.25, amount of sand = 7.0, shaking at 125 rpm.

lution of activation energy, which was obtained $33.65 \text{ kJ mol}^{-1}$ using Arrhenius plot (Fig. 7).

3.2. Adsorption isotherms

To quantify the adsorption capacity of granite sand for the removal of SDS from water, the Langmuir and Freundlich models were used. The linear form of the Freundlich model:

$$\log q = \log K + \frac{1}{n} \log C \quad (3)$$

where K and n are empirical constants. q is the amount adsorbed in mg g^{-1} . C is the concentration of solute in the solution. A plot of $\log q$ versus $\log C$ yielded a straight line (Fig. 8) with the slope $1/n$ and intercept $\log K$ confirmed the validity of Freundlich isotherm.

According to Langmuir model:

$$\frac{1}{q} = \frac{1}{q_m} KC + \frac{1}{q_m} \quad (4)$$

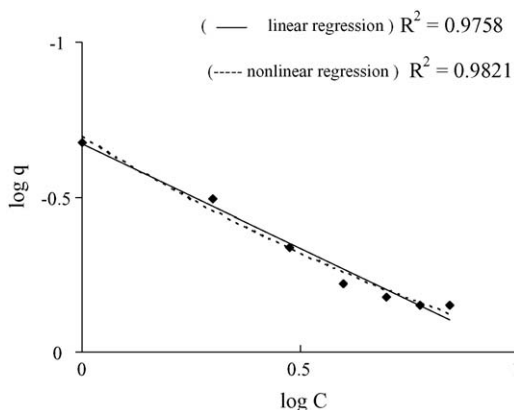


Fig. 8. Freundlich adsorption isotherm of the adsorption of SDS for initial concentration 3×10^{-3} M by granite sand. Where $\log C$ is the log equilibrium concentration of SDS and $\log q$ the log of quantity of SDS adsorbed at equilibrium. The temperature was $35 \pm 0.5^\circ\text{C}$ and pH 6.25.

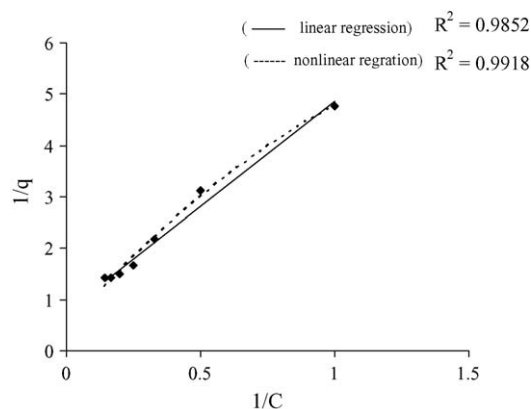


Fig. 9. The Langmuir adsorption isotherm of adsorption of SDS onto granite sand at the temperature $35 \pm 0.5^\circ\text{C}$ and pH 6.25. Where $1/C$ is the reciprocal of equilibrium concentration of SDS and $1/q$ is the reciprocal of the quantity of SDS adsorbed.

Table 2

The Langmuir and Freundlich parameters for the adsorption of SDS onto the granite sand

| Experimental Langmuir isotherm constant | | Experimental Freundlich isotherm constant | |
|---|--------|---|--------|
| q_{max} (mg g^{-1}) | 1.3070 | n | 1.4770 |
| K | 0.0192 | K | 0.1730 |
| r^a | 0.9852 | r^a | 0.9758 |
| r^b | 0.9917 | r^b | 0.9821 |

The pH of the solution of was 6.25 at $35 \pm 0.5^\circ\text{C}$ and the shaking time was 15 min at the rate 125 rpm.

^a Correlation coefficient (linear regression).

^b Correlation coefficient (non-linear regression).

where K is the Langmuir adsorption, q_m is the limiting amount of adsorbate that can be taken up per mass of adsorbent. C and q have the same meanings as in the Freundlich isotherm. The linear plot of $1/q$ versus $1/C$ shows the applicability of Langmuir isotherm (Fig. 9). The correlation coefficients obtained with both kind of regression (linear and non-linear) are given in Table 2. The values obtained from the Langmuir equation shows better correlation to the experimental results than the Freundlich equation. The validity on the Langmuir isotherm depicted the monolayer adsorption onto surface containing finite number of identical sorption. Similar behavior has been observed in a number of studies [25,26].

The free energy change ΔG , enthalpy change ΔH and entropy ΔS were calculated and listed in Table 3.

Table 3

Thermodynamic parameters for the adsorption of SDS onto granite sand at different temperatures

| Temperature (K) | $1/T \times 10^3$ | $\ln k$ | ΔG (kJ mol^{-1}) | ΔS (kJ $\text{mol}^{-1} \text{K}^{-1}$) | ΔH (kJ mol^{-1}) |
|-----------------|-------------------|---------|------------------------------------|--|------------------------------------|
| 300 | 3.33 | 5.164 | -12.88 | 0.1123 | |
| 303 | 3.30 | 5.22 | -13.149 | 0.1120 | 20.81 |
| 311 | 3.21 | 5.459 | -14.115 | 0.1122 | |

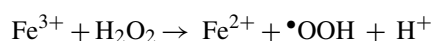
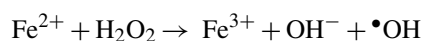
Table 4
Column experiments for regeneration of column

| No. of cycles | Washing by H ₂ O | | Washing by H ₂ O ₂ | |
|-----------------|---------------------------------------|------------------|--|------------------|
| | Amount adsorbed (mg g ⁻¹) | Percent adsorbed | Amount adsorbed (mg g ⁻¹) | Percent adsorbed |
| Fresh adsorbent | 0.461 | 48.0 | 0.461 | 48.0 |
| 1 | 0.216 | 22.5 | 0.370 | 39.0 |
| 2 | 0.144 | 15.0 | 0.292 | 30.5 |
| 3 | 0.074 | 7.75 | 0.292 | 30.5 |
| 4 | 0.007 | 0.75 | 0.215 | 22.5 |

Conditions: initial concentration of SDS, 4×10^{-3} M; pH 6.25; temperature, 35 ± 0.5 °C; volume of SDS solution, 25 ml; mass of sand to packed the column, 30 g; density of column, 2.36 g/cm³; flow rate, 0.5 ml/min.

3.3. Regeneration of column

Fenton reagent (FR) is one of the most popular advanced oxidation process (AOPs). In spite of this, environmental applications of FR are relatively recent [32]. It was first reported by H.G.H. Fenton in 1894 [33]. H₂O₂ is environmental friendly, since its decomposed into oxygen and water. Numerous research have been devoted to take advantages of the potential benefits of the use of FR as a remediation process for the treatment of wide variety of water pollutant such as surfactant [34] and commercial dyes [35]. The use of hydrogen peroxide as a washing agent converted it into FR owing to the natural occurring iron (4–6%) in the granite sand. It can be described as the generation of ·OH through catalytic decomposition of hydrogen per oxide (H₂O₂) carried out by Fe²⁺ or Fe³⁺ [32,36].



Observation shows that after four cycles, percent adsorption decreased from 48.0 to 0.76% and 48.0 to 22.5% by using H₂O and H₂O₂ solution, respectively (Table 4). This implies that the percentage recovery from the H₂O₂ solution was found to be higher than distilled water because, the production of hydroxyl radical improved the degradability of surfactant molecules and finally they released as decomposed products

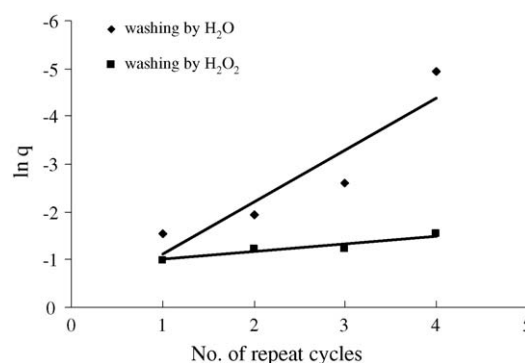
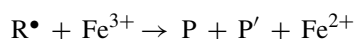
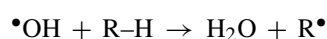


Fig. 10. First order desorption process.

from the active centers and can be perceived by the following reaction mechanism.



where R–H is the surfactant, R· is the free radical from surfactant molecule, P and P' are the decomposed fragment of surfactant.

Thus, the hydrogen peroxide solution turned out to be a better washing agent. Fig. 10 represents that desorption process by H₂O₂ solution and by H₂O followed first order kinetics.

Table 5
The effect of the interference ions (minor and trace metals) on the sand sorption of SDS

| Condition | Before adsorption, concentration of SDS ($\text{M} \times 10^{-3}$) | After adsorption, concentration of SDS ($\text{M} \times 10^{-3}$) | Percent adsorption | q (mg g ⁻¹) |
|--|---|--|--------------------|---------------------------|
| In the absence of interference ions | 4.00 | 2.08 | 48.00 | 0.461 |
| In the presence of minor metal ions ^a | 4.23 | 1.71 | 59.00 | 0.605 |
| In the presence of trace metal ions ^b | 3.97 | 3.40 | 14.14 | 0.134 |

Conditions: initial concentration of SDS, 4×10^{-3} M; pH 6.25; temperature, 35 ± 0.5 °C; volume of SDS solution, 25 ml; mass of sand to packed the column, 30 g; density of column, 2.36 g/cm³; flow rate, 0.5 ml/min.

^a Na⁺, Li⁺, K⁺, Ca⁺⁺ = 100 mg/l.

^b Cd⁺⁺, Fe⁺⁺, Pb⁺⁺, Ni⁺⁺ = 10 mg/l.

Table 6
Percent adsorption data for tested surfactant

| Surfactant | Molecular mass | Ionic type | Percent adsorption |
|-----------------------|----------------|------------|--------------------|
| SDS ^a | 288.38 | Anionic | 56.66 |
| Tween 80 ^b | 1310 | Non-ionic | 42.00 |

Conditions: amount of adsorbent = 7.0 g; Shaking time = 15 min (at 125 rpm); temperature = 26 °C.

^a pH (natural) 5.95

^b pH (natural) 6.10

3.4. Test with interference ions

The utility of the adsorbent in column chromatography has been tested by minor and trace metals. Composition and concentrations are given in Table 5.

In the presence of trace metal ions (i.e. Cd²⁺, Fe²⁺, Pb²⁺ and Ni²⁺) the adsorption capacity for SDS decreased because of the preferential adsorption of these metal ions onto the active center of adsorbent. However, on the other hand minor metals (i.e. Na⁺, Li⁺, K⁺ and Ca²⁺) enhanced the adsorption of surfactant. Because of the collective effect of these ions the formation of scum occurred, which provided a suitable adsorption conditions for SDS. These results inferred that the granite sand can also be used as an effective adsorbent for the removal of transition metals from the industrial effluents.

Adsorption studies were also carried out for a non-ionic surfactant (i.e. Tween 80) summarized in Table 6. The results revealed that the granite sand was also used for the removal of non-ionic surfactant. However, the percent adsorption was comparatively low in that case.

3.5. Removal of SDS (anionic surfactant) from ground water

In order to check the applicability of the removal technique, the optimized method was applied for the removal of SDS (anionic surfactant) from ground water. The removal efficiency was 70.11%. In the ground water sample, the capacity was found high. It was due to the presence of other major cations like calcium and magnesium, which formed scum with anionic surfactant.

4. Conclusion

Many developing countries suffer from chemical contamination of water supplies largely due to uncontrolled industrial activity and so; a water treatment technique, which has ability to deal with such pollutants in an effective way, proved valuable. Our work concerned the removal of SDS (anionic surfactant) by granite sand. The results obtained and their interpretation has been concluded:

- The adsorption capacity was decreased by raising temperature, indicating that the adsorption interactions were exothermic in nature.
- Granite sand is an appropriate adsorbent for the removal of SDS from aqueous solution. The said process was dependent

on PZNC, time of agitation, mass of sand and temperature of SDS solution. The maximum uptake has occurred at pH 2.0.

- Thermodynamic parameters have been calculated and the adsorption of SDS onto granite sand satisfied the Langmuir and Freundlich isotherms.
- Spent adsorbent was successfully regenerated using H₂O₂ solution, act as Fenton or Fenton like reagent.
- Granite sand would be useful for the treatment of water containing surfactants as well as for trace metal ions.

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