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Removal of linear alkyl benzene sulfonate from aqueous solutions by functionalized multi-walled carbon nanotubes

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ABSTRACT

This paper explores the possibility of employing Oxidized Multiwalled Carbon Nanotubes (MWCNT-COOH) for the removal of toxic Linear Alkyl benzene Sulfonate (LAS). LAS is among the most toxic industrial and house hold waste surfactants. This study discusses the feasibility of removing LAS from aqueous solutions using MWCNT-COOH. The effects of operational parameters such as solution pH, LAS concentration and contact time on the removal of LAS were studied. The four linear forms of Langmuir, Freundlich, Dubinin Radushkevich (D-R) and Temkin models were applied to determine the best fit of equilibrium expressions. Our results showed that the experimental adsorption isotherm complies with Freundlich model. The maximum adsorption capacity was determined to be 62.5 mg/g with an initial LAS concentration of 4 mg/L at pH 3 in 45 min. Fitting of the experimental results to kinetic models showed the relevance of the pseudo second-order ($R^2 > 0.99$) model for LAS. Our results confirmed that MWCNT-COOH would be promising adsorbents for LAS removal in aqueous solution.
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1. Introduction

Surfactants are used widely in household, personal care products, agriculture and industry [1,2]. Surfactants are a diverse group of chemicals with cleaning properties and consist of two heads with a different polarity or solubility in water: a polar head group, which is well solvated in water, and a non-polar hydrocarbon tail, which is not easy to dissolve in water [3,4]. Surfactants are classified by their ionic activity in water into four types: anionic, cationic, non-anionic and amphoteric [5]. LAS is the largest group of anionic surfactants (Fig. 1). Synthetic surfactants are formed of LAS and its isomers with other additives [6,7].

Anionic surfactants (AS), especially LAS, are used extensively due to their impacts on ecosystems and are usually disposed after their use into the environment [8,9]. Therefore, they represent one of the main causes of water pollution. A recent study reported the presence of LAS in different water channels in Iran [5]. The concentrations of surfactants in natural water vary from area to area and ranged from 3.780 to 1456 mg/L. This high percentage of waste chemical components should be removed from water. The conventional treatment methods such as combination of biodegradation and sorption/settling processes and Oxidative treatments and cannot remove them [10–12]. Other methods have been proposed to remove surfactants such as: (i) adsorption [13]and (ii) coagulation using polyelectrolytes with powdered activated carbon (PAC) and powdered clinoptilolite simultaneously [14].Several researchers have shown that the adsorption is an efficient process for the removal of LAS from aqueous solutions [13,16]. For this purpose, a

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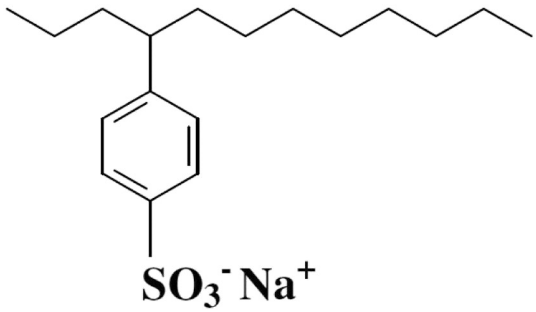

Fig. 1. Formulation of Linear Alkyl benzene Sulfonate.

variety of natural and synthetic materials have been tested as LAS, including activated carbon [17], mineral clay [17], soils [13] and others. Advanced membrane filtration techniques (*e.g.* microfiltration, ultrafiltration, nanofiltration and reverse osmosis) exhibited superior performance in treatment and removal of chemical and biological contaminants over conventional systems [18]. Hybrid methods can also be more effective than any of the previous process alone. The combination of ion exchange and ultrafiltration was more effective in surfactant removal than ultrafiltration process alone [19].

In recent years, nanotechnology has introduced different types of nanomaterials for water treatment. Nanosorbents such as carbon nanotube (CNTs) [15] and zeolites [20] proved to exhibit special adsorption properties. CNTs, in particular, received special attention for potential application in environmental protection and to work effectively against chemical and biological contaminants [15,18,21]. Single and multi-walled carbon nanotubes (MWCNTs) have been used in water and wastewater treatment [15].

The objective of this study is to investigate the adsorption mechanism of LAS using MWCNTs and to determine the optimum parameters for the maximum/efficient adsorption capacity.

2. Experimental

2.1. Materials

Multi-walled CNTs (MWCNTs) were synthesized by catalytic chemical vapor deposition (CVD) method at the Research Institute of Petroleum Industry (RIPI), Tehran, Iran. MWCNTs were used as adsorbents to study the adsorption characteristic of LAS detergent from water. The size of the outer diameter of MWCNTs was more than 10 nm while the length ranged from 5 to 15 μm. In addition, the mass ratio of the amorphous carbon of MWCNTs was less than 5%, and specific surface area was 280 m^2/g. Because of the presence of amorphous carbon in carbon nanotubes, the adsorption rate is very low. Therefore, carbon nanotubes must be purified before the adsorption process. The stock solution was stored in the refrigerator to minimize biodegradation. 10 mL of stock LAS solution was diluted to 1000 mL with distilled water; 1 mL = 10 μg LAS. Dilute solutions of concentrations 1.5, 2 and 4 mg/L LAS were prepared. The stock solution of LAS was prepared using standard methods for the examination of water & wastewater book No. 5540C [22].

2.2. Functionalization of MWCNTs

Schematic diagram of adsorbents preparation is shown in Fig. 2. In order to functionalize MWCNTs, 0.3 g of MWCNTs were scattered in 25 mL of nitric acid (65 wt.%) in a 100 mL flask equipped with a condenser and scattering was refluxed under magnetic stirring for 48 h [23]. Then, the resulting scattering was diluted in water and filtered. The filtrate was rinsed to neutral pH and the sample was dried in vacuum at 40 °C overnight to obtain MWCNT-COOH.

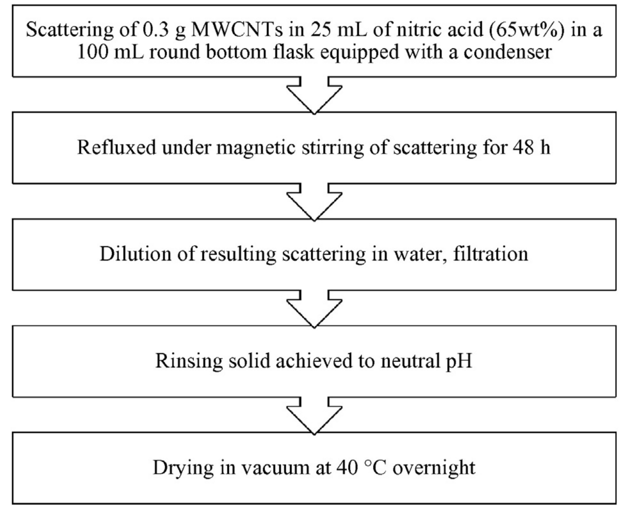

Fig. 2. Schematic diagram of adsorbent preparation.

Fig. 3 shows the probable synthesis reaction of carboxyl and amino functionalized CNTs. This figure illustrates that carboxyl group could be formed first and may have higher number of reactive sites that could lead to better interaction with polymers.

2.3. Adsorption experiments

Batch adsorption experiments were performed in glass bottles with LAS solution (250 mL) of the mentioned concentration (i.e. 4 mg/L) and 25 mg of MWCNT-COOH was added to each bottle. The amount of MWCNT-COOH was kept constant in all experimental steps. According to a previous study [5], the concentration of detergent in Tehran surface water was 0.14–3.78 mg/L. Based on LAS concentration ranging from 1.5 to 4 mg/L was selected for this study [5]. The bottles were shaken using a magnetic shaker (IKA® RCT basic) at 25 °C until equilibrium. The pH of the solutions was adjusted by adding HCl or NaOH solutions. The solution pH ranges from 3 to 9. After achieving equilibrium (corresponding to the saturation of the adsorbent), the suspension was filtered through a 0.2 μm filter and the filtrate was analyzed using NO. 5540C standard method and spectrophotometer (Perkins-Elmer Lambeda 25-UV/Vis and 652 nm wavelengths with the thickness of cell 1 cm).

2.4. Surface characterization

The morphology of the adsorbents was investigated using scanning electron microscopy (SEM, KYKY-EM3200) and X-ray diffraction (XRD, Quantachrome, NOVA2000).

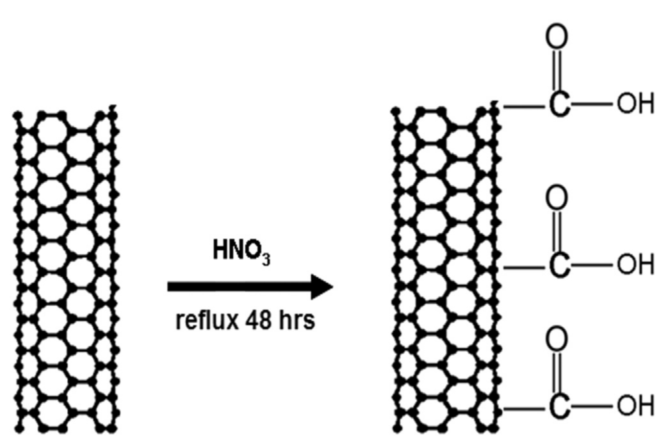

Fig. 3. Schematic diagram for chemical fictionalization of MWCNTs.

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