



# Removal of petroleum sulfonate from aqueous solution by hydroxide precipitates generated from leaching solution of white mud



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## HIGHLIGHTS

- Freshly generated hydroxide precipitates.
- Co-precipitation/adsorption process.
- Electrostatic attraction and hydrogen bonding are the main adsorption mechanisms.
- Adhesion and cohesion affect the adsorption of PS onto FGHP.

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## ABSTRACT

Freshly generated hydroxide precipitates (FGHPs), which was prepared by adding the leaching solution of white mud (LSWM) to highly alkaline solution, was used to remove petroleum sulfonate (PS) from aqueous solution. The chemical composition of the white mud and petroleum sulfonate were determined by X-ray fluorescence spectrometry and gas chromatography–mass spectrometry. The surface properties of the FGHPs and PS-FGHPs were characterized by X-ray diffraction, transmission electron microscopy, Fourier transform infrared spectroscopy and zeta potential analyzer. The FGHPs displayed excellent treatment efficiency for PS at pH 12.0. The maximum equilibrium removal efficiency of PS was reached within 60 s. The maximum adsorption capacity of FGHPs for PS was 3798.06 mg/g at 303 K and pH 12.0. The Langmuir isotherm was the best choice to describe the adsorption behavior. The kinetic data fitted the pseudo-second-order kinetic model. Thermodynamic parameters suggested that the adsorption of PS onto FGHPs occurred via physisorption and was exothermic. Electrostatic attraction and hydrogen bonding were the main adsorption mechanisms. Moreover, adhesion and cohesion also strongly affected the co-precipitation/adsorption process. Liquid bridges via hydrogen bonding, adhesive and cohesive forces linked up with  $\text{MOH}^+/\text{M}(\text{OH})_2$  particles and the surfactant molecules to form a three-dimensional network structure and lead to deposition.

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## 1. Introduction

Surfactant wastewater is one of the major pollution sources of a receiving water body around the world [1]. In the surfactant wastewater produced by the households, surface active agents or surfactants invariably exist in significant amounts due to detergents used for all kinds of washings [1]. Surfactants have also been widely used in textiles, fibers, food, paints, cosmetics, pharmaceuticals, mining, oil recovery and pulp and paper industries [1,2]. The

synthetic surfactants are of three major types: anionic, nonionic, and cationic. Anionic surfactants are the major class of surfactants and have been used widely in detergent formulations [3,4]. Petroleum sulfonate (PS) surfactants differ from relatively homogeneous conventional surfactants in that they are mixtures of sulfonated alkyl–aryl petroleum products and free mineral oils [5]. Polymers and PS surfactants are used as effective “pusher fluids” to enhance oil recovery and the PS surfactants are therefore used to improve the flooding efficiency of crude oil and widely used in exploitation [6]. Some surfactants are toxic; others are not, depending on dose, chemistry, receptors, etc. They may cause foaming in rivers and reduce the quality of water [3]. Surfactants cause short and long-term changes in ecosystems [3]. PS surfactants removal from water

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environment before they are contacted with natural water bodies is very important.

Many conventional methods exist for the removal of anionic surfactants from water environment. These involve chemical and electrochemical [7,8], membrane separation [9], photocatalytic degradation [10], adsorption [11] and biological treatment processes [12]. However, anionic surfactants having high degree of hydrophilic nature and poor biological activity, biological degradation of anionic surfactants occurs too slowly. These methods have been found to be limited, and none of them were successful in completely removing anionic surfactants from aqueous solution [5,7–13]. Hence it would be of much practical and academic interest to investigate surfactant wastewater treatment using new methods with low cost and high efficiency.

Co-precipitation/adsorption processes have high adsorption abilities and short adsorption equilibrium times and may be suitable for surfactant wastewater treatment [14–17]. For example, when metallic ions precipitate in alkaline conditions, the freshly formed hydroxides are fine particles with a larger specific surface area and higher surface free energy. As the hydroxide particles are formed, they incorporate and adsorb pollutants, so the adsorption ability can be enhanced significantly by co-precipitation/adsorption processes [14–18].

Industrial wastes are remarkable adsorbents in wastewater treatment. They provide several advantages such as their low-cost, abundant availability and possibility for sludge disposal [19–21]. The major industrial process for the production of soda ash is the ammonia–soda process. In this process, a large amount of fine-sized solid wastes known as white mud is produced. Since white mud is characterized by high water content, high alkalinity and high concentrations of soluble  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$  and  $\text{Cl}^-$  ions [5,22], it is difficult to deal with or reuse. Common treatment methods for white mud include landfill disposal or discharge to oceans [23], which causes a potential threat to groundwater, soil and the marine environment. Therefore, a new method is needed to reuse these problematic materials [22–24].

In this work, we attempt to reduce raw materials and operating costs for PS removal by industrial wastes treating PS-containing wastewater. Co-precipitation/adsorption process has been used to remove PS from aqueous solution. When the leaching solution of white mud (LSWM) is added to alkaline solution, metal hydroxides are precipitated. This freshly generated hydroxide precipitates (FGHPs) has positive surface charge, which can be used as adsorbent to attract negatively charged PS surfactant. The effect of pH, initial PS concentration, adsorbent dosage, contact time and temperature were investigated. Adsorption isotherms and kinetic data were obtained and thermodynamic parameters were determined. The adsorption mechanism between FGHPs and PS was also discussed.

## 2. Materials and methods

### 2.1. Materials

Alkaline white mud was obtained from the Shandong Aluminum Plant, dried at 103–105 °C overnight, disaggregated and sieved to 200 mesh prior to use. Its chemical composition as determined by X-ray fluorescence spectrometry (XRF, ZSX Primus II,

Rigaku Corporation, Tokyo, Japan) is shown in Table 1. The white mud was dispersed in 1.2 mol/L hydrochloric acid with a solid content of 0.1268 g/mL for 12 h and filtered to obtain the leaching solution.

The PS surfactant used in this present work was obtained from the ShengLi oilfield in China. The petroleum sulfonate surfactants are mixtures of petroleum sulfonates and free mineral oils that form stable compound. The active components of PS sample were refined by solvent-extraction method to extract PS surfactant. Pentane, absolute alcohol, isopropyl alcohol and petroleum ether (boiling range 308–338 K) could be used as extracting solvent. In this work, we used isopropanol–water mixtures (1:1, v/v) as extracting solvent to purify PS sample for further experiments. The active components of PS sample are characterized by gas chromatography–mass spectrometry (GC–MS). GC–MS analysis were performed with a Thermo Finnigan TRACE (Thermo Finnigan, San Jose, CA, USA) coupled with a TRACE MS plus (EI 70 eV) from the same manufacturer. The analyses were carried out using different fused silica capillary columns (30 m × 0.25 mm i.d.; film thickness 0.25 μm) of different polarities (DB-5 and HP-Innowax) from Agilent Company (Palo Alto, CA, USA). The oven temperature was programmed from 50 to 250 °C at 3 °C/min and held isothermal for 10 min. Injector and interface temperatures were 220 and 250 °C, respectively. The carrier gas was helium at a flow rate of 1 mL/min. Diluted samples (1/10 in ether) of 1.0 μL were injected manually and the split ratio was adjusted to 40:1. The components were identified by comparing their mass spectra with those in the NIST98 GC–MS library and these in the literature (Huang et al. [2]), as well as by comparing their retention times. The components were 53.97% PS, 39.69% free mineral oil and 4.13% inorganic salts, with a recovery rate of 96.8% [5]. NaOH/HCl was used to adjust the solution pH.

All other chemical reagents were of analytical grade and were used without further purification.

### 2.2. Adsorption experiments

Adsorption experiments for PS were carried out using a batch equilibrium technique. A 5000 mg/L stock solution was prepared in deionized water, followed by successive dilution to obtain the necessary concentrations. The solution pH was adjusted to the required values using 0.5 mol/L HCl and 2.0 mol/L NaOH solution.

Adsorption of PS was performed by adding a given amount of leaching solution into 250-mL beakers containing 100 mL PS solution at the required concentration and pH. The mixture was stirred continuously at 350 rpm using a Jintan JJ-1 motor stirrer for a given contact time for adsorption. After stirring, the mixture was allowed to settle under static conditions for 10–15 min. The mixture was centrifuged at 8000 rpm (LG10-2.4A, Beijing Medical Centrifuge Factory, China) for 15 min to separate the unsettled small particles from the supernatant after settling. The concentrations of PS in the supernatant were determined using a UV–visible spectrophotometer (UV-1601, Shimadzu, Japan) at wavelength of 652 nm corresponding to the maximum adsorbance. The effect of pH on PS removal was examined at 303 K, an initial PS concentration of 200 mg/L, and pH values were varied; the effect of adsorbent dosage at 303 K and pH 12.0 was studied by varying the adsorbent dosage (0.50–2.50 g/L) at initial PS concentration in the range of

**Table 1**  
Chemical constituents of white mud.

Constituent	$\text{CaCO}_3$	$\text{CaSO}_4$	$\text{Mg(OH)}_2$	$\text{Fe}_2\text{O}_3$	$\text{Al}_2\text{O}_3$	$\text{SiO}_2$	CaO	$\text{CaCl}_2$	NaCl
Percentage by weight (%)	36.47	6.10	24.30	1.84	2.99	9.69	4.17	7.86	2.38

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