



The effect of environmental factors on the adsorption of lubricating oil onto expanded graphite



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ABSTRACT

The development of high effective adsorbent materials is of great importance for the removal of oil spillage and chemical leakage in environmental cleanup. Herein, the uptake of lubricating oil on a new type of expanded graphite was investigated by batch techniques. The results indicated that the maximum adsorption capacity of expanded graphite can be maintained over a wide ionic strength range, from 0.001 to 1.0 mol/L of NaCl, making them suitable for lubricating oil cleanup under harsh conditions. The outstanding adsorption performance of expanded graphite is attributed to its unique physical properties such as high porosity, high hydrophobicity, low density and excellent mechanism stability. The finding presented herein indicated that the expanded graphite is a promising candidate for the uptake of lubricating oil in environmental remediation in oil spillage and chemical leakage accidents.

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1. Introduction

With the increasing utilization and transport of oils and chemical solvents, oil spillage and chemical leakage accidents frequently occur worldwide such as The Gulf of Mexico oil spill accident in 2010. Therefore, it is of great importance for the cleanup of oil spillage and chemical leakage accidents [1–4]. Currently, the removal of oil spillage has been extensively investigated by various methods such as dispersion, *in situ* burning, bioremediation and adsorption [5–10]. Among these methods, adsorption is considered as one of the most promising approaches due to its large adsorption capacity, potential recyclability, high adsorption rate, low-cost, environmentally friendly and ease operation [11–23]. Many researchers investigated the uptake of oils on minerals such as zeolites, expanded perlite and sepiolite [24–28]. However, the limited adsorption capacity and poor selectivity for the adsorption of spilled oils for these natural adsorbents. Hence, it is crucial to fabricate affordable, environmentally benign oil adsorbent with high adsorption performance and obvious selectivity, as well as good recyclability.

Recently, carbon-based materials have attracted interest in oil spillage due to its low density, high porosity and large specific surface area. Expanded graphite is expected to intercalate a variety of organic and inorganic substances, which could make these substances stable in its interlayer [29–32]. However, the removal of oil, special lubricating oil, by expanded graphite is not available in recent years [33,34].

The objectives of this study are (1) to synthesize expanded graphite and characterize it by using XRD, SEM, FTIR and potentiometric titration; (2) to investigate the effect of reaction time, ionic strength, initial lubricating oil concentration and temperature on the adsorption of lubricating oil on expanded graphite by batch techniques.

2. Materials and methods

2.1. Synthesis of expanded graphite

The expanded graphite was synthesized by the exfoliation of graphite [35]. Typically, 5.0 g of flake graphite was slowly put into the mixture solution of 100 mL concentrated H₂SO₄ and 100 mL HNO₃ under ambient conditions for 2 h under vigorous stirring conditions, then 120 mL of H₂O₂ (30 wt.%) was added. The suspension was ultrasonicated and centrifuged repeatedly in order to obtain the expandable graphite. The expanded graphite was obtained by instant heating of expandable graphite at 1100 °C for 60 s.

2.2. Characterization of expanded graphite

The expanded graphite was characterized by SEM, XRD, specific surface area and FTIR spectra. The XRD patterns were mounted from 2θ = 10 to 60° by using a Scintag Pad V X-ray diffractometer equipped with a Peltier thermoelectrically-cooled detector and a copper X-ray tube at a step scan increment of 0.2° and a dwell time of 2 s. A typical SEM micrograph was obtained using a field emission scanning electron microscope (FEI-JSM 6320F, Japan). The potentiometric acid-base titration was performed using a computer-controlled DL50 Automatic Titrator (Mettler

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Toledo, Switzerland) in the different background electrolyte under argon conditions.

2.3. Batch adsorption

The adsorption of lubricating oil on expanded graphite as a function of reaction time, salt content, initial oil concentration and temperature were carried out in polyethylene tubes by using batch technique. The stock suspensions of expanded graphite with lubricating oil were contacted for 48 h to attain the adsorption equilibrium. The cyclic operation of lubricating oil adsorption on expanded graphite was repeated under the same experimental conditions. The amount of adsorbed lubricating oil was calculated from the difference between the initial concentration (C_0) and the equilibrium one (C_e) in supernatant. All experimental data were the average of triplicate determinations and the relative errors were about $\pm 5\%$.

3. Results and discussion

3.1. Characterization

The morphology of expanded graphite was characterized by SEM techniques. SEM image reveals that the expanded graphite displays carbonaceous nanosheets self assembled into a porous and 3D framework (Fig. 1A). As shown in Fig. 1B, it is determined that the distance between graphene layers along the c -axis orientation remains $\sim 3.35 \text{ \AA}$, which is typically related to the corresponding $d(002)$ plane [31]. Hirata et al.

demonstrated that the unit lamination of EG is reduced by the thermal method [36]. Instant heating of the expandable graphite induced the vaporization of graphite intercalation and hence a significant expansion of the material along the crystallographic c -axis occurs. Results of XRD patterns show that the basal spacing of expanded graphite is enlarged by thermal treatment. A variety of oxygenated functional groups of expanded graphite can be demonstrated by FTIR spectra. As shown in Fig. 1C, GO presented the various oxygenated functional groups such as hydroxyl (at $3345, 3208 \text{ cm}^{-1}$), carboxyl (at $\sim 1678 \text{ cm}^{-1}$), $C=C$ (at $\sim 1625 \text{ cm}^{-1}$), ether or epoxy (at 1284 cm^{-1}) [37,38]. The sharp peaks centered at 1433 cm^{-1} was corresponded to the C–O vibration mode [39]. The relative intensities of peaks at ~ 990 and 817 cm^{-1} were attributed to the bending vibration of hydroxyl groups. It was quite evident from FTIR analysis that the amount of hydroxyl, carboxyl and epoxy groups was observed in the surface of GO. The surface wettability of expanded graphite was demonstrated by using a water contact angle measurement. As shown in Fig. 1D, it displays surface hydrophobicity with a water contact angle of 145.6° . Compared to hydrophobic wettability to water, the expanded graphite showed excellent oleophilic property. When a lubricating oil droplet dripped on the surface of the expanded graphite, it was absorbed immediately and a contact angle of oil of $\sim 0^\circ$ was observed. Therefore, the expanded graphite displays high hydrophobicity and super-oleophilicity, which is crucial for selective uptake of spilled oils. The as-prepared expanded graphite has a very low apparent density (*ca.* 12 mg/cm^3) due to evaporation of some volatile species during the pyrolysis process [40]. The high porosity is consistent with SEM observation. Owing to its low-

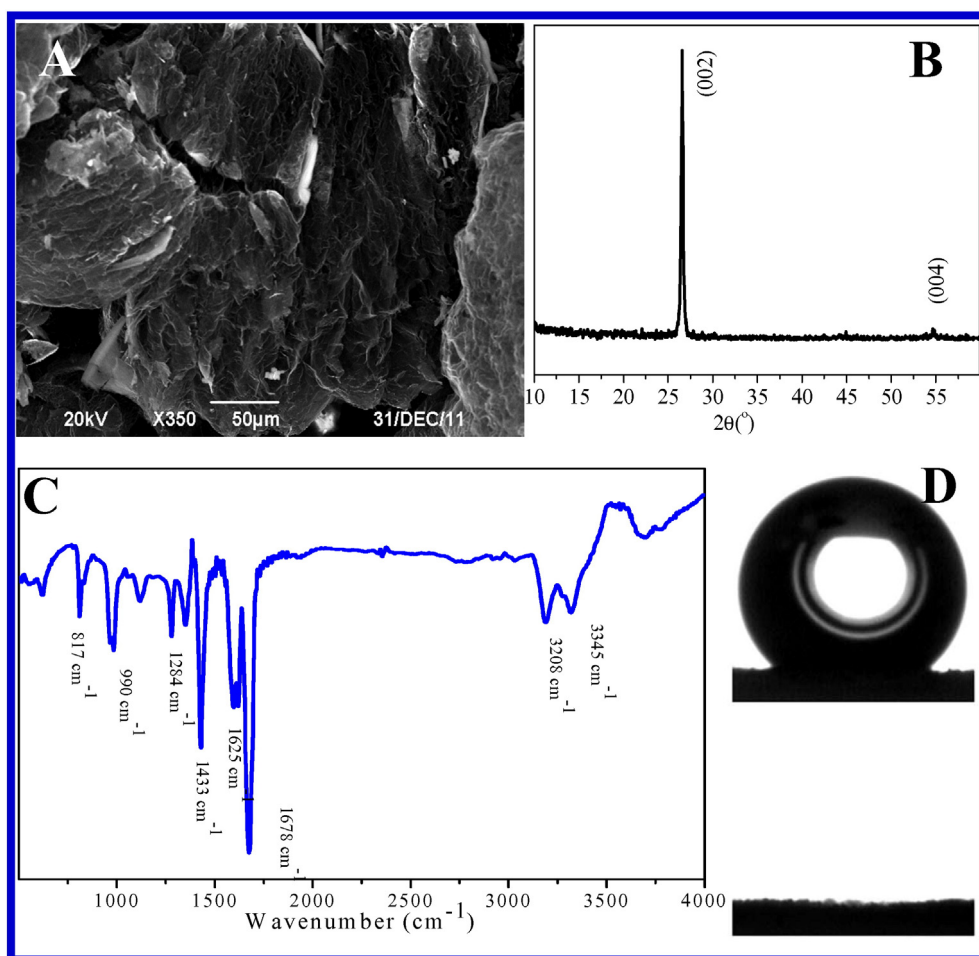


Fig. 1. Characterization of expanded graphite. A: SEM image; B: XRD pattern; C: FTIR spectra; D: wettability measurement.

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