

An Expanded Graphite Used as Oil Adsorbent Prepared with an Alternative Green Route Based on the Classical Hummer Method

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The expanded graphite (EG) has received much attention due to its wide potential applications related to its large sorption capacity; it can be used for oil adsorption in spill accidents. EG is usually produced by some methods, such as liquid-phase exfoliation, chemical vapor deposition, ultrasonic and microwave irradiation [1]. Commonly, EG is prepared by rapid heating of graphite intercalation compound at high temperature in a muffle or with a more advantageous procedure using microwave irradiation [2]. This work describes a method to obtain GE using an alternative route based on the Hummer methods [3]. Expanded graphite was produced from natural graphite (Gr) in flakes (mesh -10, 99.9% purity) and a mixture of concentrated reagent-grade sulfuric acid and hydrogen peroxide as oxidants agents. Our method implies a reduced consumption of chemicals and fewer steps, simplifying the washing procedure and generation of acid washing waters: 2.5 g Gr were mixed with 10 mL H₂SO₄ (98%) in a Spex 8000M device for 5 min, the obtained paste was ice-cooled and 3-4 mL H₂O₂ (30-32%) was added slowly and the mixture was kept for 8-10 h. The slurry was washed with 400-500 mL deionized water, vacuum filtered (Buchner) and the solid was dried at 60°C for 12 h. This EG was compared with a commercial product (Sigma-Aldrich #808121). Both powders were expanded in a commercial microwave oven (Frigidaire, model FMDL17S4GLW,700W) for 30 s (three cycles of 10 s heating / 10 s resting). And their performance was compared in the remediation of water contaminated with light oil (kerosene). Surface morphology and elemental composition were performed using an EDS detector in a Hitachi SU3500 microscope.

Fig. 1 (a-b) shows the morphology and composition of graphites before their expansion, our graphite (EG) has a significant concentration of C, O and S associated with its chemical interaction with H₂SO₄. There are no transition metal remnants derived from the mandatory use of metallic oxidizer agents in the classical Hummers route. The commercial sample (CS), besides the above elements, has traces of Mn (derived from permanganate reaction and their incomplete remotion process, shown with white arrows). During the microwave irradiation processing, both functionalized graphites showed a spontaneous expansion; Fig. 1 (c-d) micrographs exhibit their characteristic "worm-like" morphology and enhanced porosity. Both expanded graphites were tested as adsorption media for oil extraction in oily waters (Fig. 2a). EG demonstrated its efficiency, reaching an adsorption capacity of 100 grams of oil per gram of material compared to CS with a lower value as shown in Fig. 2(b).

References:

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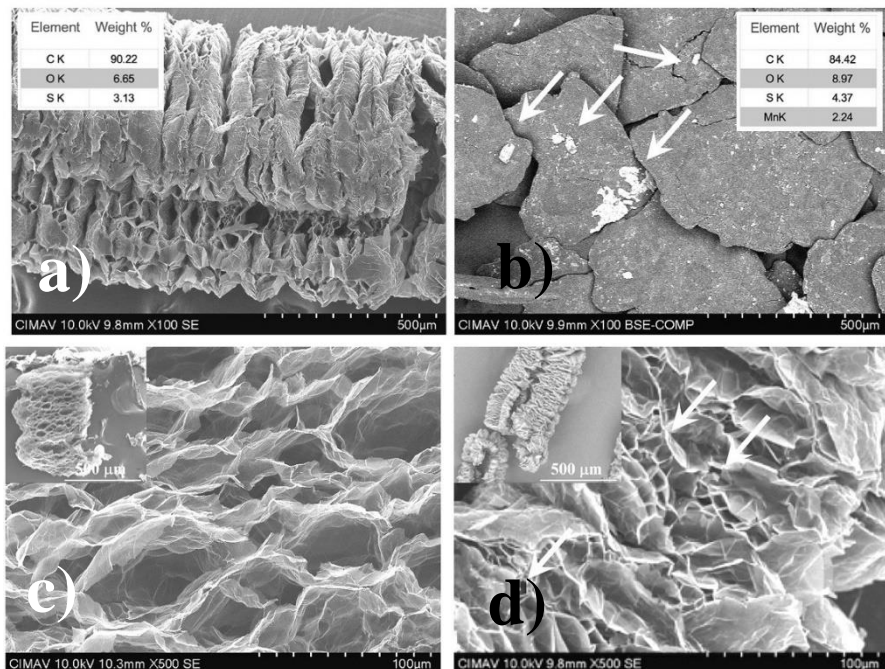


Figure 1. SEM-EDS micrographs and analyses of functionalized graphites a) EG, b) CS and expanded samples c) EG, d) CS.

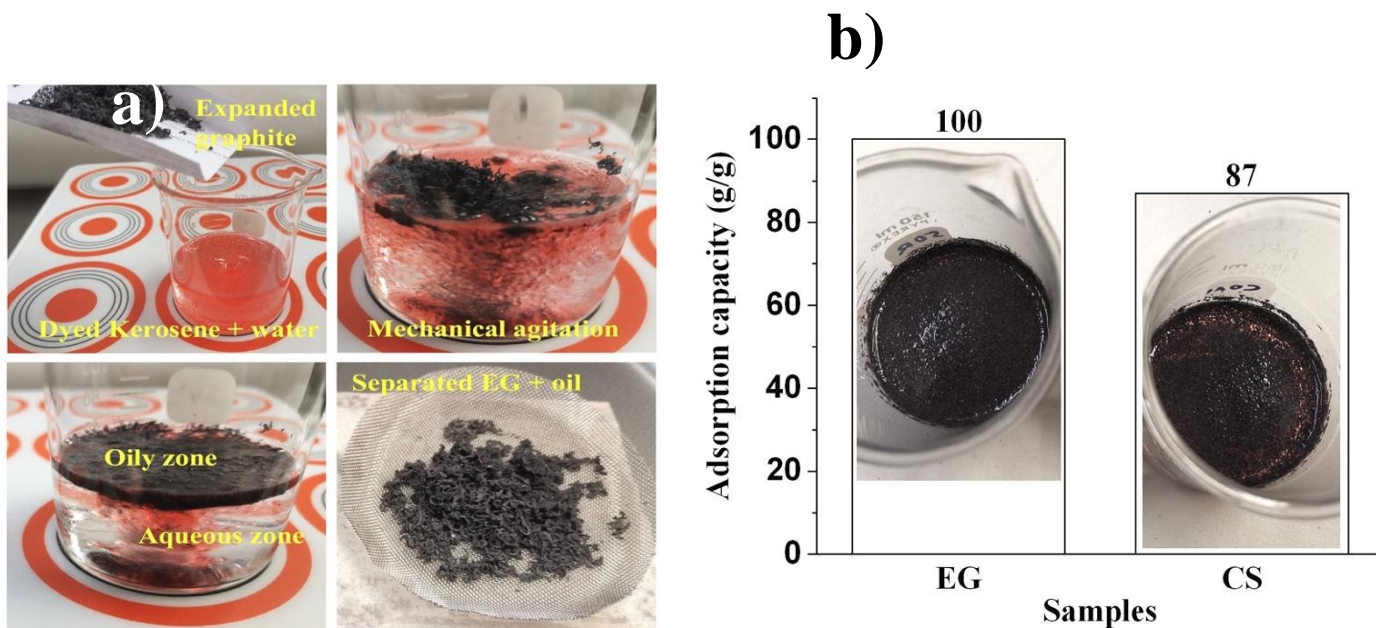


Figure 2. a) Experimental procedure for adsorption testing and b) plot of kerosene adsorption capacity of each expanded graphite.