

Supporting information

Graphene-polymer composite: extraction of polycyclic aromatic hydrocarbons from water samples by stir rod sorptive extraction

Yan-Bo Luo,^a Jin-Sheng Cheng,^b Qiao Ma,^a Yu-Qi Feng,^{*,a} and Jing-Hong Li^{*,b}

^a Key Laboratory of Analytical Chemistry for Biology and Medicine (Ministry of Education), Department of Chemistry, Wuhan University, Wuhan 430072, China

^b Department of Chemistry, Key Laboratory of Bioorganic Phosphorus Chemistry and Chemical Biology, Tsinghua University, Beijing 100084, China

*Corresponding authors: Yu-Qi Feng, E-mail: yqfeng@whu.edu.cn; Fax: +86-27-68755595; Tel: +86-27-68755595

Jing-Hong Li, E-mail: jhli@mail.tsinghua.edu.cn; Fax: +86-10-62771149; Tel: +86-10-62795270

Preparation of graphite oxide and graphene

Preparation of graphite oxide

Graphite oxide was prepared by modified Hummers method: Graphite powder (1.5 g, 325 mesh) was put into a mixture of 12 ml concentrated H₂SO₄, 2.5 g K₂S₂O₈ and 2.5 g P₂O₅. The solution was heated to 80 °C and kept stirring for 5 h by using oil-bath. In a next step, the mixture was cooled to room temperature and diluted with deionized water (500 ml) overnight. Then, the product was obtained by filtering using 0.2 micron nylon film and dried naturally. The pre-oxidized graphite was then re-oxidized by Hummers method. Pretreated graphite powder was put into 0 °C concentrated H₂SO₄ (120 ml), soon after, 15 g KMnO₄ was added gradually under stirring, the temperature of the mixture was kept to be below 20 °C by ice-bath. Successively, the mixture was stirred at 35 °C for 4 h, and then diluted with 250 ml deionized water by keeping the temperature under 50 °C. 700 ml deionized water was then injected into the mixture followed by adding 20 ml 30% H₂O₂ drop by drop. The mixture was filtered and washed with 1 : 10 HCl aqueous solution (1 L) to remove

metal ions followed by 1 L of deionized water to remove the acid. The resulting solid was dried in air and diluted to make graphene oxide dispersion (0.5% w/w). Finally, it was purified by dialysis for one week to remove the remaining metal species, the graphene oxide was obtained by filtration and dried in vacuum for 24 h at 60 °C.

Preparation of Graphene

Preparation of graphene from graphene oxide consists of two steps: in the first step, 100 mg graphite oxide was dispersed in 100 g water. After sonication for 1 hour with, a clear brown dispersion of graphene oxide was formed, 800 mg sodium borohydride in 20 ml water was then added into the dispersion of graphene oxide after its pH was adjusted to 10 with 5% wt sodium carbonate solution. The mixture was then kept at 80 °C for 1 hour under magnetic stirring. During reduction, the dispersion turned from dark brown to black. After centrifuging and extensively rinsing with water several times, the partially reduced graphene oxide can be redispersed in 100 g water via mild sonication.

In the second step, 2 g hydrazine hydrate in 5 ml water is added into the dispersion and the reaction mixture was kept at 100 °C for 24 hours under magnetic stirring. After rinsing with water thoroughly, the graphene thus prepared can be readily dispersed in water via a few minutes sonication.

Fig. 1S Schematic illustration for stir rod sorptive extraction.

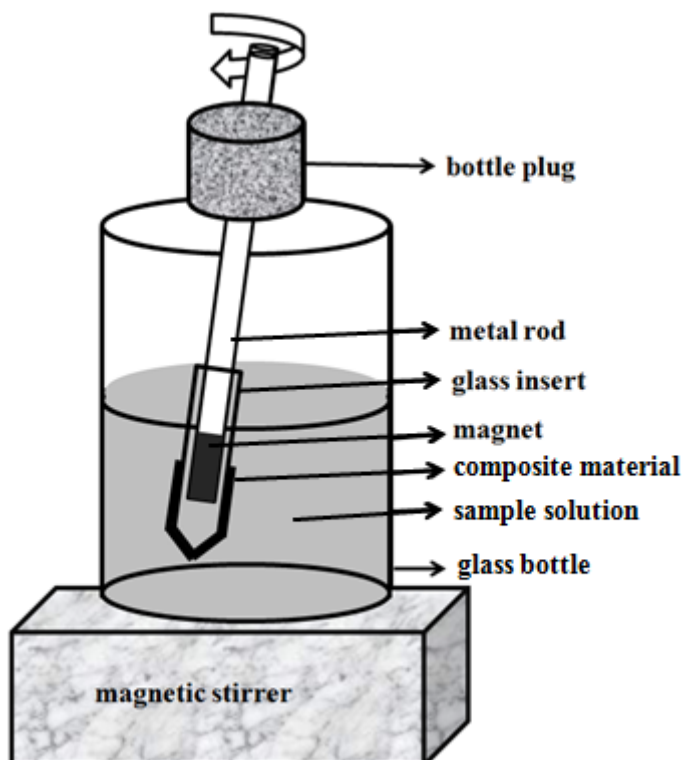


Fig. 2S Effect of different desorption solvents on the desorption of 16 PAHs. Sample solutions with 16 PAHs spiked at 1 ng/mL were prepared with deionized water. The

52 extraction time was 90 min, no additional inorganic salt and organic phase were used.
 53 Recovery = $C_d/C_s \times 100\%$, where C_d and C_s are the peak areas of 16 PAHs of
 54 desorption solution and standard solution (50 ng mL^{-1}) obtained by GC-MS analysis,
 55 respectively.

