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Synthesis and high-performance purification of functionalized graphene for water treatment

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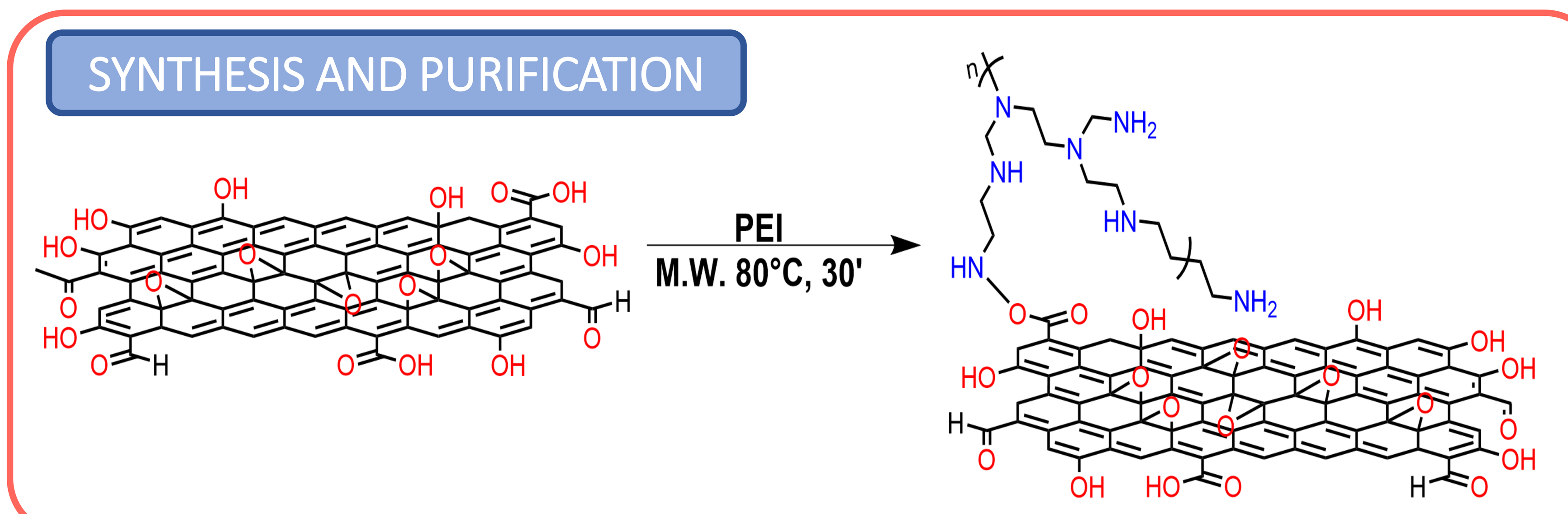
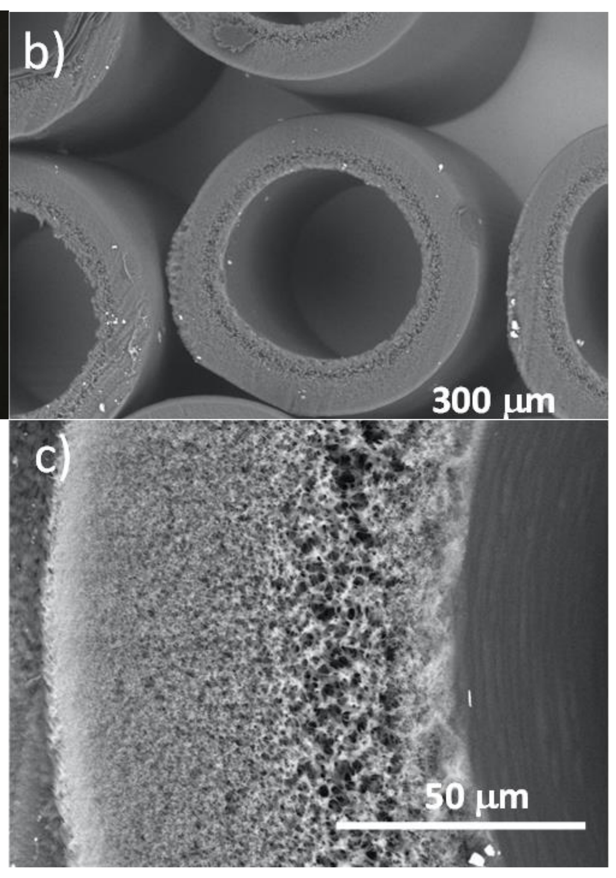
INTRODUCTION

Covalent modification of graphene oxide (GO) enables the tuning of chemo-physical properties of GO and promotes new functionalities and applications[1]. Different functionalization of GO have been recently proposed as sorbent material for water purification[2] from organic and metallic contaminants. However, for practical exploitation in water treatment large amount of materials should be prepared with high and reproducible batch purity level to avoid risks of secondary contamination caused by leaching of not-covalently bonded reagents and to guarantee reliable and stable batches performances. To this aim, here we report a new approach based on microwave (MW) assisted synthesis combined to microfiltration (MF) on commercial hollow fiber module allowing fast modification with efficient and reproducible purification of GO. As case study we selected polyethilenenimine (PEI) modified GO (GOPEI) which has revealed great potential for the removal of metal ions from water[3] and for the realization of membranes for ion sieving[4]. The so obtained GOPEI nanosheets were exploited for arsenic and lead removal from water and the performances were related to the surface properties (i.e. charge, polarity and structure) of the functionalized graphene.

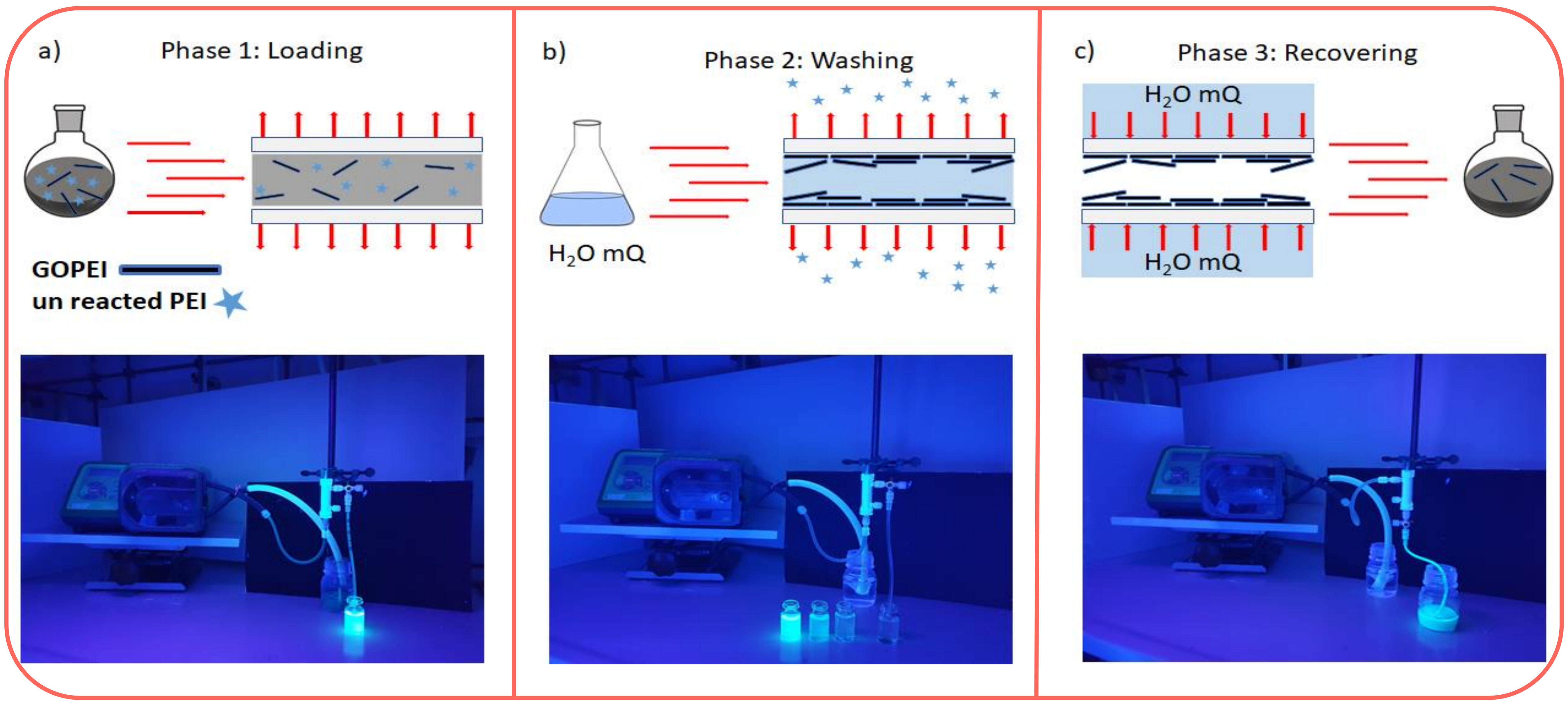
SYNTHESIS AND PURIFICATION



a) image of a PES cartridge (Plasmart 25 model, 110 mm length, Φint= 60 mm), about 275 hollow fibers, 700 mg weight (Φint= 50 µm, Φext=300 µm, cut-off= 0.1-0.2 µm); b) SEM image of the section of hollow fibers; c) pourus section of PES hollow fiber.

- GO and PEI suspension in water-EtOH was irradiated with MW at 80°C at a fixed power of 120 W for 30 minutes;
- The crude material was purified by microfiltration on commercial Versatile PES® hollow fibers modules (cut off range 100-200 nm), i.e., by forcing the solution to pass through the hollow fiber membrane section using a peristaltic pump.



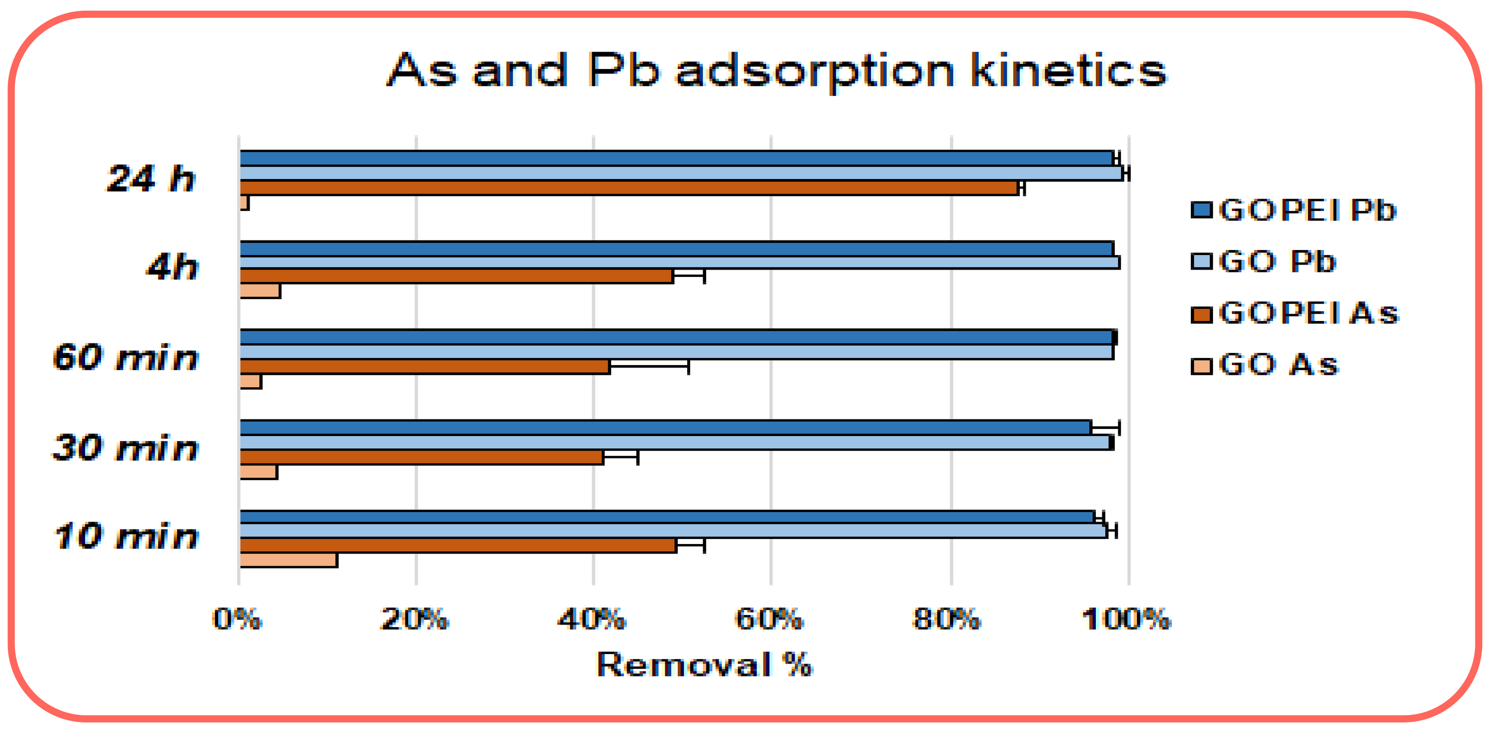
Purification mechanism:

a) Loading phase: the crude suspension is introduced in the hollow fibers passing each fiber in to out,
b) washing phase : the immobilized GO sheets (of size lower than polymer pore size) are retained on the fiber wall and washed with mQ water in the same flow direction,
c) recovery phase: purified GOPEI is collected in opposite flow direction (out-in).

To further visualize the purification process, PEI has been labeled with a fluorecent thiophene-based dye. This allowed to monitor the purification with UV-vis spectroscopy.

ARSENIC AND LEAD ADSORPTION

- GOPEI was used as sorbent for arsenic and lead contaminants, from spiked tap water (pH=7±0.5) at environmentally relevant concentration (100 µg/L each).
- GOPEI adsorbs both As and lPbead while unmodified GO adsorbs only Lead.
- GO reached 97.6 % removal for Pb after only 10 min while As adsorption was only 10%.
- GOPEI reached 49.1% removal of As after 10 min (87.5% at 24 h).
- The different selectivy can be explained considering the different surface charge of the two materials.
- The surface charges of GO are negative (Z_{pot}= - 23.18 mV) while the one for GOPEI are positive (Z_{pot}= 14.49 mV).



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