

# Versatile one-step route to synthesize high-quality graphene and graphene-nanoparticle composites based on modified polyol process

Rebecca F. Albers<sup>1</sup> (PG), Rafael A. Bini<sup>2</sup> (PQ), Laudemir C. Varanda<sup>1,\*</sup> (PQ)

<sup>1</sup> Colloidal Materials Group, Chemistry Institute of São Carlos - USP, São Carlos, SP, Brazil. \*lvaranda@iqsc.usp.br

<sup>2</sup> Federal University of Technology of Paraná, Campus Toledo, Toledo, Paraná, Brazil.

Key words: graphene, graphene/nanoparticles composite, polyol process, FePt, magnetite.

## Abstract

We described a new and versatile method to synthesize graphene and graphene/nanoparticles composites in one-step route, based on the modified polyol process. The method to synthesize graphene in the polyol medium was adjusted to synthesize graphene/FePt and graphene/Fe<sub>3</sub>O<sub>4</sub>.

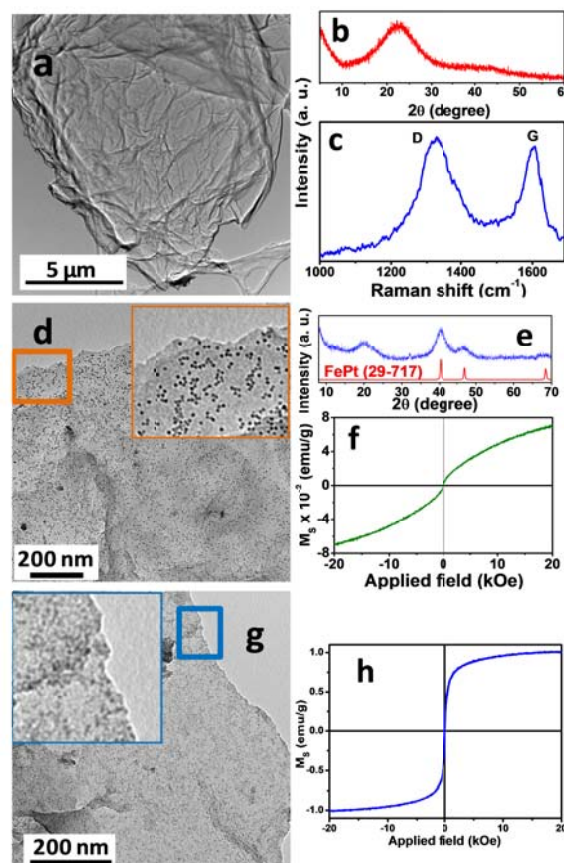
## Introduction

There is great interest to obtain composites that synergistically combine the unique properties of graphene (G) or graphene oxide (GO) with those from nanoparticles (NP)<sup>1</sup>. The modified polyol process is an efficient method to NP synthesis with morphological, structural and chemical composition controlled<sup>2</sup>. The reaction medium in the polyol promotes an environment suitable to the graphenization and to G synthesis from GO. In this work, we have described a new and versatile method for the synthesis of G, G/FePt and G/Fe<sub>3</sub>O<sub>4</sub> nanostructures in one-step route based on the modified polyol process. The method can be tuned to synthesis of many G/NP composites.

## Results and Discussion

GO was prepared using the modified Hummers method and flocculated with diethyl ether.<sup>3</sup> **G synthesis:** in a three neck round bottom flask the GO suspension (7mL, 80 mg) was mixture with the benzyl ether (BE, 20 mL), oleylamine (OAm, 2.7mmol) and 1,2-tetradecanediol (2.7 mmol). The temperature was increased to 100°C (30 min) and to reflux (120 min) under N<sub>2</sub> flux and stirring. **G/FePt synthesis:** using the same approach, Pt(II) (0.045 mmol) and Fe(III) (0.054 mmol) acetylacetonates were mixture with BE, OAm, and the diol without GO. The system was heated at 100°C (60 min) and to reflux (120 min). Temperature was decreased to 150°C and the GO suspension was injected into the system, which remains at 200°C (60 min). **G/Fe<sub>3</sub>O<sub>4</sub> synthesis:** following the G/FePt approach, OAm was replaced by oleic acid and 0.25 mmol of the Fe(III) acetylacetonate was used. **Characterization:** X-ray diffraction (XRD), transmission (TEM) and scanning (SEM) electron microscopies, Raman and UV-Vis spectroscopies, and vibrating sample magnetometry (VSM). The polyol medium was effective to promote the exfoliation and reduction of the GO leading to

obtain large graphene sheets. The G/NP composites were successful synthesized in a simple one-step route resulting in G/FePt and G/Fe<sub>3</sub>O<sub>4</sub> with NP average diameter of 5 nm and 8 nm, respectively.



**Figure 1.** Graphene: (a) TEM image, (b) XRD and (c) Raman spectrum; G/FePt: (d) TEM image (e) XRD, and (f) MxH hysteresis loop; G/Fe<sub>3</sub>O<sub>4</sub>: (g) TEM image and (h) magnetic hysteresis loop.

## Conclusions

G and G/NP composites (FePt, Fe<sub>3</sub>O<sub>4</sub>) synthesis in one-step were successfully performed by the modified polyol process. The method can be scaled to allow mass production and easily tuned for other NP already successfully reported by polyol process.

## Acknowledgements

To FAPESP for the financial support.

<sup>1</sup> Yang, W. et. al, *Carbon* **2016**, 96, 947.

<sup>2</sup> Varanda, L. C.; Jafelicci, M., Jr. *J. Am. Chem. Soc.* **2006**, 128, 11062.

<sup>3</sup> Marcano, D. C., et. al, *Acs Nano* **2010**, 4, 4806.