## Graphene Encapsulated Bimetallic Fe-Cu Nanoparticles: Synthesis, Purification and Characterization

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## Extended Abstract

Magnetic nanoparticles were used at various engineering applications such as catalysis for chemical reactions, electronic applications, biomedical applications (cancer detection and therapy), waste water treatment and oil spill cleaning [1,2]. Although, many papers were published about graphene encapsulated Fe nanoparticles, there are a few studies about the synthesis of graphene encapsulated nanoparticles with bimetallic core compositions. Cui et al. [3] synthesized the graphene encapsulated core/shell nanoparticles with binary metal (Fe/Co and Fe/Ni) core compositions. Farooq et al. [4] synthesized Fe-Cu/reduced graphene oxide (rGO) nanoparticles to use as catalysis for possible catalytic applications.

Herein, 1.25 g of Iron(III) chloride hexahydrate (FeCl<sub>3</sub>.6H<sub>2</sub>O) salts and 1.25 g of Copper(II) acetate hydrate (Cu(CO<sub>2</sub>CH<sub>3</sub>)<sub>2</sub>xH<sub>2</sub>O) salts were mixed with 6.25 g of fumed silica powders (Evonik Aerosil 380). These powder mixtures were solved into ethanol (C<sub>2</sub>H<sub>5</sub>OH, Merck, % 96) using stirrer and prepared solution were heated and stirred to prepare precursor powders. Silica impregnated Fe and Cu-salts based precursors were filled into quartz boats to locate chemical vapor deposition (CVD) system. CVD temperature, time and gas flows were changed to detect optimum CVD parameters. 900 and 950°C were selected as CVD temperatures. Also, holding time these temperatures are 60 min and outlet pressure of system is 50 mbar. The other parameters are flow rates of methane (CH<sub>4</sub>) and hydrogen (H<sub>2</sub>) gases. 50 ml/min and 100 ml/min flow rates for methane gas that used as carbon source were preferred for different CVD studies. Synthesized particles via CVD were purified using 2 M hydrofloric acid (HF) and 4 M hydrochloric acid (HCl) leaching to remove remained SiO<sub>2</sub> phase and uncoated Fe/Cu nanoparticles. Purified powders were characterized X-Ray diffractometer (XRD), scanning electron microscope (SEM), transmission electron microscope (TEM), Raman spectroscopy and differential thermal analysis (DTA)/thermogravimetric analysis (TG) and vibrating sample magnetometer (VSM).

Based on the XRD analysis, after the CVD Fe, Cu and C (graphite) phases were detected and also after leaching steps same phases were detected after leaching steps. Transmission electron microscope (TEM) images showthe graphene encapsulated morphologies. Additionally, hysteresis loops enabled from VSM measurements show the synthesized nanoparticles have soft magnetic properties. Therefore, they might be a candidate material for different biomedical applications.

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## References

[1] S. Mertdinç-Ülküseven, U. Savacı, K. Onbasli, Ö. Balcı-Çağıran, H.Y. Acar, M.L. Öveçoğlu, D. Ağaoğulları, "In-situ

synthesis of graphene encapsulated Fe/Fe<sub>2</sub>O<sub>3</sub> nanoparticles for possible biomedical applications", *Journal of Materials Research and Technology*. vol. 20, pp. 2558–2577, 2020.

- [2] D. Ağaoğulları, S.J. Madsen, B. Ögüt, A.L. Koh, R. Sinclair, "Synthesis and characterization of graphite-encapsulated iron nanoparticles from ball milling-assisted low-pressure chemical vapor deposition", *Carbon.* 124 pp. 170–179, 2017.
- [3] X. Cui, P. Ren, D. Deng, J. Deng, X. Bao, "Single layer graphene encapsulating non-precious metals as highperformance electrocatalysts for water oxidation", *Energy and Environmental Science*. vol. 9, pp.123–129, 2016.
- [4] U. Farooq, M. Danish, S. Lu, M.L. Brusseau, M. Naqvi, X. Fu, X. Zhang, Q. Sui, Z. Qiu, "Efficient transformation in characteristics of cations supported-reduced graphene oxide nanocomposites for the destruction of trichloroethane", *Applied Catalysis A: General*. vol. 544, pp.10–20, 2017.