AERONAUTICS INSTITUTE OF TECHNOLOGY

cbecimat26
26° Congresso Brasileiro de Engenharia e Ciência dos Materiais

22–26 November 2026
Costão do Santinho · Florianópolis, SC

Surface Chemical Modification and Nitrogen Enrichment of Reduced Graphene Oxide by Indirect DBD Plasma–Liquid Treatment

André Petraconi[1] · Felipe Miranda[1] · Gilberto Petraconi[1] · Rodrigo Sávio Pessoa[1] · Argemiro Soares da Silva Sobrinho[1]

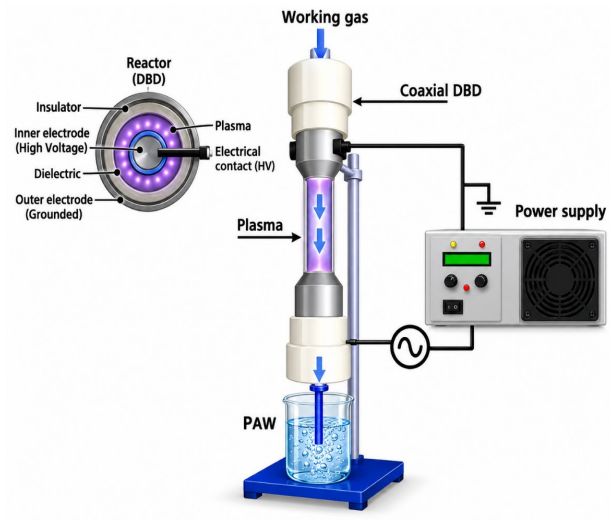
[1] Aeronautics Institute of Technology (ITA), São José dos Campos, São Paulo, Brazil

INTRODUCTION

This study presents a novel and environmentally friendly approach for the functionalization of reduced graphene oxide (RGO) nanosheets. However, conventional nitrogen modification can require high temperatures or harsh reagents that may compromise its structure. Here, a coaxial dielectric barrier discharge (DBD) reactor activated water or NH_4OH under compressed air or N_2 for indirect plasma–liquid RGO treatment, aiming to promote surface nitrogen enrichment while retaining its graphitic characteristics.

Objective: Evaluate graphene functionalization using different plasma-activated solutions.

METHODOLOGY



01 / ACTIVATE
40 mL DI water or NH_4OH (28%); air/N_2, 5Lmin^{-1} 10 min.

02 / TREAT
Add 0.1g RGO; expose for 30 min.

03 / RECOVER
Dry at 60 °C for 72 h.

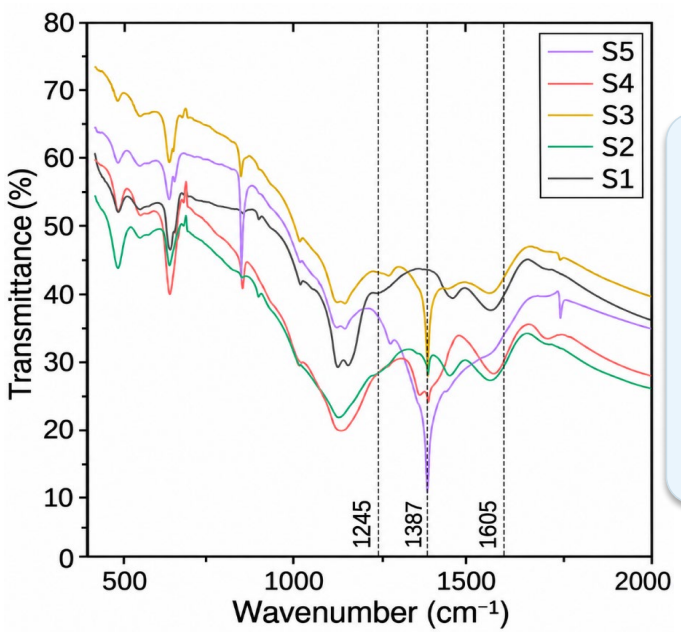
Fig. 1. Coaxial DBD reactor (adapted from Miranda et al. [2]); P = 50.70 W (air) and 66.84 W (N_2).

RESULTS AND DISCUSSION

Untreated	pH	EC[1]	ORP[2]
DI water	6.90	0.037	+8.9
NH_4OH (28%)	12.97	0.829	−346.9

Medium	pH[3]	pH[4]	pH[5]	EC[5]	ORP[5]
PAW/air	2.35	3.35	1.87	5.352	+285.4
PAW/N_2	3.91	10.22	10.18	1.871	−190
NH_4OH/air	11.08	12.30	11.02	3.420	−235.4
NH_4OH/N_2	12.52	12.43	10.80	3.063	−223.0

Table 1. pH after 10 min activation ([3]), immediately after RGO addition ([4]), and 30 min with RGO ([5]). EC: mS cm^{-1}; ORP: mV.



S1 - Pristine RGO

S2 - NH_4OH/air

S3 - NH_4OH/N_2

S4 - PAW/air

S5 - PAW/N_2

Fig. 2. FTIR spectra (KBr pellets), S1–S5, showing fingerprint-region variations near 1245, 1387 and 1605 cm^{-1}.

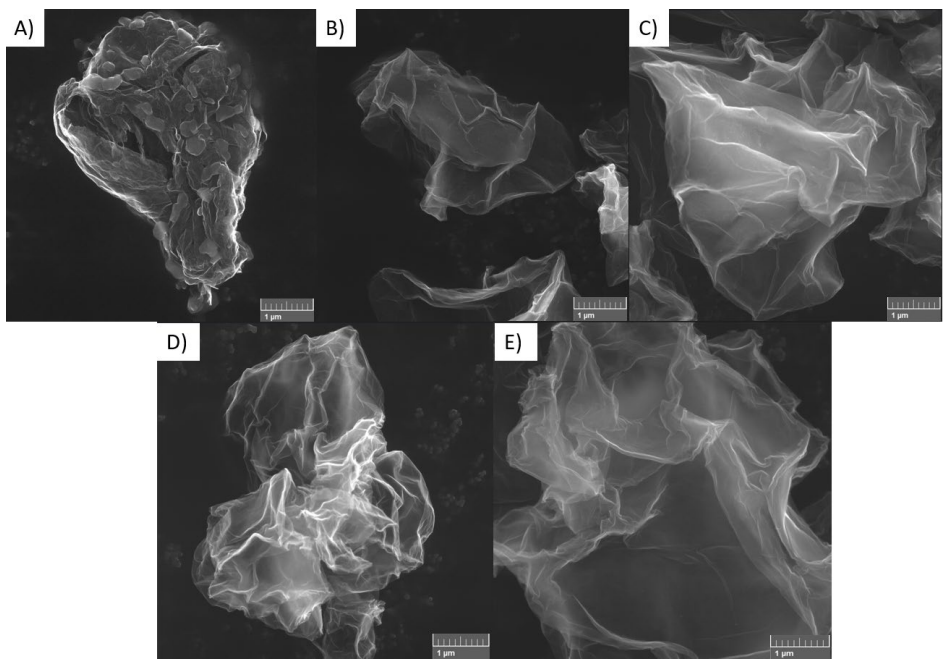


Fig. 3. FEG-SEM micrographs: (a) pristine RGO (S1), (b) NH_4OH/air (S2), (c) NH_4OH/N_2 (S3), (d) PAW/air (S4), (e) PAW/N_2 (S5).

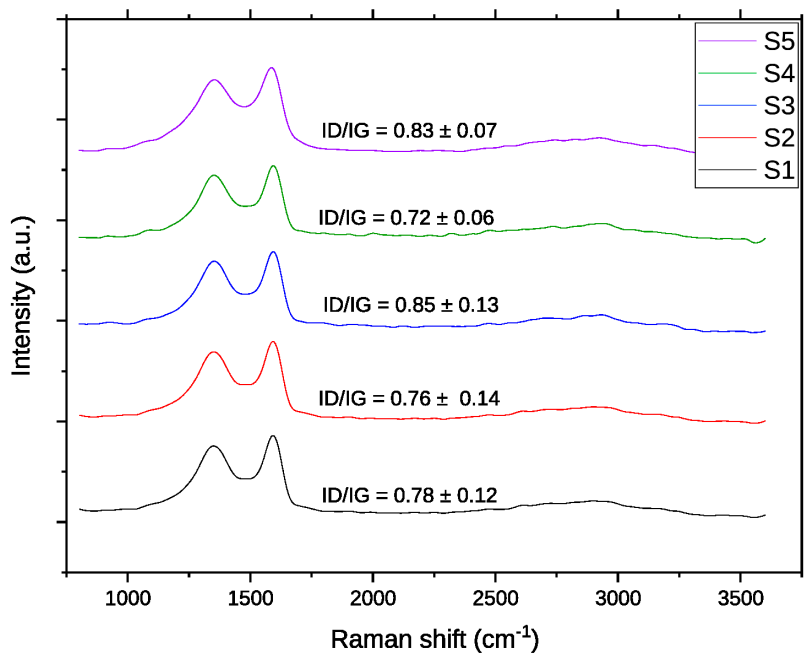


Fig. 4. Raman spectra of S1–S5 (306-spectrum averages), normalized to the G band. I_D/I_G values: descriptive mean ± SD.

Sample	C (at.%)	O (at.%)	N (at.%)
S1	78.18 ± 0.08	21.55 ± 0.25	0.27 ± 0.24
S2	84.72 ± 1.07	13.09 ± 1.05	2.19 ± 0.05
S3	85.10 ± 0.16	13.69 ± 0.21	1.21 ± 0.16
S4	80.76 ± 0.21	17.19 ± 0.35	2.05 ± 0.19
S5	85.48 ± 0.29	13.64 ± 0.40	0.88 ± 0.12

Table 2. XPS surface atomic composition (at.%, mean ± SD) for S1–S5; three surface regions per specimen.

CONCLUSIONS

Indirect DBD plasma–liquid treatment modified RGO surface chemistry while retaining characteristic graphitic spectral features. Air-fed treatments yielded the highest XPS-detected nitrogen (~2 at.%); all treated samples showed lower XPS-detected surface oxygen than pristine RGO. The results support surface-associated nitrogen enrichment and apparent partial deoxygenation/redistribution.

This novel DBD plasma-liquid method offers a scalable, tunable, and environmentally friendly route for graphene functionalization. Further studies for optimization of plasma parameters and solution composition can enhance functional group selectivity, expanding potential applications

References and Acknowledgments

[1] Pei, S.; Cheng, H.-M. The Reduction of Graphene Oxide. Carbon 2012, 50, 3210–3228. doi:10.1016/j.carbon.2011.11.010.
[2] Miranda, F. S. et al. Physicochemical Characteristics and Antimicrobial Efficacy of Plasma-Activated Water. Water 2023, 15, 4045. doi:10.3390/w15234045.
[3] Miranda, F. S. et al. Influence of Gas Type on Reactive Species Formation in Plasma-Activated Water. Plasma Chem. Plasma Process. 2024, 44, 1713–1733. doi:10.1007/s11090-024-10475-7.

FAPESP (2023/07607-0), Eneva H_2 Turquoise Project, ITA, and LPP.

Special thanks to ENEVA for funding this R&D&I project.