Supporting Information

CoFe$_2$O$_4$ Nanoparticles Grown within Porous Al$_2$O$_3$ and Immobilized on Graphene Nanosheets: A Hierarchical Nanocomposite for Broadband Microwave Absorption

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S1. Materials Synthesis

S1.1. Chemical Reagents

Ferric Nitrate Fe(NO$_3$)$_3$.9H$_2$O, Aluminium Nitrate Al(NO$_3$)$_3$.9H$_2$O, polyethylene glycol (PEG-400), Sodium Hydroxide (NaOH), and triethanol amine were purchased from Fisher Scientific, Cobalt Nitrate Co(NO$_3$)$_2$.6H$_2$O and Stearic Acid were bought from Merck India, Graphene was provided by ad nanotechnologies. Deionized water was used for all the experiments. The chemicals used in this work were of analytical grade and were used without any further purification.

S1.2. Synthesis of Porous Alumina (PA)

2.23g of Stearic acid was warmed at 80°C. An aqueous solution of Aluminium nitrate was prepared by dissolving 29.410g of Al(NO$_3$)$_3$.9H$_2$O in 40 mL distilled water and warmed it at 80°C. A TEA solution was prepared by mixing 31.2 mL of TEA with 60 mL of distilled water. An aqueous solution of TEA was mixed with stearic acid and stirred continuously for about 30 min. This mixture was then added to the aqueous solution of Al(NO$_3$)$_3$.9H$_2$O with constant stirring at 80°C for another 30 min. A white precipitate was formed. Further, the reaction mixture was stirred for 12h at room temperature. Then the gel was transferred in a Teflon-lined stainless steel autoclave, closed tightly, and aged for 24h at 90°C. Then the autoclave was cooled to room temperature after which the white gel formed was filtered, washed with distilled water, and dried at 85°C to obtain precursor powder. The precursor was then transferred into a crucible and calcined at 550°C for 3h in the air to obtain Porous Alumina (PA) powder.

S1.3. Synthesis of Cobalt Ferrite (CF)

To prepare CoFe$_2$O$_4$, Co(NO$_3$)$_2$.6H$_2$O and Fe(NO$_3$)$_3$.9H$_2$O (1:2 molar ratio) were dissolved in water and mixed with PEG-400 and water mixture (1:5 weight ratio). Then the pH of the reaction mixture was raised to ~11 by adding 2M aqueous NaOH solution dropwise. Then the
reaction mixture was refluxed at 160°C for 16h. The resultant black-coloured product was separated from the reaction mixture using a magnet followed by washing with distilled water, ethanol and then it was dried at 60°C for 10h.

**S1.4. Synthesis of PA-CF nanocomposites**

For synthesizing PA-CF nanocomposites, 1.232g of Co(NO$_3$)$_3$.6H$_2$O & 3.424g of Fe(NO$_3$)$_2$.9H$_2$O (1:2 molar ratio) were weighed and dissolved in 50 mL water and both the nitrate solutions were mixed and stirred for about 10 minutes. In a 1000 mL round bottom flask, PEG-400 was weighed and the nitrate mixture was added to it dropwise and the solution was stirred for 30 min. Meanwhile, 1.5g of Porous Alumina was well dispersed in 100 mL of water. After stirring, porous alumina was added to the solution and 2 M NaOH was added dropwise till the pH of the solution was about ~11. The solution was stirred for 30 min after which it was refluxed in an oil bath for 30 min at 80°C. Then the temperature of the reaction mixture was increased to 160 °C and it was refluxed for 16h. After cooling, the mixture was filtered and the obtained product was washed with distilled water until the pH was neutral and the product was dried in an oven at 80°C overnight.

**S1.5. Synthesis of ((PA-CF)$_x$.Gr$_y$) nanocomposites**

(60PA-40CF)-Gr nanocomposites were synthesized by a simple wet impregnation method. Required amounts of Graphene (Gr) and 60PA-40CF were weighed and dispersed in about 100 mL Methanol in a 250 mL round bottom flask. The mixture was refluxed in an oil bath at 70°C for 4 h. After cooling, it was filtered using a vacuum pump and the obtained product was dried at 80°C overnight.

**S2. Characterizations of the Materials**

The synthesized composites were characterized by X-ray diffraction (XRD) by using a powder X-ray diffractometer (Mini Flex II, Rigaku, Japan) with Cu Kα ($\lambda$=0.154nm),
thermogravimetric analysis (TGA) by using a DTA-60 (Shimadzu, Japan), Field Emission Scanning Electron Microscopy (FESEM) by using Quanta 250 FEG (FEI), Energy Dispersive X-ray Spectra (EDS) by using an EDAX ELEMENT electron microscope, Raman spectroscopy by using a Horiba via Raman microscope with a 532 nm laser excitation, Multiple point BET surface area was determined by a Surface area and porosimetry analyzer (Micromeritics Tristar 3000, USA) using N\textsubscript{2} as the probe. The cumulative pore volume and pore surface area of pores were determined from BJH desorption data. XPS measurements were carried out by using a Thermo-Scientific ESCALAB Xi+ spectrometer having a monochromatic Al K\textalpha X-ray source (1486.6 eV) and a spherical energy analyzer that operates in the CAE (constant analyzer energy) mode.

S3. Microwave absorption study

The microwave absorption analysis of the synthesized materials was carried out in the X-band (8.2–12.4 GHz) range by using a HP 8510 vector network analyzer. From the measured values of complex permeability and complex permittivity, Reflection Loss (RL) was calculated from the measured values of the complex permeability and complex permittivity. For the preparation of samples to carry out the aforesaid measurement, the nanocomposites in the powder form were mixed with 10 wt\% of binder [polyvinyl alcohol (PVA)] after which the mixture was dried. The dried mixture was ground to powder and compressed under a pressure of 10 tons to form a rectangular pellet having a size of 10.16 mm × 22.86 mm × 2 mm which fits exactly into the rectangular waveguide of the X-band (8.2–12.4 GHz).
Figure S1. XRD patterns of (a) PA, (b) CF, (c) 60PA-40CF, and (d) Graphene.
Figure S2. XRD patterns of \((60\text{PA}-40\text{CF})_{10}\text{-Gr}_{90}\), \((60\text{PA}-40\text{CF})_{25}\text{-Gr}_{75}\), \((60\text{PA}-40\text{CF})_{50}\text{-Gr}_{50}\), and \((60\text{PA}-40\text{CF})_{75}\text{-Gr}_{25}\).
Table S1. BET surface area and porosimetry analysis data of the synthesized materials.

| S. No. | Materials       | BET Surface Area (m²/g) | Pore Volume (cm³/g) | BJH Desorption average pore diameter |
|--------|-----------------|-------------------------|---------------------|-------------------------------------|
| 1      | Pure PA         | 218                     | 0.33                | 4.6 nm                              |
| 2      | 60PA-40CF       | 245                     | 0.35                | 3.5 nm                              |
| 3      | (60PA-40CF)₁₀⁻Gr₁₀ | 199.08                 | 0.21                | 3.9 nm                              |
Figure S4. FESEM micrograph of Gr.

Figure S5. EDS spectra of (60PA-40CF)$_{90}$-Gr$_{10}$. 
Figure S6. Elemental mapping images of (60PA-40CF)_{90}-Gr_{10} nanocomposite.

Figure S7. XPS survey spectrum of (60PA-40CF)_{90}-Gr_{10}.
Figure S8. Frequency dependence (a) real part and (b) imaginary part of relative complex permittivity, (c) real part, and (d) imaginary part of relative complex permeability values of the synthesized materials.
Figure S9. Typical Cole-Cole semicircles for (a) 90PA-10CF, (b) 80PA-20CF, (c) 70PA-30CF, and (d) 50PA-50CF.

Figure S10. Plots of $\mu'(\mu)^{-2}f^{-1}$ vs frequency for the synthesized materials in the frequency range 8-12 GHz.
Figure S11. 3D thickness dependent RL plots of (a) 90PA-10CF, (b) 80PA-20CF, (c) 70PA-30CF, and (d) 50PA-50CF.
Figure S12. Minimum reflection loss values of (a) PA and (b) CF.

Figure S13. Minimum reflection loss values of (a) 90PA-10CF and (b) 80PA-20CF.

Figure S14. Minimum reflection loss values of (a) 70PA-30CF and (b) 60PA-40CF.
Figure S15. Minimum reflection loss values of (a) 50PA-50CF and (b) (60PA-40CF)\textsubscript{75}-Gr\textsubscript{25}.

Figure S16. Minimum reflection loss values of (i) (60PA-40CF)\textsubscript{90}-Gr\textsubscript{10}.
**Figure S17.** Attenuation constant ($\alpha$) vs. frequency curves for PA, CF, 60PA-40CF, and (60PA-40CF)$_{90\text{-Gr}_{10}}$.

**Table S2.** Microwave absorption properties of the synthesized materials.

| S.No. | Composite    | Reflection Loss (dB) and % absorption | Frequency (GHz) | Thickness (mm) | Effective bandwidth (RL < -10 dB) (GHz) |
|-------|--------------|---------------------------------------|-----------------|----------------|----------------------------------------|
| 1     | Pure PA      | -7.44 (~82%)                           | 10.37           | 3.2            | -                                      |
| 2     | Pure CF      | -8.52 (~86%)                           | 11.23           | 2.5            | -                                      |
| 3     | 90PA-10CF    | -6.90 (~79%)                           | 11.10           | 2.8            | -                                      |
| 4     | 80PA-20CF    | -9.29 (~88%)                           | 10.93           | 2.7            | -                                      |
| 5     | 70PA-30CF    | -12.2 (~94%)                           | 11.86           | 2.3            | 10.05-12.4 GHz                        |
| 6     | 60PA-40CF    | -12.73 (~95%)                          | 11.21           | 2.3            | 10.3-12.4 GHz                         |
Table S3. Comparison of the Microwave Absorption properties of various Al₂O₃, CoFe₂O₄, Graphene, and based Composites.

| Samples                          | Minimum Reflection Loss (dB) | Corresponding Frequency (GHz) | Absorber Thickness (mm) | Effective bandwidth (RL < -10 dB) (GHz) | Ref. |
|----------------------------------|------------------------------|--------------------------------|-------------------------|----------------------------------------|------|
| Al₂O₃/TiO₂/Fe₂O₃/Yb₂O₃          | -20.18                       | 3.52                           | 3.5                     | 2.16-9.76 (7.6)                        | 1    |
| ZrB₂/Al₂O₃                      | -15.6                        | 10.8                           | 1.4                     | 10.3-12 (1.7)                          | 2    |
| TiO₂/Al₂O₃                      | -29                          | 10.6                           | 1.7                     | 8.2-18 (9.8)                           | 3    |
| NiCrAlY/Al₂O₃                   | -15.7                        | 8.9                            | 2.0                     | 8.2-9.5 (1.3)                          | 4    |
| grain-like Ni/Al₂O₃/Ni          | -28.2                        | 12.92                          | 4.5                     | - (5.5)                                | 5    |
| flower-like Ni/Al₂O₃/Ni         | -45.3                        | 11.4                           | 3.5                     | -                                      | 5    |
| Composite Material                        | Energy (eV) | Thickness (nm) | Band Gap (eV) | Reference |
|-------------------------------------------|-------------|----------------|---------------|-----------|
| KNN/Al$_2$O$_3$                           | -6.8        | -              | 0.95          | (7.1)     |
| Al$_2$O$_3$–MoSi$_2$/Cu                   | -19.09      | 10.5           | 1.4           | -         |
| Al$_2$O$_3$@PPy@rGO                       | -39.7       | 13.2           | 3.0           | 10.5-16.0 |
| FeSiAl/Al$_2$O$_3$                         | -34.2       | 9.4            | 1.9           | 9.1-11.0  |
| FeSiAl and flaky graphite filled Al$_2$O$_3$ | -15.2       | 10.6           | 1.0           | 10.0-11.2 |
| Reduced graphene oxide/Cu–Ni ferrite/Al$_2$O$_3$ | -23.2       | 15.6           | -             | -         |
| Fe$_3$Al@Al$_2$O$_3$                       | -34         | 11.5           | 2.0           | - (6.7)   |
| Ti$_3$SiC$_2$/Al$_2$O$_3$                  | -24.4       | 11.1           | 1.3           | 10.1-12.4 |
| FeSiAl@Al$_2$O$_3$@SiO$_2$                 | -46.29      | 16.93          | 2.0           | 10.14-17.45 |
| Graphene Nanosheets/ZnO/Al$_2$O$_3$       | -45         | 11.5           | 1.82          | 11.2-12.0 |
| Al$_2$O$_3$ –MoSi$_2$ – Cu                | -17.96      | -              | 1.7           | - (2.42)  |
| Coiled carbon nanotubes/CoFe$_2$O$_4$ composites | -14.44      | 13.20          | 3.0           | - (4.0)- |
| CoFe$_2$O$_4$/NiFe$_2$O$_4$                | -20.1       | 9.7            | 4.5           | 7.8-16.2  |
| CoFe$_2$O$_4$ rGO/SiO$_2$                  | -24.8       | 5.8            | 2.0           | - (1.7)   |
| CoFe$_2$O$_4$/polyaniline in paraffin      | -28.9       | -              | 2.0           | 14.0-18.0 |

Reference:

6. [Reference 6]
7. [Reference 7]
8. [Reference 8]
9. [Reference 9]
10. [Reference 10]
11. [Reference 11]
12. [Reference 12]
13. [Reference 13]
14. [Reference 14]
15. [Reference 15]
16. [Reference 16]
17. [Reference 17]
18. [Reference 18]
19. [Reference 19]
20. [Reference 20]
|                       | τ (s) | Δ (s) | Δτ/Δγ | Error (%) |
|-----------------------|-------|-------|--------|-----------|
| FeCo/CoFe$_2$O$_4$    | -15.0 | -     | 6.7    | -         |
| CoFe$_2$O$_4$-coated  | -14.8 | 13.6  | 2.0    | 12-15.6   |
| poly(acrylonitrile)   |       |       |        | (3.6)     |
| microspheres with     |       |       |        |           |
| graphene              |       |       |        |           |
| Polyaniline(PANI)/CoFe|$-30.2$| 5.6   | 3.0    | 4.2-7.8   |
| 2O$_4$/Ba$_{0.4}$Sr$_{0.6}$TiO$_3$| | | | (3.6) |
| CoFe$_2$O$_4$/rGO     | -11.1 | 17.0  | 2.5    | -         |
|                       |       |       |        | (2.3)     |
| Fe$_3$O$_4$/SiO$_2$/graphene | -27.1 | 12.2  | 1.5    | 3.3-13.6  |
|                       |       |       |        | (10.3)    |
| Nitrogen-doped graphene | -11.3 | 12.7  | 3.0    | 12.2-14.3 |
|                       |       |       |        | (2.1)     |
| GN/Carbon             | -28.1 | -     | 1.5    | -         |
|                       |       |       |        | (5.7)     |
| CoNi/graphene         | -31.0 | 4.9   | 4.0    | 9.5-16.8  |
|                       |       |       |        | (7.3)     |
| nitrogen-doped graphene | -24.6 | 8.51  | 3.0    | 7.55-12.44|
|                       |       |       |        | (4.89)    |
| NiCo$_2$/GNS          | -30.0 | 11.7  | 1.6    | -         |
|                       |       |       |        |           |
| Co$_{0.33}$Ni$_{0.33}$Mn$_{0.33}$Fe$_2$O$_4$/graphene | -24.29 | -     | 2.3    | 9.52-18.00|
|                       |       |       |        | (8.48)    |
| α-Fe$_2$O$_3$ nanorod–| -45.5 | 14.6  | 2.0    | 10.5-16.0 |
| graphene              |       |       |        | (5.5)     |
| graphene/CoFe$_2$O$_4$/Y$_3$Fe$_5$O$_{12}$ | -36.1 | 14.88 | 3.0    | 14-16     |
|                       |       |       |        | (2.0)     |
| (60PA-40CF)$_{90}$-Gr$_{10}$ | -30.68 | 10.71 | 2.0    | 8.2-12.4  |
|                       |       |       |        | (4.2)     |
|                       | -30.77 | 9.04  | 2.3    | 8.2-12.4  |
|                       |       |       |        | (4.2)     |
|                       | This work | | | |
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