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Development and Characterization of RGO Ink for Microwave Absorber based Stealth Application

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This research paper presents a method for synthesizing reduced graphene oxide (rGO) ink and investigates its morphological behavior based on sonication time. This study also explores the experimental verification of rGO's functionality as a tunable attenuator operating in the 1-8 GHz range. The synthesis process consists of three phases: synthesis of graphene oxide (GO), reduction of GO to rGO, and dispersion of rGO powder in a solvent. A comparative analysis of morphological and functional behavior is conducted by varying sonication time using X-ray Diffraction (XRD), Scanning Electron Microscope (SEM), and Energy Dispersive Spectroscopy (EDS). Results indicate that increased sonication time enhances the quality of multi-layered graphene, resulting in finer stacked layers and improved electrical properties. Also, measuring insertion loss at different biased voltages shows that r-GO ink can be used as a microwave tunable attenuator. This study provides valuable insights into the synthesis and characterization of rGO ink, highlighting its potential applications in the field of microwave electronics.

Keywords: Graphene oxide; X-ray Diffraction; Scanning Electron Microscope

1 Introduction

Now-a-days Graphene and its derivatives (Graphene oxide and reduced Graphene oxide) are gaining widespread attention from researchers, industries, and government agencies, as they explore its unique properties, synthesis methods, and potential applications in fields such as photonics, microwave absorption, sensor technology, and biomedicine[1-4].

Graphene was successfully peeled off over a decade ago; the discloser of the two dimensional Graphene element was done by Novoselov & Geim[5-7]. However, obtaining high yield graphene is very complicated and expensive process; therefore reduced Graphene Oxide(rGO) can be an affordable alternative to the miraculous material graphene. Reduced graphene oxide offers almost same electrical and mechanical properties; therefore, it can be considered as effective alternative to graphene mainly for preparation of nanocomposite material which can further be used in microwave industries[8].

Graphene is a two dimensional mono layered material having sp^2 bonded carbon atoms densely arranged in hexagonal lattice, it will be appropriate to call graphene basic building block as it can be wrapped in 0-D Fullerenes, rolled in 1-D Carbon Nano Tubes(CNTs) or can be stacked in 3D material graphite shown in Fig. 1[7].

Graphene is capable of potentially replacing silicon in electronic and microwave devices due to its unique and superior properties compared to silicon: high electron mobility, exceptional electrical conductivity, flexibility and mechanical strength, high thermal conductivity, scalability and integration with existing technology. Since graphene has the capability of altering the place of silicon based electronic and the microwave device[9-12], therefore this article is significant for the following: synthesis of grapheme oxide, reduced graphene oxide and reduced graphene oxide ink using cost effective graphite powder. And a comparative study of structural behavior of rGO ink on the basis of altering the sonication time.

In previous research articles[13-16] the application of graphene for implementation of RF component has been studied theoretically only. RF component tunable attenuator studied in[17] operated at 5GHz operating frequency.

The synthesized multi-layer crystalline rGO obtained from chemical reduction method was drop casted in stacked form in 13-14 layer over the

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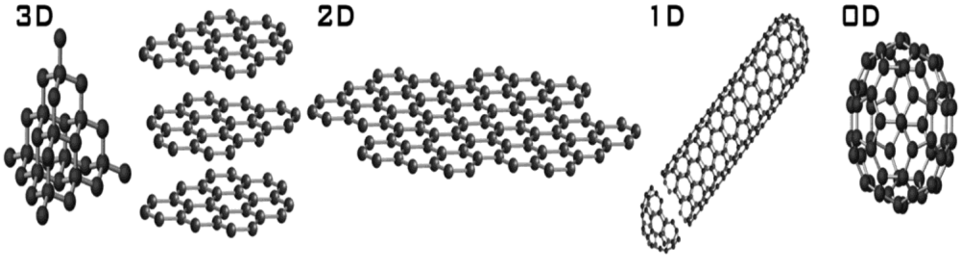


Fig. 1 — Crystal structures of various allotropes of carbon (Left to right): 3D structure of diamond and graphite; 2D graphene; 1D nanotubes and 0D buckyballs[7]

substrate surface in between micro-strip lines. A comparative analysis of electric field emission through microstrip lines on few-layered rGO was done on parallel plate type setup as shown in[18-20].

Novelty of Work

Tunable Attenuator Functionality: The research experimentally verifies rGO's functionality as a tunable attenuator in the 1-8 GHz frequency range. This application of rGO in microwave electronics is innovative, especially as it provides insights into how rGO's electrical properties can be manipulated through biased voltages to control insertion loss. This aspect of the study highlights a practical application of rGO ink, which is relatively underexplored in the existing literature.

Integration of Synthesis and Functional Testing: By not only synthesizing rGO ink but also testing its application in microwave electronics, the research integrates material synthesis with practical device applications. This holistic approach of moving from material creation to functional testing in a specific, high-frequency domain is a significant contribution, especially in the context of rGO's use in tunable attenuators.

2 Materials and Synthesis

This section presents the materials used and process followed for synthesis of graphene ink. Various methods areavailable in literature to develop layers and thin film graphene. The synthesis of graphene can be accomplished by basically two approaches, that is bottom-up and top-down approach, as shown in Fig. 2[4]. Bottom up approach comprises the synthesis from different carbon sources while on other hand top-down approach involves the separation of easily available graphite layers to single sheets.

2.1 Material used

The process of synthesis is started with raw graphite as base material with fixed 99% of carbon content. Along with the base material, potassium per magnet, sodium nitrate, 98% sulphuric acid, 5% hydrochloric acid, 44% hydrogen peroxide, 75% of hydrazine hydrate and PVP of analytical quality were used. During the processes, distilled water with resistivity 18.25 MΩ/cm is used.

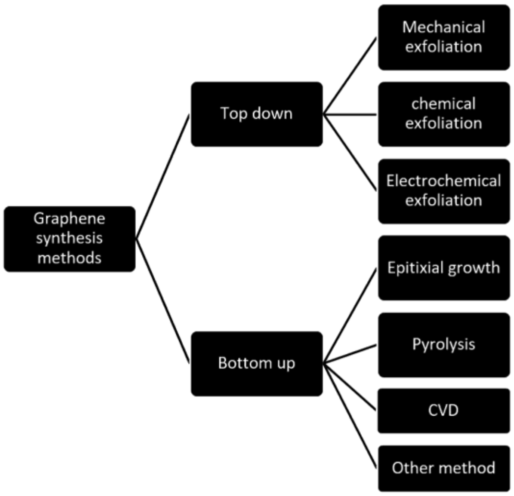


Fig. 2 — Schematic representation of different methods of graphene synthesis[4]

2.2 Synthesis Process

This article emphasis on the preparation of graphene ink, which can be obtained in basic three phases. Phase I, includes synthesis of grapheneoxide (GO), which can be obtained by reacting graphite powder with concentrated acid in presence of a strong oxidizing agent. The complete process followed in Phase-I is shown in Fig. 1. Phase II includes the reduction of graphene oxide by removal of oxygen.

Phase I (Oxidation, Intercalation and Exfoliation)

Figure 1 the preparation of graphene oxide (GO) begins with the reaction of 1 g each of raw graphite powder and sodium nitrate ($NaNO_3$), continuously stirred with 46 mL of sulfuric acid (H_2SO_4) in an ice bath for 4 hours. This results in the formation of a dark green solution.

Subsequently, 6 g of potassium permanganate ($KMnO_4$) is gradually added to the solution, which is then stirred for 1 hour while maintaining the temperature at 50 °C. An exothermic reaction occurs as 220 mL of distilled water is added dropwise with continuous stirring.

Following this, the ice bath is removed, and the temperature is raised to 35 °C for 45 minutes. Afterward, 10 mL of hydrogen peroxide (H_2O_2, 30%) is added dropwise while stirring at 800 rpm, leading to the formation of a bright yellow suspension. The resulting high-yield graphene oxide paste is then washed three times with 5% hydrochloric acid (HCl) using a centrifuge, followed by repeated washing with distilled water. Finally, the graphene oxide paste is dried in a hot air oven for 12 hours at 50 °C.

Phase II (Process to obtain reduced Graphene Oxider GO)

The graphene oxide obtained from Phase I is reduced using hydrazine monohydrate as a reducing agent. A 300 mg sample of GO powder is dispersed in 100 mL of distilled water and sonicatedfor 3, 4, and 5 hours using an ultrasonic bath cleaner. After sonication, 3 mL of hydrazine monohydrate is added to the solution, which is then heated in an oil bath at 100 °C for 24 hours. This process yields black solid precipitates of reduced graphene oxide (rGO), which are separated via vacuum filtration. The resulting product is washed three times with 5% ethanol and distilled water, using a centrifuge for thorough cleaning. Finally, the washed residue is collected and dried in a hot air oven at 70 °C for 24 hours.

Phase III (r GO ink preparation)

Figure 4 the 25 mg of rGO obtained in phase-II is added with 40 mg of PVP (Polyvinylpyrrolidone) [MW:40,000] dispersed in 10 ml isopropyl alcohol followed by sonication for 2 hours,3 hours, 4 hours and 5 hours. Sonication time can lead to changes in XRD peaks by altering the intensity, position, and broadness of the peaks. These changes are indicative of modifications in the material's crystallinity, layer spacing, and degree of exfoliation or disorder[21-23]. Finally, a black colored rGO ink is formed.

3 Characterization Methods

As discussed in phase-III of synthesis process, four samples with different sonication time of 2hrs, 3hrs, 4 hrs and 5 hrs respectively are prepared and labeled as Sample-1, Sample-2, Sample-3 and Sample-4. (as shown in Fig. 4). These samples are then analyzed through X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS).

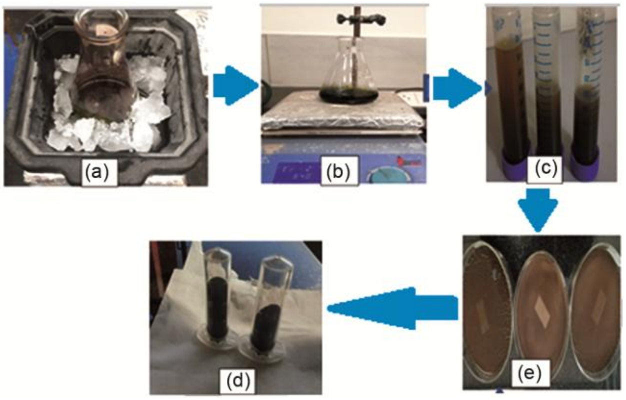


Fig. 3 — Phase I:GO synthesis (a) raw graphite powder and Sodium nitrate ($NaNO_3$) with Sulphuric acid (H_2SO_4) under ice bath (b) mixing Potassium per magnet ($KMnO_4$) gives sluggish brown solution (c) brown solution centrifuged (d) dried in hot air oven at 70°C (e) powdered GO

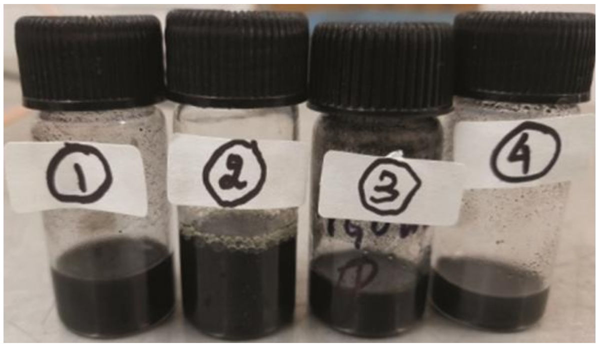


Fig. 4 — Final samples in the form of rGO ink

The X-ray diffraction patterns of four samples of powdered rGO with different sonication time were recorded using HyPix-400(2D HAPD)detector (SmartLab SE Model, Rigaku, Japan) for 1° rotation per minute in the 2θ range of 5° to 90° with Cu –Kα radiation ($\lambda = 1.5418$ Å) to analyze the interlayer spacing (d-spacing; distance between two consecutive planes) of rGO. Fig. 5 shows the comparative results of all the samples 1, 2, 3 and 4, which exhibits sharp dominant peaks at approximately 2θ = 24.94°, 28.54°, 28.44° and 24.38° respectively with d-spacing of 0.3nm, 0.36nm, 0.36nm and 0.37nm respectively. Second relatively weak peak for all samples obtained at approximately 2θ = 44.21°.

Due to intercalation and diffusion of oxygen group after reduction which creates defects, XRD pattern shows the crystalline structure of few layered graphene only. Sample 1 has low intensity first peak as compared to the other sample due to the presence of tightly bound oxygenatom inrGO and increase in sonication time did effectively helped in its reduction[13-14].

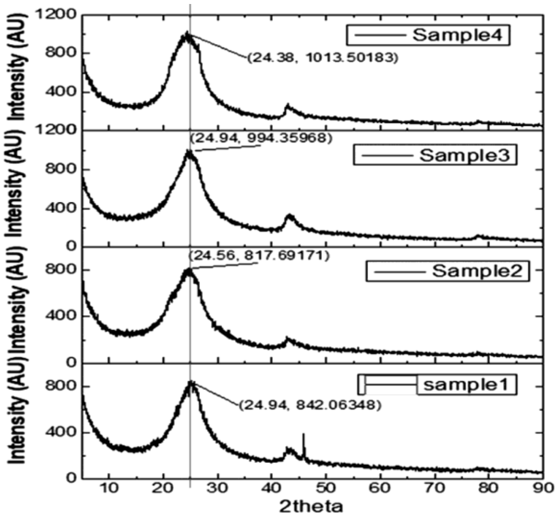


Fig. 5 — XRD comparison plots for various rGO samples

Figure 6 shows the SEM (Scanning Electron Microscope) micrographs of the all the four samples at a magnification of (a) at 2μm and (b) 500nm .The variation in the number of stacked layers can be easily observed in figure which indicates that increase in sonication time supports forming fine stacked layers due to defects created by the easily reduced oxygen molecule.

In sample 3 (Fig. 6(a)), some random beautiful flower like structure can be observed, which actually are the unsettled defects created by reduced oxygen. Fig. 7 shows the EDS plots for sample 3 and 4 which shows the energies of present molecules. Reason for choosing these two samples is the presence of some flower like unknown structure and of the best sample result obtained during SEM testing.

EDS results shows the percentage of effectively reduced oxygen in the sample 4 (4 hours sonication time) ,observed oxygen percentage in sample 3 is 22.09% whereas in sample 4 it reduces to 12.77%. The results shows the increase in sonication time causes the effective reduction of oxygen in GO. It

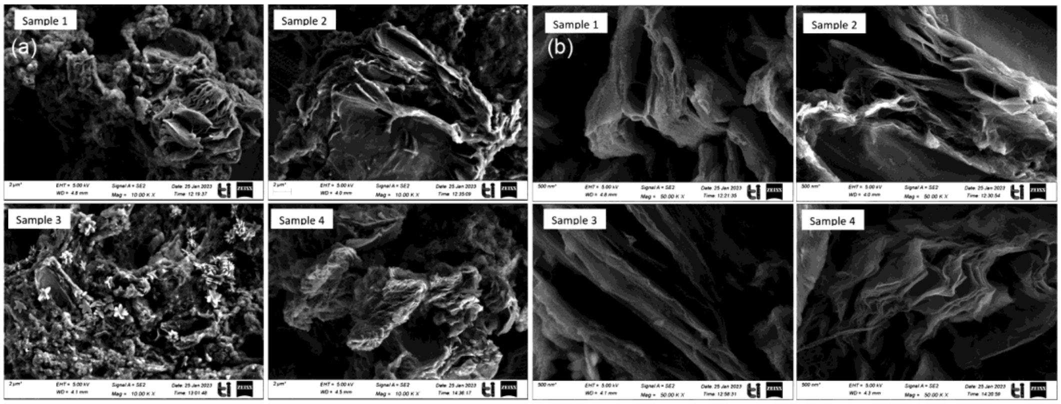
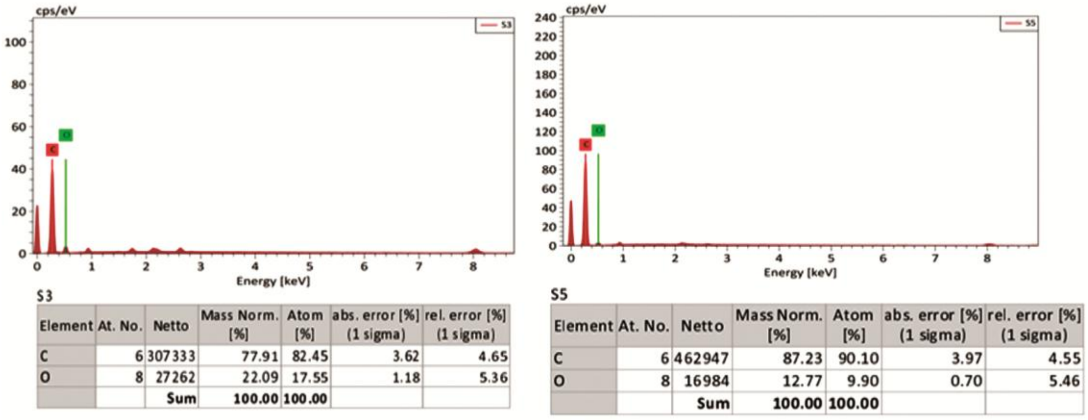


Fig. 6 — (a): SEM micrographs of samples at 2 μm and (b) 500nm



S3

Element	At. No.	Netto	Mass Norm. [%]	Atom [%]	abs. error [%] (1 sigma)	rel. error [%] (1 sigma)
C	6	307333	77.91	82.45	3.62	4.65
O	8	27262	22.09	17.55	1.18	5.36
		Sum	100.00	100.00		

S5

Element	At. No.	Netto	Mass Norm. [%]	Atom [%]	abs. error [%] (1 sigma)	rel. error [%] (1 sigma)
C	6	462947	87.23	90.10	3.97	4.55
O	8	16984	12.77	9.90	0.70	5.46
		Sum	100.00	100.00		

Fig. 7 — Energy – dispersive X-ray spectroscopy (EDS) of sample 3 and sample 4

also been observed that the unknown flower like structure in sample 3 is the exfoliated oxygen molecule.

4 Tunable MW attenuator

A microwave attenuator is a device used to reduce the power of microwave signals without distorting the waveform. rGO's conductive and dielectric properties allow it to interact with electromagnetic waves. When rGO ink is integrated into an attenuator, it can absorb and dissipate microwave energy, providing tuneable attenuation across a frequency range[24-25].

The rGOink (synthesized and characterized as discussed above) is experimental verified in tunable MW attenuator whose operating range is 1-8GHz. The dielectric constant (permittivity) and conductivity of materials like rGO are frequency-dependent. As the frequency increases, these properties change, affecting how the material absorbs and reflects electromagnetic waves. The microwave energy interacts differently with the material at different frequencies, leading to varying degrees of signal attenuation.

A prototype of tunable attenuator has been fabricated to analyze the electromagnetic performance of rGO ink. The rGO based microstrip attenuator consists of gap of length L in between two microstrip lines, where the few layered graphene RGO is stacked[15-17]. The circuit was designed on a FR4 dielectric substrate with thickness h=254μm, ϵ=4.4, tanδ=0.03, thickness t=17 μm.The stacked rGO filled area was of length 500 μm. The schematic diagram of experimental setup is shown in Fig. 8. It consists of a microstrip line with a gap in which few layered rGO was stacked. The circuit is attached with two bias tees for proper biasing.

DC and RF measurements was performed with the help of setup shown in Fig. 9, which includes an Anritsu test fixture UTF 3680–20 and two bias tees SigaTek SB14D2.Various DC voltages were applied to the bias tees and insertion loss has been observed. For each value of dc voltage, insertion losses were measured using VNA.

The insertion loss for various DC voltages has been measured using VNA at minimum range of -38.9dB at 1 GHz also a maximum range of 31.94 dB at 7 GHz is shown in Fig. 10. This shows r-GO ink can be used as microwave tunable attenuator.

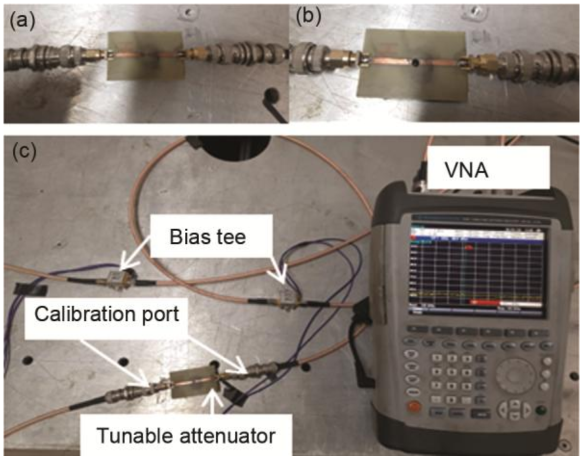


Fig. 9 — Attenuator circuit (a) without rGO ink (b) with stacked rGO ink (c) Experimental setup rGO-based microstrip attenuator

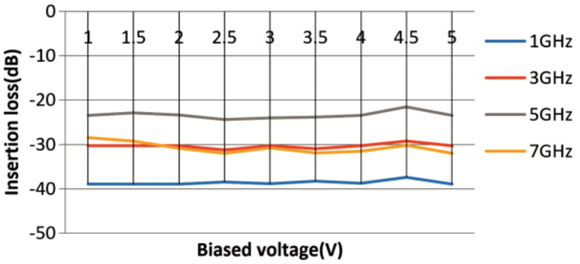


Fig. 10 — Measured insertion loss for various biased voltage

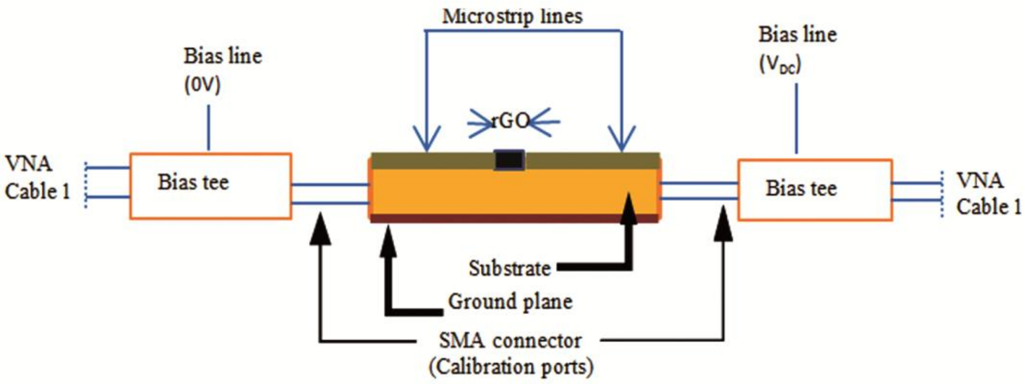


Fig. 8 — Schematic diagram of the measurement setup of the rGO-based microstrip attenuator

5 Conclusion

This article presents an efficient method for synthesizing rGO ink, along with an analysis of its morphological behavior based on sonication time, and the experimental verification of rGO as a tunable attenuator operating in the 1-8 GHz frequency range. The synthesis of reduced graphene oxide ink was successfully carried out in three main phases. Phase I involved the synthesis of graphene oxide (GO) by reacting graphite powder with concentrated acid in the presence of a strong oxidizing agent. Phase II focused on reducing the graphene oxide by removing oxygen, and Phase III involved dispersing the rGO powder in a solvent.

A comparative study of the morphological and functional properties of samples with varying sonication times (Samples 1, 2, 3, and 4) was conducted using X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), and Energy Dispersive Spectroscopy (EDS). The results indicate that increasing sonication time improves the quality of multilayered graphene, resulting in finer stacked layers, which in turn enhances the electrical properties of the material.

Additionally, insertion loss measurements at different biased voltages demonstrated that rGO ink can be effectively used as a tunable microwave attenuator.

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