STM JOURNALS

Journal of
Polymer & Composites

ISSN: 2321-2810
Volume 12, Issue 5, 2024
DOI (Journal): 10.37591/JoPC

http://engineeringjournals.stmjournals.in/index.php/JoPC/index

Research JoPC

Electro-chemical Treatment of Graphite Oxide: Preparation and Characterization of High Quality Graphene Material

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Abstract

With the enlargement of flexibility and suitability in material industries, high-performance thermal management materials are demanded with high thermal conductivity and electrical insulation are growing. In recent years, graphene has received a numerous focus from science community due to its high thermal conductivity and electrical insulation. Fabrication of light weight graphene and its derivatives as graphene Oxide (GO) and reduced graphene oxide (rGO) via facile processing method is yet a challenge for industries. The preparation of graphene through the wet exfoliation process of graphite leads to a several outstanding features of graphene. By thermal chemical reduction method, is simple and ecofriendly process used for the intercalate of graphene from graphite in a mass amount in the presence of aqueous solution H_2SO_4 and H_2O_2 through electrolysis process at high voltage (60 Volt) with electrolyte heating approach (60-70°C). This study investigates the structure and composition of graphene produced at different temperature as 650°C, 750°C and 850°C by thermal reduction of graphene oxide (GO) for short range of time (5 minutes). Thermal reduced graphene oxide (TrGO) diffraction peak, inter planar distance, crystallite size and structural disorder are all the significantly impacted by magnetic field and cyclic thermal loading, according to the FESEM and Raman spectra. TEM, UV analysis and TGA analyses demonstrated the significant influence of magnetic field and cyclic heating loading on the surface topography and micro structural characteristics. The thermal stability and functional groups contain oxygen seem to be unaffected by the magnetic field and cyclic thermal loading.

Keywords: Thermally reduced graphene oxide, Electro chemical exfoliation, electrolyte, thermal reduction, green synthesis

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Received Date: June 07, 2024
Accepted Date: June 24, 2024
Published Date: July 05, 2024

Citation: Vishwa Deepak Dwivedi, Pradeep Kumar Singh, Pankaj Kumar Singh. Electro-chemical Treatment of Graphite Oxide: Preparation and Characterization of High Quality Graphene Material. Journal of Polymer & Composites. 2024; 12(5): 25–36p.

INTRODUCTION

For developing wonder materials [1] i.e. light in weight, more flexible, stronger and easy in cost through smart way with better-quality properties, Nature always insists us. In a straight forward, a systematic graphene is a two dimensional crystal structure, compiled of single layer of carbon atoms, set up in a honeycomb arrangement with hexagon members hooped structure. An allotrope of carbon i.e. graphene containing sp^2 form covalent σ bonded with the adjoining carbon atoms separated by a distance of 1.42 Å from each other [2]. Graphite is formed due to the combined form of graphene layers on top of each other. Due to its low budget, easy access and widespread ability, for the volatile growth of interest in the scientific world, Graphite is of great interest convert to graphene.

The thinnest material initiate in nature, is Graphene and having 2D structure with only 0.3354 nm of a single layer thickness. The carbon's simplest form is Graphene and Graphene is the thinnest material than other carbon nanomaterials produced in nature. For the advancement of both on hand technologies and progress of new technologies, Graphene "wonder material" [3] that can be used. graphene oxide (GO), Reduced graphene oxide (rGO) a graphene sheets and layered graphenes are the graphene family members. Through the exfoliation of graphite oxide, a single layer of graphene oxide (GO) is achieved by the acid-base treatment of GO following Sonication process [4]. On the surface of GO, several functional groups are present as carboxyl group, hydroxyl group, epoxies group, oxygen group and phenol group. The existence of carbon atoms bound to oxygen atoms, is the main difference between graphene and graphene oxide. Graphene Oxide is the product of the hydrophilic derivative of graphene. Both the two types of domains as sp^2 hybridized (i.e. aromatic) and sp3 hybridized (i.e. aliphatic) of GO can easily assist the surface interactions [5].

Reduced graphene oxide (rGO) is the derivative of graphene family. Reduced graphene oxide (rGO) is a product of graphene oxide(GO) where in removing its oxygen functional groups, rGO is obtained from the thermal or chemical reduction process of graphene oxide (GO). The intermediate structure between ideal graphene sheet and highly oxidized graphene sheet is obtained as reduced graphene oxide (rGO). To renovate the. thermal strength and electrical conductivity of graphene, the removal of epoxy, carboxyl and hydroxyl [6] is too much beneficial Due to the unique chemical and physical properties [7] such as electrochemical properties, electrical properties, thermal properties, mechanical properties, huge specific surface area and high transparency, graphene has been attracted strong scientific and technological interest since its discovery in 2004 [8]. Graphene has also distinctive antibacterial properties. All the entire remarkable properties of the material, combined collectively can take a innovative revolution in the world. Graphene has exposed enormous prospective applications in various fields, consists of sustainable energy conversion and storage (batteries, solar cells, fuel cells, ultra capacitors), bioscience/biotechnologies, nanocomposite materials, electronic devices, tissue engineering. graphene based materials as graphene foam, graphene electrode, graphene fiber, graphene aero gel, and graphene film, have focused many scientists' interests due to its significant properties in acoustics, optics, electrics, mechanics, and thermology. Now a days, graphene membranes are mainly used to filter the water for safe drinking purpose but not only the filtration of various gases. For the production of graphene batteries for improved charging capacity than traditional lithium battery, graphene energy can be utilized [9]. A latest research for human use is graphene ink can get regarding a resolution in the area of printed electronics, graphene shoes and clothing, rubber bands, disease detectors, graphene glasses and paints respectively. The graphene based materials with unique properties has been obtained by the microstructures of graphene sheets such as folds, kinks and ripples. Hence, the micro-structural exploitation of the material has play a vital role in the improvement of their performance and application fields [10]. On the way to the applicability of graphene, in a broad variety of applications and promising novel approaches, researchers are searching a low cost, simple path processing, more competent and better yielding method for processing of graphene., that can be scaled up extremely compared to current approaches and be reasonably suitable for commercial and industrialized applications. A number of unique properties of graphene make it a novel and wonder material for limitless applications.

Carbon-based materials [11] have been developed in arranged form and applied since ending the 21st century. Initially, the lamellar structure of thermally reduced graphite oxide had been discovered by Collins Brodie in 1859. Through the powder diffraction technique, graphene structure was analyzed in 1916 and in 1924 through the Single crystal X-ray diffraction technique, the internal structure graphene had been examined successfully. A thin film of graphite were synthesized by mechanical exfoliation process in 1990s. Finally, in 2004, the first information by two scientists Prof. Geim and Prof. Novoselov, at the Manchester University had been found about single layer (monolayer) of graphene through micromechanical peeling approach [1] (tear tape or scotch tape method) by the segregation of graphene from graphite. In 2007, Meyer et al represented an idea about the single layer graphene with the thickness of one atom only could freely hang up on the micro fabricated scaffold in air or vacuum. Actually at room temperature single layered graphene could exist in a stable phase. By the cleavage approach, the first remark had been observed in the field of graphene through anomalous quantun Hall effect [12]. This smart way of development of graphene layers from graphite can only be used for fundamental science and is not appropriate for the huge volume production of graphene. Through long year of research, graphene and its derivatives have become widely utilized as for mass volume production within most efficient cost and simple formation now a days. By the use of oxidizing agents, a graphite can be exfoliated to obtain graphene oxide (GO) is a most common technique. With a atomic ratio of carbon/oxygen 2.0-3.0; Graphene Oxide is an oxidatively modified graphene derivative. To prepare graphene various approaches has been identified as electrochemical technique, chemical exfoliation, liquid phase exfoliation, chemical vapour deposition, Electro-magneto-chemical exfoliation, thermal exfoliation, Supercritical fluid exfoliation techniques. But the major challenge is how to prepare high quality graphene in a mass volume for various applications. Chemical reduction of graphene oxide (GO) is the one of the most efficient approach for the synthesis of graphene on a mass scale. By the chemical reduction heat treatment technique, The group contains oxygen atoms are removed on the carbon atomic layer of graphene Oxide (GO) and to produce reduced graphene Oxide (rGO), the best suitable method for mass volume formation of graphene for commercial and industrial applications [13].

To improve the definite properties of graphene oxide (GO) with high rate of interest, the reduction process of graphene oxide has been performed, make it easy for the various application areas with great performance rate. Through the reduction mechanism [14], a GO modification can take a new interesting path for the removal of a group contains oxygen atoms in a organize way itself. By the various reduction conditions, the composition, structure and morphology of the reduced graphene oxide (rGO), achieved through various reduction processes, is properly arranged in a controlled manner.

In this work, the influence of reduction temperature on the structural properties of graphene is studied by Raman Spectroscopy, UV-visible spectroscopy and Scanning electron microscopy (SEM) analysis with dispersive x-ray spectroscopy (EDS) [15] signifies the micro structural and surface behaviour impact of thermally reduced graphene oxide. It also identifies the impact of oxygen generated molecules and thermal behaviour of GO under thermal heating. The impact of molecules produced by oxygen and the thermal behaviour of graphene oxide (GO) under thermal heating are also identified. Images from field emission scanning electron microscopy (FESEM) with EDS revealed the morphology of the single and multi layer graphene. FESEM images of GO and rGO showed wrinkly, flaky structures and the Energy dispersive x-ray spectroscopy (EDS) provided information about their composition. Raman spectroscopy [16] that clearly varies with an increase in layer membership. The shape, width and position of the D peak second order alter as the number of layers increases, signifying a shift in the electron bands causes by a double resonant Raman process. The G peak has slightly decreased in magnitude. This enables the clear and non-destructive identification of graphene layers, a competence required in the emerging field of research. The GO and rGO were successfully synthesised, as demonstrated by XRD spectroscopy, which displayed peaks at 7.95 nm and 29.96 nm respectively. The absorption peak of GO (230 nm) and rGO (280 nm) respectively were discussed in the UV- visible spectroscopy at various locations. The researcher's objectives were therefore met, and this environmentally friendly [17] method can be used as a stand-in to lessen dependency on chemicals.

EXPERIMENTAL SETUP

Sample Preparation

A pencil lead made of hetero structured graphite is also utilized. For the task, different grade of pencil (H,B and 6B) are taken. Pencil lead made of graphite and clay is often an intercalated compound in conductive graphite that contains a percentage of metal oxides with silica. Pencil graphite core with clay particles is an intercalated composite in conductive graphite with a small amount of metal oxides and a large amount of silica [18]. The range of 9H to 9B in the ratio of clay to graphite is typically used to identify pencil graphite where B and H stand for darkness (amount of clay) and hardness (Quantity of graphite) respectively. Higher grade pencils appear to have a higher percentage of graphite in them. The exfoliation process can be accelerated by applying a positive or negative charge to the material using a graphite electrode, which encourages the intercalation of ions with opposite charges. For the electrolysis process, pencil lead is used as the anode and cathode electrodes since it has the same length but a different diameter. H_2SO_4 is used as an electrolyte in the electrolysis process from pencil lead. The effects of pencil lead are observed through the exfoliation of graphite.

Exfoliation Process through Electrochemical Method

Initially, during the electrochemical exfoliation process, graphite pencil lead was used as anode and a cathode rod (derived from an exhausted dry cell battery). The electrode (graphite pencil lead) and cathode were both put in an aqueous solution containing 8% H_2O_2 and 1 molarity H_2SO_4 correspondingly. In order to obtain a larger yield of graphene oxide (GO). Between the two electrodes, a 30 millimeter space is kept during the exfoliation procedure. The exfoliation process requires a supply of 60 Volt direct current (DC) power. The electrolyte's temperature regular increased as a result of electrode polarization and resistance formed in the process. The internal resistance will decrease as the temperature rises, weakening the electrolyte's ionic connection in the process. In Figure.1, the experimental setup was displayed. For six hours, the product was sonicated using a piranha solution (1 molarity H_2SO_4 and 4 % H_2O_2). Subsequently, add a small amount of H_2SO_4 and H_2O_2, sonicated for three hours, then boil the mixture and filter again using a membrane. Following the exfoliation process, the floating product is removed from the electrolyte's top, filtered via a membrane, and a vacuum-filtered for 12 hours at approximately 800°C to dry it.

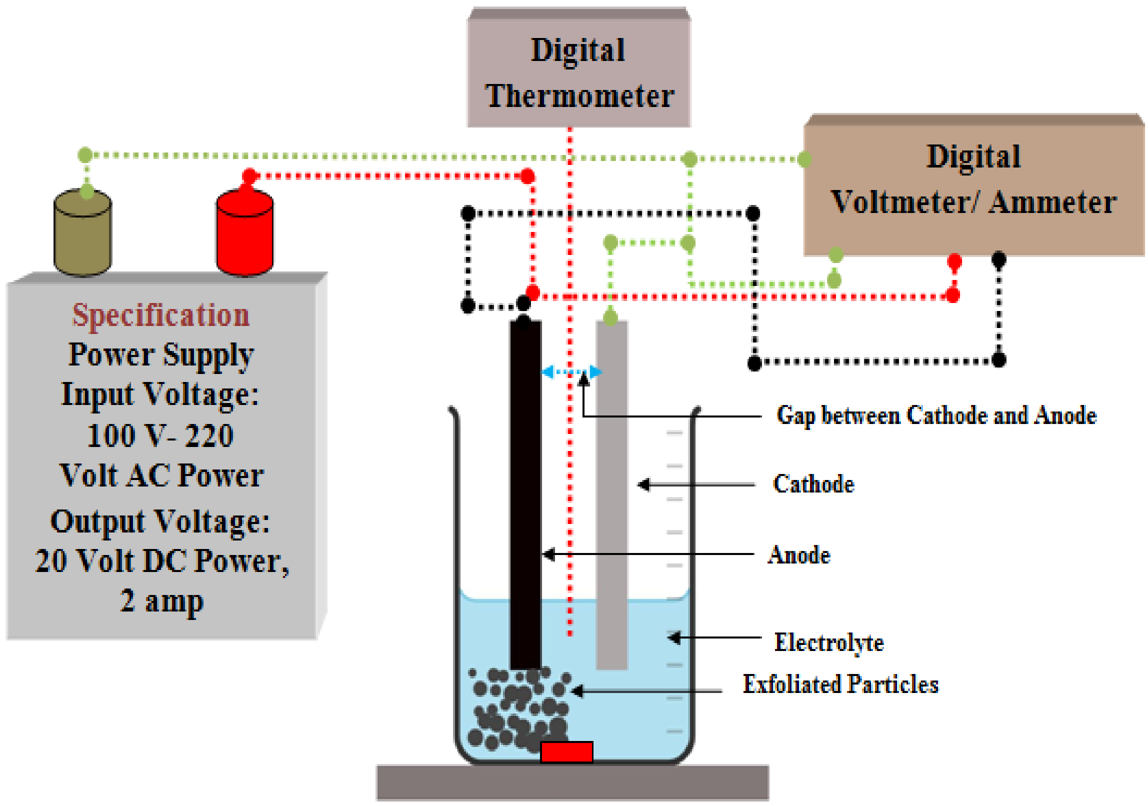


Figure 1. Systematic diagram of exfoliation process of graphite.

Following the completion of the Sonication procedure, graphene oxide (GO) was collected during the process in either a colloidal or solid condition. Previously, the high yield thermally reduced graphene oxide (TrGO) was produced by cyclic temperature action of synthesized graphene oxide (GO) [20].

Figure 2. (a) Synthesis of graphene oxide through Electrochemical method, (b) Sonication process of graphene oxide using Bath sonicator, (c) Drying of graphene oxide after exfoliation process.

Before the graphene oxide (GO) was free of gaps, it was placed in a beaker within a muffle furnace (for furnace cooling) and heated to 650^{o}C, 750^{o}C and 850^{o}C for 10, 15 and 20 minutes respectively. Graphene Oxide (GO) is transformed into thermally reduced graphene oxide (TrGO) during the drying process, and is designated as TrGO-650^{o}C, TrGO-750^{o}C and TrGO-850^{o}C in that order.

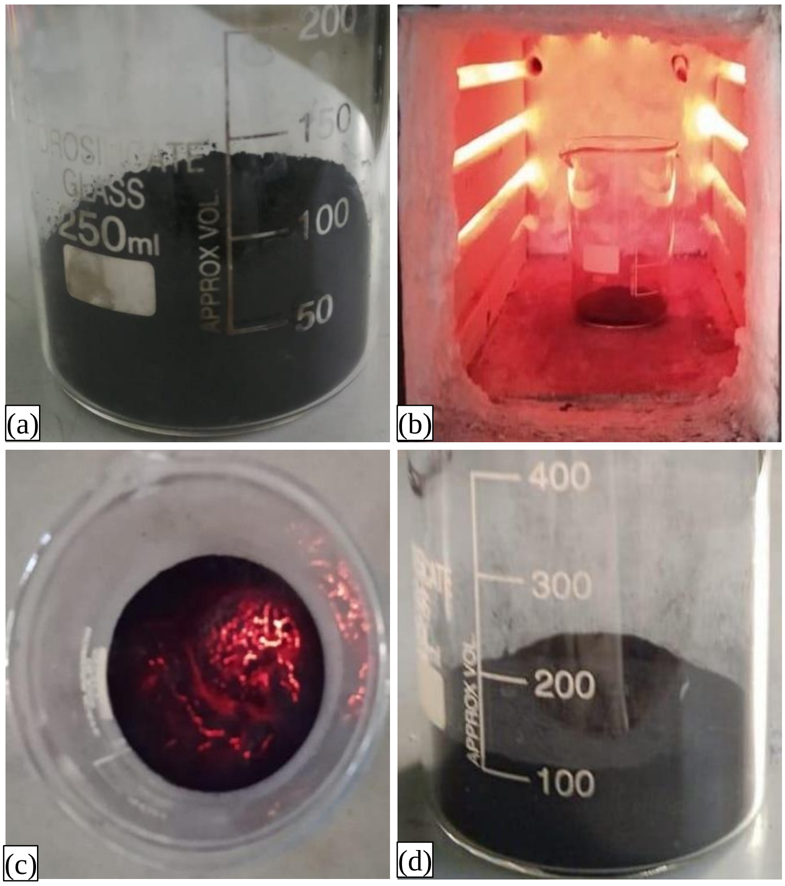


Figure 3. (a) Image of exfoliated graphene oxide sample, (b) Image of thermal reduction of graphene oxide using muffle furnace, (c) Top view of sample during thermal reduction process, (d) Image of thermally reduced graphene oxide (TrGO).

By using the Raman spectral analysis to examine phase purity, crystallinity, and the diffraction peak of thermally reduced graphene oxide (TrGO) during cyclic heating, the morphology of the material was determined. The structural behavior of thermally reduced graphene oxide (TrGO) preserves at various temperature scale is observed by the FESEM investigation. Thermal stability and the quantity of oxygen functional groups in thermally reduced graphene oxide (TrGO) at various temperature scales are determined by TGA and FTIR analysis [19].

RESULT AND DISCUSSION

Field Emission Scanning Electron Microscopy

Thermally reduced graphene oxide (TrGO) has been shown to have a structural and surface characterization [20] at different temperature scales such as TrGO-650ºC, TrGO-750ºC and TrGO-850ºC, respectively. The exfoliated graphene oxide is studied in a magnetic field to reveal an expanded, thinner, porous microstructure and a space between layers where as carbon layers in graphene oxide are layered and related when it is thermally reduced. When the sample is repeatedly subjected to temperature loading, it exhibits rapid volumetric expansion, porous microstructure, separation of the carbon layers, fractured carbon layers, rise in surface area and prolonged gap between carbon layers.

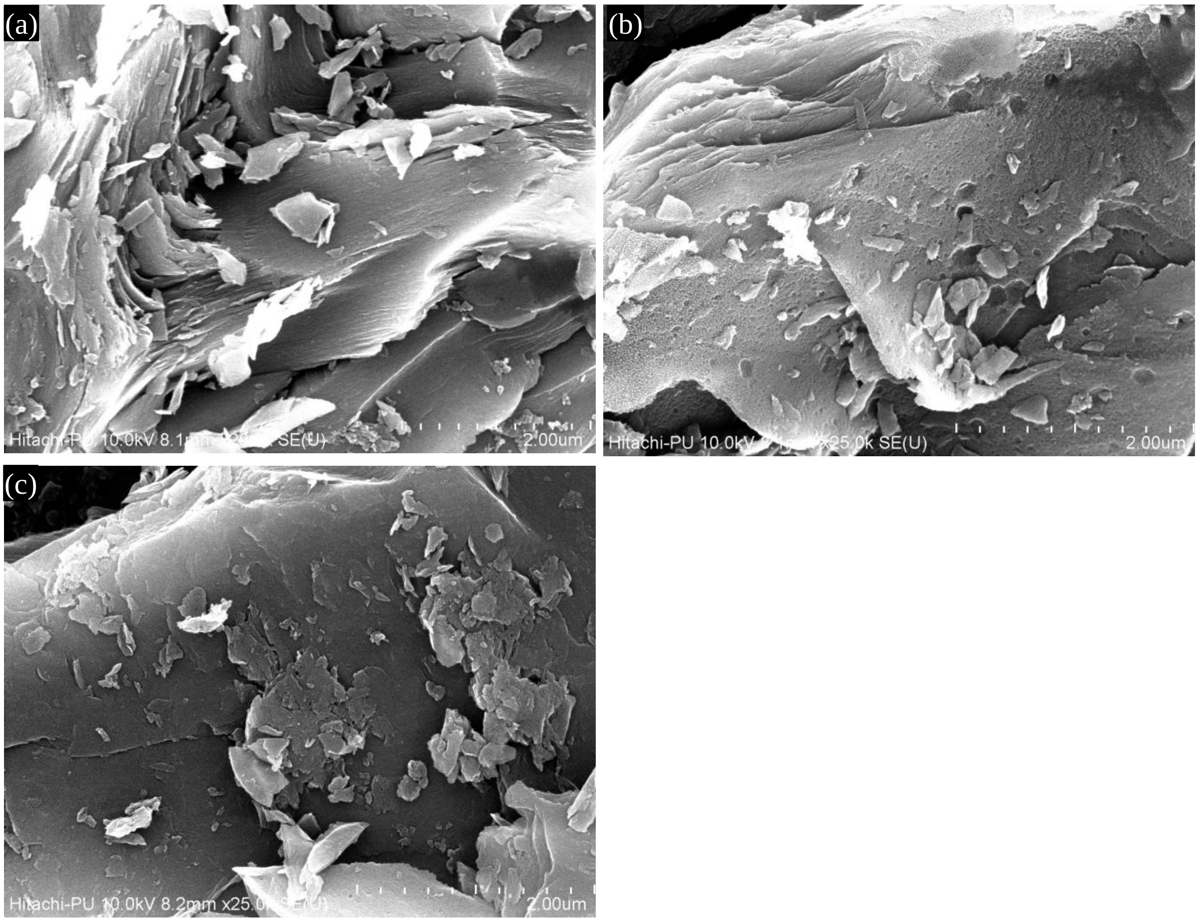


Figure 4. FESEM with EDS images of (a) TrGO-650ºC, (b) TrGO-750ºC, (c) TrGO-850ºC at 2 μm.

Raman Spectroscopy

A better method for allocating the equivalent changes in graphene based on peak position and intensity in Raman analysis, which is a spectroscopic, vibrational and optical method for characterizing the structural disorder, chemical composition and crystal structure [21] of thermally reduced graphene oxide (TrGO). In Raman analysis, the G band indicates the graphitic nature of the crystal while the D band indicates the extent of flaws of thermally reduced graphene oxide (TrGO).

The higher the D peak intensity, the more defects, there are in the sample. The imperfections have caused the sheet's edges to be uneven. A high G band indicates that the sample is crystalline. Thermally reduced graphene oxide (TrGO) exhibits three distinct peaks denoted as D, G and 2D peaks at varying temperature scales. Via the D bands the crystal disorder or anarchy that includes the oxygen function, edges and defects connected to the graphene surface addressed. G band discusses the first order spreading of the E 2 G mode. The 2D peak has recorded the graphene quality. The molecular bond length is related to the maximum and minimum wave numbers of the peaks in Raman spectra [22].

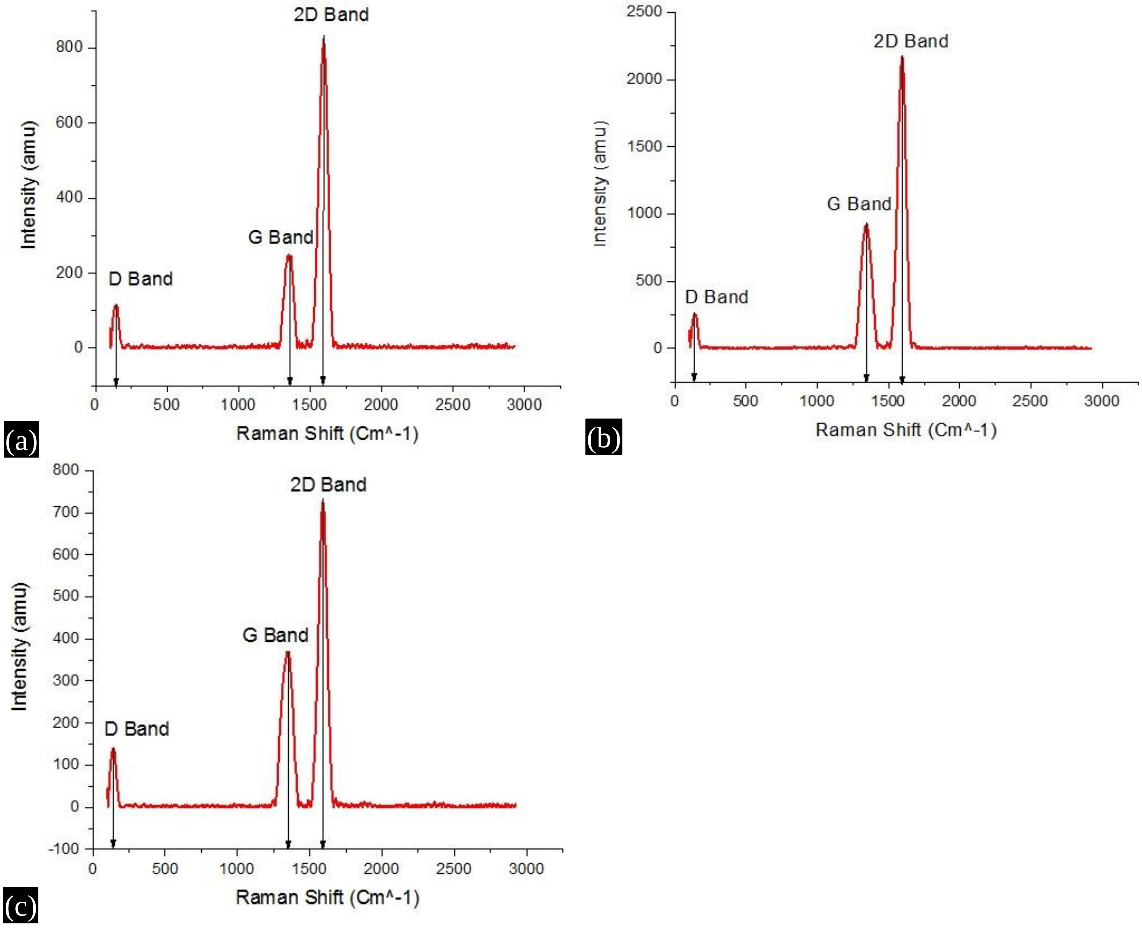


Figure 5. Raman spectra identified for (a) TrGO-650ºC, (b) TrGO-750ºC, (c) TrGO-850ºC.

Transmission Electron Microscopy

TEM is a useful microscopy technique in which an ultra thin object is examined and interacted with using an electron beam [23]. Graphene's thickness and low carbon atomic number make it totally transparent to an electron beam. The small beam contact with the hexagonal carbon monolayer produces a well defined signal that can be readily replaced based on the ensuling image and diffraction patterns [24]. Consequently, the TEM technique is becoming more and more well liked as a helpful tool for comprehending the structure and morphology of graphene. In Figure 6 displays the TEM examination of TrGO at different temperature scales as 650ºC, 750ºC and 850ºC respectively. The findings demonstrates that TrGO sheets are made up of several thin layers, layered one on top of the other with multiple wrinkled carbon layers and folding.

UV-Visible Spectroscopy

UV- visible spectroscopy s a widely used analytical technique to investigate the interactions between ultraviolet (UV) and visible light and materials. It provides significant information about the properties and electronic structure of molecules and materials [25]. This approach measures the absorption, transmission and reflection of light, in UV-visible portion of the electromagnetic spectrum. The visible (400-800 nm) and ultra violet (200-400 nm) wavelengths, which fall between 200 and 800 nm, are included in UV-visible spectrum. Within this spectrum, different molecules and compounds display distinctive absorption patterns because of their different electronic configurations and energy levels [26].

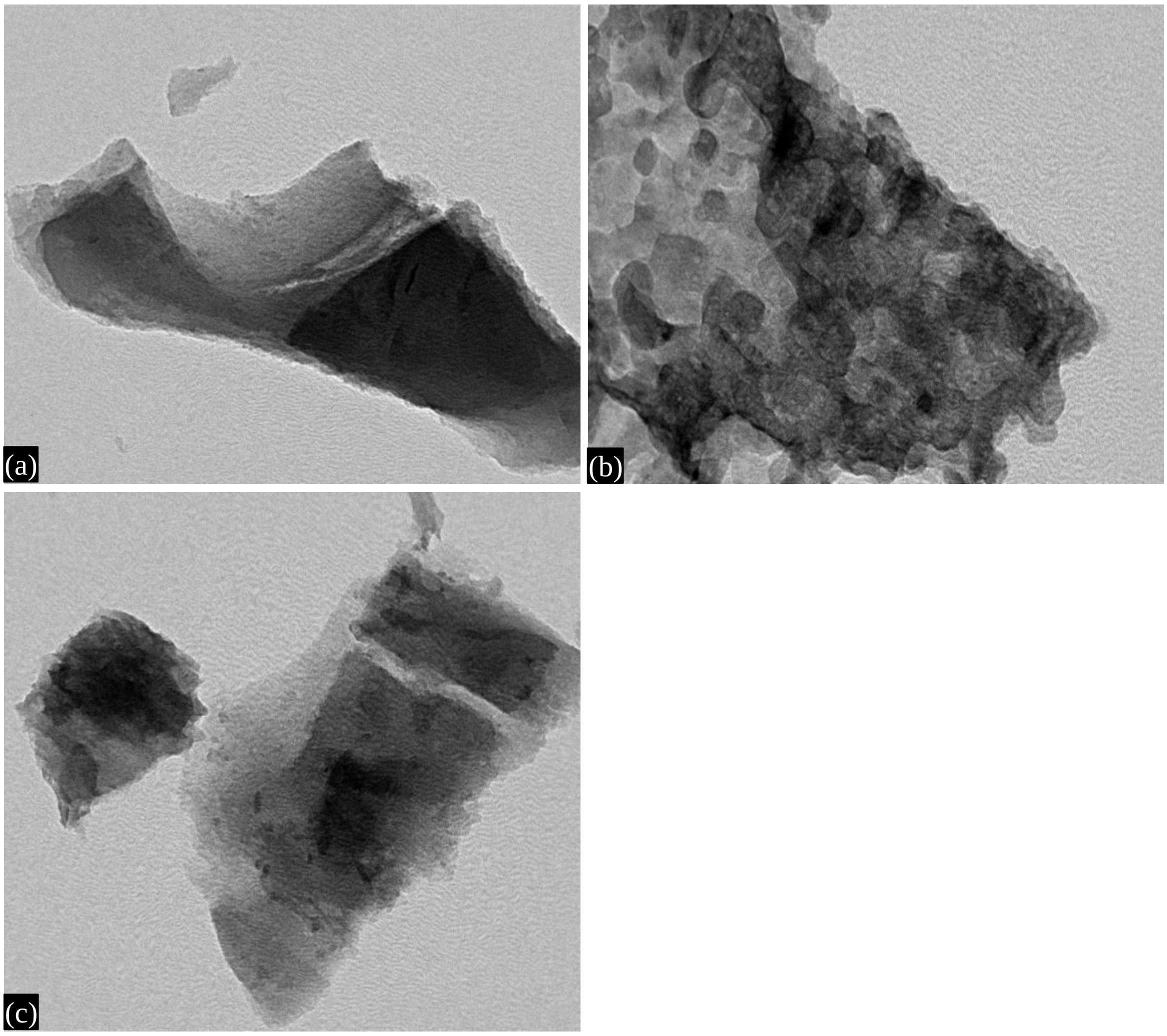


Figure 6. TEM image identified for (a) TrGO-650ºC, (b) TrGO-750ºC, (c) TrGO-850ºC.

It is often used to assess the quantity or quality of substances, such as nucleic acids, proteins and others compounds that absorb UV light. UV-visible spectroscopy measures how much of a material absorbs or transmits visible and UV light. It make it possible to characterize a variety of materials, such as organic compounds, dyes and pigments using their UV and visible absorption spectra.

Thermal Stability Analysis

Thermo gravimetric analysis (TGA) is an analytical technique used in numerous disciplines, including as material science, chemistry and engineering sections [27]. By evaluating a sample at a controlled temperature, thermal gravimetric analysis (TGA) offers important insights into the thermal stability [28] and break down behaviour of compounds. Figure 8 (a, b, c) displays the thermal behaviour of reduced graphene oxide at various temperature scale 650ºC, 750ºC and 850ºC evaluating the performance characteristics.

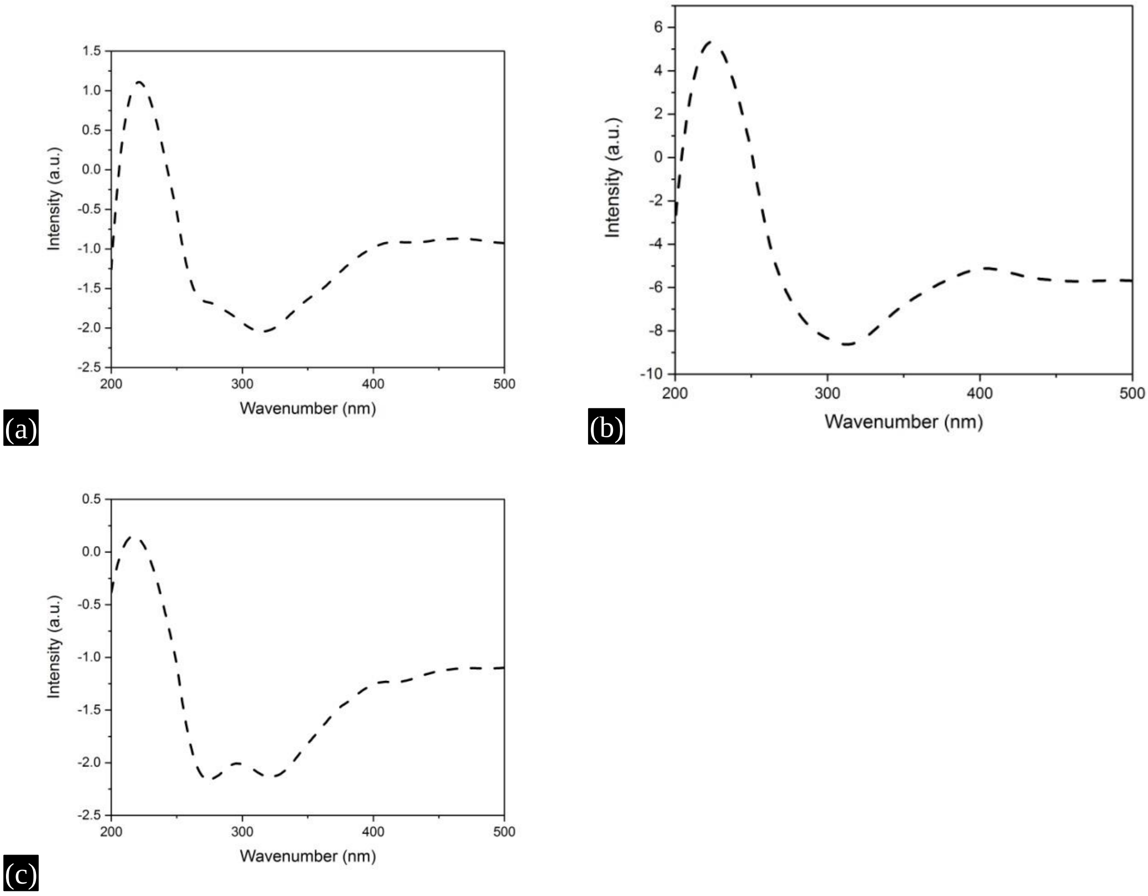
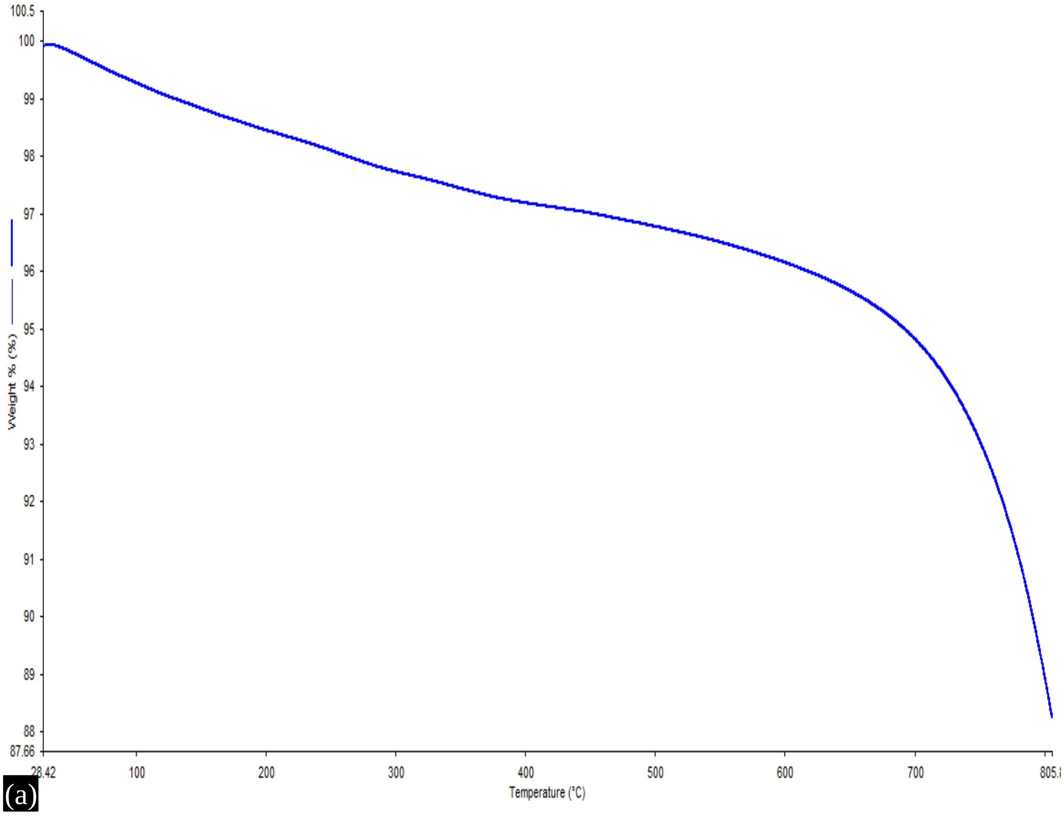


Figure 7. UV-visible spectra of (a) TrGO-650ºC, (b) TrGO-750ºC, (c) TrGO-850ºC.



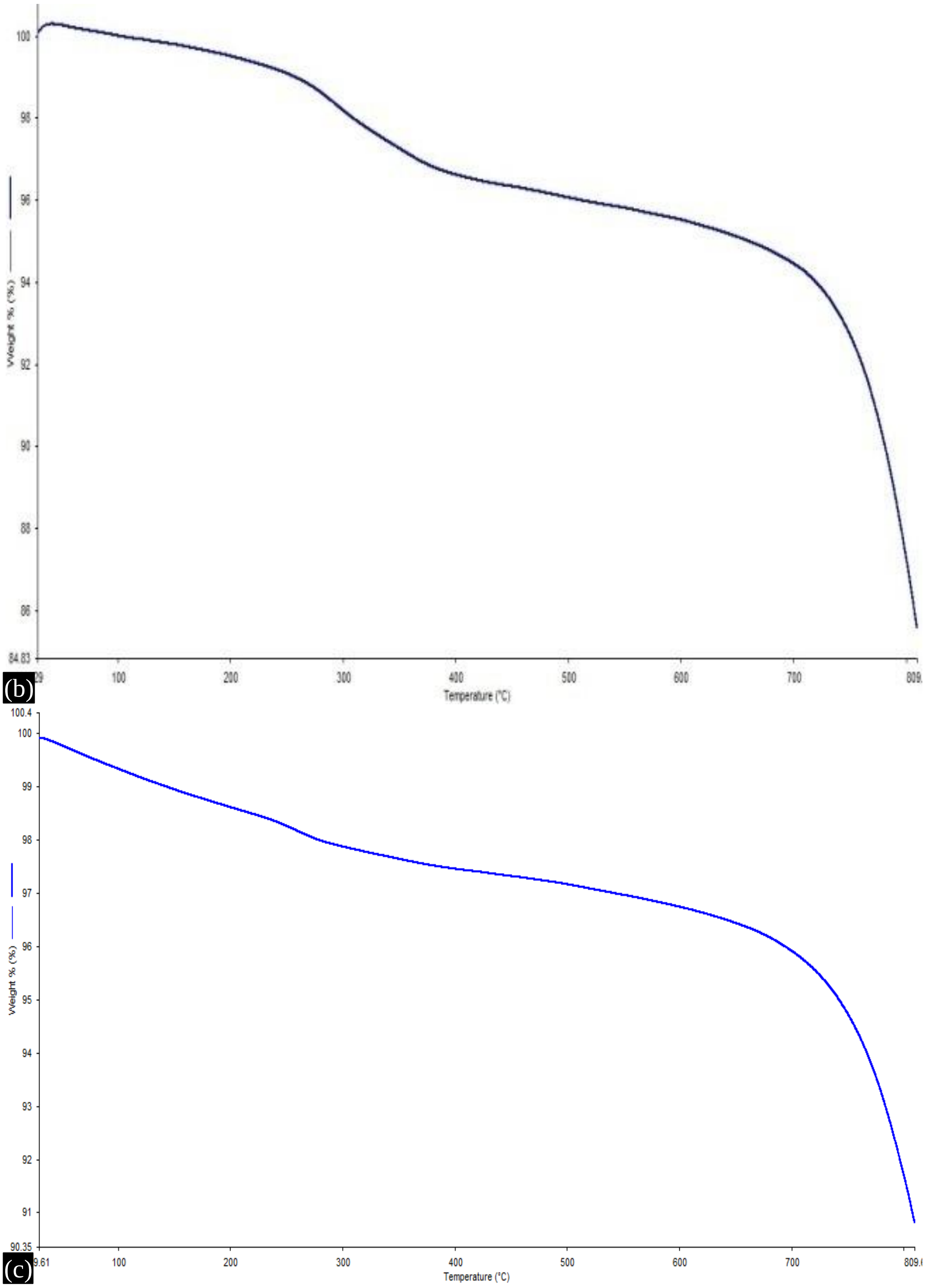


Figure 8. Thermal stability behaviour of (a) TrGO-650ºC, (b) TrGO-750ºC, (c) TrGO-850ºC.

CONCLUSION

The properties and behaviour of thermally reduced graphene oxide (TrGO) at various temperature conditions can be ascertained by a number of characterization techniques, including Field emission scanning electron microscopy (FESEM) with EDS, Raman analysis, UV-visible spectroscopy and TEM analysis. A common method for examining the structural and chemical behaviour of graphene and its derivatives is Raman surface spectra investigation. Through Raman spectroscopy, graphene exhibits two Raman peaks; the G band which is approximately 1370 cm^{-1} and the 2D band which is around 1580

cm^{-1} respectively. The intensity ratio of the G band to the D band, which is associated with faults, can be used to determine the magnitude of reduction. The surface and structural properties of thermally reduced graphene oxide (TrGO) at various temperature scale such as TrGO-650ºC, TrGO-750ºC and TrGO-850ºC have been investigated using FESEM analysis. When exfoliated graphene oxide is examined in a magnetic field, it reveals a thinner, more expansive microstructure with gaps between the layers, where as thermally reduced graphene oxide (TrGO) exhibits stacked and connected carbon layers. Thermal gravimetric analysis (TGA)offers crucial insights in the thermal stability and break down behavior of TrGO. A method is used for assessing the chemical structure of a material, using UV-visible spectroscopy. The UV-visible analysis is used to identify the quantity of functional groups of oxygen containing in TrGO-650ºC, TrGO-750ºC and TrGO-850ºC, respectively. TEM is a useful microscopy technique in which an ultra thin object is examined and interacted with using an electron beam. Graphene's thickness and low carbon atomic number make it totally transparent to an electron beam.

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