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<http://www.AANBT.dormaj.com>[https://doi.org/10.47277/AANBT/2\(4\)46](https://doi.org/10.47277/AANBT/2(4)46)**Green Synthesis and High-efficiency Method for Reduced Graphene Oxide by Urtica Dioica Extracts****Negar Javanmardi¹, Nooredin Goudarzian^{2*}, Mingzhu Xia¹ and Fengyun Wang¹**¹School of Chemistry and Chemical Engineering, Nanjing University of Science and Technology²Chemistry Department, Islamic Azad University, Shiraz Branch, Shiraz, Iran

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ABSTRACT

There are various methods to produce nanoparticles, but utilizing the plants for nanoparticle synthesis due to their environmentally friendly nature and low cost is very highly considered. Here, we report the green synthesis of reduced graphene oxide with high efficiency using extracts of *Urtica Dioica* for the first time. In this study, *Urtica Dioica* extracts were utilized as a reducing material for the synthesis of nano-graphene oxide. The reduced graphene oxide was analyzed for the determination of size and structural properties using XRD, FTIR, Raman spectra, and TEM.

KEYWORDSGreen synthesis, *Urtica Dioica*, Reduced graphene oxide, Graphene oxide**1. Introduction**

These days, the craving to create and utilize nanomaterial's with expanding enthusiasm for these materials is expanding. Therefore, there are strategies for the arrangement and manufacture of nanoscale materials, including electrical circular segment (plasma), (chemical compound decrease) (laser cut and microwave rays). The recuperated graphene oxide achieved from these techniques and utilization of materials has hazardous radiation. The reactions under specific conditions (temperature and pressure) are costly and tedious and have potential dangers for the earth, thus the requirement for a minimal effort, non-dangerous and nonlethal strategy. One of the strategies for the creation of production of reduced graphene oxide is green recovery, which is these days centered around. This technique needs graphene oxide has been reestablished with this strategy is significantly expanded [1-5]. The most widely recognized types of crystal are carbon particle, graphite, and diamond. Graphite is a three-dimensional carbon allotropic, which has

a layered structure, in which four carbon atoms are associated with three other carbon atoms through three covalent bonds and comprise a six-center system structure. Each one of these layers is known as a layer or graphene layer [6-7].

Each sheet is parallelized to another sheet and creates a customary system with the AB arrangement. Along these lines, the fourth electron capacity, through the development of a van der Waals bond, makes the sheets bond together. Because of the shortcoming of the connections between the graphene sheets (van der Waals), the above sheets can without much of a stretch slip over one another. So graphite is a useless strong. Not at all like diamonds (other carbon allotropes), graphite is a conductive electric current (Figure 1). The reason is that in the graphite of every carbon atom, three electrons from four electrons frame a layer of their ability for the development of covalent bonds, and an electron stays discontinuous. This electron can cause electrolytic conductivity of the graphite so that the electric equilibrium of the graphite crystal system crashes into the

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entry of an electron through the present bearer wire, and an electron is promptly expelled from the contrary side to keep up the electric charge balance. This is the most vital and most utilized graphite in the throwing and liquefying of metals. It's different uses incorporate the production of batteries, plant refueling, hardware, pencils, refractories, and oil [8].

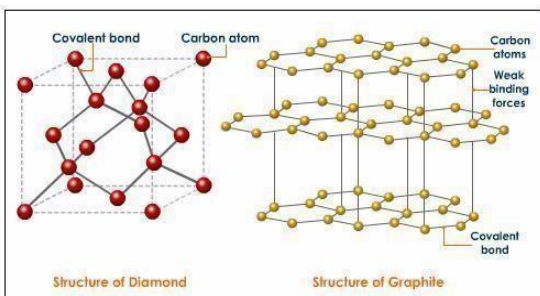


Figure 1: Graphite and carbon structure

Graphite oxide was first created by Hummer and Hoffman in 1860. It is a mixture of carbon, hydrogen, and oxygen molecules. Graphite oxide comprises a layered structure of graphene oxide sheets that is extremely hydrophilic. The most widely recognized technique for integrating graphite oxide with a synthetic strategy is to utilize at least one concentrated acid within the strong oxidizers. Out of the blue, Brody utilized this strategy in 1859 [9-12]. In the oxidation reaction, oxidizing an electron is supplanted by a chemical reaction. It is important that utilizing a mixture of graphite oxide and water for quite a while, the material can be changed into a mixture of suspended aquatics of graphene oxide sheets in a totally layered frame. It depends on carbon atom that will make an incredible upset in innovation and make the universe without bounds more brilliant, quicker, and more exact. Graphene oxide is produced using a carbon atom layer and makes a honeycomb-formed network. It is a thin layer of graphite, with a thickness of around two-tenths of a nanometer. At the point when the oxidizing operator is responded with graphite, the interstitial space between the layers is expanded and, therefore, the separation of these layers, every one of which is currently a graphene oxide layer, is encouraged. The restoration of graphene oxide implies the separation of functional groups and the loss of waterborne graphene oxide, which altogether encourages its washing [13-15]. A few strategies for the generation of graphene have been created so far that can be for the most part isolated into two classes: Top-Down and Bottom-Up. Today, graphene oxide has turned out to be a standout amongst the most questionable subjects in the field of physical science and materials science since with the numerous abilities it has in this article, it is foreseen that the improvement of new electronic instruments will make a gigantic advancement and as a substitute for Silicon in future gadgets. Researchers trust that graphene oxide is a standout amongst the toughest materials at any point known, because of its carbon-to-carbon bond strengths. Graphene oxide has one of kind properties, for example, nanometric size, high hardness, and mechanical quality, high electrical and warm conductivity, adaptability, and attraction. Along these lines,

numerous utilizations will be made, incorporating applications in nano electronic, sun oriented cells, vitality stockpiling gadgets, for example, batteries and supercapacitors [10-12]. Gravity oxide has altered therapeutic imaging, sensor building, and security apparatuses. . Because of their particular nuclear cosmetics in graphene and graphene oxide, these materials have high conductivity and are extremely reasonable for use in circuits and electronic gadgets. Graphene oxide is not diamondlike and has been broadly utilized for it. Because of its flexible properties and high weight course, it is foreseen that there will be a considerable measure of improvement in the business. Graphene oxide is generally utilized as an appropriate retentive in water cleaning and photograph synergist responses because of its numerous applications and its different composites [16]. Graphene is as of now the most consumable type of graphene, which, in spite of auxiliary contrasts, is utilized as a rule rather than graphene, and even now and again, for example, electromagnetic assimilation, graphene is best. Graphene is the most grounded substance at any point considered, a substitute for silicon, and its abnormal properties, for example, the most astounding electrical conductivity among known materials, have shaken the universe of science and the media. Graphene is an exceedingly carbon-bearing material and high-density atomic material. The strange mix of its properties, for example, high mechanical quality and hardness, high electrical and warm conductivity and high warm conductivity, superior optical and elite properties, are of specific worry to the scientists, and the way that scientific experts can scarcely discover a substitution for graphene. It has numerous utilizations in nano hardware, solar cells, and vitality stockpiling gadgets, for example, batteries and supercapacitors. Graphene is the hardest and most slender substance at any point created by individuals. This material, in spite of the fact that it has a thick structure, is because of its little thickness, which is equivalent to the thickness of a carbon atom [17-19].

1.1 Reduced graphene oxide production methods

Concoction vapor deposition (CVD) and coarse development: For around four decades, utilizing the CVD strategy, hydrocarbons have been set up on the surface of intermediate metals of thin films of graphite, be that as it may, a lot of carbon source adsorbed onto the surface, The outcome is that thick crystals of graphite are shaped. As of late, specialists have prevailing with regards to creating mono-and multi-layers of graphene by enhancing this procedure. A case of this is the separation of ethylene on nickel surfaces [20-21].

1.2 Production method of graphite oxide

From the nineteenth century, when graphite oxide was first created, the Brody, Hammer, and Stoudmeyer techniques have been utilized to deliver this material [12, 14, and 20]. Each of the three of these techniques incorporates the oxidation of graphite within the strong acids and solid oxidizers. The oxidation surface, in the

extent to the strategy utilized, can have distinctive reaction conditions and graphite. Numerous examinations have been done to illuminate the structure of graphite oxide, and a few models are talked about in the papers [10-12]. This technique for making graphene is better with vast scale creation. In this technique, the graphite oxidized and in a strong acidic condition, it isolates the layers of graphene oxide. This oxidation procedure produces a great deal of oxygenated, carboxyl, epoxy, and hydroxyl groups on the surface of graphene. The nearness of these polar factor groups, now and again, surface ionization, unequivocally influences graphene oxide, and consequently, it is effectively spread in water and polar organic solvents.

The drawback of graphene oxide is that the nearness of functional groups at the surface expels graphene from its interesting properties.

Graphene oxide is an electrically conductive structure whose layers are fundamentally bended by the nearness of carbon-carbon bonds. Consequently, they reestablish graphene oxide with reducers, for example, hydrazine to convey it closer to the first graphene structure. In spite of the fact that the recovery procedure restores a lot of graphene conductivity and leveling, yet the last item still has critical measures of carbon-oxygen bonds and is a long way from graphene's primary properties. This technique still has every one of the hardships and issues of the previously mentioned strategies and, as of now stated, critical measures of carbon-oxygen bonds stay, in organic strategies, utilizing mild reactors, notwithstanding decreasing the selectivity dangers, and higher recuperation rates will leave [12,22].

1.3 *Urtica Dioica* as a reducing agent

Urtica dioica (family *Urticaceae*), also known as stinger, nettle, nettle leaf, stinging nettle (although not all plants of this species sting), or common nettle, is a herbaceous perennial flowering plant that is native to western North Africa, much of temperate Asia, and Europe [23]. Nowadays, it is globally found anywhere, e.g. North America, as well as Iran, Shiraz (Mount Sivand, mountains neglecting the Maharlou Lake, between the backwoods, between Jahrom and Mansurabad, near Taram Darab).[24-26] This species consists of 6 subspecies, 5 of which have hollow stinging hairs named trichomes on the stems and leaves that act like hypodermic needles and inject histamine and other chemicals and cause a stinging sensation upon contact ("contact urticaria", a form of contact dermatitis).[27-28] It has long been used as textile raw material, tea, food, and traditional medicine [23-29].

The fresh leaves of *Urtica dioica* contain about 82.4% water, 17.6% dry matter, 7.1% carbohydrates 5.5% protein, and 3.3-0.7% fat.[30] Mature leaves contain about 40% α -linolenic acid, a valuable omega-3 acid.[30-32] The leaves of this plant contain carotene, xanthophyll, lutein, and carotenoids. This plant also contains Vit E (tocopherols), Vit K1 (pantothenic acid), Vit B2 (riboflavin), Vit C (ascorbic acid), and zeaxanthin. [30, 33] Leaves have the highest contents of vitamins [30,34-49].

Studies have demonstrated that the mixture of green graphene oxide reduced by the utilization of *Urtica Dioica* extracts is less known in the present researches. In the present study, the green reduction of graphene oxide from *Urtica Dioica* species was considered [16, 34].

2 Experimental

2.1 Preparation of Graphene oxide powder

Graphene oxide was prepared by oxidation of regular graphite powder by Modified Hummer's technique [9-12]. In the ordinary strategy for preparation, 5 gr graphite and 5 gr NaNO_3 (sodium nitrate) were added to 200 ml of H_2SO_4 (on Ice and water) and bit by bit 30 gr of KMnO_4 was added to this blend, and the procedure temperature was kept at 30°C (since the response is exothermic). The reaction mixture at that point was mixed for 18 hours at room temperature with the goal that thick glue was shaped. The glue at that point was filled with 400 ml of distilled water and following 20 minutes another 1 liter of distilled water was added to the blend. 30 ml of 30% H_2O_2 arrangement was added to the reaction mixture was dropped insightful to reduce the abundance KMnO_4 . The arrangement was transformed into light-dark colored in the wake of including H_2O_2 . The solution was expelled in a decanter and centrifuged and after that washed out with distilled water to achieve the pH of 7 and afterward dried under vacuum for 24 hours to frame GO powder [12].

2.2 Preparation of *Urtica Dioica* leaf extracts

Leaves of the *Urtica Dioica* plant were gathered from different regions of Shiraz, Fars, Iran, washed independently with water and dried at room temperature for seven days (NOVIKOV, PG and KAPELEV 1984). 10g of dry leaves were washed and grounded (with a blender) and added to 200 ml of deionized water in a 500ml Erlenmeyer flask. The mixture was boiled for 60min before sifted under encompassing conditions. The concentrates were separated and kept at 4 °C and utilized within seven days [35, 36].

2.3 Reduction of graphene oxide

50mL Urtica Dioica leaf extract was added to 200mL of a homogeneous scattering of graphene oxide (1mg/mL) and was shaken and sonicated for 20 min, the mixture was warmed in an oil shower at 98 °C under reflux for 24 hrs. The subsequent dark scattering was centrifuged at 15000 rpm for 60 min. Then, the pellet was washed 3 times with DI water to evacuate the undesirable materials. In the end, the reduced graphene oxide (RGO) was dried at 70 °C for 24 hrs [37, 46-51].

3 Results and Discussion

3.1 Optimization of reaction condition for the reduction of GO

The reduction of graphene oxide with Urtica Dioica extricates was checked by an adjustment, an expansion in absorbance at 600nm and tends to be observed by optical thickness estimation at 600nm [38-39]. Serial dilution checked the straight connection between absorbance at 600 nm and the concentration of graphene suspension. We examined the impact of Urtica Dioica concentrates on various fixations on the reduction of GO. Figure 2 demonstrates the adjustment in absorbance at 600 nm with time, for the response between various concentrations of Urtica Dioica extracts (5, 10, 15, 20, 25, and 30 % v/v) and 0.5mg/mL of GO suspension at 98°C for 45 hrs.

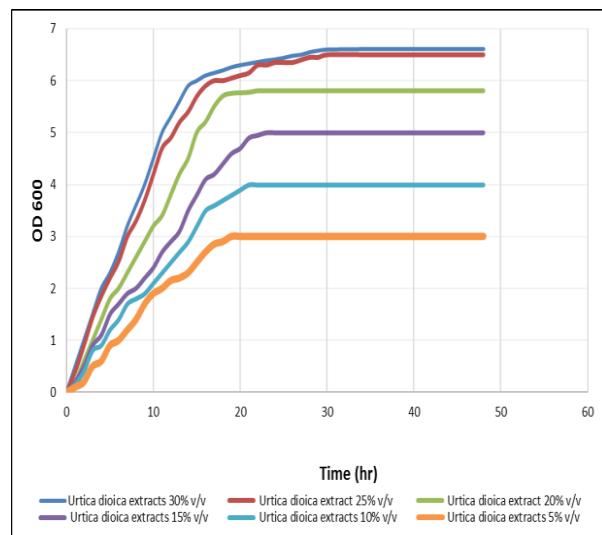


Figure 2: Effect of Urtica Dioica extract concentration on the reduction of (0.5 mg/mL) graphene oxide at 98 °C. The level of reduction seen to increment with a concentration of the Urtica Dioica removes from 5% to 30%. No further increment in absorbance was discovered when the concentration of the Urtica Dioica extracts expanded to 30%. Thus, 25% of Urtica Dioica extracts were utilized for future tests.

Figure 3 demonstrates the adjustment at 600 nm for the reaction between 25% Urtica Dioica extracts and

0.5mg/mL GO at six distinct temperatures (25, 40, 50, 60, 80, and 98 °C). As the temperature elevated from 25 to 98 °C, the level of reduction likewise expanded.

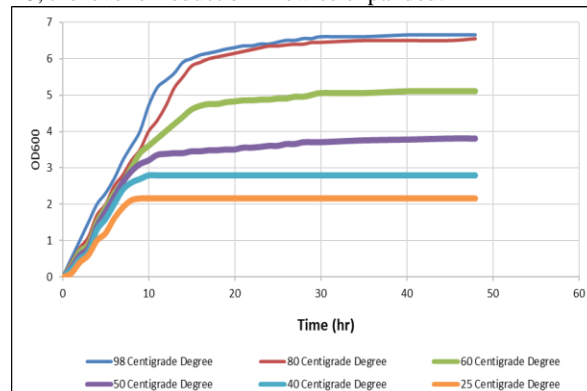


Figure 3: Effect of temperature on the reduction of graphene oxide (0.5mg/mL) using 25% v/v Urtica Dioica extracts

Both the values of final absorbance and reaction rate were higher at 98 °C compared to those at 25, 40, 50, 60, and 80 °C.

3.2 Infrared spectra

Graphite powder was utilized to make graphene oxide, which was acquired from Merck Inc. The aftereffects of the graphite infrared spectroscopy test are watched. The graphite contains no carbonyl and epoxide group. The adsorption in the locale of around 3422 cm⁻¹ is identified with the hydroxyl group of the atom water consumed by graphite [40].

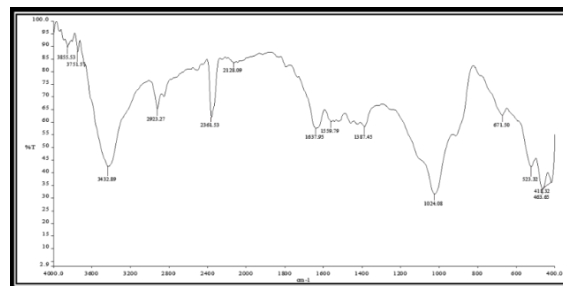


Figure 4: Infrared spectra of graphite

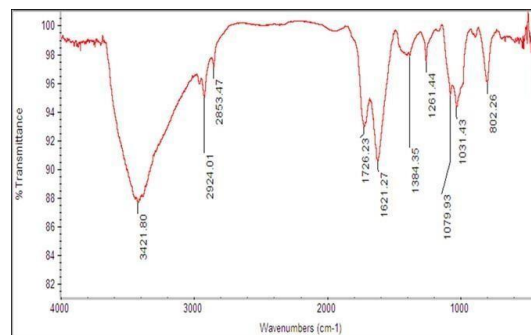
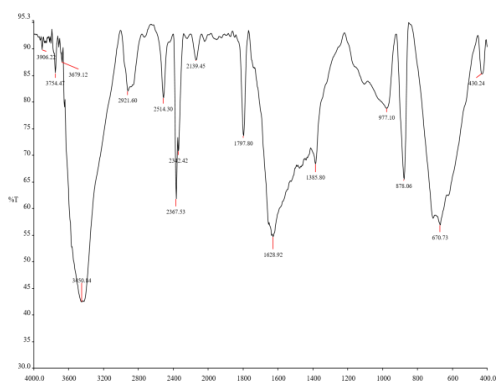


Figure 5: Infrared spectra of graphene oxide

Graphene oxide prepared by modified Hummers method. The FT-IR spectroscopy analysis of graphene oxide demonstrates an absorption frequency of 1720 cm^{-1} relative to the tensile vibrations of the carbonyl group ($\text{C}=\text{O}$) and additionally strong absorption in the 3421 cm^{-1} of the hydroxyl group (OH), 1384 cm^{-1} group of the flexural vibrations of the hydroxyl group (OH), $1200\text{-}1210\text{ cm}^{-1}$ for C-O-C stretching,

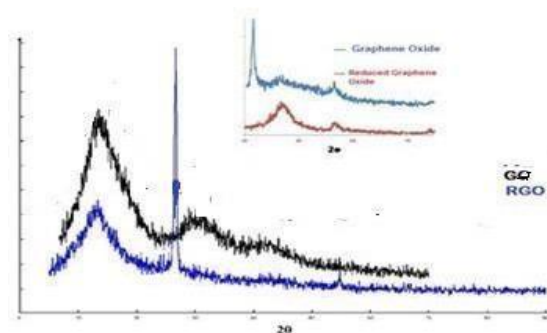
1049 cm^{-1} for C-O stretching and absorption in the 1031 cm^{-1} locale of the epoxide tensile frequencies that indicate the presence of functional groups such as carbonyl, carboxylic, epoxy and hydroxyl groups in GO. Likewise, the absorbance peak in 1621 cm^{-1} is identified with the functional groups ($\text{C}=\text{C}$) staying on graphene plates, which is not oxidized [40]. At that point, the concentrate of leaf extract of *Urtica Dioica* was added to arranged GO, refluxed then sonicated, and at the last stage, the suspension was centrifuged and the RGO dark shaded washed with water twice and went away in the stove. The examination of the FT-IR spectra in GO and RGO demonstrates that in GO there is a tensile vibration of $\text{C}=\text{O}$ in cm^{-1} of 1720 cm^{-1} , yet in RGO the ductile vibration of $\text{C}=\text{O}$ isn't so serious and moved to 1797 cm^{-1} , and in addition the power of the vibration retention of OH in 3421 cm^{-1} and 1384 cm^{-1} which at RGO diminished fundamentally after reduction and achieved the estimations of 3350 cm^{-1} and 1100 cm^{-1} , so it tends to be presumed that amid the recovery procedure (planning of RGO), the current oxygen species On the surface of graphene sheets, the concentrate of *Urtica Dioica* plant has been decreased and graphene oxide has been reduced to graphene.

**Figure 6:** Infra-red spectra of reduced graphene oxide

3.3 XRD analysis

The XRD test was used to check the structure of graphene oxide, graphene, and oxidized graphene. As shown in figure 7, XRD for GO demonstrated a peak at $2\theta = 10.5$, which shows the nearness of oxygen-containing

functional groups in void spaces between graphene oxide plates. As found in Figure 7, the graphene XRD example of this peak has been exchanged to the range $2\theta = 13$ and speaks to the graphene cover after the way toward recovering and emptying water particles and oxygen bunches from the spaces between the layers of graphene oxide [39-44].

**Figure 7:** XRD spectra of reduced graphene oxide and graphene oxide

3.4 Raman study

Raman scattering is a valuable device to portray graphene and graphite as this dissipating emphatically relies upon the electronic structure. Raman range of GO was observed to change after reduction (Figures 8 and 9). In the RGO and GO spectra, 2 crucial vibration bands were seen in the scope of $1250\text{-}1750\text{ cm}^{-1}$. The G vibration mode, inferable from the first-order scattering of E_2g phonons by sp^2 carbon of GO, and RGO was discovered at 1587 and 1592 cm^{-1} separately, whereas the D vibration band acquired from a breathing method of j-point photons of $\text{A}_{1\text{g}}$ symmetry of RGO and GO showed up at 1345 and 1350 cm^{-1} separately. After RGO the force proportion of the D and G band (ID/IG) was expanded essentially. As D band emerges due to sp^2 carbon group, higher power of D band proposed the nearness of more detached graphene space in RGO in contrast with GO and expulsion of oxygen moieties from the former. It is notable that the 2-phonon (2D) Raman dissipating of graphene-based materials is an important band to separate the monolayer graphene from twofold layer/multilayer graphene as it is profoundly discerning to graphene layers stacking. For the most part, a Lorentzian peak for the 2D band of the multi-layer graphene sheets seen at 2700 and 2900 cm^{-1} [40-42]. This demonstrates RGO has a multilayer structure. After RGO, the 2D band is

extraordinary, which proposed graphene layers stacking. As GO has diverse kinds of functional groups that may avoid graphene layers stacking yet after reduction because of an abatement of such functional groups a couple of graphene layers are stacking and framed multi-layer RGO[39-40].

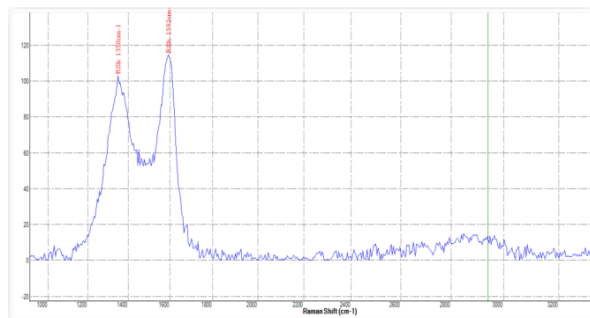


Figure 8: Raman spectra of GO

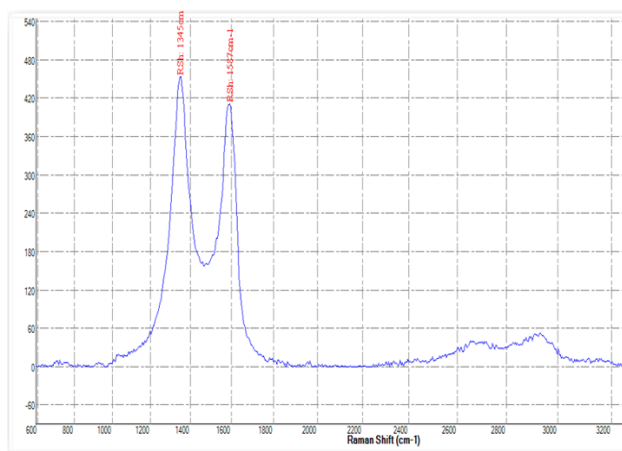


Figure 9: Raman spectra of RGO

3.5 Transmission Electron Microscopy (TEM)

Figure 10 displays TEM images of the RGO sheet reduced with *Urtica Dioica* leaf extract. The appearance of RGO silky and transparent sheets in TEM images verify its stability under a high-energy electron bar. The high-resolution TEM images are utilized to get to the quantity of layers in numerous areas. The edges of the suspended graphene films tend to crease back, permitting a cross-sectional perspective of the films. The collapse of a couple of layers at the edges of the films shows up as a couple of dim lines, individually. The arrangement of a few layers of RGO is unmistakably obvious. It has likewise been discovered that the forces of all the diffraction spots are not equivalent and sharp enough. Additionally, the diffraction spots are related to some uncertain spots. Every one of these perceptions additionally supporting the development of few layers RGO.

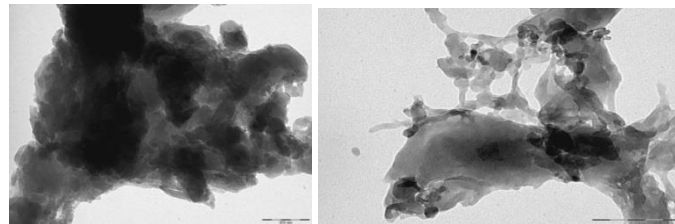


Figure 10: TEM images at 200nm magnifications, showing the formation of the few layers of reduced graphene oxide

In addition, for the first time, a facile approach is reported for effective RGO using *Urtica Dioica* extracts as green and environmentally friendly reducing agents. The Elemental Analysis of C, H, N, S, and O (CHNS/O-2400 series II- Perkin Elmer) was used to characterize the de-oxidation efficiency and morphology of reduced graphene and the results proved that *Urtica Dioica* extracts could effectively reduce GO with a high C/O ratio (6.95), compared with that of GO reduced by reported hazardous chemical reducing agents and other natural reducing agents [43-44]. The RGO production mechanism is probably due to the active components in *Urtica Dioica* extracts, which have a high binding affinity to the groups containing oxygen to form their oxides. This method avoids the use of any hazardous chemicals, thus facilitate the mass production of RGO [39-41].

4 Conclusion

We showed that the study Phytoextracts had tremendous potential to be utilized as agents for reducing GO with an environmentally-friendly synthetic protocol. GO was made from graphite powder by a modified method of Hummer followed by ultra-sonication. The optimized reaction condition for GO reduction was at 98 °C for 45 hrs and using 25% v/v of *Urtica Dioica* extracts.

We showed that the considered phytoextracts can possibly be utilized as diminishing operators for the reduction of GO with an ecological amiable engineered convention. GO was prepared from graphite powder utilizing the modified strategy of Hummer trailed by ultra-sonication. The optimized reaction condition for GO reduction was at 98 °C for 45 hrs and using 25% v/v of *Urtica Dioica* extracts. The most imperative favorable circumstances of the phytochemicals are their wealth in nature, cost-viability, and simple item segregation after reduction as they extricated from non-palatable or squander plant items. The estimations of particular capacitance, high C/O proportion (6.95), and high electrical conductivity of the phyto-extract RGO are adequate. In this way, this green strategy can be utilized for the vast scale generation of RGO. An eco-accommodating and naturally considerate reduction framework by utilizing *Urtica Dioica* removes as a

biocatalyst for GO reduction is portrayed. The reduction was done in a watery medium at an alternate temperature. TEM uncover the development of a few-layer of reduced graphene oxide. FTIR and X-ray examination give proof to the end of labile oxygen usefulness from the GO surface. De-oxygenation and the development of deformities in the RGOs were affirmed by Raman spectroscopy. The primary points of interest of this system over the conventional synthetic reduction are the cost-adequacy, ecologically agreeable methodology, and basic item confinement process. This ecological amicable decreased graphene oxide can possibly be utilized in different territories, for example, biomedical applications.

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Conflicts of Interest: The authors declare that they have no conflicts of interest to report regarding the present study.

Ethical issue

Authors are aware of and comply with, best practices in publication ethics, specifically about authorship (avoidance of guest authorship), dual submission, manipulation of figures, competing interests, and compliance with policies on research ethics. Authors adhere to publication requirements that submitted work is original and has not been published elsewhere in any language.

Authors' contribution

All authors of this study have a complete contribution for data collection, data analyses, and manuscript writing.

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