Facile Green Synthesis of Reduced Graphene Oxide in L-cysteine Solution and its Structural, Morphological, Optical and Thermal Characteristics

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Abstract: The current research reports a cost-effective, efficient ad green reducing agent (L-cysteine) to reduce the graphene oxide (GO) for large-scale reduced graphene oxide (rGO) synthesis. Fabrication of rGO was performed by the reduction of GO using different concentrations from L-cysteine. Synthesis of rGO was noticed by change in color of GO solution from brown to black. For additional confirmation, the structural, morphological, optical and thermal properties of synthesized rGO were analyzed using powder X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, scanning electron microscope (SEM), energy dispersive X-ray Spectroscopy (EDX) atomic force microscopy (AFM), thermogravimetric analysis (TGA) and ultraviolet-visible spectrophotometer (UV-Vis). The XRD, FTIR and EDS results showed the oxygen-containing groups such as hydroxyl, carbonyl, and epoxy. The UV-Vis spectrum for GO exhibited an absorbance peak at 233 nm which undergoes a red shift of the absorbance peak to 265, 273 and 278 nm due to reduction of GO to rGO using 5, 8 and 10 mg/L of L-cysteine respectively. XRD patterns demonstrated the disappearance of the characteristics peak of GO (11.1) and reinforced this appearance of peak around ~26, indicating the efficient reduction of GO and restoration of graphene sp2 hybridized structure. Furthermore, FTIR spectroscopy showed the gradual disappearance of band at 1745 cm-1 assigned to GO as L-cysteine concentration was increased. The thermal stability of the GO was much lower than those of all the rGO powders where the increased concentration of L-cysteine resulted in enhanced more thermal stability and higher C/O ratio in rGO. The SEM images confirmed the successful structurally exfoliation of two dimensional rGO sheets and showed the folded, curled and flake-like morphology of the graphene nanosheets.

Keywords: GO, Green synthesis, rGO, nanosheets, L-cysteine, SEM, TGA.
1. Introduction
A quasi-two-dimensional material, graphene is one of the allotropes of carbon which has a hexagonal structure of carbon atoms has become a very popular material in recent years with a wide range of applications [1]. Graphene has been experimentally studied for long time, but Geim and Novoselov have been identified and analyzed the graphene nanosheets by mechanical exfoliation of pyrolytic graphite using Scotch tape [2]. Graphene exhibited a variety of unique and exceptional properties including high surface area, excellent thermal, superior optical, remarkable electrical and unusual mechanical characteristics [3]. Owing to these unique and fascinating properties, graphene has attracted significant attention in various areas of applications.

Graphene exhibits tremendous potential and unlimited possible applications in the fields of materials science, physics, chemistry and biology [4]. The remarkable characteristics make graphene a very promising material for use in the biomedical, electronic, catalysis, optics, environmental, agricultural, food, energy storage and other fields [5,6]. Till date, several innovative methods including chemical vapor deposition [7], arc discharge [8], chemical reduction of graphite oxide [9], aerosol pyrolysis [10], mechanical exfoliation [11], liquid-phase exfoliation [12], hydrothermal [13], solvothermal [14] and laser reduction of graphite oxide [15] have been developed to for the synthesis of thin layered graphene. Synthesis of graphene for large scale applications is mainly depends upon the carbon precursors and efficient and economical preparation method [16]. Fabrication of high-quality graphene in high yield and large-scale cost-effective in simple and eco-friendly manner is a huge challenge facing scientists [17].

Among different methods, synthesis of graphene via chemical reduction of graphite oxide is recognized as most favorable method to produce high quality graphene due to easy, scalable, facile and inexpensive synthesis rout [18]. Various chemical reducers have been utilized to fabricate graphene nanosheets, including hydrazine, hydrazine hydrate, sodium iodide, sodium bisulfite, dimethylhydrazine, hypophosphorous acid, sodium hydroxide, pyrogallol, hydroquinone, sodium borohydride and hydriodic acid [19-22]. Unfortunately, the highly toxic and corrosive nature and instability of all these reducing agent limit its widespread use. Therefore, there is a growing need to develop new and innovative methods which are safe for the environment and can be adopted to synthesis graphene with excellent properties. For this reason, interest has been focused toward environmentally friendly, cost-effective processes for synthesis graphene nanosheets with less toxicity and less harm to the environment [23].

The use of green routs in rGO fabrication is highly attractive owing to the environmental sustainability, lower production and disposal cost and high carbon content [24]. Green methods for the synthesis of graphene and graphene-based materials are far superior to those fabricated via classical methods [25]. Recent efforts have mainly focused on the substitution of toxic reducing agents by natural analogs to produce graphene and graphene-based materials [26, 27]. A number of approaches have been reported for green synthesis of rGO and to achieve the previous requirement including Rose water [28], Wild carrot root [29], Sabdarriffa [30][31], green tea [32], ascorbic acid [33] L-cysteine [34,35], Lantana camara [36], grape seeds [37], Anthos squamosal [38], Fenugreek Seeds [39] and ginkgo biloba [40]. In the current paper, we demonstrate a one-pot green facile synthesis of rGO using L-cysteine as a natural reducing and capping agent.

2. Experimental Work
2.1. Materials
Graphite powder (5 μm) was supplied from B.D.H, potassium permanganate (KMnO₄, 99.0%), sulphuric acid (H₂SO₄, 97%), hydrochloric acid (37%), potassium permanganate (KMnO₄, 99.0%) sodium hydroxide (99%) and hydrogen peroxide (H₂O₂, 30%) were supplied by Scharlau, sodium nitrate (NaNO₃, 99%) was supplied by Merck, L-cysteine (C₃H₇NO₂S, 97%) was received from Sigma-Aldrich. All of the chemicals were analytical grade and were used exactly as received. De-ionized water (DI) was used in the synthesis of samples and other solutions.
2.2. Instruments

The XRD analysis of GO and rGO samples was performed on a Shimadzu- XRD 6000 diffractometer, with CuKα radiation of 1.54056 Å; the XRD pattern was recorded in range 10°-75°. Fourier transform infrared spectroscopy (Shimadzu FT-IR 8400S/Japan) was used to study the functional groups present in the GO and rGO using KBr pellets method in the wavelength number from 500 to 4000 cm⁻¹. Scanning electron microscopy (MIRA3 TESCAN - Czech) was used to analyze the morphology of the Go and rGO samples and elemental composition was determined from energy dispersive X-ray diffraction (EDX) joined to SEM. Optical absorption spectra of GO and rGO (aqueous solutions) were obtained using a UV-vis spectrophotometer (Shimadzu, UV-1650). The mass loss of GO and rGO was carried out on a TG 209 F1 Netzsch Iris instrument (Netzsch, Germany).

2.3. Synthesis of GO

The GO nanosheets was synthesized by a facile and scalable chemical route via conventionally-modified Hummer’s method using graphite powder as a starting material [41,42]. Typically, graphite powder was oxidized into graphite oxide under harsh conditions using a mixture of H₂SO₄ (to disperse graphite), KMnO₄ and NaNO₃(oxidant agents). The next step is rinsing graphite oxide with H₂O₂ (30 %) and HCl (10 %) in subsequent order. The developed brown color product was repeatedly washed with DI until the pH of decanted water reached ~7. Then the GO is directly dispersed into DI and sonicated for 30 min to produce a homogeneously dispersed GO. Afterwards, the resultant GO aqueous dispersion was separated by centrifugation at 5000 rpm for 30 minutes. The obtained brown product was dried at 45 °C overnight using vacuum oven.

2.4. Reduction of GO with L-cysteine

Typically, GO (0.5 mg/mL) was dispersed in 100 mL DI followed by a sonication for 20 min to obtain a homogeneous suspension solution. Different amounts of L-cysteine powder (5, 8 and 10 mg/L) were added into the GO aqueous dispersion and shaken thoroughly at 40 °C for 20 h [35]. During the reduction process, homogeneously dispersed brown-colored GO was finally turned to light dark color material indicated high level of reduction and successful deoxygenation of GO [31]. It is well known that the L-cysteine has a lower solubility in water and alcohol. However, it dissolves in dilute solutions of alkali hydroxides, hence, a certain amount of 0.1 mol L⁻¹NaOH aqueous solution was added into the GO suspension to enhance L-cysteine solubility [43]. Black slurry obtained from the above solution after being centrifuged at 10000 rpm for 20 min was washed several times with water and ethanol up to pH = 7.0. Lastly, the resulting wet rGO was dried under vacuum at 50 °C for 24 h to obtain it in the black powder form. The rGO samples were designated as rGO1, rGO2 and rGO3 based on 5, 8 and 10 mmol/L L-cysteine concentration, respectively. The reduced graphene oxide steps are shown in Scheme 1.
3. Results and Discussion

3.1. X-ray Diffraction Analysis

The crystalline phase of GO and rGO powders has been analyzed by the XRD technique. Figure 1 displayed the XRD pattern of GO and rGO samples. The GO pattern exhibited a strong peak at 2θ = 11.10 Å (001) [44]. After GO reduction, the (001) peak had disappeared, and the XRD diffraction peak (002) for rGO appeared at 2θ = 25.85°, 25.92° and 26.14° for rGO1, rGO2 and rGO3 on using L-cysteine concentration at 5, 8 and 10 mmol. L⁻¹, respectively. The d-spacing between the crystal lattice and wave field was calculated using following Bragg’s law [45]:

\[ \lambda = 2d\sin(\theta)/n \]

Where, \( \theta, \lambda, n, \) and \( d \) are the angle of scattering, the wavelength of the incident x-rays (1.54 Å), the positive integer (order of reflection) and the interlayer distance, respectively. The interlayer distance of GO ~ 7.65 Å showed an agreement with previous works [6, 30]. High d-spacing value of GO is attributed to strong electrostatic repulsion forces among adjacent GO layers induced by the abundant oxygen functional groups and inserted water molecules [46]. Moreover, the interlayer space of 3.78 Å, 3.69 Å, and 3.62 Å were calculated for the rGO1, rGO2 and rGO3 samples respectively. However, a narrowed d-spacing between the rGO layer caused by the removal of oxygen functional groups. The decreasing in interlayer separation is an important evidence of a successful GO reduction. Generally, the interlayer spacing of the rGO can be adjusted by the controlled reduction.
3.2. FTIR Analysis

FTIR analysis is a suitable tool to obtain functional groups that present in sample. Figure 2 displays the FTIR spectra of GO and rGO samples. In the GO spectrum, the characteristic peaks appear at 1051, 1315, 1580, 1760 and cm$^{-1}$, can be assigned to C=C, C-O (epoxy), -OH and C=O bonds respectively [22]. The broad peak centered at 3432 cm$^{-1}$ corresponds to O-H stretching vibrations of –COOH functional groups and adsorbed water [47]. Above peaks did not appear in the graphite spectrum, confirming that the oxidation process introduced a high level of oxygen-containing functional groups, such as -COOH and C = O located at the GO sheet edges, and epoxy and hydroxyl groups on the basal planes of the GO sheets [48]. However, for the rGO samples, the intensities of O–H, C=O and C-O peaks seem to be slightly weaker as compared to GO spectrum which indicate the deoxygenation and hence successful reduction of GO and the obtained results are almost similar to the pristine graphite. It was expected that rGO will has less oxygen-related functional group.

3.3. UV-Visible Analysis

The UV-Visible analysis was employed for evaluation of optical properties of the synthesized materials. The spectra for GO and rGO solutions were recorded as shown in the Figure 3. The absorption spectrum of GO dispersions exhibits a peak at 233 nm which corresponds to the electronic π- π* transition of the C=C bonds [49]. Similar noticeabilities have been observed for the reduction of GO with other green reducers reported in a previous literature [50, 51]. The energy band gap can be calculated using blank's equation ($E = \frac{hc}{\lambda}$). The study demonstrates that the value of the GO band gap energy was 5.39 eV. Furthermore, a shoulder was also observed around 300 nm for GO sample, which is attributed to n- π* transition of the C=O.
groups [52]. On reduction of GO, the C=C peak at 230 nm is gradually red-shifted to a longer wavelength (265, 273 and 278 nm) in case of rGO1, rGO2 and rGO3 respectively. This red shift indicates a possible deoxygenation of the GO and restoration of the electronic conjugation sp2 within the graphene sheets after the promotion of GO reduction by L-cysteine [53]. Additionally, it can also be seen the disappearance of the C=O shoulder at 300 nm which indicates the GO has been successfully reduced by L-cysteine.

![FTIR spectra of G, GO and rGO powders.](image)

**Fig. 2.** FTIR spectra of G, GO and rGO powders.

![UV-Vis spectra of GO and rGO solutions.](image)

**Fig. 3.** UV-Vis spectra of GO and rGO solutions.

### 3.4. FE-SEM Analysis

Morphology and topography of the synthesized GO and rGO samples were elucidated through SEM, as represented in Figure 4. Owing to the exfoliation and restacking processes, SEM analysis is clearly exhibiting the course, wrinkled,
crumpled and wave-like structure in GO sheets forming clusters with roughened surface where the few layers of graphene sheets clearly seen. With the exfoliation of graphite into GO, the surface of GO exhibits a soft carpet-like morphology, which may be due to the interaction of residual H$_2$O molecules and oxygen functionalities besides carboxyl groups with GO. In contrast, upon reduction from GO to rGO, SEM analysis exhibits partially folded, curled and flake-like structure with some individual sheets closely stacked. With the removal of oxygen-containing functional groups from the basal planes as well as from the GO sheet edges, the graphene sheets were then allowed to closely associate and stack via the interlayer van der Waals forces and strong $\pi$–$\pi$ stacking [54]. This observation is in good agreement with the XRD results, where the interlayer distance for rGO or is smaller than GO, enhancing the stacking of rGO sheets.

3.5. EDX Analysis

Energy-dispersive X-ray spectroscopy (EDS) was also performed to evaluate the chemical composition of GO and rGO powders. Figure 5 displays the EDX spectra of GO and rGO materials. Furthermore, the C:O ratios of GO and rGO samples are listed in Table 1. As results show, EDS spectrum of GO confirms that the material contains only oxygen (O) and carbon (C) which can be considered as evidence of the successful oxidation of graphite. As GO is the most oxidized form of graphite, its C:O decreased as the oxygen content increased due to high oxygen content, whereas in rGO, high decrease of the oxygen content and larger C:O ratios are observed indicates the efficient contribution of L-cysteine as an eco-friendly reduction agent. According to EDS measurements of the rGO samples, oxygen content decreased from 27.98 % to 20.03 % on increasing of L-cysteine concentration from 5 mmol/L to 10 mmol/L, respectively. According to EDS data, the atomic C/O ratio changed from 1.69 to 3.73 after reduction of GO, indicating a high degree of reduction.
Fig. 4. SEM images of GO and rGO samples.

Fig. 5. EDX spectra of rGO3 sample.
Table 1. C/O atomic ratio of GO and rGO samples.

| Samples | C %  | O %  | C/O |
|---------|------|------|-----|
| GO      | 58.38| 34.50| 1.69|
| rGO1    | 60.32| 27.98| 2.15|
| rGO2    | 66.93| 24.52| 2.72|
| rGO3    | 74.84| 20.03| 3.73|

3.6. AFM Analysis

AFM is widely used to qualitatively examine surface topography and sheet thickness of synthesized materials, and the main results of samples are shown in Figure 6 and summarized in Table 2. The roughness average of a pristine GO is 0.88 which due to increase of active sites that relate with attachment of covalently bonded oxygen(C-O) which enhances the porosity of surface. In contrast, after the reduction process, the roughness of rGO was significantly higher than the roughness of GO and this is attributed to the elimination of, carboxyl, hydroxyl and epoxy groups on GO surface. One of the most important parameters of AFM analysis is surface skewness. The skewness index that indicates the asymmetry of the probability distribution of peaks and valleys [55]. As Table 2 shows, the enhanced surface roughness is supported by negative values of skewness corresponding to higher valleys which mean more rounded peaks with sharper valleys. Also, kurtosis parameter is a statistical measure used to describe the flatness of the height distribution. For spiky surfaces, Sku >3; for bumpy surfaces, Sku <3; perfectly random surfaces have kurtosis of 3 [56]. As Table 2 show, the results of Sku values corresponds to spiky surfaces, of all GO and rGO samples.

Table 2. Surface topographic parameters of synthesized carbonaceous materials.

| Samples | Roughness average, nm | Surface skewness | Surface kurtosis | Thickness, nm |
|---------|------------------------|------------------|------------------|---------------|
| GO      | 0.88 nm                | -0.353           | 4.68             | 231.91        |

Fig.6. AFM 2D and 3D image of rGO3.
3.7. TGA Analysis

The thermal properties of GO and rGO powders was tested using TGA analysis whereas the main results are shown in Figure 7 and listed in Table 3. The thermal treatment in argon was carried out at heating rate of $10 \, ^\circ C \, min^{-1}$ from room temperature to $600 \, ^\circ C$. TGA of GO shows a major weight loss up to $200 \, ^\circ C$ owing to evaporation of absorbed water molecules [57]. At temperatures ranging from $210$ to $600 \, ^\circ C$, a weight loss of $18.2\%$ was observed which can be ascribed to deoxygenating process pyrolysis of CO and CO$_2$ groups in the unstable carbon remaining in the graphitic structure [58], whereas the total weight loss of GO $\sim 90 \%$. In general, the TGA results show that the less weight loss was observed with rGO samples are lower than GO. TGA curves of rGO powders suggest that a large proportion of oxygen functionalities were vanished from the surface of GO upon reduction.

![Fig. 7. TGA curves of G, GO and rGO powders.](image)

| rGO sample | TGA temp, °C | Weight loss, % |
|------------|--------------|----------------|
| rGO1       | 340.613 594.248 | 30.70 15.92 |
| rGO2       | 338.437 594.721 | 6.46 3.34 |
| rGO3       | 335.882 595.001 | 5.19 3.37 |
Conclusions

The rGO nanosheets have been successfully fabricated via an easy, inexpensive, eco-friendly and dependable rout at mild conditions. Aqueous solution of L-cysteine has been efficiently obtained for the reduction of GO into rGO. The L-cysteine exhibited a promise capacity a reducing, capping and stabilizing agent in the same time which may possibly attributed to the existence of the sulfhydryl groups. The synthesized rGO was analyzed by XRD, UV-Vis, FTIR, SEM, EDX, AFM and TGA techniques and the results confirmed the oxygen removal from GO surface. The results suggested that the utilized synthesis method could be advantageous for large-scale preparation of rGO. The results demonstrated that increasing the L-cysteine concentration induced a lowering of the O content in GO and hence improved the rGO optical, morphological and thermal properties.

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