# GREEN SYNTHESIS OF GRAPHENE AND ITS FUNCTIONALIZATION USING DES

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# CANDIDATE'S DECLARATION

We, Shyam (2K23/MSCCHE/40) and Aditya Saraswat (2K23/MSCCHE/03), hereby certify that the work which is being presented in the dissertation entitled "**Green synthesis of Graphene and its functionalization using DES**" in partial fulfillment of the requirements for the award of the Degree of Master of Science in Chemistry, submitted in the Department of Applied Chemistry, Delhi Technological University is an authentic record of our own work carried out during the period from 2024 to 2025 under the supervision of Dr. Richa Srivastava.

The matter presented in the thesis has not been submitted by us for the award of any other degree of this or any other Institute.

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# CERTIFICATE

Certified that Shyam (2K23/MSCCHE/40) and Aditya Saraswat (2K23/MSCCHE/03) have carried out their research work presented in this thesis entitled **“Green synthesis of Graphene and its functionalization using DES”** for the award of Degree of Master of Science in Chemistry from Department of Applied Chemistry, Delhi Technological University, Delhi, under my supervision. The dissertation embodies the results of original work, and studies are carried out by the students themselves, and the content of the thesis does not form the basis of the award of any other degree to the candidate or to any other body else from this or any other University/Institution.

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# ABSTRACT

The increasing need for sustainable and environmentally friendly materials has induced the search for ecologically-clean synthesis methods for advanced nanomaterials. This dissertation describes a green synthetic route to graphene with the further modification by Deep Eutectic Solvents (DES). To start with, via a modified Hummers procedure (GO) have been synthesized. This method allows for better control over oxidation levels and particles dispersion properties. Then the GO was reduced by a plant-based phytoextract which is a nontoxic and eco-friendly alternative to conventional chemical reducers. The RGO thus obtained was further characterized by Fourier Transform Infrared Spectroscopy (FTIR), Ultraviolet-Visible (UV-Vis) spectroscopy, and Thermogravimetric Analysis (TGA) to check the extent of reduction and to evaluate thermal stability.

In the next step a DES was prepared by mixing the naturally obtained hydrogen bond donors and acceptors. This DES is then used for the modification of the RGO surface in order to improve its chemical reactivity and application. The functionalized graphene was characterized. The FTIR experiment reveals the nature of the mutual interaction of the DES components and the graphene surface. The results indicate that DES is not only the functionalizing agent but also the stabilizing agent of the graphene sheets phenomenon that can lead to the utilization of green sectors such as chemistry and nanotechnology.

This work highlights the possibilities of using green chemistry principles to produce and modify graphene-based materials, thus creating a more environmentally friendly way of conducting research in the field of material science.

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**Shyam** **Aditya Saraswat**

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# ABBREVIATIONS

GO : Graphene Oxide

RGO : Reduced Graphene Oxide

DES : Deep Eutectic Solvent

UV–Vis : Ultraviolet–Visible Spectroscopy

FTIR : Fourier Transform Infrared Spectroscopy

TGA : Thermogravimetric Analysis

HBD : Hydrogen Bond Donor

HBA : Hydrogen Bond Acceptor

# CHAPTER 1

# INTRODUCTION

## 1. Introduction

The single sheet of carbon(C) atoms organised in a hexagonal lattice, graphene has captured immense interest because of its extraordinary electrical conductivity, mechanical strength, and thermal stability [1]. Among the various methods of synthesis, "green" techniques are gaining in popularity as environmentally acceptable and sustainable alternatives to traditional chemical treatments, which are often carried out with hazardous substances. In particular, the use of plant-based extracts to reduce graphene oxide (GO) is in line with the principles of green chemistry and is a very cheap way of obtaining reduced graphene oxide (RGO) that is still high performance [2-4].

### 1.1 Graphene Oxide: Synthesis via Modified Hummer's Method

GO is very often produced by oxidising graphite, and on account of improvements in reaction efficiency and repeatability, the modified Hummer's method has become especially popular [4]. The process involves using strong oxidising agents such as potassium permanganate and sulfuric acid, though changes like controlled addition rates and lower temperatures reduce the generation of by-products and safety issues are improved [5]. GO is a versatile source for further chemical modifications due to the presence of oxygen-containing functional groups (epoxy, hydroxyl, and carboxyl) that are plentiful in the material [6].

### 1.2 Green Reduction of Graphene Oxide to RGO

Traditional GO reduction usually means using hydrazine, which is a highly toxic reducing agent. On the other hand, phytoextracts (water-soluble plant parts) have natural polyphenols and reducing sugars that can go through a green process for the reduction of graphene oxide (GO) to reduced graphene oxide (RGO) [7]. In our project, we use a clearly-identified phytoextract for the purpose of going green while obtaining a very high level of reduction. Spectral methods, e.g., ultraviolet-visible

(UV–Vis) and Fourier-transform infrared spectroscopy (FTIR), along with thermogravimetric analysis (TGA) for thermal stability, are procedures that confirm the reduction process [8-9].

### 1.3 Deep Eutectic Solvents (DES): Green Functionalization Media

Deep eutectic solvents (DESs) represent a new type of solvents that are constructed by the combination of hydrogen bond donors and acceptors, which results in a eutectic mixture with a melting point lower than each of the components [10]. The fact that they are tunable, biodegradable, and have a low level of toxicity makes DESs the best media for the green functionalization of nanomaterials [11]. In this dissertation, we produce a choline chloride-based DES and try to get the best out of its characteristics to functionalize RGO, thus allowing not only the introduction of specific functional groups but also the conservation of graphene's structural integrity.

### 1.4 Rationale and Objectives

Functionalizing RGO with DES offers multiple benefits: enhanced dispersibility in polar and nonpolar media, enriched chemical reactivity for subsequent applications, and improved compatibility in composite materials [12-13]. The use of DES not only facilitates covalent or non-covalent modification of the graphene surface, but also influences thermal and structural stability of the resulting hybrid materials, as evidenced by thermogravimetric analysis and FTIR studies [14-15]. Functionalized graphene's like these are more and more important in the areas of energy storage and also in biomedical applications.

# CHAPTER 2

# LITERATURE REVIEW

The rapid expansion of nanotechnology has propelled the exploration and utilization of novel materials with extraordinary properties. Among these, graphene and its derivatives have garnered extensive attention due to their exceptional physical, chemical, and mechanical characteristics. Graphene, a single sheet of carbon atoms in a hexagonal lattice, stands out for its high electrical conductivity, remarkable tensile strength, excellent thermal conductivity, and large surface area. Its derivatives, including GO and RGO, offer enhanced versatility, particularly in solution-based processing and functionalization, making them suitable for various advanced applications.

However, traditional methods for synthesizing graphene materials often involve hazardous chemicals, high-energy consumption, and environmentally damaging processes. The increasing global focus on sustainability and green chemistry has steered research towards the development of eco-friendly synthesis methods. This literature review provides an in-depth examination of the green synthesis of graphene using modified Hummer's method for GO production, followed by phytoextract-mediated reduction to RGO, and subsequent functionalization using Deep Eutectic Solvents (DES). It also covers comprehensive characterization via UV-Vis, FTIR, and TGA, and elaborates on the multifaceted applications of graphene and its derivatives.

## 2.1. Graphene and Its Derivatives: Structure and Properties

Graphene is an allotrope of carbon comprising a two-dimensional sheet of $sp^2$-hybridized carbon atoms. Due to its unique structure, graphene exhibits superior electrical conductivity (~200,000 $cm^2/V \cdot s$), mechanical strength (~130 GPa tensile strength), and thermal conductivity (~5,000 $W/m \cdot K$) [16]. These qualities make graphene very suitable for use in electronic devices, photonics, sensors, and energy storage systems [17].

In general, graphene oxide (GO) is produced by the oxidation of graphite which thus leads to the formation of oxygen-containing functional groups such as hydroxyl, epoxy, carbonyl, and carboxylic acids. It is these groups that make GO more hydrophilic and more easily dispersed in polar solvents while at the same time they turn it into a very versatile go-to for further chemical modifications [18]. RGO obtained by either chemical, thermal, or biological reduction of GO is a version that partially restores the conjugated π-network thus improving the electrical conductivity and chemical stability through the reduction process [19].

The existence of functional possibilities on GO and RGO makes it very easy for further diversification and the merging of these materials with others in this way the range of their possible applications will be reaching far and wide to new composite materials, biomedical and catalysts. It is worth noting that the extent of the process of both oxidation and reduction is of prime importance in defining the properties of those materials [20].

## 2.2. Synthesis of Graphene Oxide Using Modified Hummer’s Method

The traditional Hummer's method for preparing GO is a reaction of graphite oxidation with mixture of potassium permanganate ($KMnO_4$), sodium nitrate ($NaNO_3$), and concentrated sulfuric acid ($H_2SO_4$). Although being quite successful, this method is also very polluting since it creates nitrogen oxides and manganese residues as a result of the reaction [21]. In turn, environmentally friendlier, safer and more productive modified Hummer’s methods are now available.

Besides that, a sodium nitrate may be substituted with phosphoric acid which is much safer since no toxic gases are formed and additionally, a pre-oxidation step may be performed so as to increase intercalation and oxidation efficiency [22]. Several upgraded approaches have shown that they can efficiently improve graphite exfoliation and generate high-quality GO with large lateral size and elevated oxidation degree [23]. The GO obtained by a modified Hummer’s procedure is very stable in water and also has a large number of reactive sites which make it possible to perform reduction and functionalization in a subsequent step [24].

The oxidized nature of GO enables its use in water purification membranes, dye-sensitized solar cells, drug carriers, and as a precursor for RGO-based materials in energy storage devices [25-26]. The flexibility and scalability of this method support its adoption for large-scale production, aligning with green chemistry principles [27].

### 2.3. Green Reduction of GO Using Phytoextracts

Traditionally, to reduce GO to RGO, people usually employ reducing agents, such as hydrazine, sodium borohydride, or hydroquinone, which are dangerous, corrosive, and environmentally hazardous. However, plant extracts have been recognized as a new and eco-friendly source of reducing agents for this purpose. The extracts are phytochemical content, including polyphenols, flavonoids, and other antioxidants, can provide electrons, thus helping GO be reduced through oxygen functionalities.

To this end, an array of extracts of plants such as *Camellia sinensis* (green tea), *Azadirachta indica* (neem), *Punica granatum* (pomegranate), *Ocimum sanctum* (tulsi), and *Terminalia chebula* have been shown to be highly effective in eliminating GO [30-33]. Several factors, such as biocompatibility, low environmental impact, and affordability, make the use of these extracts even more attractive. On the other hand, adsorption of some agents onto the RGO surface, which may bring about better dispersion stability and biological activity, is one of the tasks that phytochemicals can perform.

Researches have indicated that the chemical performance of green-reduced RGO regarding the electric conductivity of the chemically resected RGO is not significantly different, as well as the green-reduced RGO exhibits more thermally stable and lower defect density [35]. Additionally, these substances can be very good performance indicators while working on the development of electrochemical sensors, supercapacitors, and photocatalysis [36].

## 2.4. Characterization Techniques: UV, FTIR, and TGA

To evaluate the synthesis, reduction, and functionalization processes, multiple characterization techniques are employed:

### 2.4.1 UV-Visible Spectroscopy

GO typically exhibits an absorption peak near 230 nm (π-π* transitions of C=C bonds) and a shoulder around 300 nm (n-π* transitions of C=O bonds) [37]. Upon reduction, the absorption peak shifts to ~270 nm, indicating restoration of the conjugated π-system [38]. This shift provides qualitative confirmation of reduction.

### 2.4.2 FTIR Spectroscopy

FTIR spectra reveal the presence and transformation of functional groups. GO displays strong peaks for O-H (~3400 $cm^{-1}$), C=O (~1720 $cm^{-1}$), and C-O (~1050 $cm^{-1}$) groups. After reduction with phytoextracts, these peaks diminish, while C=C (~1620 $cm^{-1}$) peaks increase in intensity, indicating partial deoxygenation and reconstruction of the sp2 carbon network [39].

### 2.4.3 Thermogravimetric Analysis (TGA)

TGA evaluates thermal stability and decomposition behavior. GO shows significant weight loss (~50%) around 200–300°C due to the decomposition of oxygen functionalities. In contrast, RGO exhibits reduced weight loss and improved thermal stability, reflecting successful reduction [40].

## 2.5. Deep Eutectic Solvents (DES): Synthesis and Green Chemistry Perspective

DES are emerging as green alternatives to conventional solvents. They are formed by hydrogen bonding between a hydrogen bond donor (HBD) and a hydrogen bond acceptor (HBA), typically at a specific molar ratio [41]. Common combinations include choline chloride (HBA) with urea, glycerol, or lactic acid (HBD) [42].

The synthesis of DES is simple: heating and stirring the HBA and HBD until a homogeneous, stable liquid is formed. DES are non-volatile, biodegradable, and non-toxic, with tunable physicochemical properties such as viscosity, polarity, and conductivity [43]. Their ability to solubilize various organic and inorganic materials makes them highly suitable for green synthesis and functionalization processes [44-45].

DES are used in catalysis, metal extraction, polymer synthesis, electrochemistry, and as reaction media in organic transformations [46]. Their wide electrochemical window and ability to stabilize reactive intermediates also facilitate applications in electrodeposition and energy storage [47-48].

### 2.6. Functionalization of RGO Using DES

Functionalization is key to enhancing the stability, processability, and reactivity of RGO. DES can be used both as reaction media and functionalizing agents. The hydrogen-bonding network in DES facilitates non-covalent interactions and the introduction of functional groups such as amines, hydroxyls, or carboxyls onto the RGO surface [49].

Studies have shown that RGO functionalized with DES (e.g., choline chloride-urea, choline chloride-lactic acid) exhibits improved dispersion in polar solvents and enhanced interaction with polymer matrices and metal ions [50-51]. FTIR spectra of DES-functionalized RGO reveal new peaks corresponding to C–N, O–H, and N–H stretching, confirming successful grafting [52]. These modifications significantly expand RGO's utility in composite materials, biosensors, and environmental applications.

Furthermore, DES-functionalized graphene has demonstrated enhanced electrochemical properties, enabling its use in advanced supercapacitors, fuel cells, and catalytic systems [53-54]. The biocompatible and non-toxic nature of DES also supports the integration of such materials in biomedical applications.

### 2.7. Applications of Graphene and Its Derivatives

The exceptional physicochemical properties of graphene and its derivatives have opened avenues for applications across various fields:

### 2.7.1 Energy Storage Devices

Graphene-based materials serve as high-performance electrodes in lithium-ion batteries, sodium-ion batteries, and supercapacitors. Their high conductivity and surface area facilitate rapid charge transfer and ion diffusion [55]. Functionalization with DES enhances surface wettability and improves electrolyte accessibility, resulting in higher specific capacitance and cycling stability [56-57].

### 2.7.2 Environmental Remediation

GO and RGO are efficient adsorbents for the elimination of heavy metal ions, dyes, and organic impurities from wastewater. Their large surface area and functional groups enable deep interactions with contaminants [58]. RGO functionalized with DES shows better selectivity and reusability thus providing a potential ministration for the technologies of water treatment [59].

### 2.7.3 Sensors and Biosensors

Graphene derivatives have high surface area to volume ratios and outstanding electron mobility, which make them very suitable for sensing applications. The functionalized RGO is employed for glucose sensors, DNA detectors, as well as gas sensors [60]. DES-modified graphene improves the sensitivity and the selectivity through the creation of the selective binding sites and the better dispersion in the sensor matrices.

### 2.7.4 Biomedical Applications

RGO that is combined with green synthesis inherits the advantages of non-toxicity and surface functional groups that make it suitable for applications in drug delivery, bioimaging, and tissue engineering. DES-functionalized RGO can be tailored for targeted drug delivery and antimicrobial coatings [61]. Its tunable surface properties improve drug loading efficiency and release kinetics.

### 2.7.5 Catalysis and Photocatalysis

Graphene and RGO serve as catalyst supports and active catalysts in hydrogenation, oxidation, and photodegradation reactions. DES-functionalized graphene materials enhance catalytic performance by stabilizing active sites and facilitating electron transfer [62]. Applications include dye degradation, $CO_2$ reduction, and hydrogen production.

### 2.7.6 Composite Materials

Graphene is incorporated into polymers, ceramics, and metals to enhance mechanical strength, thermal conductivity, and electrical properties. Functionalized RGO forms strong interfacial bonds with matrix materials, leading to high-performance composites for aerospace, automotive, and electronics applications [63].

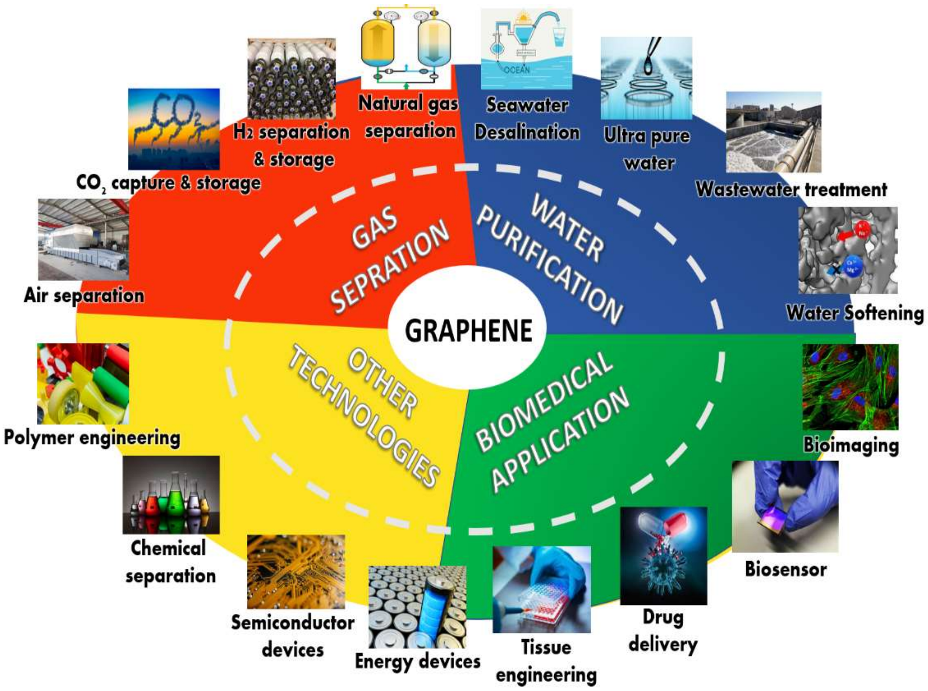


**Fig 2.1 Applications of Graphene**

## 2.8 Research Gap

Graphene and its derivatives continue to be at the forefront of material science research due to their unique physical, electrical, and chemical properties. However, the traditional routes to synthesizing these materials, particularly reduced graphene oxide (RGO), still largely rely on toxic chemicals such as hydrazine or sodium borohydride.

These chemicals pose serious environmental and health risks, making them unsuitable for large-scale or biomedical applications [64].

While plant-based phytoextracts have recently been explored as green alternatives for reducing GO, there remains a lack of consensus on the optimal conditions, efficiency, and reproducibility of such reduction methods. Furthermore, most studies focus narrowly on the reduction aspect without integrating it into a broader sustainable synthesis and functionalization framework [65-66].

Another underexplored area is the use of Deep Eutectic Solvents (DES) for the functionalization of graphene. DES are recognized for their green chemistry credentials—biodegradability, low toxicity, and easy preparation—yet their potential for modifying graphene's surface chemistry remains insufficiently characterized [67]. There is also limited data on how DES influence the stability, dispersion, and reactivity of RGO in real-world applications [68].

In addition, existing literature lacks comprehensive characterization using multiple techniques such as UV-Vis, FTIR, and TGA in a single integrated green synthesis pipeline. Most studies tend to assess only one or two aspects, which limits the holistic understanding of structure–property relationships [69].

Finally, there is a scarcity of research that links the green functionalization of Graphene using DES to specific application enhancements in areas like sensors, catalysis, or biomedicine. This disconnect highlights a critical opportunity for research to not only develop sustainable methods but also validate their practical impact [70].

Thus, this thesis addresses these gaps by:

- Employing a phytoextract-mediated, non-toxic approach for GO reduction.
- Utilizing DES for post-synthesis functionalization.
- Conducting comprehensive characterization.
- Evaluating the functional properties of the final product, creating a sustainable and application-driven roadmap for graphene synthesis.

## 2.9 Objectives

The overarching objective of this research is to develop a sustainable and environmentally benign approach for the synthesis and functionalization of graphene using green chemistry principles. Specifically, the study aims to:

1. Synthesize Graphene oxide (GO) using a modified Hummer’s method that minimizes the use of hazardous reagents and enhances oxidation efficiency.
2. Reduce GO to reduced Graphene oxide (RGO) employing plant-based phytoextracts as green reducing agents, thereby replacing toxic conventional chemicals with biodegradable, natural alternatives.
3. Characterize the synthesized RGO using UV-Visible spectroscopy, Fourier Transform Infrared Spectroscopy (FTIR), and Thermogravimetric Analysis (TGA) to confirm successful reduction and structural restoration.
4. Synthesize Deep Eutectic Solvents (DES) using non-toxic components and evaluate their potential as green functionalizing agents for RGO.
5. Functionalize RGO using DES to enhance its physicochemical properties and application potential, with structural confirmation provided by FTIR spectroscopy.
6. Discuss the green-synthesized and DES-functionalized graphene materials with the potential to be used in the environmental, electrochemical, and biomedical fields.

Combining green synthesis methods with advanced material characterization, this study is a part of the sustainable nanotechnology community and it paves the way for the development of environmentally friendly graphene-based materials.

# CHAPTER 3

# EXPERIMENT

## 3.1 Synthesis of Graphene Oxide and Its Reduction Using Phytoextract

The graphene oxide (GO) synthesis was done in a laboratory using a modified Hummer's method. At the beginning, natural graphite powder and sodium nitrate ($NaNO_3$) were carefully added into 50 mL of 80% concentrated sulfuric acid ($H_2SO_4$) in a conical flask. The flask was put into an ice bath and the mixture was stirred vigorously to ensure uniform dispersion. After a short time, the mixture became a thick black slurry. At this point, potassium permanganate ($KMnO_4$) was slowly added to the slurry while the temperature was kept under 20°C to prevent overheating and possible thermal runaway.

Then the reaction was allowed to go on for about 4 hours with continuous stirring after the addition of $KMnO_4$. At the end of this time, the mixture was gradually diluted with distilled water and heated at 70 to 80°C for 2 hours to improve oxidation. To stop the reaction and neutralize the medium, more water was added and then hydrogen peroxide ($H_2O_2$) was slowly introduced. The colour of the mixture went from brownish-black to bright yellow, which confirmed that graphene oxide was formed. The mixture was thoroughly rinsed with distilled water to get rid of any ions and by-products. The homogenous slurry of pure graphene oxide was obtained after the mixture was filtered and dried and it was stored for further use.

Furthermore, the process of turning GO to reduced graphene oxide (RGO) was done by means of a natural and eco-friendly method that is based on plant extracts. The roots of wild carrot were selected to be the reducing agent. The roots were washed, chopped, and then they were added to the GO dispersion that was made in deionized water. This mixture was kept at room temperature initially and then heated gently for about 48 hours. Over time, a noticeable change in the color of the dispersion was recorded, transitioning from yellowish-brown to a darker hue, indicating the successful reduction of GO to RGO by the phytochemicals present in the wild carrot root.

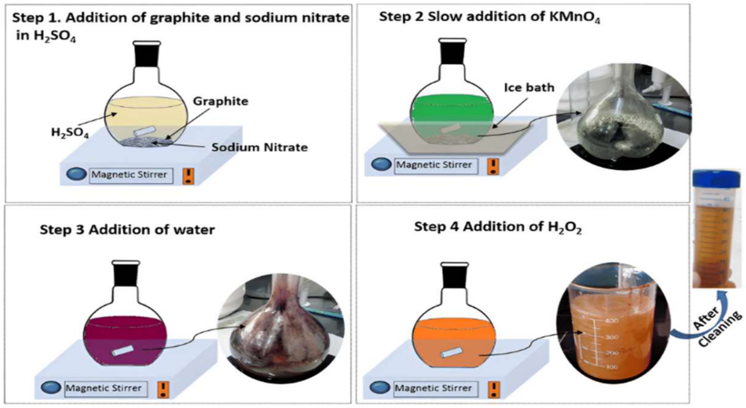


**Fig 3.1 Modified Hummer's method for GO synthesis**

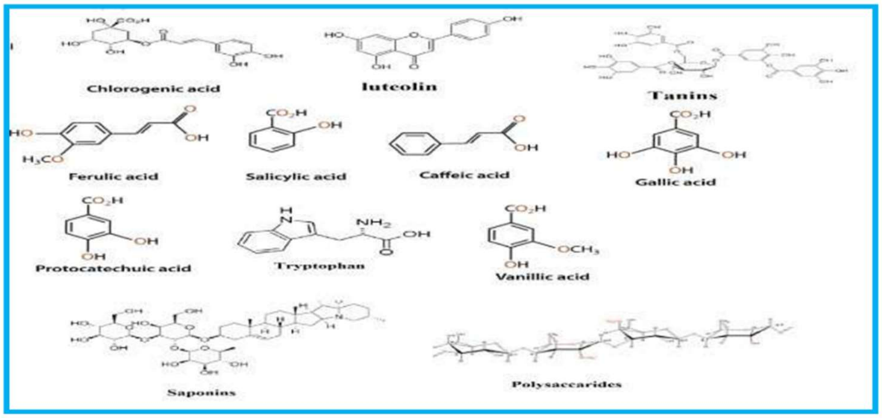


**Fig 3.2 A variety of bioreducing compounds derived from diverse natural green reducing agents. (Picture Courtesy: Google Images)**

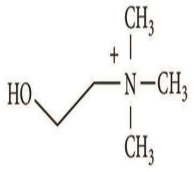
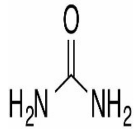
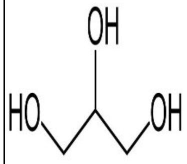
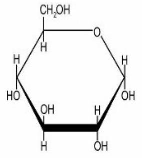
### 3.2 Synthesis of DES

Before initiating the synthesis process, all solid reactants were carefully dried in a vacuum oven maintained at 60 °C to eliminate any residual moisture. The chosen salts and hydrogen bond donors in the specific molar ratios outlined for each formulation were mixed. The temperature of the reaction vessel was set and maintained at a minimum of 70 °C throughout the synthesis. This temperature facilitated the uniform fusion of the components and ensured complete formation of the eutectic mixture. The reaction proceeded at atmospheric pressure, and stringent moisture control was implemented to prevent unwanted water absorption during mixing. Each mixture was stirred continuously, and the reaction was typically completed within three hours.

Once the mixtures turned into clear, homogenous liquids, they were immediately transferred into airtight, amber-colored vials to avoid any potential degradation due to light exposure or atmospheric moisture. We ensured that freshly prepared DES samples were used for all characterization and subsequent experiments to eliminate the possibility of contamination or chemical alteration over time.

In total, five distinct DESs were synthesized. These combinations were carefully chosen to explore the influence of different component chemistries on the physical and functional properties of the DESs. A detailed summary of the components used, their molar compositions, and the shorthand notations employed for each DES formulation is presented in Table 3.2.

**Table 3.1. Summary of the reagents utilized, including their molecular formulas and designated short forms.**

| Name of material | Abbr. | Formula (Structure) | Name of material | Abbr. | Formula (Structure) |
|---|---|---|---|---|---|
| Choline chloride | ChCl |  | Urea | U |  |
| Glycerol | Gly |  | D-(+)-Glucose | Glu |  |

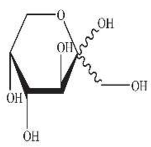
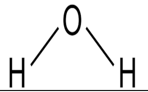
| | | | | | |
|---|---|---|---|---|---|
| Ethylene glycol | EG |  | D-(–)-Fructose | Fru |  |
| | | | Water | W |  |

**Table 3.2 List of the deep eutectic solvents synthesized, with associated molar ratios and abbreviated labels.**

| DES | Molar Ratio (salt : HBD ) | Abbreviation |
|---|---|---|
| ChCl:Gly | 1:2 | DES 1 |
| ChCl:EG | 1:2 | DES 2 |
| ChCl:U | 1:2 | DES 3 |
| Glu:ChCl | 2:5 | DES 4 |
| ChCl:Fru | 5:2 | DES 5 |

### 3.3 Functionalization of Graphene using DES

For the functionalization process, 200 mg of the pre-oxidized graphene obtained from the above procedure was weighed and transferred to a clean glass vial. To this, 0.7 mL of a selected Deep Eutectic Solvent (DES) was added. The mixture was then placed in an ultrasonic bath maintained at 60 °C and sonicated for 3 hours to facilitate surface interaction and bonding between the graphene and the DES components.

After sonication, the suspension was filtered under vacuum, and the retained solid was thoroughly washed with distilled water until the effluent was completely transparent and exhibited a neutral pH comparable to the influent water. This ensured the removal of any loosely bound DES residues.

A total of 5 different DESs were employed in separate experiments to evaluate their efficiency in functionalizing graphene.

Once filtration was complete, the functionalized graphene samples were dried in ambient air at 100 °C for a minimum of 3 hours. The dried materials were then transferred to a clean, airtight desiccator and allowed to cool down to room temperature. These prepared samples were subsequently used for further characterization.

# CHAPTER 4

# RESULT AND DISCUSSION

## 4.1 UV spectra of GO and RGO

The UV–Visible spectroscopy results provided clear insights into the optical behavior of the synthesized samples. Initially, the spectrum of graphene oxide (GO) displayed a prominent absorption peak near 230 nm, which corresponds to the π–π* electronic transitions of aromatic C–C bonds. A weaker shoulder peak around 301 nm was also observed, likely arising from n–π* transitions associated with the carbonyl (C=O) functional groups present in GO.

Following the reduction process using the plant extract, a noticeable shift in the main absorption peak was detected. The characteristic peak at 230 nm gradually shifted toward higher wavelengths, stabilizing around 280 nm. This red shift signifies a major change in the electronic structure, hence indicating the reduction of GO to RGO. The red shift also indicates the partial restoration of the $sp^2$-hybrided carbon network which is the main feature of the reduction process.

Hence UV–Vis spectral analysis is one of the methods that show the reduction of GO to RGO. The spectral changes registered in the spectra are a direct reflection of the transformation and the reactivation of the conjugated π-system thus, it is consistent with the behavior that occurs during the successful reduction process.

## 4.2 FTIR spectra of GO and RGO

Firstly, to reverify the chemical transformation consistent with the reduction process, the FTIR analysis was undertaken in the case of graphene oxide (GO) to the reduced graphene oxide (RGO) substances. The graph of GO unveiled various characteristic absorption ranges that directly indicated the existence of oxygen-containing functional groups. Most noticeably, a sharp signal near 1720 $cm^{-1}$ was recorded, which relates to C=O vibrations of the carbonyl or carboxylic groups. Further, the signals at 1204 $cm^{-1}$ and 1049 $cm^{-1}$ were in the first place assigned to C–O–C (epoxy) and C–O vibrations, correspondingly. Also, a wide signal in the neighbourhood of 3400 $cm^{-1}$ was registered, which is normally related to the –OH vibrations that imply the existence of the hydroxyl groups in GO.

Upon treating GO with the plant extract reduction, the FTIR chart showed various modifications of a high degree. In particular, the high-intensity bands that corresponded to the oxygen functionalities, mainly for the C=O and C–O groups, were at most largely reduced or totally missing. The wide –OH band at 3400 $cm^{-1}$ was also much weaker, which meant that the amount of hydroxyl had been reduced. These spectral variations confirm the main idea of the deep elimination of oxygen-containing parts and the general process of the reduction of GO being carried out.

The implication is that the reduction has not been entirely rid of the oxygen functionalities. The leftover existence of some oxide groups, though highly reduced, points to a partial but considerable reduction, which is a common aspect in phytochemical-based processes. The FTIR results first and foremost strong evidence of the deoxygenation of GO, the fact that the plant extract is the most suitable reducing agent that can be compared to the $sp^2$ carbon network in graphene, and therefore the solving of the graph.

### 4.3 TGA graph

The thermal properties of the graphene-based products which were synthesized in the experiment were experienced for their stability under heat. The thermogravimetric analysis (TGA) was a direct comparative between graphene oxide (GO) and the reduced form (RGO) and also identified the structural differences that arise after the reduction process.

The TGA curve of GO clearly indicated a two-stage mass loss pattern. The loss of mass was initiated at approximately 175 °C to 190 °C which we corresponded to the experiment that the only contribution to this loss is from the physically adsorbed water and the decomposition of the oxygen-containing functional groups that are the most the hydroxyl and epoxy groups. This process thus far has contributed to the loss of mass in the range of 12-15%. With the rising temperature, it was found that at about 450 °C, the second step of the mass loss, being the highest, occurred. This observation was explained as the disappearance of very stable oxygen functionalities that were still present in the GO structure. The process of the mass loss was necessary in reaching the 45% of the thermal decomposition of GO. The aforementioned thermal processes point out to the abundant oxygen groups in GO which consequently lead to low thermal stability.

On the contrary, the RGO contract was depicted with a much more stable heat profile. Just a very small loss of weight which was about 8-9% was detected at 250°C and it was far less in magnitude than the case of GO. This shows that the reduction process was most likely to be responsible for the removing of oxygen groups, thus improving the thermal resistance of the material. The lower percentage of degradation is also in agreement with the re-formation of the conjugated carbon network which results in a higher integrity of RGO when it is exposed to high temperatures.

Further analysis indicated that the significant degradation in GO was mainly the result of the production of gases like CO, $CO_2$ and water vapor, as well as the oxidative decomposition of the carbonaceous matter that was left. In the instance of RGO, the lesser number of these easily removable oxygen groups was the main reason for the improved thermal stability of RGO.

The thermal analysis showed that RGO has higher thermal stability than GO which is a clear demonstration that the reduction process is effective in improving the material's structural integrity and in increasing its capability for hot applications.

### 4.4 FTIR analysis of functionalized Graphene using DSE

FTIR spectroscopy provides valuable insight into the chemical modifications that occur when graphene is functionalized using deep eutectic solvents (DESs). The appearance and shifts in specific infrared absorption bands help in identifying the nature of the functional groups introduced or removed during treatment.

In general, DES-functionalized graphene tends to show new absorption peaks in regions typically associated with nitrogen- and oxygen-based functionalities. For instance, the broad absorption bands between 3326–3186 $cm^{-1}$ are commonly attributed to N–H stretching vibrations, indicating the presence of amine ($-NH_2$) and secondary amine ($-NH-$) groups. These features are often observed when DESs contain nitrogen-rich components such as urea or amine-based hydrogen bond donors.

Additionally, a prominent peak near 1605 $cm^{-1}$ is generally linked to in-plane N–H bending or C=N/C=C stretching within aromatic or conjugated systems. This peak suggests possible interactions between DES components and the graphene backbone, particularly when covalent bonding or π–π stacking occurs.

In samples where oxygenated groups are present or altered, peaks in the range of 1720–1740 $cm^{-1}$ typically correspond to C=O stretching vibrations, indicating the presence of carboxylic or carbonyl functionalities. These peaks may weaken or disappear entirely after DES treatment, especially if the solvent system facilitates reduction or purification of the graphene surface.

Similarly, bands between 1040–1250 $cm^{-1}$ are often linked to C–O stretching vibrations, including those from epoxy or alkoxy groups. A reduction in intensity or disappearance of these peaks is often interpreted as successful removal of surface oxygen functionalities, particularly when DESs exhibit acidic or reductive behavior.

Interestingly, certain DESs can also act as surface cleaning agents. For example, a highly acidic DES may significantly reduce the number of observable FTIR peaks, resulting in a nearly flat spectrum with minimal absorption bands. In such cases, the reduction in peaks—especially those related to O–H, C=O, and C–O bonds—can be taken as an indicator of surface purification and increased graphitic purity.

In summary, FTIR spectral analysis reveals a wealth of information regarding the functional groups introduced or removed during DES-based treatment. The peak positions, intensities, and overall spectral changes serve as direct indicators of how the graphene surface chemistry evolves through the functionalization process. This information is crucial for designing graphene materials with specific chemical, electrical, or mechanical properties tailored by the choice of DES.

# CHAPTER 5

# PROSPECTS , CONCLUSION & SOCIAL IMPACT

## 5.1 Conclusion

This thesis has disclosed an environmentally aware and reproducible method for the green creation, the reduction, and the functionalization of graphitic materials in order to provide a practical response to the rather aggressive chemical industry. The research began with the modified Hummer's method to produce graphene oxide (GO) and accomplished a more controlled and environmentally conscious oxidation process, thus being the groundwork for the further modification of the substance.

The reduction of GO to RGO by the use of the phytoextract from the wild carrot root was a demonstration of the natural and plant-based reducing agents' case and their efficiency. Emissive and heat experiments together served as oxygen groups' disappearance and the incomplete recovery of the conjugated $sp^2$ carbon network. The RGO obtained was the one with better structural stability and electrical properties which were the green reduction plan.

The functionalization of the RGO by the use of a couple of the newly synthesized DESs (Deep Eutectic Solvents) was the part of the research that was done next. The green solvents were thus the ones that could, apart from helping the binding of functionalities such as amine and carbonyls ones, also play the part of the surface cleaner and, in some cases, the ones that contributed to the increase of pure purity of the material. While changes in absorption profiles indicated the successful surface modification achieving this without the damage of the graphene framework as the FTIR spectra confirmed the introduction of new chemical moieties, the incorporation of DESs as both reaction media and functionalizing agents opens promising directions for tailoring graphene surfaces in a controlled, application-specific manner.

## 5.2 Future Prospects

While this paper explores the green synthesis routes for technical feasibility and environmental benefits, it also enables future research in different fields. One of the immediate application areas is the refinement of DES recipes to introduce chosen functional groups in a selective way thus, materials being more compatible with polymers, metals or the biological world interface.

Additionally, the systems which are compatible by nature and they are both plant extracts and DESs could likely be conceived in a single step reduction-functionality here the process of the material is not only facilitated but also the surface reactivity is increased. In addition, these hybrid methods could be the unlocking of new features by utilizing the diversity of phytoextracts and structural modify of DESs.

With regard to the actual use, the nature-based RGO materials made via this environmentally friendly pipeline represent a significant step forward in the development of high-performance energy storage systems of the future, especially as the electrode materials for supercapacitors and lithium-ion batteries, in which the surface chemistry is a critical determinant of the performance. Moreover, the materials' biocompatibility enhancement makes them a top candidate place in the biomedical area, such as drug delivery, tissue scaffolding, and biosensing.

On the other hand, although up-scaling the synthesis process to an industrial level without losing environmental friendliness and reproducibility is still a significant challenge and also an opportunity, it is very much a part of the game. Future work is expected to focus on stabilizing the materials for a longer period, making them more cost-efficient, and life-cycle assessment of these green graphene materials to help it become completely ready for market implementation besides still being research-based in laboratory settings.

To sum up, the partnership of green chemistry principles and nanoscale material science, as a case illustration in this technical paper, propels not only the advancement of graphene research but is also a significant move towards better, cleaner, and more responsible material technologies on a global scale.

### 5.3 Social Impact

There's this growing urgency to shift towards sustainable technologies, and that’s really putting green chemistry in the spotlight these days. This research is making a real difference by offering an eco-friendlier way to create and modify graphene—a material that holds so much promise in areas like energy storage and biomedical engineering.

One of the coolest things about this work is how it moves away from the hazardous chemicals that have typically been used in making graphene. Instead, it opts for wild carrot phytoextract and Deep Eutectic Solvents (DES), which are both non-toxic and biodegradable. This change not only helps reduce health risks for workers in labs but also shrinks the environmental impact of producing nanomaterials. And let’s be honest, this is especially important in places where regulations might not be as strict.

Plus, the approach in this thesis really embraces the idea of a circular economy. By using materials that come from nature and cutting down on waste, it could actually help local farming communities. Think about it—there's potential for creating demand for plant-based byproducts, which could lead to new, eco-friendly value chains and support rural livelihoods.

What’s even more exciting is that this research opens doors for technologies that are inclusive. That green-synthesized, DES-functionalized graphene could be used in affordable solutions like low-cost water purification systems, eco-friendly sensors, and biocompatible medical devices. How great would it be for under-resourced communities that struggle with access to clean water, health check-ups, or sustainable energy.

By proving that DES can be used for functionalizing graphene, this study is paving the way for safer materials in both medical and electronic devices, which is so important for ensuring public health and safety. The methods they’ve laid out here aren’t just scalable; they can be adapted for schools and research institutions all over the globe. That means future scientists can innovate while keeping environmental ethics in mind.

At the end of the day, this work isn’t just about creating a new material. It’s really a commitment to responsible science. It showcases how research that’s green, scalable, and socially relevant can play a direct role in building a more sustainable, fair, and health-focused society.

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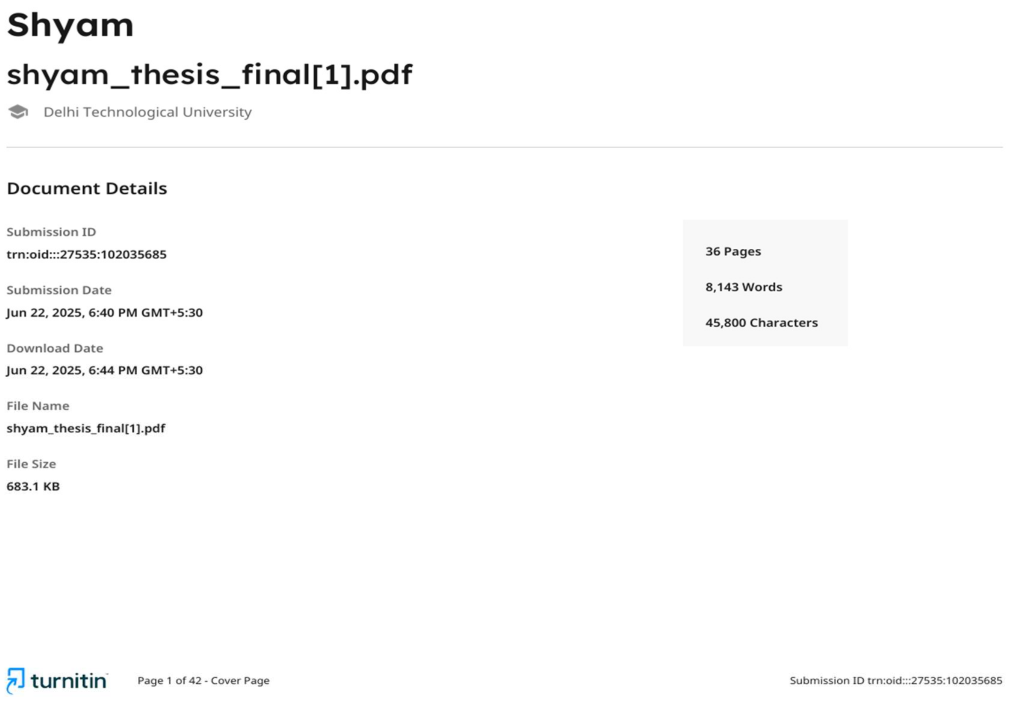
# PLAGIARISM REPORT



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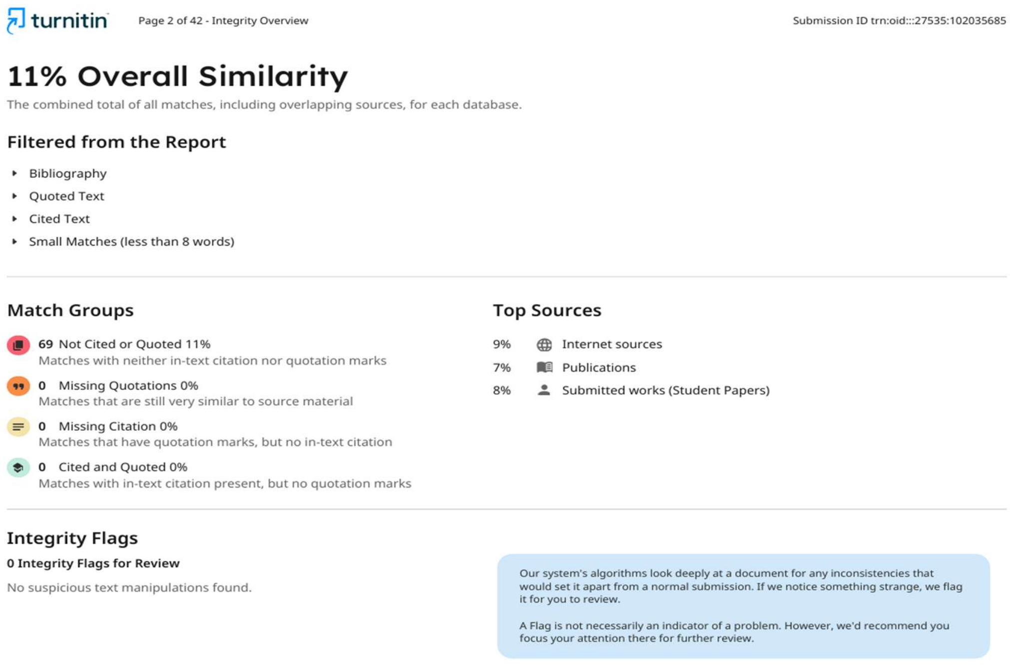

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