

# Green approach for preparation of reduced graphene oxide decorated with gold nanoparticles and its optical and catalytic properties



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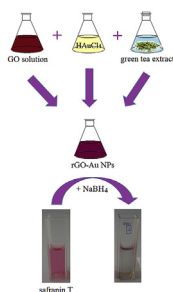
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## HIGHLIGHTS

- Reduction of GO was performed by an environmentally friendly approach.
- Gold nanoparticles were prepared by self-assembly on the graphene oxide surface.
- Surface properties were enhanced after the formation of gold nanoparticles.
- Optical properties have been changed after the graphene reduction and formation of gold nanoparticles.
- The decolorization of safranin T was observed within 60 min.

## GRAPHICAL ABSTRACT



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## ABSTRACT

Graphene oxide (GO) was reduced and modified by gold nanoparticles using aqueous leaf extract of green tea. Successful formation of gold nanoparticles (AuNPs) on graphene oxide surface was determined by scanning electron microscopy (SEM). Raman spectroscopy, X-ray photoelectron spectroscopy (XPS), and Fourier transform infrared analyses (FT-IR) have been used to demonstrate the behavior of complex of reduced graphene oxide with gold nanoparticles (rGO-AuNPs), the removal of oxygen-containing groups from the graphene, and subsequent formation of reduced graphene oxide (rGO). We also demonstrated the change of optical properties of GO after the reduction and formation of gold nanoparticles on its surface by UV–vis spectroscopy and fluorescence spectroscopy. The positive impact of rGO-AuNPs composite on safranin T reduction in the presence of NaBH<sub>4</sub> without light irradiation was examined, as well. The dye decolorization was observed within 60 min which highlights the exceptional catalytic potential of the rGO-AuNPs.

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## 1. Introduction

Graphene, two-dimensional sheet of carbon atoms bonded through sp<sup>2</sup> hybridization, and its related derivatives, due to their unique properties, have great potential for various applications in

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many technological fields such as nanoelectronics, engineering materials, energy technology (e.g. fuel cell, supercapacitor, and hydrogen storage), sensors, and catalysis [1,2]. The high potential for graphene application resulted in increasing demand for mass production of graphene (G) and graphene oxide (GO), as well. Graphene has been prepared by a variety of techniques, including micromechanical cleavage, chemical processing, epitaxial growth [3], and so on. One of the promising chemical routes for graphene preparation is the chemical reduction of graphene oxide [4] which is a large-scale and low-cost method that includes noncovalent and covalent functionalization of reduced graphene oxide, respectively. The chemical reductants such as hydrazine, hydroquinone, sodium borohydride, sodium hydrogen sulfide, and hydroiodic acid have been investigated for the chemical reduction of graphene oxide [5–7], but most of them are toxic, corrosive, and highly explosive. Therefore, nontoxic or natural products that offer effective and harmless approaches to the reduction of GO are currently under investigation. Recently, the application of some natural reducing agents such as leaf extract [8,9], wild carrot root [10], saccharides [11], vitamin C [4,12], amino acids [12,13], and bacteria [14,15] for reduction of GO has been studied.

Graphene-based materials have already been employed as a substrate for supporting various nanoparticles (Ag, Au, ZnO, CuO, TiO<sub>2</sub>, and Fe<sub>3</sub>O<sub>4</sub>) [16–21]. Recent studies have shown that prepared hybrid materials were characterized by new properties as a result of combination of behavior of both materials which lead to the application in different fields. A number of methods have been reported for synthesis of nanoparticles-graphene composites which are usually based on the simultaneous reduction of the metal salt and GO. NaBH<sub>4</sub> and hydrazine hydrate are the most widely used reducing agents for the synthesis of the composite of metal nanoparticles and graphene. Due to their harmful effect on the human health and environment, the search for an efficient, less toxic, and eco-friendly reducing agent is of great significance for the preparation of nanoparticle-graphene materials. Recently, the preparation of nanoparticles assembled on graphene has been demonstrated in the presence of tannic acid [22], citrate [23], saccharides [24], and gallic acid [25].

In this study, graphene oxide decorated with gold nanoparticles was prepared by green and facile approach at room temperature using tea phytochemicals (especially polyphenols as the most effective contribution [26,27]) as a reducing agent and chemically exfoliated graphite oxide and HAuCl<sub>4</sub>·3H<sub>2</sub>O as precursors. The morphology of prepared composite and successful formation of gold nanoparticles on GO surface was characterized by SEM, Raman and FT-IR spectroscopy. The impact of gold nanoparticles on optical properties of GO was determined, as well. We also demonstrated that rGO-AuNPs composite can be used as an efficient catalyst for safranin T (chemical structure is presented in Fig. 1) reduction

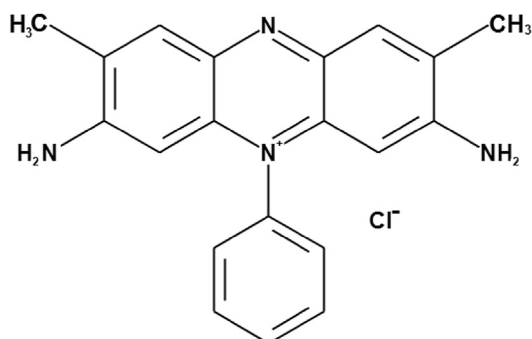


Fig. 1. Chemical structure of safranin T.

reaction.

## 2. Materials and methods

Graphite powder was purchased from NGS Naturgraphit GmbH (Germany). Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>, 95.0–98.0%), potassium permanganate (KMnO<sub>4</sub>, ≥99.0%), sodium nitrate (NaNO<sub>3</sub>, ≥99.0%), hydrogen peroxide solution (H<sub>2</sub>O<sub>2</sub>, ≥30%), gold(III) chloride trihydrate (HAuCl<sub>4</sub>·3H<sub>2</sub>O, ≥99.9%), sodium borohydride (NaBH<sub>4</sub>, 99.99%), and safranin T (C<sub>20</sub>H<sub>19</sub>ClN<sub>4</sub>) were purchased from Sigma Aldrich (USA). Ultrapure water was used throughout the course of investigation.

For the tea extract solution, the green tea powder (95% purity) was purchased in the local market. 10 mL of ultrapure water heated to 80 °C was added to the 1.2 g of green tea powder. After 5 min, the solution was filtered through a 0.22 μm cellulose membrane and cooled to room temperature.

### 2.1. Synthesis of rGO-AuNPs composite

GO was prepared from graphite powder by a modified Hummers method [28]. In our synthesis, 0.5 g of graphite and 0.25 g of NaNO<sub>3</sub> were added into 11.5 mL of H<sub>2</sub>SO<sub>4</sub>. The mixture was kept below 10 °C using an ice bath, and 1.5 g of KMnO<sub>4</sub> was slowly added into the mixture. Temperature was increased to 15 °C and the mixture was stirred for 45 min and then heated to 30 °C and stirred for another 90 min. After that, 80 mL of ultrapure water was slowly added into the mixture during a period of 30 min to keep temperature below 50 °C. Finally, 60 mL of water and 8 mL of 30% H<sub>2</sub>O<sub>2</sub> were added into the mixture to stop the reaction.

The obtained sample was collected by centrifugation (Minispin Eppendorf; 30 min, 12 000 rpm, 25 °C) and washed by ultrapure water several times for removing of unexploited graphite and subsequently dried at 80 °C. Finally, the purified graphene oxide was sonicated at frequency of 20 Hz and power of 600 W (VibraCell VC600, Sonics & Materials, USA) for 30 min at concentration of 1 mg mL<sup>-1</sup> to obtain the GO solution for further use.

12.5 mL of 1 mM HAuCl<sub>4</sub> solution was added to the 25 mL of GO suspension and mixed together by vortex mixer (IKA Werke, Germany) for 2 min. Subsequently, 1 mL of tea extract was added drop by drop to the resulting solution and left in darkness for 24 h. Finally, the mixture was collected by centrifugation (Eppendorf 5804R centrifuge; 5 min, 15 000 rpm, 25 °C) and washed with ultrapure water several times to remove unassembled nanoparticles and then redispersed in 25 mL of ultrapure water.

The whole process of reduction of GO and subsequent formation of gold nanoparticles on its surface is illustrated in Fig. 2.

### 2.2. Characterization of rGO-AuNPs

The successful formation of gold nanoparticles on the graphene oxide surface, their morphology and size was examined by means of Scanning Electron Microscope LYRA 3 XMU (Tescan, Czech Republic) operated under an accelerating voltage of 30 kV.

Raman spectra were excited using several traps to trap of micro graphene particles and complexes of graphene with gold nanoparticles to reduce the mobility particles, respectively. The trapping was performed using a homemade optical tweezer (OT) composed of Spatial Light Modulator X10468-03 (Hamamatsu, Japan) and trapping fiber laser IPG YLM-10-LP-SC (IPG Photonics, Japan) with maximal output power of 10 W at wavelength of 1070 nm. OT cooperates with a micro-Raman spectrometer Shamrock SR 303i (Andor, England) and low noise camera Newton DU970P (Andor, England) cooled at -80 °C. 532 nm laser Torus 532 (Laser Quantum, England) with a 320 mW maximal output power was used to the

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