

# Morphological, Structural and Optical Studies of Graphene Oxide Thick Films Fabricated by Screen Printing

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## Abstract:

Graphene oxide (GO) thick films were successfully fabricated on glass substrates using a simple and cost-effective screen-printing technique and systematically investigated for their morphological, structural and optical properties. Scanning electron microscopy revealed a crumpled, wrinkled, and flake-like morphology with loosely stacked GO sheets, forming a porous surface network. The specific surface area measured by the Brunauer–Emmett–Teller (BET) method was found to be  $3.29 \text{ m}^2 \text{ g}^{-1}$ , indicating partial restacking of GO layers in the thick-film configuration. Energy-dispersive X-ray spectroscopy confirmed the presence of carbon and oxygen elements, verifying the successful formation of graphene oxide without detectable impurities. X-ray diffraction analysis showed a characteristic (001) diffraction peak of GO, consistent with JCPDS data, confirming the layered structure with expanded interlayer spacing. The average crystallite size, calculated using the Debye–Scherrer equation, was found to be 65.84 nm. Fourier transform infrared spectroscopy revealed prominent oxygen-containing functional groups such as hydroxyl, epoxy, carbonyl, and alkoxy groups, confirming the oxidized nature of the GO films. UV–Visible absorption studies exhibited a strong absorption peak in the ultraviolet region attributed to  $\pi$ – $\pi^*$  transitions, along with a shoulder corresponding to  $n$ – $\pi^*$  transitions of C=O bonds. The optical band gap estimated using the Tauc method indicated the semiconducting nature of the GO thick films. The combined results demonstrate that screen-printed graphene oxide thick films possess suitable structural and optical characteristics for potential applications in optoelectronics, sensing, and energy-related devices.

**Keywords:** Graphene oxide; Thick films; Screen-printing technique; Structural characterization; Optical properties; BET surface area; Semiconducting materials.

## 1. INTRODUCTION

Graphene is a two-dimensional (2D) carbon nanomaterial composed of a single layer of  $sp^2$  hybridized carbon atoms arranged in a hexagonal lattice, has attracted tremendous attention due to its extraordinary electrical, mechanical, thermal and optical properties. Since its first successful isolation in 2004, graphene has emerged as one of the most promising materials for advanced electronic, sensing, and energy applications [1]. However, the large-scale production and processability of pristine graphene remain challenging because of its hydrophobic nature and strong tendency to agglomerate. To overcome these limitations, graphene oxide (GO) is an oxidized derivative of graphene containing abundant oxygen functional groups, has been widely investigated as an alternative material with excellent dispersibility in water and other polar solvents [2]. Graphene oxide possesses a layered structure similar to graphene but

contains various oxygen-containing functional groups such as hydroxyl ( $-OH$ ), epoxy ( $C-O-C$ ), carbonyl ( $C=O$ ), and carboxyl ( $-COOH$ ) groups on its basal planes and edges. These functional groups significantly modify the physical and chemical properties of graphene oxide, making it hydrophilic and easily dispersible in aqueous solutions [3]. The presence of these oxygen functional groups also introduces tunable electronic and optical properties, enabling GO to behave as a semiconducting material. Furthermore, the large specific surface area, excellent chemical stability, and high mechanical strength of GO make it suitable for numerous technological applications including gas sensors, optoelectronic devices, energy storage systems, catalysis, transparent conductive films, and environmental monitoring [4]. Among various fabrication methods for thin and thick films, the screen printing technique has gained considerable interest due to its simplicity, cost-effectiveness, scalability, and suitability for mass production. Screen printing is a versatile deposition technique widely used for the fabrication of functional films in electronic devices, sensors, and photovoltaic applications. The method allows the deposition of uniform films with controlled thickness over large areas using a thixotropic paste containing the active material [5]. In comparison with other thin-film fabrication techniques such as chemical vapor deposition, spin coating, and vacuum deposition, screen printing offers advantages such as low processing cost, ease of fabrication, and compatibility with various substrates including glass, ceramic, and flexible materials. These advantages make screen printing particularly attractive for the development of thick films used in sensing and optoelectronic applications [6, 7].

Graphene oxide thick films prepared using screen printing techniques provide a porous and interconnected network structure that enhances surface-related phenomena such as adsorption, charge transfer, and catalytic reactions [8, 9]. The morphology of GO films typically consists of wrinkled and crumpled sheets that stack loosely, forming a high surface area structure. Such structural features play an important role in determining the functional performance of the material, especially in applications such as gas sensing and photodetection [10, 11]. Therefore, detailed investigation of the morphological characteristics of GO thick films is essential for understanding their surface properties and optimizing their performance in practical applications.

In recent years, several researchers have explored graphene oxide-based materials for various technological applications including chemical sensors, biosensors, flexible electronics, and energy storage devices [11]. The combination of excellent electrical conductivity (after reduction), tunable band gap, and high surface reactivity makes GO a promising material for next-generation functional devices [12]. However, the performance of GO-based devices strongly depends on the fabrication technique and the resulting microstructure of the films. Hence, it is essential to study the fundamental morphological, structural, and optical properties of GO films prepared by scalable fabrication methods [12, 13].

In the present work, graphene oxide thick films were fabricated on glass substrates using a simple screen-printing technique. The prepared films were systematically characterized to investigate their morphological, structural, and optical properties. Scanning Electron Microscopy (SEM) was used to study the surface morphology of the films, while Energy Dispersive X-ray Spectroscopy (EDS) was employed to confirm the elemental composition. The crystalline structure of the GO films was analyzed using X-ray Diffraction (XRD), and the functional groups present in the material were identified using Fourier Transform Infrared (FTIR) spectroscopy. The optical properties of the films were investigated using UV-Visible spectroscopy, and the optical band gap was estimated using the Tauc method. Additionally, the specific surface area of the films was determined using the Brunauer-Emmett-Teller (BET) method. The comprehensive investigation of these properties provides valuable insight into the structure-property relationship of screen-printed graphene oxide thick films. The results of this study may contribute to the development of GO-based materials for potential applications in sensing, optoelectronics and energy-related devices.

## 2. EXPERIMENTAL WORK

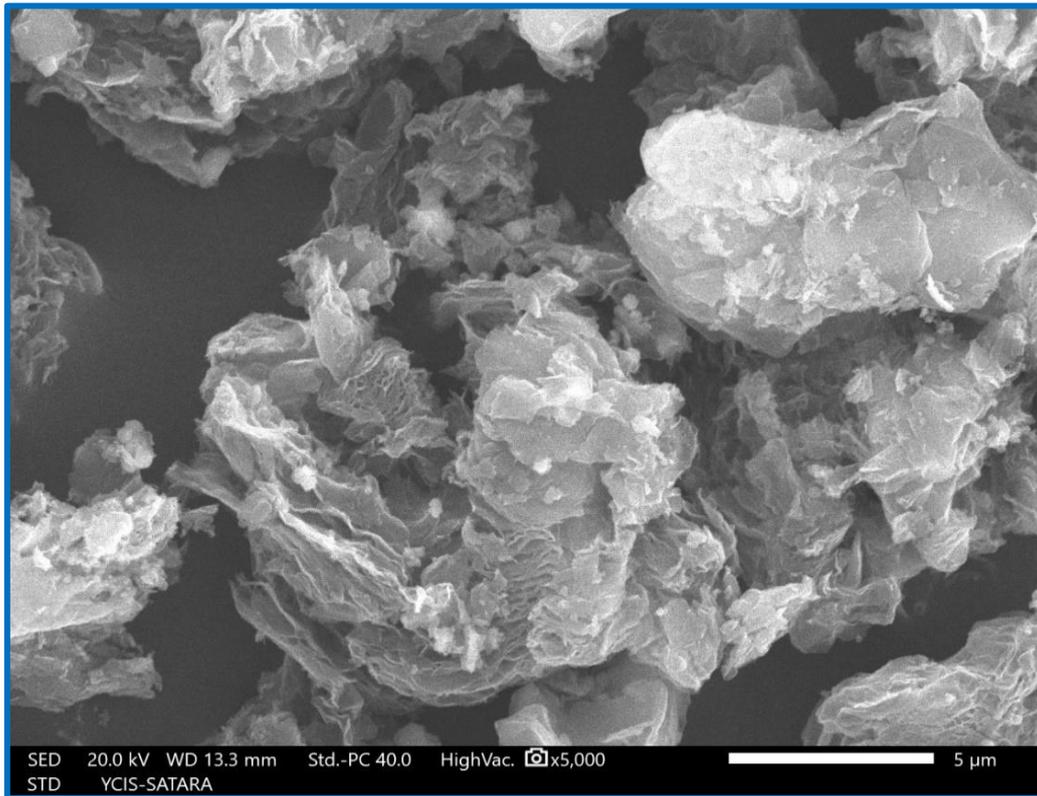
The thick films of graphene oxide were prepared using the screen-printing technique due to its simplicity, reproducibility and suitability for large-area film fabrication. Commercially available graphene oxide powder was used as the starting material for the preparation of thick films. In order to prepare the printable paste, the GO powder was mixed with a suitable organic binder system consisting of ethyl cellulose (EC) as a binder and butyl carbitol acetate (BCA) as a solvent to obtain a thixotropic paste with appropriate viscosity for screen printing [14-17]. The mixture was thoroughly ground in an agate mortar for sufficient time to ensure homogeneous dispersion of GO particles and uniform consistency of the paste. The prepared thixotropic paste was then deposited onto clean glass substrates using a stainless-steel screen with a suitable mesh size. During the screen-printing process, the paste was forced through the patterned mesh using a rubber squeegee, resulting in the formation of a uniform thick film on the substrate surface. After printing, the films were allowed to dry at room temperature for a 30 minutes to evaporate the excess solvent. Then, the printed films were calcinated at 150 °C for 2 hrs in the muffle furnace to remove the remaining organic components and to improve the adhesion of the film to the substrate [17].

The prepared graphene oxide thick films were then used for further characterization studies. The morphological characteristics of the films were examined using scanning electron microscopy (SEM), while energy dispersive X-ray spectroscopy (EDS) was used to confirm the elemental composition. The structural properties were analyzed using X-ray diffraction (XRD), and the functional groups present in the GO films were identified using Fourier transform infrared (FTIR) spectroscopy. The optical properties of the films were investigated using UV–Visible spectroscopy. These characterization techniques were employed to understand the morphological, structural and optical behavior of the screen-printed graphene oxide thick films.

## 3. RESULT AND DISCUSSION

### 3.1 Scanning Electron Microscopy

Figure 1 shows the SEM micrograph of the graphene oxide thick films prepared by the screen-printing technique. The micrograph reveals a highly wrinkled, crumpled, and flake-like morphology composed of randomly oriented graphene oxide sheets. These sheets appear loosely stacked with visible intersheet gaps and folded edges, which originate from the exfoliation of graphite during oxidation and subsequent film formation [18, 19]. The observed morphology is characteristic of graphene oxide and arises due to the presence of oxygen-containing functional groups that prevent complete restacking of the graphene layers. The overlapping GO flakes form a porous network with interconnected voids distributed throughout the film surface. However, compared to GO powders or aerogel-like structures, the degree of porosity in the thick film is relatively moderate [19, 20]. This is attributed to partial restacking and agglomeration of GO sheets during paste preparation, screen printing, and drying processes.

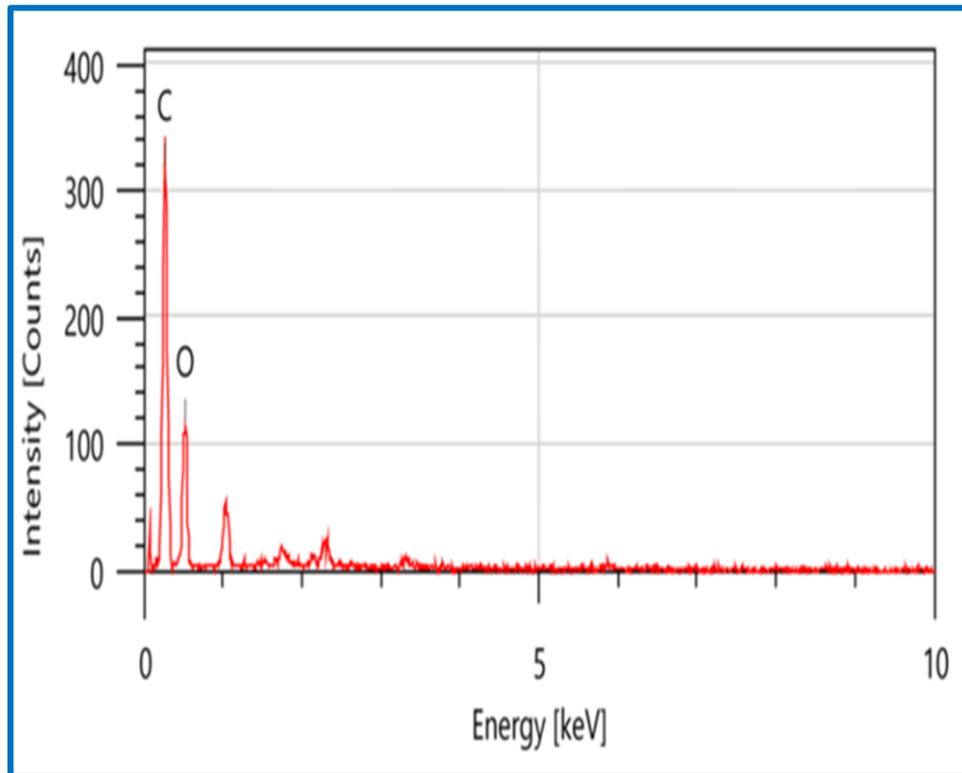


**Fig. 1.** SEM micrograph of GO thick films

The mechanical compaction inherent to thick-film fabrication tends to reduce accessible surface area by limiting the availability of internal pores. The specific surface area of the GO thick films, measured using the Brunauer–Emmett–Teller (BET) method, was found to be  $3.29 \text{ m}^2 \text{ g}^{-1}$ . This comparatively low surface area, relative to highly exfoliated GO powders, confirms the dense packing and layered assembly of GO sheets in the thick-film configuration. The reduction in surface area is mainly caused by interlayer interactions, van der Waals forces, and hydrogen bonding between oxygen functional groups, which promote sheet restacking during film formation [20, 21]. Despite the moderate BET surface area, the wrinkled morphology and exposed sheet edges observed in the SEM micrograph provide a significant number of active sites on the film surface. These structural features are advantageous for applications such as sensing and optoelectronics, where surface reactivity and charge transport pathways are more critical than extremely high surface area. The combined SEM and BET results demonstrate that the screen-printed GO thick films possess a stable, porous morphology with sufficient surface accessibility, making them suitable for practical device fabrication [15, 17].

### 3.2 Energy-dispersive X-ray

Figure 2 presents the energy-dispersive X-ray spectrum of the graphene oxide (GO) thick films fabricated by the screen-printing technique. The spectrum shows two prominent peaks corresponding to carbon (C) and oxygen (O), confirming that the films are primarily composed of graphene oxide. The strong carbon peak originates from the  $\text{sp}^2$ -hybridized carbon framework of graphene sheets, while the oxygen peak indicates the successful incorporation of oxygen-containing functional groups during the oxidation process.



**Fig. 2.** EDX spectrum of GO thick films

The presence of oxygen in the EDX spectrum provides direct evidence for the functionalization of the graphene lattice with groups such as hydroxyl ( $-\text{OH}$ ), epoxy ( $\text{C}-\text{O}-\text{C}$ ), carbonyl ( $\text{C}=\text{O}$ ), and carboxyl ( $-\text{COOH}$ ), which are characteristic of graphene oxide. The relative intensity of the oxygen peak suggests a significant degree of oxidation, which disrupts the extended  $\pi$ -conjugation of pristine graphene and contributes to the semiconducting behavior observed in optical studies [18, 20]. No additional impurity-related peaks were detected in the spectrum, indicating the high purity of the GO thick films and the absence of residual metal catalysts or contaminants. Minor signals, if any, may be attributed to the glass substrate or conductive coating used during SEM analysis.

### 3.3 X-ray diffraction

Figure 3 illustrates XRD pattern of the graphene oxide (GO) thick films prepared by the screen-printing technique, along with the corresponding standard data from the JCPDS card. The diffraction pattern exhibits a distinct and broadened peak centered at  $2\theta \approx 10.02^\circ$ , which is indexed to the (001) crystallographic plane of graphene oxide. This reflection is a characteristic signature of GO and arises due to the expansion of interlayer spacing caused by the intercalation of oxygen-containing functional groups and water molecules between the graphene layers [17, 19].

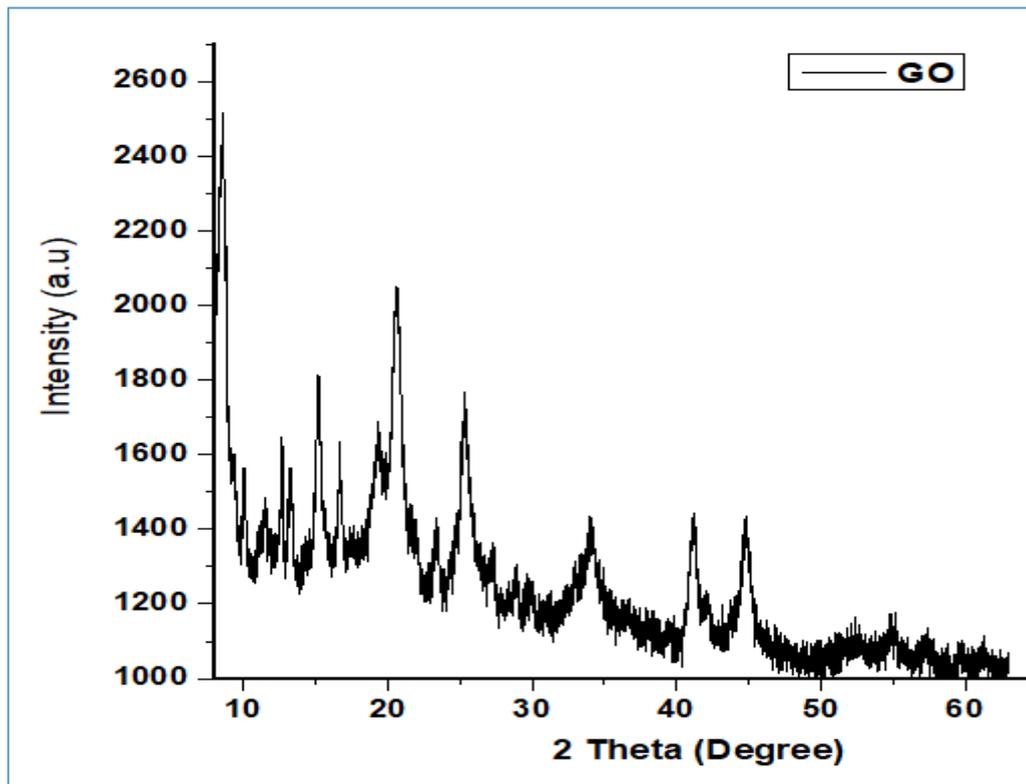
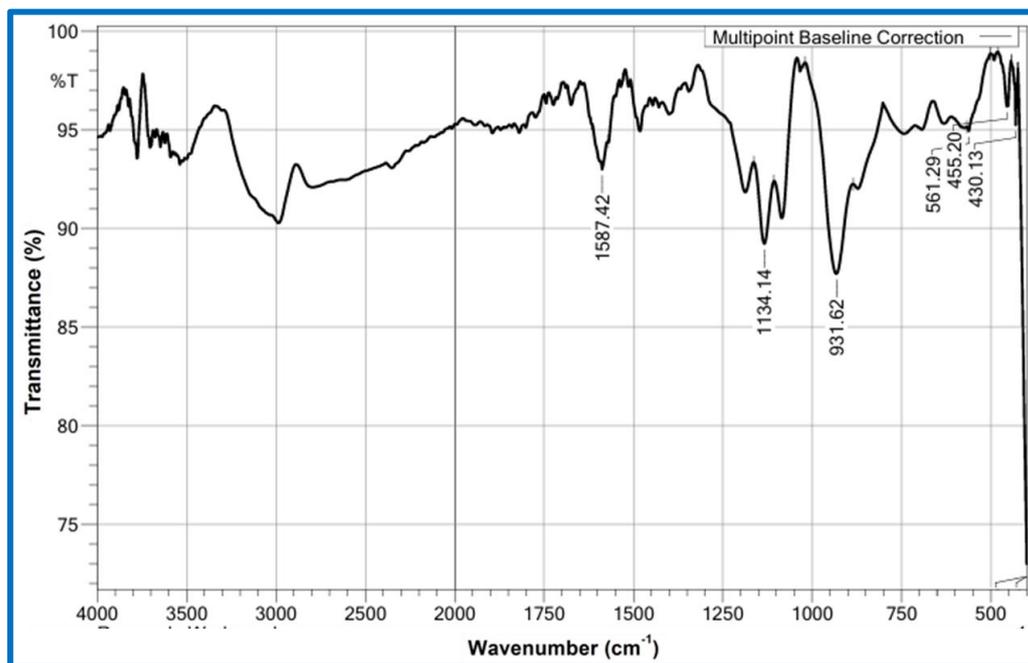


Fig. 3. XRD pattern of GO thick films

The average crystallite size ( $D$ ) of the GO thick films was calculated to be 65.84 nm using the Debye–Scherrer equation, based on the full width at half maximum (FWHM) of the dominant (001) diffraction peak. The relatively large crystallite size suggests the presence of stacked GO domains consisting of multiple graphene oxide layers with coherent scattering regions. The peak broadening observed in the XRD pattern is attributed to lattice strain, structural defects, and the presence of oxygen functional groups, which collectively reduce long-range crystallinity [20, 21].

### 3.4 Fourier transform infrared (FTIR)

Figure 4 shows the Fourier transform infrared spectrum of the graphene oxide (GO) thick films prepared by the screen-printing technique, recorded in the wavenumber range of 4000–500  $\text{cm}^{-1}$ . The spectrum clearly reveals the presence of characteristic oxygen-containing functional groups, confirming the successful oxidation and functionalization of graphene sheets [21, 22]. A broad absorption band observed in the high-wavenumber region around 3200–3500  $\text{cm}^{-1}$  is attributed to the stretching vibrations of hydroxyl ( $-\text{OH}$ ) groups and adsorbed water molecules, which are commonly present in graphene oxide due to its hydrophilic nature. This broadness indicates extensive hydrogen bonding between oxygen functional groups and interlayer water [17, 21].

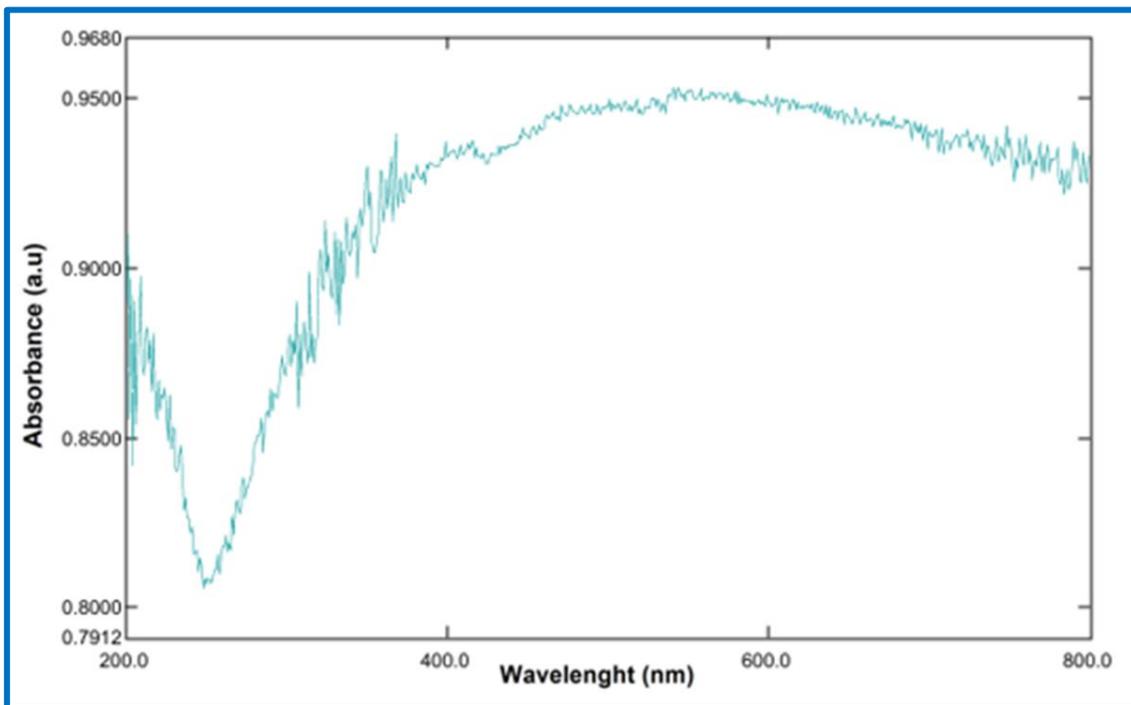


**Fig. 4.** FTIR spectra of GO thick films

The absorption band appearing near  $\sim 1580\text{--}1620\text{ cm}^{-1}$  corresponds to the skeletal vibration of aromatic C=C bonds in the  $\text{sp}^2$ -hybridized carbon framework. The persistence of this band suggests that partial graphitic domains remain intact even after oxidation, which is important for charge transport in GO-based films [14, 19]. The distinct peak around  $\sim 1700\text{--}1730\text{ cm}^{-1}$  (if present or as a shoulder) is associated with C=O stretching vibrations of carbonyl and carboxyl groups located at the sheet edges. Additionally, the band near  $\sim 1220\text{--}1260\text{ cm}^{-1}$  is assigned to C–O–C epoxy stretching, while the peak observed around  $\sim 1050\text{--}1150\text{ cm}^{-1}$  is attributed to C–O stretching vibrations of alkoxy groups [16, 20]. These bands collectively confirm the extensive functionalization of graphene layers.

### 3.5 UV-Vis spectroscopy

Figure 5 shows the UV–Visible absorbance spectrum of the graphene oxide (GO) thick films prepared by the screen-printing technique, recorded over the wavelength range of 200–800 nm. The spectrum exhibits a strong absorption peak in the ultraviolet region, typically centered around 230–240 nm, which is attributed to the  $\pi\text{--}\pi^*$  electronic transitions of aromatic C=C bonds within the  $\text{sp}^2$ -hybridized carbon network of graphene oxide [20–22]. This characteristic absorption confirms the presence of conjugated graphene domains in the GO structure.



**Fig. 5.** Absorbance versus wavelength plot of GO thick films

In addition to the main absorption peak, a noticeable shoulder or broad absorption band appears in the ~290–320 nm region. This feature is associated with  $n-\pi^*$  transitions of C=O bonds, arising from oxygen-containing functional groups such as carbonyl and carboxyl moieties introduced during the oxidation process [23, 24]. The presence of this shoulder further validates the functionalized nature of the GO thick films, in good agreement with the FTIR results. The absorbance gradually decreases with increasing wavelength toward the visible region, indicating reduced photon absorption at longer wavelengths. This behavior is characteristic of graphene oxide and reflects the disruption of the extended  $\pi$ -conjugation system due to oxidation-induced defects [24–26]. Unlike pristine graphene, which exhibits nearly wavelength-independent absorption, the GO thick films show a semiconducting optical response.

## CONCLUSIONS

Graphene oxide thick films were successfully fabricated on glass substrates using a simple and cost-effective screen printing technique. The prepared films were systematically investigated for their morphological, structural, and optical properties. The SEM analysis revealed a crumpled and wrinkled sheet-like morphology consisting of loosely stacked graphene oxide layers, which form a porous surface network. Such morphology provides a relatively large active surface area that is beneficial for surface-related applications. BET analysis indicated a specific surface area of  $3.29 \text{ m}^2 \text{ g}^{-1}$ , suggesting partial restacking of GO sheets in the thick-film configuration. The elemental composition confirmed by EDS analysis showed the presence of carbon and oxygen elements, verifying the successful formation of graphene oxide without noticeable impurities. X-ray diffraction studies exhibited a characteristic (001) diffraction peak, confirming the layered structure of graphene oxide with expanded interlayer spacing due to the presence of oxygen-containing functional groups. The average crystallite size calculated using the Debye–Scherrer equation was found to be 65.84 nm, indicating the nanoscale nature of the GO crystallites. FTIR spectroscopy further confirmed the presence of various oxygen functional groups such as hydroxyl, epoxy, carbonyl, and alkoxy groups, which are characteristic features of graphene oxide. UV–Visible spectroscopy revealed a strong absorption peak in the ultraviolet region corresponding to  $\pi-\pi^*$  transitions of aromatic C=C bonds along with a shoulder band associated with  $n-\pi^*$  transitions of C=O groups. The optical band gap estimated using the Tauc method confirmed the semiconducting behavior of the graphene

oxide thick films. The results demonstrate that screen-printed graphene oxide thick films possess suitable morphological, structural, and optical characteristics, making them promising candidates for applications in optoelectronic devices, sensors and energy-related technologies.

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