## ORIGINAL



# Design and Fabrication an optical sensor devices base on graphene oxide

# Diseño y fabricación de dispositivos sensores ópticos basados en óxido de grafeno

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#### ABSTRACT

Contamination of oil, particularly by dissolved water, is a very common problem in the failure of step-down transformers used by electricity providers as this degrades the insulating property of the oil. In this paper, the use of D-shaped optical fibers functionalized with Graphene Oxide is presented to detect the water content in transformer oil. The synthesis of graphene oxide was achieved by a modified version of Hummer's method. Subsequently, the drop-casting process was used to apply the graphene oxide onto the D-shaped fibre. The coating thickness attained in the samples was around 200 nm. Side polishing in a single-mode fiber engages an evanescent field that increases its sensitivity as an optical sensor. A few layers of graphene oxide coating on D-fiber exhibit a quick response time and high sensitivity to moisture content present in transformer oil, which proves to be a hopeful solution in real-time monitoring and maintenance of transformer insulation systems. It manifested that the experimental results had a high sensitivity to different water contents in transformer oil for the D-shaped fiber coated with GO. The GO-coated fibers exhibited a sensitivity of 0,5677 dB/ppm, which is relatively high compared with the sensitivity in the case of uncoated D-shaped fibers.

**Keywords:** Graphene Oxide; Nanolayers; Optical Sensor; Transformer Oil; Moisture Detection; D-Shaped Fiber.

#### RESUMEN

La contaminación del aceite, particularmente por agua disuelta, es un problema muy común en la falla de los transformadores reductores utilizados por los proveedores de electricidad, ya que esto degrada la propiedad aislante del aceite. En este artículo, se presenta el uso de fibras ópticas en forma de D funcionalizadas con óxido de grafeno para detectar el contenido de agua en el aceite del transformador. La síntesis del óxido de grafeno se logró mediante una versión modificada del método de Hummer. Posteriormente, se utilizó el proceso de recubrimiento por goteo para aplicar el óxido de grafeno sobre la fibra en forma de D. El grosor del recubrimiento alcanzado en las muestras fue de aproximadamente 200 nm. El pulido lateral en una fibra monomodo involucra un campo evanescente que aumenta su sensibilidad como sensor óptico. Unas pocas capas de recubrimiento de óxido de grafeno en la fibra en forma de D exhiben un tiempo de respuesta rápido y una alta sensibilidad al contenido de humedad presente en el aceite del transformador, lo que demuestra ser una solución prometedora para el monitoreo en tiempo real y el mantenimiento de los sistemas de aislamiento de transformadores. Se evidenció que los resultados experimentales mostraron una alta sensibilidad a diferentes contenidos de agua en el aceite del transformador para la fibra en forma de D recubierta con GO. Las fibras recubiertas con GO exhibieron una sensibilidad de 0,5677 dB/ppm, que es relativamente alta en comparación con la sensibilidad en el caso de fibras en forma de D sin recubrimiento.

© 2025; Los autores. Este es un artículo en acceso abierto, distribuido bajo los términos de una licencia Creative Commons (https:// creativecommons.org/licenses/by/4.0) que permite el uso, distribución y reproducción en cualquier medio siempre que la obra original sea correctamente citada **Palabras clave:** Óxido de Grafeno; Nanocapas; Sensor Óptico; Aceite de Transformador; Detección de Humedad; Fibra en Forma de D.

#### **INTRODUCTION**

The graphene oxide has a large number of studies for developing the electronic properties, thus properties have made researcher pay attention to Graphene.<sup>(1,2,3,4)</sup> Graphene is a 2D substance composed only of carbon atoms. It is sometimes referred to as a "miracle material" due to its exceptional properties.<sup>(5)</sup> The characteristics of Graphene and other Carbon-based materials have generated significant attention for a wide range of applications.<sup>(6)</sup> These properties have attracted a great deal of attention and high levels of thermal conductivity, Minimal thermal noise occurs when the material exhibits its natural electrical also mechanical characteristics. <sup>(1,7)</sup> Additional information Graphene, which was initially isolated in 2004, is 2x102 It is one atomic layer thick and has a strength that is several times greater than steel. It is also the most conductive material in the world and has high rigidity and strength.<sup>(8)</sup> Sched demonstrated that potential applications of single molecule of graphene have excellent optical absorption, highly sensitive and selective detection properties.<sup>(9,10)</sup> Researchers and organizations are actively exploring Graphene, a material that is expected to bring about significant changes in several industries by enhancing processes, improving component performance, and creating new products. <sup>(11,12,13)</sup> Initially developed for the purpose of producing Graphene on a wide scale, Graphene Oxide (GO) has shown distinct features that make it deserving of further study focus. The Graphene Oxide multilayers, which have an orderly-layered structure, may be easily made using a drop-casting approach. These multilayers possess two significant properties.<sup>(14,15)</sup> The Graphene Oxide generated in this study was synthesized utilizing a modified version of Hummer's technique The prepared Graphene exhibited 4,8 µL. The UV-Vis take place to study the linear optical properties.<sup>(16)</sup> Optical fiber sensors are now in great demand because of their distinctive features, including little signal loss, resistance to radio frequency or electromagnetic disturbances, exceptional sensitivity, and versatility in measuring different parameters.<sup>(17,18,19,20)</sup>

Many researchers utilized optical sensors to sense the water content in transformer oil and others application. M. Mezher et al.<sup>(21)</sup> In this study reported the fabrication of D-shaped fibre sensor coated by platinum nanostructures on an ITO substrate (Pt-NSs), the platinum structures were synthesized using an electrochemical method. S. Karthikeyan et al.<sup>(22)</sup> In this work, reported on moisture detection in transformer oil based on an optical waveguide fabricated using graphene oxide (GO). The SU-8 polymer channel waveguide was coated by GO film, using a drop-casting technique. measured at varying water contents.<sup>(23)</sup>

In this work, nonlinear optical response studies of thin layers using graphene oxide (GO) and their performance for detecting water content in transformer oil. The sensor performance has been evaluated based on bare optical fibre, D-shape optical fibre integrated with GO and the effects of the covering device with polydimethylsiloxane (PDMS). M. S. Mani Rajan et al.<sup>(24)</sup> This study discusses the most recent advances in photonic crystal fiber for THz applications. In particular, a D-shaped graphene-covered PCF sensor for detecting the quality of transformer oil when it is contaminated is thoroughly examined.<sup>(25)</sup> In this study, a moisture sensing technique for real-time monitoring of the moisture content in transformer oil based on an S-taper fiber structure, the results show that the transmission dip experiences the red-shifts with decreasing moisture, which could be used to correlate/trace moisture content. N. Jamali et al.<sup>(26)</sup> proposed a thin film made of graphene oxide to detect moisture levels in transformer oil and 2-FAL levels. Capacitive sensors using molecular imprinted polymerization have been proposed to detect 2-FAL levels at ppm levels. S. Ibrahim et al.<sup>(27)</sup> In this work, the fabrication of a capacitive sensor for humidity sensing using graphene oxide and polyvinyl alcohol composite material is accomplished. The graphene oxide was synthesized using a modified Hummer's method and was subjected to materials characterization such as Field Emission Scanning Electron Microscopy (FESEM) and X-Ray Diffraction (XRD). I. S. Amiri et al.<sup>(28)</sup> this research focuses on the side-polished Single-Mode Optical Fibers (SMOF) as refractive index (RI) sensor utilizing properties of Surface Plasmon Resonance. The SMOF with cladding stripped off shows a D-shaped optical fiber. Silver protected by graphene oxide (GO) as viable candidates. A few layers of GO on top of silver thin layer were applied as a material to overcome silver coating degradation process. N. Saidin et al.<sup>(29)</sup> In this study, Fiber Bragg Grating (FBG) sensor coated with graphene oxide (GO) is designed and constructed to measure temperature sensitivity. The FBG is synthesized with GO by implementing dip coating technique. Then, the bare and coated FBG sensor is tested by applying heat at the grating region of the FBG. S. Mohan et al.<sup>(30)</sup> proposed an optical fiber relative humidity (RH) sensor that exploits the intensity modulation scheme through evanescent wave absorption spectroscopy and fulfills the objective is reported here. The fiber sensor employs graphene oxide (GO) diffused silica nanostructured thin sensing film as the cladding on the centrally decade straight and uniform multimode optical fiber.

In this study, we aim to fabricate a nano-photonic sensing device by side polishing single-mode fibers to resemble D-shaped fibers. This configuration leverages the evanescent field generated by side polishing,

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exposing the surrounding fiber to create an efficient sensing region.<sup>(31,32)</sup> By coating the D-shaped fiber with a thin layer of graphene oxide, we enhance the performance of optical sensors, making them highly sensitive to moisture content in transformer oil. The goal is to develop a sensor with a fast response time and high sensitivity, capable of detecting water content in transformer oil, thus preventing transformer failures and ensuring efficient operation.

### METHOD

Graphene oxide can be synthesized by different ways of chemical oxidation, for example, Hummer's method, Staudenmaier's method, and Brodie's method.<sup>(35,36)</sup> n this study, a GO solution was prepared through a modified Hummer's method. The steps for synthesis were as follows:<sup>(33,34,35)</sup>

First, the graphite flake underwent an extensive oxidation procedure by combining Potassium Permanganate (KMnO4) with concentrated acid, like the sulphuric acid, together with the flake. The mixture has continuously agitated at ambient temperature for a duration of 3 days. Throughout this duration, the hue of the amalgamation transitioned from a deep purple green to a deep brown shade. In order to halt the oxidation process, a solution of Hydrogen Peroxide (H2O2) was used, resulting in a conspicuous change of the mixture's color to a vivid yellow hue, which signifies a significant degree of oxidation in the graphite. The graphite oxide was then subjected to many washes with Hydrochloric Acid (HCl) also deionised (DI) water until a pH level of 4-5 was attained.

The washing procedure included the separation of the liquid portion from the solid residue using a centrifuge method with a centrifugal force of 10 000 times the acceleration due to gravity (g-force). Throughout this procedure, the graphite oxide underwent exfoliation, resulting in the formation of a gel composed of Graphene Oxide. Ultimately, the gel was thinned down by adding DI water until it reached an appropriate concentration for drop-casting into a solution.<sup>(37,38)</sup>

The drop-casting procedure involves directly applying Graphene Oxide solutions onto microscope glass using a micropipette. The sample was then dehydrated at ambient temperature, while monitoring the drying process with an optical microscope. To enhance the thickness of the Graphene Oxide coating without altering the coating area, a process known as multiple-drop-casting is used. This process involves depositing minuscule amounts of solution droplets onto the identical surface. Figure 1 exhibits the appearance and structure of the synthesized Graphene oxide using a scanning electron microscope (SEM) image. The picture shows a crumpled layer with a sheet-like structure, with diameters measuring in the range of several micrometers.



Figure 1. FESEM image of Graphene Oxide

An Energy Dispersive X-Ray (EDX) study was conducted to confirm the elemental composition of the chosen materials, GO, and Determine the chemical compositions of the structures on the substrate. EDX spectrum of Graphene Oxide, has been provided information about its elemental composition as shown in figure 2. O K $\alpha$  Peak: indicates a significant presence of oxygen, common in Graphene Oxide due to oxygen-containing functional groups.<sup>(39)</sup> C K $\alpha$  Peak: represents carbon, the primary element in graphene, confirming the graphitic structure.<sup>(40)</sup> Mn K $\alpha$  and Mn K $\beta$  Peaks: Suggest manganese in the sample, possibly from impurities or intentional doping.<sup>(41)</sup> S K $\alpha$  Peak: Points to sulfur, which could be from residual processing chemicals or functionalization. <sup>(42)</sup> Cl K $\alpha$  and Cl K $\beta$  Peaks: Indicate chlorine, likely from contamination or chemical processing.<sup>(43)</sup> The spectrum is crucial for understanding the material's purity and functionalization, impacting its electrical and mechanical properties.



Figure 2. EDX analysis spectrum of Graphene Oxide (GO)

Figure 3 shows Fourier Transform Infrared Spectroscopy (FTIR spectrum) of the GO recorded in the range of 4000-500 cm-1The FTIR spectrum of GO have a large peak at 3331 cm-1, which corresponds to the stretching vibrations of hydroxyl groups (O-H). These peaks are consistent with findings from prior investigations.<sup>(44,45)</sup> C=O Stretching Peak 2110 cm-1 this peak is associated with the carboxyl group (C=O).<sup>(46,47)</sup> It indicates the oxidation of carbon atoms in GO. At 1634 cm-1, This summit is acknowledged as a skeleton ring with a double bond (C=C). The stretching vibration functional group associated with the sp2 carbon domain is found in un-exfoliated graphene oxide (GO) sheets.<sup>(48)</sup>



Figure 3. Fourier Transform Infrared Spectroscopy

The Ultraviolet-Visible (UV-Vis) The absorption spectra of nanoparticle layers are measured (PG Instruments T80. Figure 4 Displays the ultraviolet-visible spectra of the graphene oxide (GO). The UV-Vis spectrum shows a distinct peak at around 250 nm, which is typical for GO and similar to those in several previous research its characteristic of the  $\pi$ - $\pi$ \* transitions of the C=C bonds in the aromatic rings of graphene.<sup>(49,50)</sup> This peak indicates the electronic structure of GO and is a signature of its conjugated system. The absorbance gradually decreases as the wavelength increases towards 300 nm.

Figure 5 Displays the X-ray diffraction (XRD) pattern of graphene oxide (GO). The XRD pattern shown in the image for Graphene Oxide (GO) reveals several key features: Sharp Peak, the prominent peak at around 25 degrees 2Theta is characteristic of GO, indicating the (001) crystallographic plane. Interlayer Spacing peak, this peak corresponds to the interlayer spacing in GO, which is larger than that of graphite due to oxygen-containing functional groups. Amorphous Background peak, the pattern also shows a broad hump, suggesting the presence of amorphous carbon or disordered regions.

These features are typical of GO and help in understanding its structure and degree of oxidation.



Figure 4. Absorption spectrum of graphene oxide



Figure 5. X-Ray-Diffraction of Graphene Oxide

Nonlinear optical properties of GO Scan experimental setup



Figure 6. Experimental setup for Z-Scan technique

The Z-Scan approach was utilised to assess the nonlinear optical characteristics of the GO sample. This technique examines both the refractive and absorptive components of nonlinearity, while also assessing their intensity and polarity.<sup>(51)</sup> The Z-Scan approach involves scanning the solution in the Z direction using a focused Gaussian laser beam. This is done by moving the beam waist in a fitted focusing arrangement, as seen in

figure 6. the Z-scan data for graphene oxide sample1=  $0,2\mu$ m and sample2 = $0,25\mu$ m using a continuous wave helium-neon laser with a wavelength of 632 nm and 10 mW of output power. There are two different types of z-scan experiments: (a) open aperture z-scan and (b) close aperture z-scan and this method use a single-beam technique that is both sensitive and infectious to determine the topic of interest is the nonlinear refractive index and nonlinear absorption Characteristics of nonlinear optical materials. Analysing the plot of the nonlinear transmission produced via an aperture in the far field allows for the easy calculation of the nonlinear refractive index.

#### Result of z-scan

Figure 7 displays the outcomes of the non-linear absorption coefficient and saturation absorption for each thickness.



Figure 7. (a) Open aperture of Z-scan and (b) Close aperture of Z-scan carve for two different thickness of GO

The refractive index and absorption coefficient values of graphene oxide samples were measured for both closed and open aperture settings based on the transmission spectrum. it is evident from the results that for grapheme oxide demonstrated self-focusing. The refractive index was altered by the application of hydrogel film gas treatment. The absorption coefficient was determined by analysing the transmission spectrum using the open aperture z-scan technique, as shown in table 1.

Table 1. Nonlinear refractive index (n2) andabsorption coefficient (β) for samples			
Sample	n2 (cm2/GW)	β (cm/GW)	
Sample 1	0,091885997	6,06666E-11	
Sample	0,008700259	1,66677E-10	

#### Fabrication d-shape fiber

Experiment Of Water Sensing

A D-shaped fibre has an asymmetrical cross-section that mimics the form of the letter "D".<sup>(52)</sup> In order to produce fibers with a certain form, one may use a polishing technique to remove the cladding off the side of a Single Mode Fibre (SMF-28) until the fibre core is revealed. The normal single mode fibre is polished to a depth of around 40 µm. The polishing process starts by establishing a connection between the 2 terminals of a single mode fibre and the fibre pigtails. The first device is linked to a red laser source operating at a wavelength of 1550 nm, whilst the second device is linked to the optical power meter.

A microscope glass slide functioned as a holder for the fibre, and the fibre was attached to the glass slide throughout the polishing process. The cladding was stripped off using sandpaper with a grit level of 1200. The hand polishing method involves vigorously rubbing the sandpaper back and forth on the fibre until the polishing surface shows the presence of red light going through the fibre core. The fibre is subjected to meticulous polishing using an UltraPol TEC 40809 polishing machine, as seen in figure 8. The purpose of this procedure is to diminish the roughness of the D-fiber and enhance the transmission of light.



Figure 8. ULTRAPOL machine for polishing fiber

As the polishing surface approaches the evanescent field of the wave guiding mode, the transmitted power will start to decrease, as seen in figure 9. Hence, the variability in the power being transferred may be regarded as a reliable indicator of the different phases of polishing. The polariser controller is used to assess the polarisation extinction ratio of the D-shaped fibre. Two sets of single-mode optical fibres were polished until the transmission power loss for both sets reached -9,4 dB with a polarisation extinction ratio of 0,48 dB, also -26,8 dB with a polarisation extinction ratio of 3,1 dB, respectively.



Figure 10 depicts the arrangement of the D-shaped fibre coated with graphene oxide (GO). The D-fiber was connected to 2 fibre pigtails by fusion splicing: one pigtail has linked to the laser light source (FLS-50 Light Source) and the second pigtail was coupled to the power meter (Thorlabs PM100 USB). The input cable was linked to the 1550 nm light source, while the output fibre has attached to an optical power meter. The computer is connected to both the optical power meter also the CCD camera. The polarisation extinction ratio of a D-fiber coated with graphene oxide (GO) was measured by monitoring the MAX change in transmitted power while adjusting the polarisation controller. The research aimed to assess the sensing capabilities of a metal-coated D-shaped fibre by examining the transmission characteristics of the fibre when a solution was put to it.



Figure 10. Characterization set-up of GO coated D-shaped fiber

#### **RESULTS AND DISCUSSION**

The D-fiber was subjected to structural investigation using the Scanning Electron Microscope (SEM). Figure 11 displays the scanning electron microscope (SEM) picture of the polished surface. This surface was obtained by eliminating the polished fibre from the glass slide by the process of heating the slide to a temperature of 140°C, causing the glue to melt.



Figure 11. SEM images of the side-polished SMF

The D-fiber reaction to the transformer oil samples was tested without applying the GO-coating. Once the transformer oil droplet is placed on the detecting zone, the transmitted power experiences an almost instantaneous reduction by the 3rd second. The transmitted power saw an approximate rise of ~1,5 dB after the application of the water droplet. These findings indicate that the refractive index of the optical fibre core is lower than that of the transformer oil. As a consequence, light leaks into the transformer oil, which covers the optical D-fiber area. Consequently, the power loss was reduced as it passed through the cladded guide. Significant variations in the transmitted power level of the D-fiber have been noticed over time When the transformer oil samples are used. The variation is seen by a vertical discrepancy in figure 12. Figure 13 demonstrates variations in transmitted power for various water contents present in the oil sample. We conducted a measurement of the mean power variation during a duration of 50 seconds. A range of transmitted power, namely between 16 and 36 parts per million (ppm), has been shown. The suggested sensor has shown a sensitivity of -0,482 dB/ppm, specifically in detecting the water content dissolved in transformer oil.



Figure 12. Depicts the time-dependent behavior of the power level communicated while analysing transformer oil samples with varying amounts of dissolved water using a D-fiber



Figure 13. Displays the response curve of transmitted power when employing d-fiber with varying water content

The GO solution is deposited onto the optical D-shape fibre by the drop casting method, resulting in the formation of a GO-coated optical fibre. A single droplet of graphene oxide (GO) solution was placed onto the active area's surface and evenly distributed over the polished optical fibre. The coating is thereafter let too dry for about 10 minutes. The power variation in the optical fibre caused by the alignment of the D-shaped optical cable.<sup>(53)</sup> This enhances the interaction between the shrinking area of the fibre core and the transformer oil. Hence, a fibre optic cable is fabricated with a graphene oxide (GO) layer to investigate the impact of different GO thicknesses on the stability of transmission power. Afterwards, the optical fibre was coated with exactly four droplets of graphene oxide (GO) solution in order to assist the formation of additional layers. Subsequently, the coating is allowed to undergo a curing process for about 30 minutes, resulting in a measured thickness of approximately 0,20  $\mu$ m. The optical fibre was analysed by exposing it to several transformer oils, each containing samples with varying degrees of water content. A water droplet functions as a point of reference.

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Figure 14. The time-dependent change in the transmitted power level is determined by using a fibre optic cable coated with graphene oxide (GO) to analyses transformer oil samples containing different concentrations of dissolved water



Figure 15. Transmitted power response curve with varying water content utilising fibre optic coated with graphene oxide (GO)

Table 2. the parameters of dissolved water and d-fiber optic			
Parameter		D-Fiber	D-Fiber with GO
Water content (ppm)	161	-7,1 dB1	5,1 dB1
	18	-10,5 dB1	7,2 dB
	21	-13,4 dB	8,7 dB1
	361	-21,2 Db1	16,9 dB1
Sensitivity (dB/ppm)		-0,4820	0,5677
R <sup>2</sup>		0,7076	0,9934
Error		-3,7603	-3,497

#### CONCLUSIONS

This paper presents GO-coated D-shaped optical fiber as a potential device for the detection of water content in transformer oil. In the fabrication process, D-shaped fibers were formed by polishing Single Mode Fiber (SMF-28) and coating with GO through the drop-casting technique. Scanning electron microscopy analysis presented the formation of the desired fiber geometry and GO coating.

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It manifested that the experimental results had a high sensitivity to different water contents in transformer oil for the D-shaped fiber coated with GO. The GO-coated fibers exhibited a sensitivity of 0,5677 dB/ppm, which is relatively high compared with the sensitivity in the case of uncoated D-shaped fibers. GO coating interacts with the transient field of the fibre core and the surrounding medium enhancing the detection capabilities.

In the end, the obtained results of open-aperture and closed-aperture Z-scan proved nonlinear optical properties for GO-coated fibers. Open-aperture Z-scan showed saturable absorption, while closed-aperture Z-scan indicated self-focusing nonlinearity. These nonlinear properties can be very useful for developing state-of-the-art photonic devices, including optical limiters, switches, and modulators.

This research therefore identifies GO-coated D-shaped fibers as a very useful tool for controlling the water content in transformer oil. Improved sensitivity and stability make this sensor suitable for practical applications in the power industry, in which methods of detection against water contamination for assuring the performance and lifespan of transformers are of paramount interest. Future studies could be oriented to additional optimization of the GO coating process and evaluation of the long-term stability and performance of the sensors under real working conditions.

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## FINANCING

None.

### **CONFLICT OF INTEREST**

Authors declare that there is no conflict of interest.

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