

UNIVERSITI TEKNOLOGI MARA

**SYNTHESIS OF 3D HYDROGEL
GRAPHENE QUANTUM DOTS
NANOCOMPOSITES MODIFIED
FIBRE OPTIC FOR BIOSENSORS
APPLICATION**

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ABSTRACT

In this study, a novel fibre optic sensor towards biosensing application was developed by introducing polymeric supporting membranes of hybrid hydrogels (HH) and nanomaterials of graphene quantum dots (GQDs) as coating materials. HH was formed by introducing chitosan (CH) as an additive that would further aid in introducing the amide group that was lacking in agarose (AG) hydrogel. GQDs were synthesised using a chemical reduction method transforming the precursor graphene oxide (GO) into GQDs nanostructures. GO was synthesised prior to that using a modified Hummers' method. GQDs were optimised by varying the parameters in the chemical reduction method with the optimum condition for synthesis ~ 2 nm of GQDs was obtained by using 25 μ L of dispersed in 5 mL of sulfuric acid, 0.014 g of potassium permanganate and 0.1 mL of hydrogen peroxide. The optimum GQDs were found to be having an average size of 2.481 ± 0.649 nm as confirmed by transmission electron microscopy, subtle x-ray diffraction peak at around $2\theta=26^\circ$, loaded with hydroxyl (OH), carbonyl (C=O) and carbon bonds found from Fourier transform infrared analysis reflect that GQDs have many oxygenated functional groups making them soluble in water and stable fluorescence stability for up to 90 days with a quantum yield of 48%. A conventional fibre optic cable was used and exposed its core region made from silica by using mechanical etching via a fibre optic cable stripper. Before that, signal validation for the uncladded region (1, 2 and 3 cm) was performed with 2 cm of uncladded and was found to give the signal of 1660.74 a.u. and efficient uncladded length to be spliced. The fibre optic core coated HH was then let to be coated with the optimum coating thickness of HH on fibre optic was found at 122.18 ± 0.3 μ m by 2% total concentration of HH (2% w/v of AG with 2% v/v of CH) with confirmation by signal validation from the spectrometer with laser as a light source. After that, the coated HH (2% w/v of AG with 2% v/v of CH) probe was further coated with 2 nm GQDs via the drop-cast method and let to dry overnight. In enhancing the surface binding between the coating materials to the sensor probe, the unclad probe was further treated with sodium hydroxide, chromic acid, silane or ethanol prior coated to HH-GQDs. These coated probes were further tested for their sensing performance in the detection of refractive index (RI) changes (Δn) using glycerol solutions. Fibre optic pre-treated with chromic acid coated to HH-GQDs was found to obtain the lowest baseline error of 0.026% which improves the adhesive force between HH and the fibre optic core surface. The sensitivity was then calculated at 17358.0 ± 0.21 RIU⁻¹ with a coefficient of determination; R^2 of 0.94. As a proof of concept for biosensor applications, the protein of *streptavidin* (0.1 mg/mL) was successfully immobilised by incubation for 15 minutes on the fibre optic core treated with chromic acid and coated with HH-GQDs. 10 mg/mL of *bovine serum albumin* (BSA) was used as a blocking agent after incubation for 10 minutes on the HH-GQDs matrix. The sensor was successfully able to detect the *biotin* (0.1 mg/mL) after incubation for 15 minutes and washing with PBS. The binding affinity in respect of the change in peak wavelength ($\Delta \lambda$) of HH-GQDs matrix to the protein was measured at 90.445 a.u.nm⁻¹ with R^2 of 0.933. The sensor also shows excellent specificity, hence holding a bright potential as a highly sensitive biosensor.

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