Investigation on Structural, Optical, and Thermal Properties of Graphene Oxide (GO) Nanoparticles

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Abstract:

The aim of the study , A nanoparticles of graphene oxide (GO) was synthesized by modified Hummer's method. A new approach to synthesis graphene is oxidizing graphite powder with mixture with concentrated Sulphuric acid (H_2SO_4), Sodium Nitrate ($Na\ NO_3$) and Potassium permanganate ($KMnO_4$), Currently,an improved method for the preparation of graphene Oxide was most common one. The Graphene Oxide (GO) was characterized by X-Ray diffractration, FT-IR Spectroscopy, UV Visible Spectroscopy , Raman Spectroscopy and TGA analysis. The X-ray diffractration (XRD) results of Graphene oxide nanoparticles was found to be 12.9 nm with a hexagonal crystal structure.

Key Words:

Graphene oxide, Hummer's method,Graphite, X-ray diffratcration, FI-IR analysis,UV-Visible spectroscopy ,Raman spectroscopy and TGA-DTA analysis

1.Introduction:

Nanoscience and Nantechnology primarily deals with synthesis, characterization, exploration,and expansion of nanomaterials. Carbon , one of the most common atoms on Earth, occurs naturally in many forms and as a compound in countless substances which are called allotropes of carbon. Graphene oxide (GO) is of great interest due to its low cost, easy access and widespread ability to convert graphene. Graphene oxide is graphite that has been oxidized to intersperse the carbon layers with oxygen molecules,and then reduced, to separate the carbon layers completely into individual or few layer graphene. The completelyoxidized compound can then be dispersed in a base solution such as water,and graphene oxide is then produced[1-17]. As early as 1947, Wallance [18] first propsed the concept of graphene and studied the electronic properties of graphene using tight-binding model. It is concluded that graphite is semiconductor without activation Energy, because of the small portion of valance band of graphite extended to the conduction band, in which data established a foundation for the use of physical properties of graphite[19].

Currently, many methods had been explored to prepare graphene. Novoselov and Gim[20] firstly observed a single layer of graphene from highly oriented pyrolytic graphite using micromechanical

exfoliation method. It is a simple way to prepare graphene, but the yield of graphene was very low, and you cannot achieve high-quality industrial production. However, the low yield of graphene was still.Redox method[21-24] is the most popular method to prepare graphene and graphite oxide. During the oxidation process, graphite crystal was treated with strong oxidizing agent carried oxygen-containing functional groups into graphite oxide sheets.

In this study successfully synthesized graphene oxide (GO) by the modified Hummers method,as the precursor for preparing graphene. The structures of the GO were measured by Fourier-transform infrared spectra analyzer(FT-IR) and X-ray diffraction (XRD). A UV spectrometer(UV-Vis) was used to measure the optical absorption properties of the GO. Thermal stability of GO was determined through the thermal gravimetric analyzer(TGA).

2.Experiment and Methods:

Chemicals and materials: Graphite flakes(natural 98%), Sodium nitrate(98%, SRL chemicals), Pottasium permanganate(99%), Hydrogen peroxide(40%,Emplura), Sulphuric acid(98%, ACS), and Hydrochloric acid(35%),. All chemicals were of analytical grade and used as received.

Synthesis of Graphene Oxide (GO): Graphene oxide was synthesized by Hummers method through oxidation of graphite[25-27]. In details, 2g graphite flakes,2g of Na_2NO_3 wree mixed with in 50ml of H_2SO_4 in a 1000ml volumetric flask kept under the ice bath(0-5°C) with continuous stirring. Next, 6 g $KmnO_4$ were slowly added so that the temperature of the mixture remained below 15°C.The ice bath was removed and mixture was stirred at 35°C until it became pasty brownish and kept under stirring for 3 days. It is then diluted with slow addition of 100ml water. The reaction temperature was rapidly increased to 98°C with effervescence, and color changed to brown color. Deionized water was further added so that the volume of suspension was 200ml. of water was continuously stirred. The solution is finally treated with 20ml H_2O_2 was added after 10 min, to terminate the reaction by appearance of yellow color. The reaction product was centrifuged and washed with deionized water and 10% HCL solution repeatedly. After filtration and drying under vacuum at room temperature. Finally, the product was dried at 60°C muffle furnace, the graphene oxide(GO) was obtained as a powder.

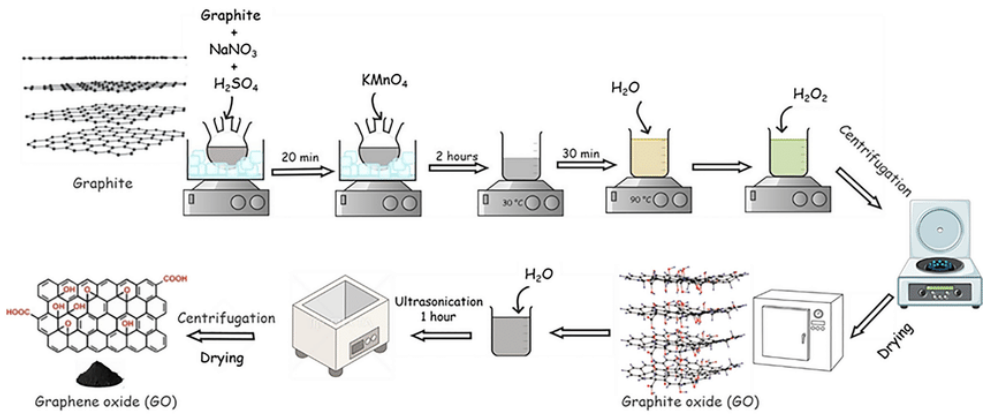


Figure 1.Graphica abstract of Synthesis of Graphene oxide NPs.

3.Results and Discussion:

3.1.Crystalline Structure Identification by PXRD Studies: The crystalline structure and phase of the synthesized sample were investigated by XRD pattern as shown in fig(2). The XRD analysis was used to determine the average crystalline properties of the GO powder. The GO powder was prepared showed a very strong peak at 2θ=10.8° which is in good agreement with the literatures[28].The results of XRD initially proved the successful synthesis of GO powder. Scherrer's formula, which is used to determine the average crystalline size of the nanoparticles is given below[12.9nm].

$$D=K\lambda / \beta \cos\theta \quad \ldots\ldots\ldots\ldots\ldots(1)$$

Where D is the size of the crystal, λ is the wavelength of X-ray radiation (0.15418 A°), β is the full-width at half maximum, θ is the angle of diffraction, and K is the a constant(0.98).

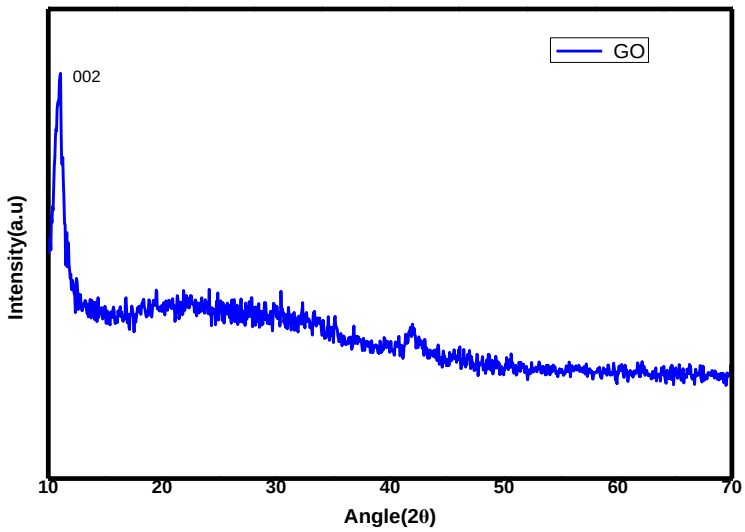


Figure 2. XRD spectrum of GO nanoparticles

3.2.FT-IR analysis:

The FTIR spectra analysis was performed to investigate the structure and functional groups of the materials, as shown in figure(3). The spectra show the presence of oxygen containing functional groups in GO. The peak at 3450 cm^{-1} GO and 3445cm^{-1} shows the stretching of hydroxyl group C=O carbonyl stretching at 1630 cm^{-1} and the C-O epoxide group stretching at 1096 cm^{-1} . High intensity of major peak in Go reveal that large amount of oxygen containing groups are present after oxidation process. The peak at 1623 cm^{-1} is related to the skeletal stretching of C=C alkene group. After reduction of GO peak intensities due to alkoxy and hydroxyl groups were decrease significantly and C=C phenol ring stretching at 1623 cm^{-1} was present. Further Confirmed that the graphite indeed was oxidized into GO was and with literatures[29,30]. The presentation of C=C groups showed that even graphite had been oxidized into GO.

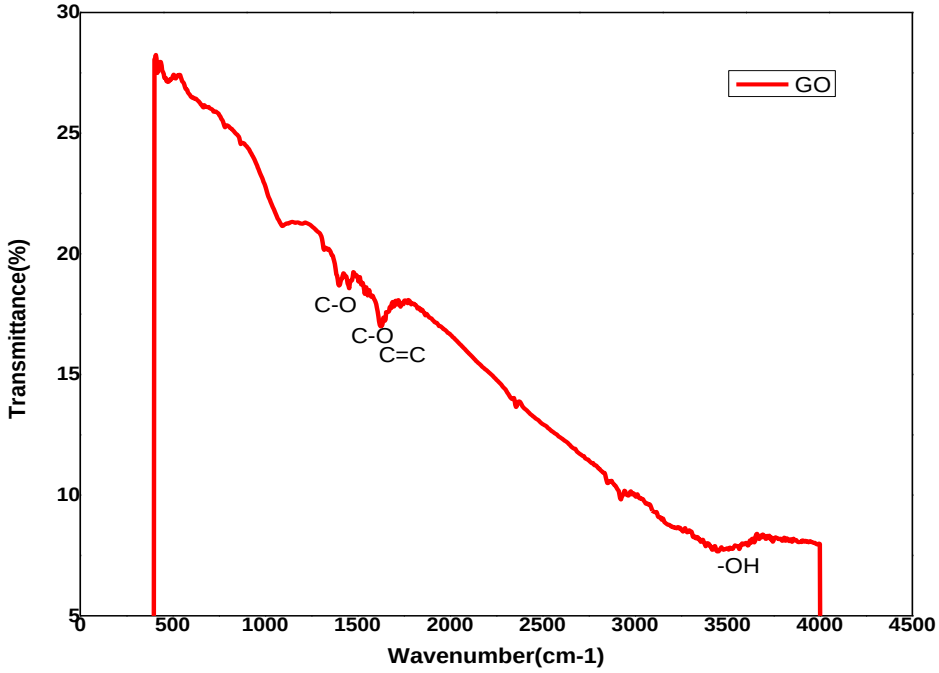


Figure 3. FTIRspectrum of GO nanoparticles

3.3. UV-Visible spectroscopy analysis:

The Optical Absorption of GO:The energy bandgap of the synthesized of graphene oxide nanoparticles was determined from their UV-Vis absorption spectra. Figure (4) shows the indicated that graphene oxide possessed a good absorption in the visible range (200~700 nm), but absorption in the ultraviolet range was also slightly decreased.The energy band gap (E_g) values can be obtained from the absorption coefficient α, which can be calculated as a function of Photon energy (hν). The relationship between the absorption coefficient and photon energy can be expressed as [31].

$$(\alpha h\nu)^2 = C\,(h\nu - E_g) \quad \ldots\ldots\ldots\ldots\ldots\ldots (2)$$

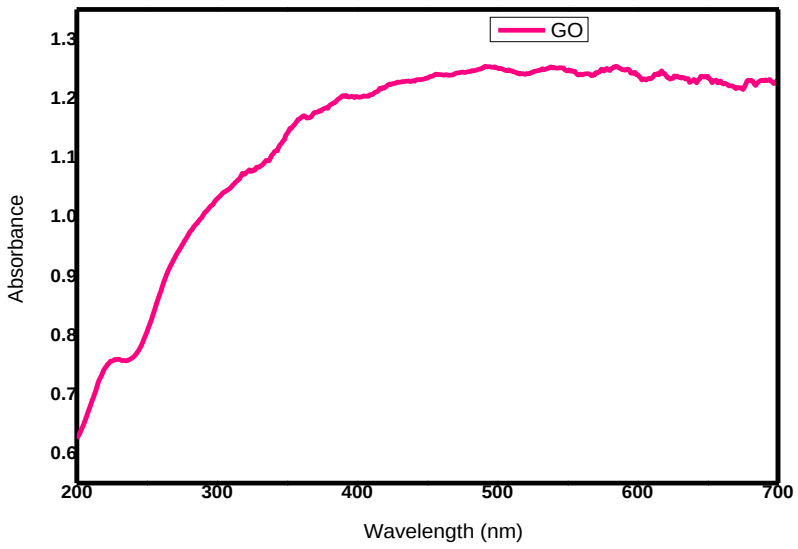


Figure 4. Optical Absorbance spectra of GO nanoparticles

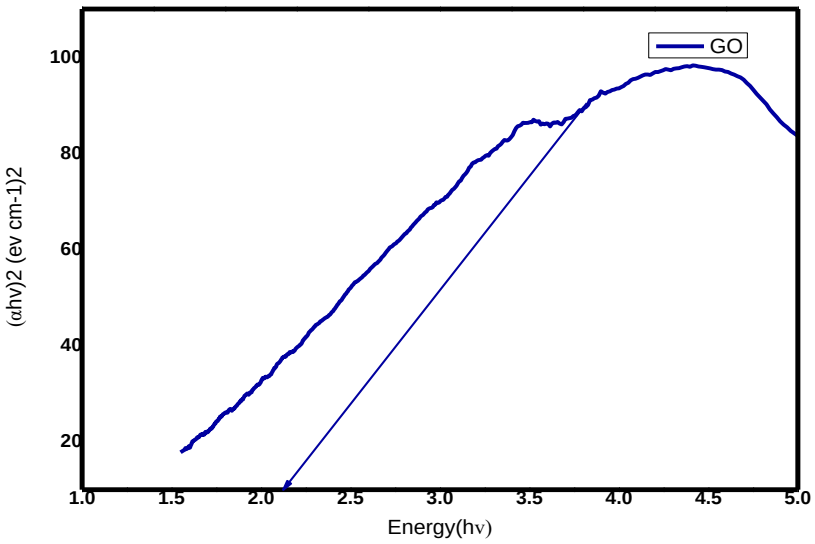


Figure 5. Tauc plot of GO nanoparticles

3.4. Raman Spectroscopy Analysis:

Raman Spectrum of GO nanoparticles was used to be observe structural changes in the samples the oxidation and reduction process. Raman Spectroscopy is widely used for the characterization of carbon products, especially considering the fact that conjugated & double carbon bonds lead to high Raman intensities. Raman spectroscopy is a non destructive technique that is widely used to obtain structural information about carbon –based materials. The main Features in Raman spectra of graphitic the carbon based materials are the G and D peaks and their overtones. The first –order G and D peaks, both arising from the vibration of Sp^2 carbon, appear at around 1602 cm^{-1} and 1339 cm^{-1},respectively.The D peak represents the breathing mode of aromatic rings arising due to defect in the sample. The D-peak intensity is therefore often used as a measure for the degree of disorder. The Shift and shape of the overtone of the D peak, called as 2D peak around 2678 cm^{-1} can be correlated to number of graphene layers.

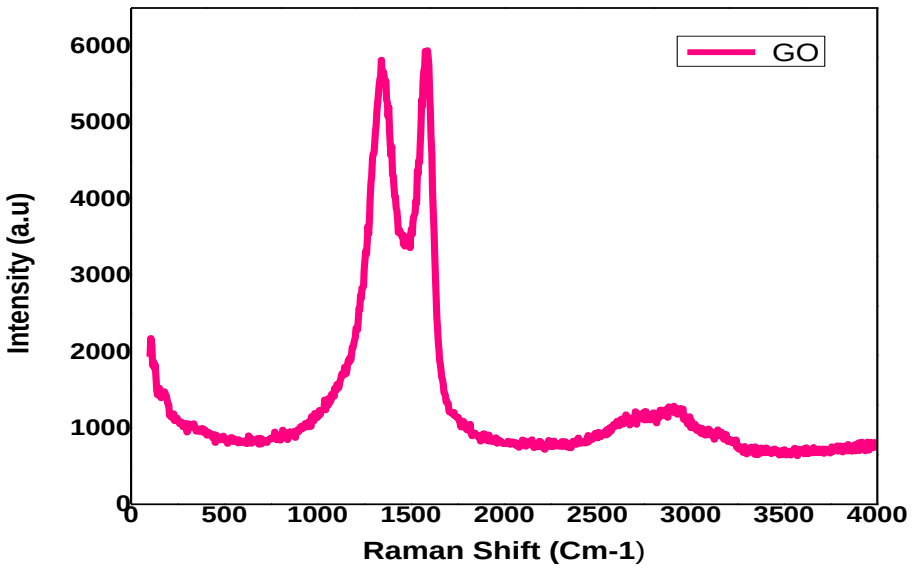


Figure 6. Raman spectrum of GO nanoparticles

3.5. TGA Analysis :

TGA is the technique to examine the thermal stability of the synthesized nanoparticles with aid of weight change against variability in the temperature. In this technique is also used to examine the thermal stability for different types of fabrics and other materials. Fig (7) show the TGA curve o Graphene Oxide nanoparticles , exhibits of weight loss 52.24%(1.438 mg).

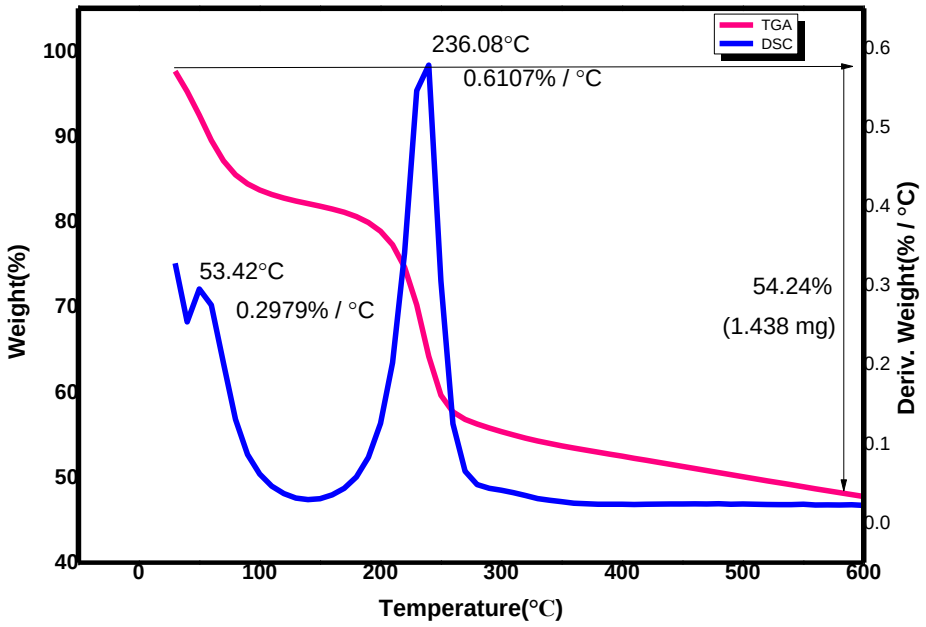
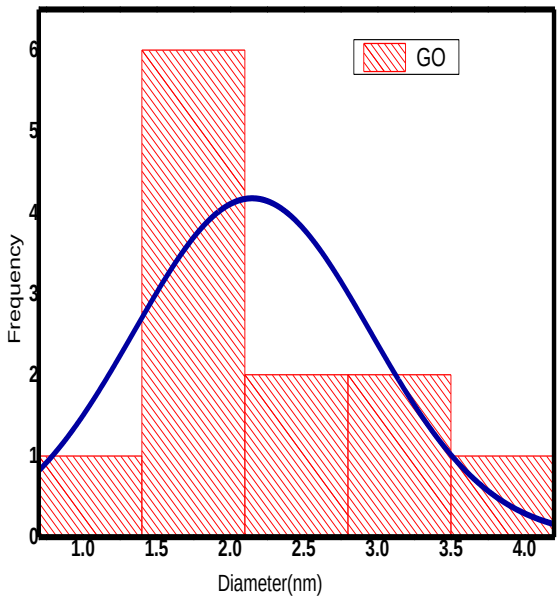


Figure 7. TGA spectrum of GO nanoparticles

3.6. Morphological Analysis:

The microstructure of the prepared materials was characterized by Scanning Electron Microscopy (SEM) analysis. The SEM micrographs of graphene oxide nanoparticles show in Fig (8) . From the figure, it can be observed that graphene oxide has layered structure, which affords ultrathin and homogeneous graphene oxide powders.The figure shows the a thickened nanostructure with rough and from the SEM micrographs of GO nanoparticles of average diameter Value 2.02nm. It is noted that GO nanoparticles which is also confirmed by the XRD and FTIR results. The SEM image also confirm that functionalized on the surface of the graphene oxide nanoparticles was identified by an energy-dispersive x-ray spectroscopy (EDX) study. The EDS spectrum of GO nanoparticles show in figure (8) confirmed the presence of carbon (52.33 %) and Oxygen (47.67%) in the Table. The results of the EDX spectrum also confirm the successful formation of GO nanoparticles.



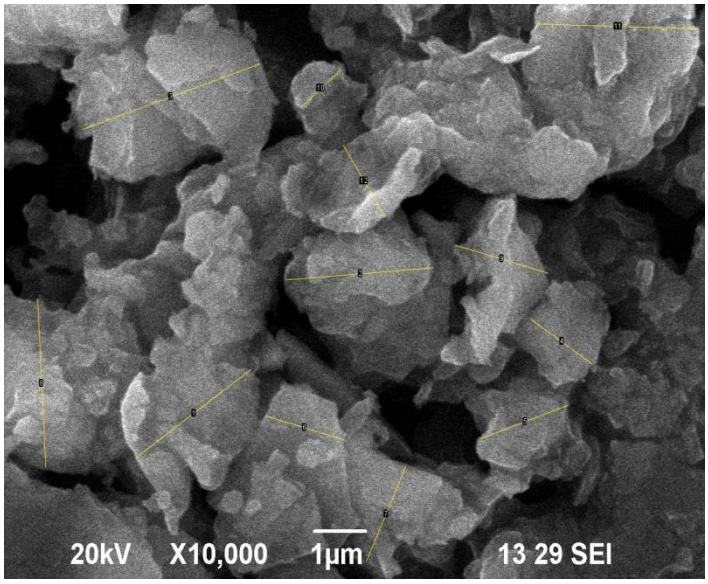


Figure 8. SEM image of GO nanoparticles

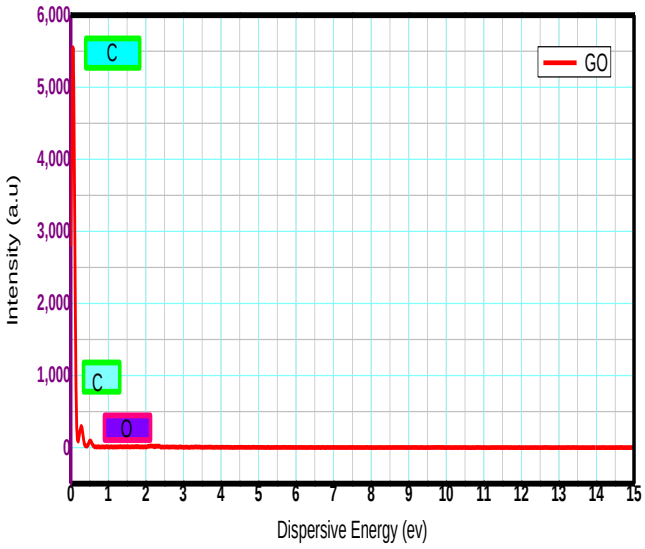


Figure 9. EDAX spectrum of GO nanoparticles

Table 1 :The elemental composition of Graphene Oxide Nanoparticles in EDX spectrum

Element	Weight %	Atomic %
C K	45.18	52.33
O K	54.82	47.67

4.Conclusion:

We have successfully developed a simple, efficient and free toxic method to synthesized of Graphene Oxide (GO) nanoparticles. In this study of GO nanoparticles ,structure,morphology and optical properties of prepared GO nanoparticles were prepared. FTIR analysis were carried out the identify functional groups and to confirm the formation of Nanomaterials.An average of optical absorption in the wavelength range 200 -800nm the Tacuc's graph plotted and revealed that the optical bandgap measured from the absorption spectrum GO nanoparticles 12 ev.In PL emission spectrum peaks were observed at different wavelength at 360,356,458,495,505 nm are detected.

Conflict of intrest:

The authors declare no conflict of intrest.

Authors' Details:

N.Thangaraj: First author,conceptualization,methodology,Investigation,Writing-Original Draft, Formal analysis.

Dr.N.Joseph John : Validation,Visualization, Supervision, Writing-review &Editing.

C.Gnana Sambandam : Validation,Visualization, Co-Supervision, Formal analysis.

Authors' contribution:

The authors declares that: The manuscript has not been previously published is not currently submitted for review to any other journal and will not be submitted elsewhere before a decision is made by this journal.

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