

Conductivity and dielectric properties of heterostructures based on novel graphitic carbon nitride and silver nanoparticle composite film for electronic applications

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Graphitic-carbon nitride (g-C₃N₄) (GCN) has emerged as the most promising material for the fabrication of next generation cutting-edge electronic and optoelectronic devices, due to its excellent and promising electrical and physicochemical properties. In this work we report on strategies to enhance the electrical conductivity of GCN thin films through the formation of heterostructures using silver nanoparticles (Ag). The synergistic effect of silver nanoparticles doping on structural and transport properties of GCN thin films was studied in detail. The structural and morphological changes in GCN polymer owing to the inclusion of silver nanoparticles were examined through Scanning electron microscopy, Fourier transform spectroscopy and X-ray diffractometer techniques. The conformational modifications in GCN polymer chains due to silver nanoparticles doping greatly enhanced the electrical conductivity of GCN thin films. The inclusion of silver nanoparticles in GCN polymer matrix decreases the barrier energy and enables the charge carrier hopping easily leading to improved electrical conductivity. The electrical conductivity of GCN-Ag composite thin film was enhanced by two folds due to the silver nanoparticles inclusion in comparison to conductivity of the pristine GCN thin film. The presence of Ag nanoparticles in the composite film plays a substantial role in improving the dielectric attributes of the pure GCN. Therefore, the doping by using silver nanoparticles might be a suitable strategy for effectively tailoring the electrical conductivity and dielectric properties of GCN thin films and can be used as flexible conducting electrode material towards fabrication of electronic and optoelectronic devices.

(Received May 22, 2022; Accepted October 3, 2022)

Keywords: Graphitic Carbon Nitride, Electrical conductivity, Silver nanoparticles, Dielectric properties, Electric modulus, Optoelectronic devices

1. Introduction

Graphitic carbon nitride commonly known as (GCN) has gained a lot of attention by researchers for its technological importance due its stability and optical transparency over a wide range of wavelengths [1–2]. In this context, the fabrication of heterostructures based on two-dimensional nanomaterial's has received much attention in science and technological applications due to their novel characteristic properties like high aspect ratio, thin layers, and very easily tuneable particle sizes [3]. In the family of two-dimensional semiconducting polymers, graphitic

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<https://doi.org/10.15251/DJNB.2022.174.1089>

carbon nitride (GCN) has captured more attention by material researchers and material physicists across world-wide owing to its superior physical and chemical properties like tuneable band gap, good level of electrical conductivity, larger thermal and chemical stability, enhanced opto-electronic properties and easy method of synthesis [4-5]. GCN has a two-dimensional conjugated polymer structure consisting of carbon and nitrogen in their polymer backbone. GCN is prepared from numerous carbon materials by replacing carbon atoms with the nitrogen atoms [6-7]. The GCN also exhibits a lamellar structure with physio-chemical characteristics that can be employed for potential electronic applications. Generally, the sp^2 hybridized nitrogen atoms of the GCN polymer matrix consists of a single pair of electrons which help to improve the adsorption of organic gas molecules [8-9]. GCN is one of the attractive two-dimensional nanomaterials, being extensively used in numerous technological applications such as opto-electronics, energy storage and for gas sensors [10-12]. Moreover, pure GCN exhibits poor electrical conductivity, small volume to surface area that hinders many scientific and technological applications [13-15]. Hence, to overcome these limitations, the need for modification in functionalization, structure and properties of GCN has emerged as an effective method [16-17]. Unfortunately, the electrical conductivity of pristine GCN is lower compared to that of indium tin oxide. This problem could be resolved to some extent by a method called secondary doping in GCN [18-19] by which the electrical conductivity can be increased to few orders more of their existing magnitude. Even in literature, a smaller number of publications are available which address this problem of improving the electrical conductivity of the GCN thin films. Hence, at present it could not meet the standard expectations of industrial demands. In this proposed research work we report the preparation of GCN based thin film using dopant like silver nanoparticles, which could be used as a potential candidate for electronic applications with improved conductivity and dielectric properties.

Silver nanoparticles help in functionalizing the composite material, thereby enhancing the thermal and electrical properties. The silver nanoparticles are generally employed as capping agents for opto-material applications. Doping of silver nanoparticles in polymer matrix leads to the enhancement in charge conjugation length and also delocalization of charge carriers, which effectively leads to improved electrical and dielectric properties of the host polymer. Thus, silver nanoparticles were selected as an effective doping material which could largely affect the electrical conductivity and dielectric properties. GCN belongs to family of soft polymer having an excellent film forming characteristic after doping [20]. The addition of silver nanoparticles causes strong embedment in the GCN polymer matrix resulting in the formation of high quality and stable thin films. To the best of our knowledge for the first time we report the electrical properties of composites films based on GCN and Ag NPs with excellent conductivity and dielectric attributes for possible electronic applications.

In this research work, the authors made an effort to understand the effect of silver nanoparticles doping on structure, morphology and transport properties of GCN thin film. This paper deals in detail with the surface morphological changes in the pure GCN film because of secondary doping using silver nanoparticles via SEM, FTIR and XRD analysis. In detail the transport properties of these prepared thin films were examined by using a temperature dependent conductivity as well as dielectric and impedance studies. In this work, it was found that the electrical conductivity of pure GCN increased by two orders of magnitude because of the inclusion of silver nanoparticles. Owing to high level conductivity and improved dielectric properties, these silver nanoparticles doped GCN thin films might find extensive use in opto-electronic device applications.

2. Materials and Methods

2.1. Experimental

The thin films were prepared by dip coating method (Model: Dipcoater-134 DCU). The morphology of the synthesized samples was investigated by scanning electron microscopy (SEM) (Model: Zeiss Ultra-60). The analysis of functional groups and chemical composition existing in the prepared samples were carried out by Fourier transform infrared spectroscopy (FTIR) (Model: Thermo Nicolet IR-370). The structural confirmation tests were done by X-ray diffraction studies

using X-ray diffractometer Cu-K α ($\lambda = 1.5418 \text{ \AA}$) radiation at a scanning rate of 6° per minute and 2θ angle range of 10° – 80° respectively. The electrical conductivity (temperature dependent) of the prepared set of samples was carried out by using four-probe method (Model: Keithley I-V meter). The dielectric properties of pristine GCN and GCN-Ag thin films were investigated by two-probe method in the applied frequency range of 10Hz–2MHz employing LCR impedance analyzer (Model: Wayne Kerr 6500B Series).

2.2. Synthesis of graphitic carbon nitride (GCN)

The graphitic carbon nitride (GCN) was prepared via thermal decomposition method as reported in the earlier work [21]. In the synthesis process of GCN, 2 gm of melamine was thoroughly dissolved in 5 ml of deionized water and the prepared solution was maintained at 90°C all throughout the night. The obtained powder was kept in the muffle furnace and vigorously heated up to 500°C for 3 hours with the ramping time of $5^\circ\text{C}/\text{min}$. The collected yellow coloured graphitic carbon nitride (GCN) was gently grinded and was used for further studies.

2.3. Synthesis of GCN-Ag nanocomposite thin film (GCN-Ag)

The GCN-Ag composite thin film was prepared by simple dip coating technique. Initially, 5 grams of GCN powder was dissolved in 10 ml of N-Methyl-2-pyrrolidone (NMP) and ultrasonicated for a duration of 2 hours to achieve a homogenous dispersion of GCN solution. In order to prepare GCN-Ag composite, 1 grams of Ag and 50 milligram of urea was added to the GCN solution and sonicated for duration of 1 hour to achieve a homogenous solution of GCN and Ag. The final homogeneously dispersed solution of GCN and Ag was coated on glass substrate of dimension (2cm \times 2cm) by using dip coating unit. After the preparation of GCN and GCN-Ag composite thin film, the films were annealed at 90°C in a dynamic oven to remove the absorbed moisture in the prepared thin film.

3. Results and discussions

3.1. Scanning electron microscopy (SEM)

The surface morphology of pristine GCN and GCN-Ag composite are shown in Figure. 1 (a) and (b) respectively. The pristine GCN sample exhibits more irregular granules on the surface with the formation of micro dimensional pores and huge agglomerations in the morphology. The morphology of the pure GCN sample shows micro-grains that are not well resolved, which may lead to obstruction of charge carriers thereby resulting in low electrical conductivity and dielectric attributes [22].

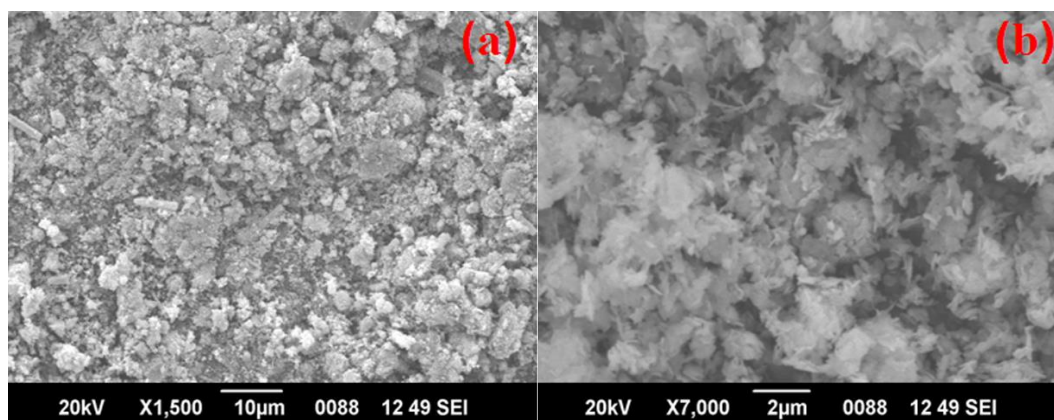


Fig. 1. SEM images of (a) pure GCN (b) GCN-Ag composite thin film.

The surface morphology of GCN-Ag composite (Figure. 1(b)) exhibits comparatively uniform surface with good and well resolved micro-grains in the GCN polymer matrix. This type of surface morphology helps for easy migration of charge carriers, which leads to enhanced electrical conductivity and dielectric properties of the GCN-Ag composite [23]. The Ag NPs embedded in the GCN matrix shows particle size ranging from 70 to 90 nm. The uniform distribution of silver nanoparticles in GCN matrix and strong interaction between Ag and GCN are supportive for improvement in the conductivity and dielectric properties.

3.2. Fourier transform infrared spectroscopy (FTIR)

The different functional groups and chemical compositions present in the prepared samples were recorded using Fourier transform infrared spectroscopy. The FTIR spectra of pristine GCN and GCN-Ag composite samples are shown in the Figure. 2(a) and (b) respectively. The pristine GCN sample shows major characteristic peaks at 1750cm^{-1} , 1250cm^{-1} , 1200cm^{-1} , 840cm^{-1} and 802cm^{-1} and are generated because of stretching frequencies of C-N, N-H and C=N molecular bonds [24]. Also, GCN-Ag composite spectra exhibits important peaks at the stretching frequencies of 2250cm^{-1} , 1250cm^{-1} and 1080cm^{-1} which are attributed to the C-N, Ag-O and Ag=O molecular vibrations respectively. The prominent peak near stretching frequency of 3130cm^{-1} present in both the pristine and composite samples is due to the O-H stretching (absorbed moisture in the samples). The FTIR characterization studies reveal the fact that silver nanoparticles are strongly embedded in the GCN matrix with the effective formation of nanocomposites.

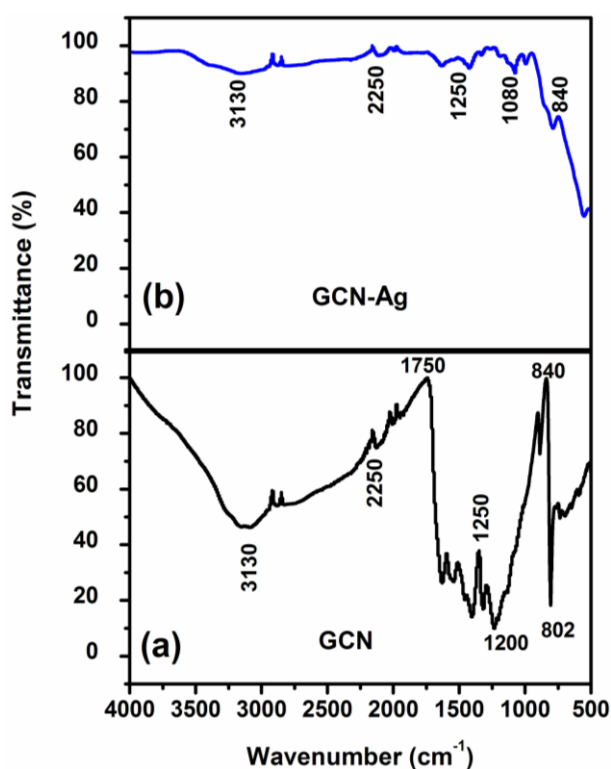


Fig. 2. FTIR spectra of (a) pure GCN (b) GCN-Ag composite thin film.

3.3. X-ray diffraction studies (XRD)

The structural features of pristine GCN and GCN-Ag composite examined by employing X-ray diffraction studies are illustrated in Figure. 3. The pure GCN sample shows prominent peaks distributed around $2\theta=15^\circ$ to 60° (JCPDS card number of GCN .74-1182) [25]. The X-ray diffraction spectrum of GCN-Ag composite shows the existence of silver nanoparticles having a characteristic nature of the tetragonal crystal structure. The nanocomposite sample exhibits major peaks at the position of $2\theta=15^\circ$, 28° , 40° , 45° , 66° and 78° corresponding to the crystal planes

(100), (200), (210), (211) (300) and (310) respectively. This X-ray diffraction study of the prepared samples shows the existence of the silver nanoparticles with their structural features being present even after the formation of the composite.

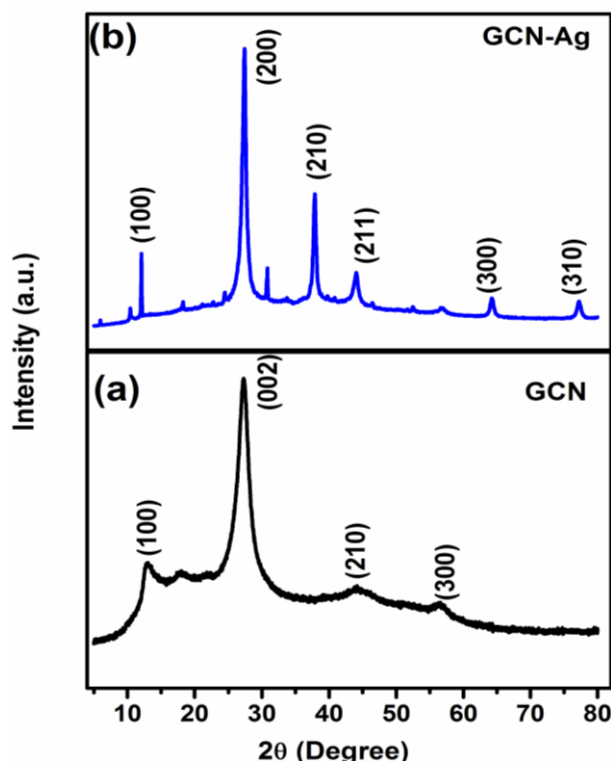


Fig. 3. XRD spectra of (a) pure GCN (b) GCN-Ag composite thin film.

3.4. Transport properties

3.4.1. Electrical conductivity

Highly conductive nanocomposite film shows superior performance in most of the opto-electronic applications as well as electrode material for device fabrication. The trend of variation in electrical conductivity for pristine GCN and GCN-Ag composite are shown in Figure. 4. The prepared samples exhibit two stage variations with respect to the applied temperature. The first stage variation is in-between the temperature range of 25 °C to 125 °C and is found to be negligibly small change in the conductivity and in the second stage between the temperatures of 150 °C to 300 °C there was a faster variation in conductivity and the trend was found to be linear with respect to temperature. The faster increment in electrical conductivity at high temperature range is because of the thermally supported hopping of charge carriers (both polarons/bipolarons) in the two-dimensional semiconductor nanostructure network. The inclusion of silver nanoparticles in the GCN polymer matrix exhibits serious effects of morphological changes of the composite and this kind of change in morphology helps to establish well conducting islands for the smoother transport of charge carriers amid their neighbouring sites. Furthermore, inclusion of silver nanoparticles in the GCN polymer matrix leads to the synergistic effects like conformational changes from coil structure to linear structure and this kind of chain structure enables easy migration of charge carriers (both polarons/bipolarons) in the nanocomposite film [26]. In general, the existence of silver nanoparticles produces a large number of π - π delocalized charge carriers in GCN-Ag composite film that enables high electrical conductivity in two-dimensional semiconducting nanostructures. With the strong embedment of silver nanoparticles in the GCN polymer matrix, the GCN-Ag nanocomposite shows superior conductivity of the order of 187.23 S/cm in comparison with a pure GCN sample of about 17.45 S/cm.

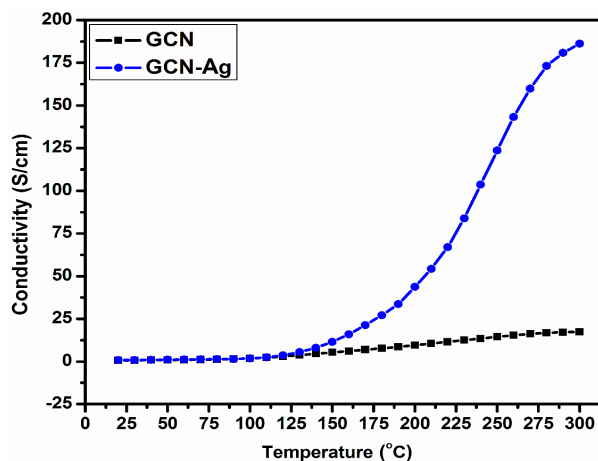


Fig. 4. Temperature dependent conductivity of pure GCN and GCN-Ag composite thin film.

The current voltage (I–V) characteristics of pure GCN and GCN-Ag composite film are shown in Figure. 5. The I–V variation of both pristine and composite thin films are almost linear (Ohmic nature) for both forward and reverse bias voltages of 4 V applied. Therefore, both pristine and composite thin films exhibit almost constant conductivity across the applied bias voltage [27]. The linear variation of I–V characteristics reflects the well formation of thin films with the combination of GCN and silver nanoparticles exhibiting superior work-function.

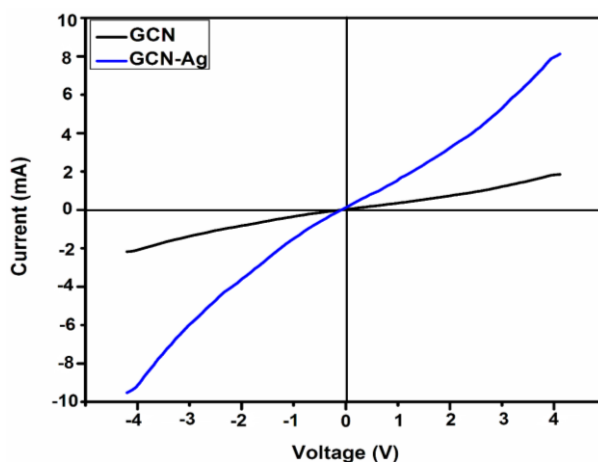


Fig. 5. Current-Voltage characteristics of pure GCN and GCN-Ag composite thin film.

3.4.2. Dielectric studies

The variation of real and imaginary part of the dielectric constant as a function of applied frequency recorded at ambient temperature for pristine GCN and GCN-Ag composite thin film are depicted in the Figure. 6 (a and b). From the plot, it can be inferred that both the real and imaginary part of dielectric constant shows almost non-linear variation. Both the values of real and imaginary dielectric constant decreases with the applied frequency up to the frequency range of 10^3 Hz and the trend becomes almost constant at higher frequencies. The strong frequency dependent dispersion of the dielectric permittivity observed at the lower frequency range can be attributed to the dipolar relaxation along with the Maxwell–Wagner–Sillars (MWS) polarization [28]. The prepared samples exhibit larger dielectric permittivity at very low frequency due to increase in the polarization of the charge carriers (both polarons/bipolarons). At the same time at higher frequencies beyond 10^3 Hz the dielectric permittivity attains almost constant value due to increased electrode polarizations at the region of grain boundaries. Among the prepared samples,

the GCN-Ag nanocomposite film exhibits the minimum value of dielectric constant due to the existence of a large number of charge carriers accumulated at the grain boundaries thereby hindering the polarization mechanism.

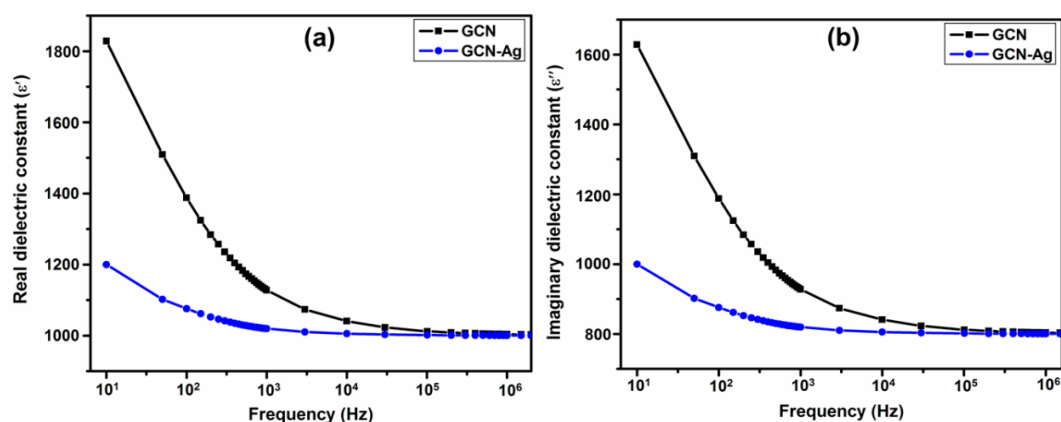


Fig. 6. (a) Real part of dielectric constant (b) Imaginary part of the dielectric constant of pure GCN and GCN-Ag composite thin film.

The variation of real and imaginary component of the electric modulus (M' and M'') in case of pure GCN and GCN-Ag composite thin films recorded as a function of frequency at room temperature are shown in Figure. 7 (a) and (b). Both real and imaginary parts of electric modulus show a very small increment in electric modulus in the frequency range of 10^1 to 10^3 Hz. This behaviour might be due to non-formation of auxiliary magnetic field around the silver nanoparticle domains. In the high frequency range between 10^3 to 10^4 Hz, the gradual rise in the electric modulus is due to the formation of an auxiliary magnetic field surrounded around the silver nanoparticle domains [29]. In contrast with the GCN-Ag composite film, pure GCN sample shows a very low value of the electrical modulus in accordance with the results obtained from the plots of real and imaginary dielectric constant.

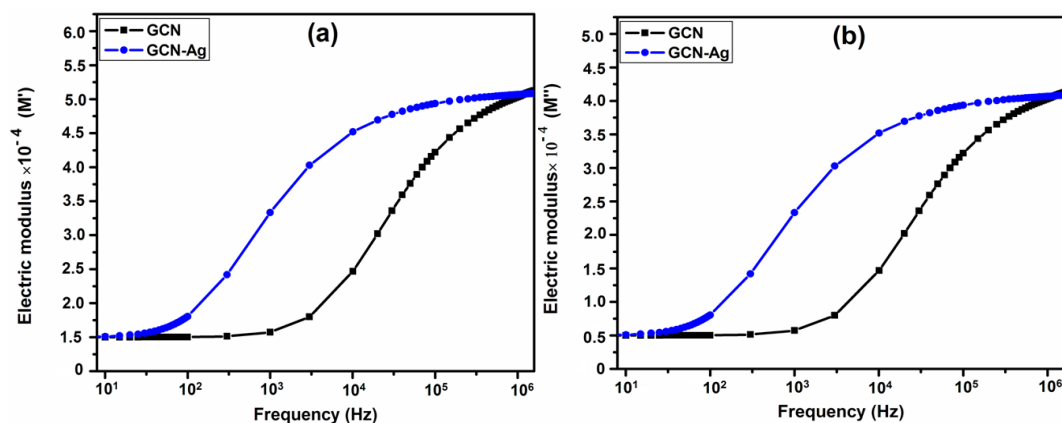


Fig. 7. Real part of electric modulus (b) Imaginary part of electric modulus of pure GCN and GCN-Ag composite thin film.

The variation of tangent loss as a function of applied frequency for pristine GCN and GCN-Ag nanocomposite film recorded at room temperature are shown in the Figure. 8. The tangent loss (dielectric loss) values significantly varied in the range of 0.87 to 0.75 for prepared thin film samples. From the plots it is observed that at lower frequencies between 10^1 and 10^2 Hz, all the prepared samples show a slightly larger dielectric loss. The high dielectric loss at very low frequencies could be due to the creation of auxiliary magnetic field around the edge of grain

boundaries. The dielectric loss slightly decreases with higher frequencies between 10^3 Hz to 10^6 Hz. Among the prepared thin film samples, GCN-Ag nanocomposite film exhibits least tangent loss (0.75) because of the non-Debye's type of relaxation mechanism occurring in the nanocomposite. Due to the existence of very small dielectric tangent losses, these nanocomposites could be possibly used in most of the electronic and opto-electronic device applications [30].

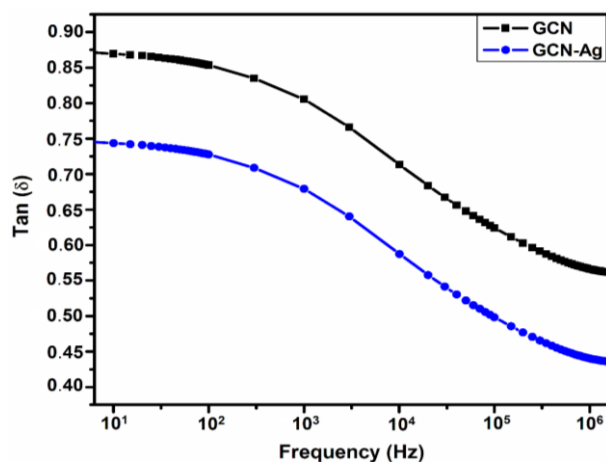


Fig. 8. Tangent loss of pure GCN and GCN-Ag composite thin film.

The impedance response of GCN and GCN-Ag composite film has been recorded by using impedance spectroscopy. The resulting plots obtained by computing real and imaginary part of impedance (Z' and Z'') are called Nyquist plots. The Nyquist plots of pure GCN and GCN-Ag composite film are illustrated in Figure. 9. It can be inferred from Nyquist plots that the resistance of GCN-Ag composite film decreases considerably in comparison with pristine GCN film [31]. GCN doped silver composite film exhibits lowest resistance value in comparison to pristine GCN sample. Both real and imaginary parts of impedance mainly depend on grain and total resistance in the LCR circuit as seen in the inset Figure. 9. The doping of silver nanoparticles in the GCN polymer matrix reduces the grain and total resistance of the polymer as shown in the complex LCR circuit. It is observed from the Nyquist plots that the area under the curve decreases for the composite sample indicating reduction in the bulk resistance of the composite sample which may be directly correlated to its electrical conductivity. It is also pointed out that, the value of series resistance and capacitance reduces leading to the enhancement of overall conductivity of GCN-Ag composite thin film.

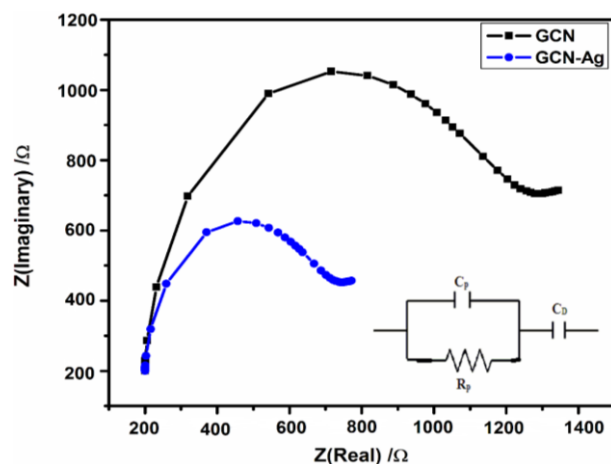


Fig. 9. Nyquist plots of pure GCN and GCN-Ag composite thin film.

5. Conclusion

The pure GCN and GCN doped silver nanoparticles based organic thin films were prepared employing simple dip coating method. The prepared thin films were characterized by employing SEM, FTIR and X-ray diffractometer characterization techniques. The synergistic effect of silver nanoparticles doped into the GCN polymer matrix on the physio-chemical features of GCN were analysed by using these characterization techniques. The temperature dependent electrical conductivity study reveals the fact that the doping of silver nanoparticles into the GCN polymer matrix considerably improves the electrical conductivity of pure GCN thin film. The dielectric and impedance studies of pure GCN and GCN doped silver nanoparticles suggest the fact that, the inclusion of silver nanoparticles in GCN polymer matrix has a greater impact on material characteristic properties. Owing to the easy method of preparation of thin films, optical transparency, high flexibility and higher value of electrical conductivity, these silver nanoparticles doped GCN organic thin films could be used as potential candidate especially in the fabrication of electrode materials for opto-electronic devices.

Acknowledgements

The authors would like to acknowledge financial support for this work, from the Deanship of Scientific research (DSR), University of Tabuk, Tabuk, Saudi Arabia, under Grant No. S-1441-0157.

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