Enhance the Electrical Properties of the Novel Fabricated PMMA-PVA/ Graphene Based Nanocomposites

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Abstract

Graphene has unique electrical properties used to improve the electrical properties of nanocomposites. Polymathic methacrylate (PMMA) and polyvinyl alcohol (PVA) were used as the polymers model. Novel acoustic-solution casting method was succeeded after applied various parameters to mix the PMMA dissolved in Dimethylformamide (DMF) with PVA dissolved in distilled water (DW) and DMF. New (PMMA-DMF)-(PVA-DW-DMF)/ Graphene oxide (GO) nanocomposite was successfully fabricated for the first time with different dissolved solutions and the addition of various ratios of GO nanosheets. Fine desperation and homogeneity of matrix and GO distribution in samples were achieved using a range of factors using the new method as presented by the image of the optical microscope (OM). The most functional group of polymers and GO with strong interfacial interaction were illustrated using Fourier-transform infrared spectroscopy (FTIR). The A.C. Electrical Conductivity was measured by the electrical properties of samples. The finding of electrical properties of the PMMA-PVA/GO nanocomposites exposed an enhancement in most of the electrical properties, for instance, dielectric constant, A.C conductivity and dielectric loss up to 50%, 96.3 % and 96.7 % with the increase of concentrations ratio of GO by 0.09, 0.18 and 0.27 wt. % respectively. These results helped to discover these new promising nanocomposites for wide electronic devices and applications.

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Key words: graphene oxide, PMMA, PVA, electrical properties, nanocomposites.

1   Introduction

Polymer nanocomposites have gained important research and industrial interests due to their excellent possibility for variable applications. The field of nanotechnology has been a very exciting topic and it can make our lives better. Polymer nanocomposites consist of a copolymer or polymer having nanoparticles or nanofillers scattered in the polymers matrices [1], [2]. The installation of the polymer-based nanocomposite is an essential feature of nanotechnology of the application of the polymers. The contribution of the nanometric inorganic as nanofiller, polymer-based nanocomposites represent a newfangled that different from the traditionally addition of the polymer [3]. Polymers have limited use for the industry of goods and structures on their own because their properties are not very rising when compared to other materials, e.g., most metals and clay [1], [4]. The characteristic of polymers improves and subsequently this has many applications. These are depended on the inorganic filler that is present inside the polymers. Considered solvent casting methods are simplest way for the installation of polymer nanocomposites and is less time-consuming.

Graphene is an essential topic of polymer-graphene based-nanocomposites [5], [6] that have been established and exhibit interesting improvements in the physicochemical properties [7]. Nanotechnology is exhibited promising developed and enhanced specifically in nanomaterials[8], graphene is considered the best potential nanofillers these days [5], [9], with his excellent and unique electrical [10], [11], mechanical properties [12], [13] and thermal [14], [15], etc. Recently, graphene an excellent applicant has been applied in many positional applications for instance, solar cells [5], transparent electronics [16] and sensors [6], [17], etc. Developed and improving the material properties depends on many elements such as, strong interfacial interactions between components of the nanocomposite, fine dispersion of the nano-filler in the matrix, component properties and the nanomaterial contribution, etc. [9]. These influences are considered the most significant led to impartment improving in the properties of the nanocomposites if it is achieved [18], [19].

PMMA is preferred material by researchers and engineers because it is easily hydrolyzed ester groups, it makes a low resistance to several chemicals [20]. PMMA is durable and transparent as well as environmental stability, etc. It uses in TV screens, vehicle appliances, lenses for glasses and outdoor applications, etc. [21].PMMA was blended with various materials using different methods polymers and nanofillers for improving the optical [22], [23], electrical [24], [25], thermal stability [26], mechanical [27] and others [28] properties of PMMA, whereas the electrical properties of PMMA-PVA blend were reported by only two investigations [25], [29].

S. Rajendran and O. Mahendran [25] were reported solvent applying a casting technique to blend PMMA and PVA with load $LiC10_4$. The samples were studied applying the finding of the impedance that was in the presence of ionic conductivity. The reported the highest conductivity that is $0.06*10^3$ $S.cm^{-1}$ at 303 K of the sample at room temperature. This makes the PMMA-PVA encouraging for lithium battery applications. PVA and PMMA as a composite polymer hydrogel were investigated by G. M. Joshi and K. Deshmukh, in (2014) [29] using a Boric acid as agent crosslinking. Their results exhibit an enhanced in the dielectric property, $\varepsilon = 590$ for samples as a gel in comparison to 400 of the PVA sample. The study helped to control and a better understanding of the electrical parameters using impedance tool. It giving specimens to manipulate the electrical parameters for the desired effect in the magnitudes and could be promising in several biomedical applications. An aqueous suspension was applied by P. P. Peregrino *et al.*, [30] to the caste of the acetylated starch (ST) with PVA and different addition of GO to prepare Bio-nanocomposite films.

Increasing the ratio of loading GO after converted to RGO present an improved thermal and electrical properties.Raman spectroscopies and Fourier transform infrared with reflectance (ATR-FTIR) were existed to record the interactions at the molecular-level between components as the main hydrogen-bonding. The findings result of both electrical resistivity and thermal diffusivity were improved by ST-GO nanocomposites after the photochemical treatment. H. J. Salavagione et al, [31] reported the addition of reduced graphite oxide to Poly(vinyl alcohol) to prepare nanocomposites.

The changes in the thermal behavior in the nanocomposites is related to the formation and interactions of hydrogen-bonding for a remarkable improvement.At the concentrations 7.5 wt% of RGO, high electrical conductivity is reached (0.1 $S cm^{-1}$) and 0.5-1 wt. % of the percolation threshold. K. Sa, et. al. in (2018) [32] investigated the electrical, thermal, and mechanical properties of multi-walled carbon nanotubes (IL-MWCNTs) with rGO and PMMA. The solvent-casing method was used to prepare these samples and the result showed a significant influence of IL-MWCNTs on samples properties. The mechanical properties were enhanced by up to 119 % and 58 %, respectively. Meanwhile, thermal properties occurred more stability after the incorporation of IL-MWCNTs in the nanocomposites.

Moreover, the electrical conductivity of IL-MWCNTs-RGO/ PMMA nanocomposites exhibited better than the composite IL-MWCNTs/ PMMA composite that without RGO. Where, strong interaction were formed between the IL-MWCNTs with RGO for side and with the matrix from another side. H. Fan, et, al. in (2019) [33] fabricated PMMA/GO as an Organic field-effect transistor (OFET) as gas sensors of ammonia applying solution-processed hybrid dielectric. PMMA and GO were dissolved in anisole and dimethylformamide (DMF) with 2:10 ratio respectively, applying ultrasonic processed.

30 nm thickness of film of GO/PMMA nanocomposites was designed using a spin-coating approach (2000 rpm) to get GO/PMMA film with 30 nm thickness the layer of gold (Au) that was thermally deposited with 50 nm thick on the semiconducting film as a source and drain electrodes under 3×10^{-3} Pa.The results presented a notable enhancement of sensing performance of the different concentrations of NH3 by increasing the ratio of GO in the matrix. This finding exhibited the qualified to the powerful bonded between functional groups of GO nanosheets and molecules gas of NH3. G. Kandhol, et, al. in (2019) [34] reported the preparation of PVA with various loading ratios 1, 1.5 , and 2 wt. % of rGO.

The solution-mixing method with and evaporative casting technique were applied to synthesis the nanocomposites. This study was focused on the influence of the frequency with the range between 100 Hz to 1.5 MHz on the temperature-dependent dielectric properties. The finding reported an improvement in the ac conductivity and dielectric constant through improve in the rGO loading ratio in the nanocomposites. The same behavior was achieved by other researchers [35], [36] after increasing the ratio of loading GO with PMMA or PVA.

Despite all the above studies, researchers [30], [31], [35], [36] reported the effect of graphene on only PMMA or PVA separately. This paper is the first study that aims to investigate the influence of addition GO of newly fabricated PMMA-DMF with the PVA-DW-DMF as nanocomposites. The developed mixing-sonication-acoustic method was applied to achieve a fine homogeneous mixture among the components in the matrix. Nanocomposites films were successfully prepared and characterized the structure and electrical properties using FTIR, OM, and the A.C. Electrical Conductivity.

The manuscript is structured as follows: section II exhibits the experimental section included the materials, Synthesis of Graphene Oxide, Purification of the nanocomposites and characterization, Section III describes the theoretical part, Section IV presents the Results and Discussion section. Finally, section V demonstrates the conclusions for this study.

2 Experimental Part

2.1 Materials

PMMA (99% purity, 20000-18000 g mol^{-1} and 213 ºC melting point) and PVA (99% purity, 18000-12000 g mol^{-1} and 230 ºC melting point) were provided by Tuttlingen Company and Panreac Company, Spain, respectively. graphite ($\leq$ 39μm), Sodium nitrate (NaNO3), sulfuric acid (H_2SO_4) (99.5% analytical grade), hydrogen peroxide, potassium permanganate (KMnO4) and hydrochloric acid (35%) were purchased to synthesis graphene oxide form Sigma-Aldrich, United Kingdom.

2.2 Synthesis of Graphene Oxide

Our group was predicated the GONs and the full characterization was available in the literature [6], [37].

2.2.1 Purification of the Nanocomposites

The various ratio of PMMA-DMF and PVA-DW/DMF was applied to get the appropriate methods to mix these two polymers with different solvents. Finally, we reported the methods that accessed not only mixed two of these materials as a fine homogeneous matrix but also with good GO dispersion as briefly in the following steps:

1-   PVA (1 g) was dissolved in 50 ml DW, were stirring for 1 h 80 ± 3 C°, meanwhile, PMMA was dissolved in DMF with 6 g in 50 ml (DMF) using a magnetic stirrer for 3h at 80 ± 3 C°. At the same time, GO nanosheets were dispersed in DMF with 0.09-0.27 wt.% in 50 ml using a magnetic stirrer and sonication bath as an acoustic solution method for good dispersion of GO in the solvent. All these steps were carried out separately before mixing the materials.

2-   After completely dissolved of the PVA in DI, 10 ml of DMF were added to 30 ml of PVA-DI solution then were mixed using magnetite stirrer for 0.5 80 ± 3 C° for better homogenous.

3-   5 ml PVA-DIS-DMF were added to 25 ml of PMMA-DMF solution and mixed for 4 hours using a magnetic stirrer at 80 ± 3 C° and the polymers matrix was fine homogenous during this time.

4-   Then the temperature of the PVA-PMMA matrix was reduced to about 35 ± 3 C° before adding three ratios of GO-DMF to prepare three nanocomposites.

5-   After the addition of GO, the mixing-sonication methods were applied. Where the mixing of the PVA-PMMA/GO matrix was continued for another 3 h then it was sonicated for 15 minutes.

6-   This procedure was repeated four times to get fine homogenous and dispersion of GO in the polymers matrix. Table 1 demonstrates the summarization of the fabrication method and preparation of (a) GO, (b) PM-PV1, (c) PM-PVGO2, (d) PM-PVGO3, and (e) PM-PVGO4 nanocomposites.

Table 1: Summarized the purification method of (a) GO, (b) PM-PV1, (c) PM PVGO2,(d)PM-PVGO3 and (e)PM-PVGO4 nanocomposites.

Sample ID	Concentration Wt. %			Total Time h		Drying method
	PVA	PMMA	GO	Sonication	Mixing	
GONs	0	0	100	4	4	Freeze drier
PM-PV1	4	96	0	0	4	40 ± 3 C° under air
PM-PVGO2	3.91		0.09	45	12	
PM-PVGO3	3.82		0.18	60	12	
PM-PVGO4	3.73		0.27	75	12	

2.2.2 Characterization

Nikon, Olympus model 73346, was recorded the Optical Microscope (OM) images. Fourier transforms infrared (FTIR) spectra (Vertex 701) from Bruker, Germany, was characterized the samples in the region between 4000 - 400 cm^{-1}. The A.C. electrical conductivity was recorded out using LCR meter (Hi TESTER, HIOKI 3532-50, Japan) with a range of variable frequencies (100Hz-5MHz) that was applied to characterizes the measurement for all samples under room temperature.

3 Theoretical Part

The dielectric constant (ε') of (PMMA-PVA/GO) nanocomposites can be considered using the following equation [38].

$$\varepsilon' = \frac{C_p}{C_o}$$

(1)

C_P means the parallel capacitance, C_o means the vacuum capacitor, and dielectric loss (ε''): is the dissipated energy as heat due to the absorbed the electric energy through the material that when the dielectric material is exposed to the A.C voltage, It can be calculated for (PMMA-PVA/GO) nanocomposites using the following equation [39].

$$\varepsilon'' = \varepsilon' D$$

(2)

D means the factor of dispersion of (PMMA-PVA/GO) nanocomposites. The conductivity of A.C electrical (σ_{AC}) was determined by applying the following equation [39].

$$\sigma_{AC} = \omega \varepsilon_0 \varepsilon''$$

(3)

ω: Angular frequency ($\omega = 2\pi f$, ε_o)means the vacuum permittivity and σ_{AC} means the generated temperature measurements in the isolated materials.

That caused by the charges vibration or dipoles rotation in their positions, as a consequence of the alternating of the field [40].

4   Results and Discussion
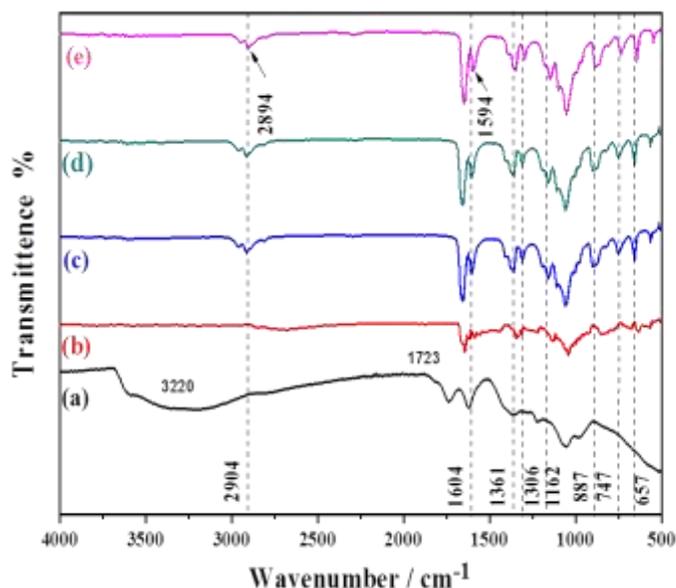
Figure 1: FTIR spectra of (a) GO, (b) PM-PV1, (c) PM-PVGO2, (d) PM-PVGO3 and (e) PM-PVGO4 nanocomposites.

The FT-IR spectrums of GO, PM-PV1 and PM-PVGO nanocomposites were illustrated in Figure 1 that was documented in the range between 4000-500 cm^{-1}. In Figure 1 (a), GO FTIR spectrum has revealed a wide peak at 3220 cm^{-1} that is related to the hydroxyl groups (O–H) stretching vibrations. The peak at 1723 cm^{-1} is linked with carbonyl groups (C=O) stretching [6]. A skeletal ring (C=C) is presented at 1604 cm^{-1}. That is a stretching vibration functional group of the sp2 carbon domain as un-exfoliated GO sheets. Additionally, at 1374 cm^{-1} an epoxy (C–O–C) and 1040 cm^{-1} a C-O functional groups are also presented in the FTIR spectra in agreement with the literature [41].

Several peaks were presented of the PMMA-PVA blend spectra as shown in Figure 1 (b), these peaks were CH_2 at 1646 cm^{-1}, C-H stretching at 1604 cm^{-1}, O-H bending 1361 cm^{-1} and O-H bending and C-O (ester bond) stretching vibration between 1260-1000 cm^{-1}. Where these peaks were associated with the net of both PMMA [42] and PVA [43].

New strong functional peaks were presented in the nanocomposites due to the contribution of the GO, these new strong bands were at 2904, 1604 and 887 cm^{-1} as well as below that clearly displayed in Figure 1 (c, d, and e).

Moreover, increasing the loading of GO presented an increase in the intensity of other peaks which also clear exhibited in the spectra of nanocomposites.Where it was noticed that functional peaks of PM-PVGO4 were shifting in up to 10 cm^{-1} of the most functional groups with increasing the ratio of the GO to the highest ration that was 0.27 wt %.This also demonstrated an increase in the intensity peaks because of the addition of the nucleating agent GO/PMMA-PVA.

The infrared spectra indicate significant changes and strong interaction formed between GO and polymer. This could reveal to the formulation of the hydrogen bonds between the O-H and the C=O in the GO and the PMMA-PVA, respectively [44].
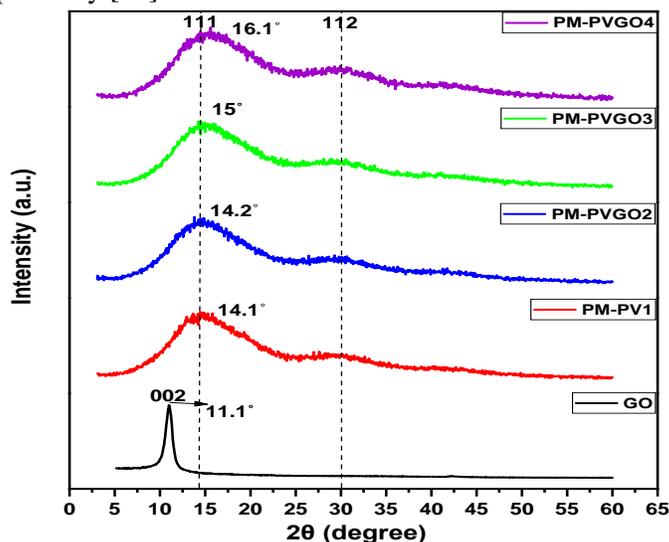

Figure 2: XRD patterns of GO, PM-PV1 and PM-PVGO nanocomposite.

The XRD patterns of GO, PM-PV1, PM-PVGO2, PM-PVGO3 and PM-PVGO4 nanocomposites illustrated in Figure 2. XRD peak at $2\theta = 26.5^o$ is reported of graphite and later the strong oxidation of GONs, this peak was a move to lower the interlayer spacing, where at $2\theta = 11.1^o$, a sharp peak is presented with 002 diffractions of GONs, the Bragg equation was used to calculate the interlayer spacing that is 0.79 nm of GONs. This shifting confirmed the strong oxidation of graphite sheets and expand the interlayer spacing by intercalated of functional group and produced of GONs in agreement with the literature [28][45].

At $2\theta = 14.1^o$ and 30°, two broad and sharp peaks of PMV1 with (111) and (112) diffractions were exhibited and related to the PMMA in agreement with the literature [28][46]. It was clearly observed that the wide and sharp intensity pattern of PMMA between 7° - 23° was covered in the area of both patterns of the GO and PVA located at $2\theta = 11.1^o$ and $2\theta = 19.8^{o,}$ respectively. It was expected the both GO and PVA will not present and could overlap with PMMA.

Whereas, the contribution of GONs exhibited shifting in peaks of the PMMA from $2\theta = 14.1^o$ to 14.2^o, 15° and 16.1^o with increasing the loading ratio of GONs from 0 wt.% to 0.09, 0.09 and 0.09 wt % in the PM-PVGO2, PM-PVGO3 and PM-PVGO4, respectively. The expansion of the volume in the macro could be the main reason for this shifting as well as the increment of interplanar crystal spacing [47].

The XRD results are agreement the FTIR finding of the formulation of strong interfacial interaction in the matrix between both polymer and GONs, besides that was not an influence on the crystal structure of the polymers in the nanocomposites in strong agreement with the finding in the literature [11][47].
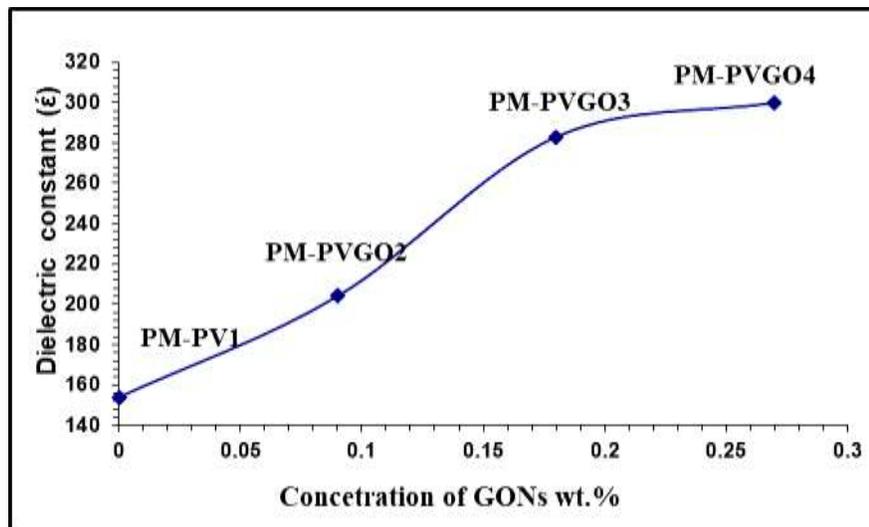

Figure 3: The dielectric constant with GONs concentration of PM-PV1 and PMMA-PVA/GO nanocomposites.

Figure 3 demonstrates the influence of adding graphene oxide nanosheets (GONs) on the dielectric constant at 100Hz under room temperature that was (27 C$^\circ$ ± 3). The increase in the concentration of GONS led to an improvement of the results of the dielectric constant that is attributed to the development of clusters. This led to higher space charge polarization, which is extended the most value to dielectric constant. the presented results indicated a significant improvement in dielectric constant, up to 25 %, 48.3 % and 50 % with the increase of concentrations ratio of GO to 0.09, 0.18 and 0.27 wt. % respectively, in the nanocomposites. The same behavior was reported in the literature [33][34].
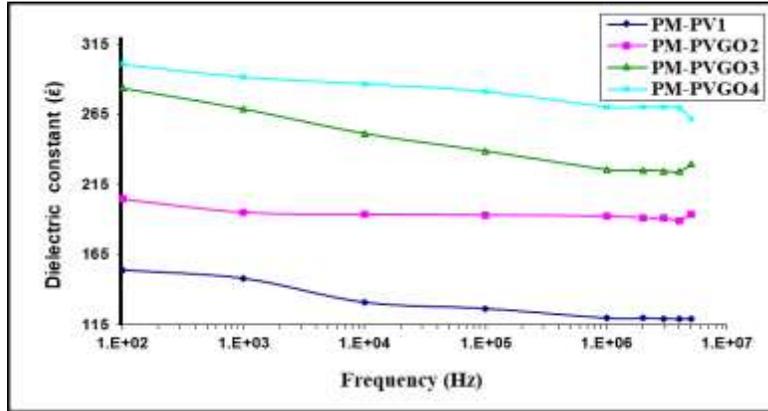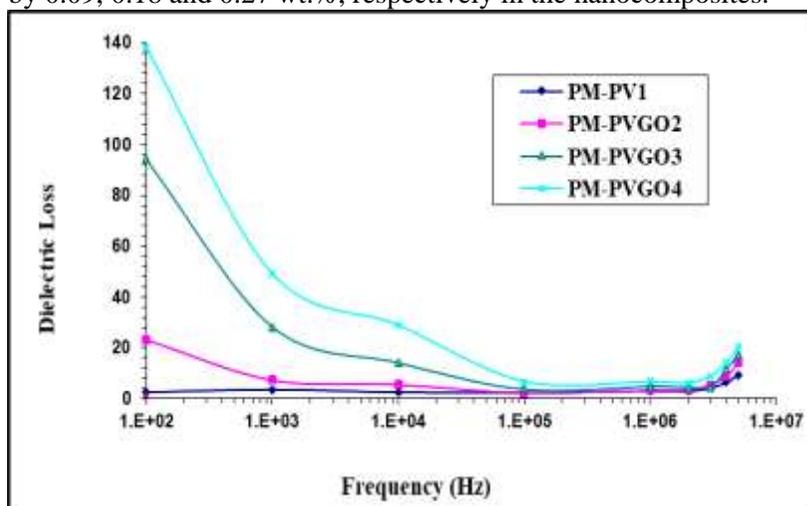
Figure 4: The dialectical constant with the frequency of PM-PV1 and (PMMA-PVA/GO) nanocomposites.

The dielectric constant with the frequency of PM-PV1 and (PMMA-PVA/GO) nanocomposites was indicated in Figure 4. At low frequency, the dielectric constant values were high. The high value of the low-frequency dielectric constant was described by the space charge presence. This led to the growth of the carriers of the charge near the electrodes.Whereas, at higher frequencies, Dielectric constant with frequency observed because of the periodical reversal of the field. The charge carriers are difficulty can to orient themselves in the field direction as a consequences of the reduction in values of the dielectric constant [48]. At a low concentration of graphene oxide nanosheets (GONS), the dielectric constant was weak. The dielectric constant was increased with a rise in the concentrations of graphene oxide nanosheets with an abundance of functional groups. Where the presented results indicated a significant improvement in variation dielectric constant, Where it enhanced up to 24.3%, 44.7 % and 48.4 % with the increase of concentrations ratio of GO by 0.09, 0.18 and 0.27 wt.%, respectively in the nanocomposites.
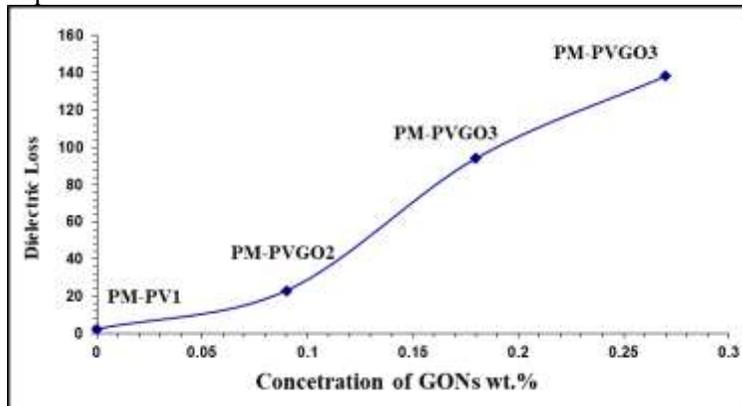
Figure 5: The dielectric loss with GO nanosheets concentration pf PM-PV1 and (PMMA-PVA/GO) nanocomposite at (100) Hz.

Figure 5 shows the effect of adding graphene oxide nanosheets (GONs) in the nanocomposites on the dielectric loss. Dielectric loss exhibited notable increases with the rose loading ratio of GONs. The increase in the dielectric loss and the content of the nan-filler ratio can be qualified to improve in the electronic charge, which can be increased due to an increase in the content of the GONs. Graphene sheets are well known that have unique electrical properties that exhibited a direct effect on the electrical properties that led to improving the dielectric loss of the nanocomposites. It was notable enhanced up to 79.2%, 94.5 % and 96.3 % with the increase of concentrations ratio of GO by 0.09, 0.18 and 0.27 wt.%, respectively in the nanocomposites.

Figure 6: The dielectric loss with the frequency of PM-PV1 and (PMMA-PVA/GO) nanocomposite.

Figure 6 illustrated the dielectric loss against the frequency of PM-PV1 and (PMMA-PVA/GO) nanocomposite. The figure demonstrates the decreasing of dielectric loss values of nanocomposite with a rise in the frequency of applied electrical field. This activity has indorsed the reduction in the contribution of space charge polarization and the dielectric loss, exhibited a high result for the samples with low frequency, which is decreased with the increasing in the frequency. Whereas, the contribution of GO significantly improves the dielectric loss of nanocomposite PMMA-PVA/ GO nanocomposite with increasing the loading ratio of GONs, which is associated with an increase in the number of the charge carrier's. At lower loading ratio of graphene oxide nanosheets, it forms clusters shape, when the ratio of (GONS) become a high, the (GONS) result a continuous network in the nanocomposites. Where the presented results indicated a significant improvement in variation dielectric constant, Where it arrived up to 66.7%, 70 % and 94.3 % with the increase of concentrations ratio of GO by 0.09, 0.18 and 0.27 wt.%, respectively in the nanocomposites in agreement with the previous investigation [49].
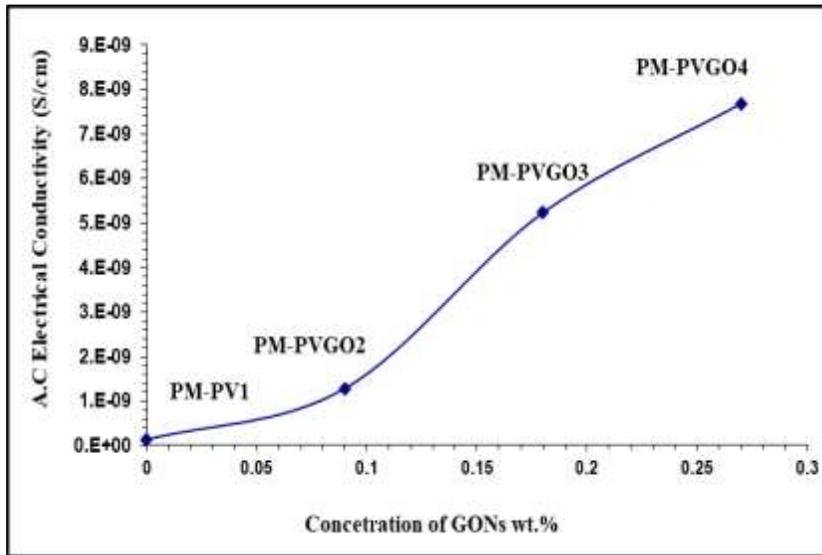
Figure 7: A.C electrical conductivity with the concentration of GONs at 100 Hz of PM-PV1 and nanocomposite.

Figure 7 shows the A.C conductivity and graphene Oxide concentration of PM-PV1 and PMMA-PVA/GO nanocomposite at 100 (Hz). The A.C conductivity increased with rise the loading ratio of GONs. GONs have energy sites on the nanosheets instead of these in the polymers led to this improving in the charge carrier's number then the transfer of the charge. The presented results indicated a significant improvement in variation dielectric constant, Where it significantly improved up to 80%, 95.3 % and 96.7 % with the increase of concentrations ratio of GO by 0.09, 0.18 and 0.27 wt.%, respectively in the nanocomposites, These results agree with other researchers' results [32][50].
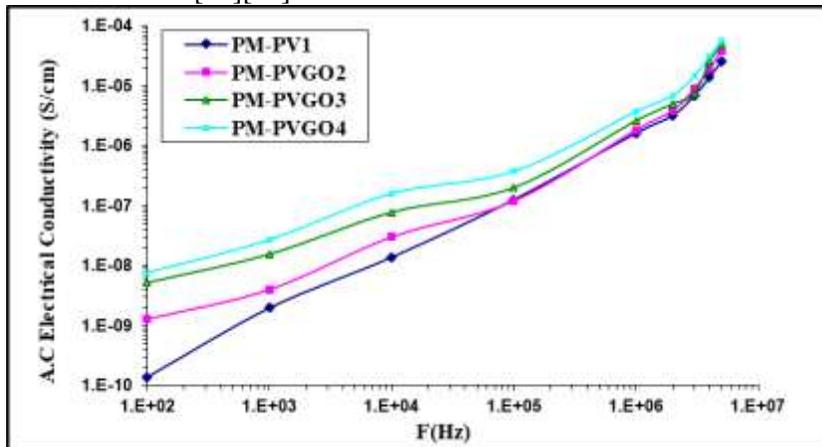

Figure 8: The A.C electrical conductivity with the frequency of PM-PV1 and (PMMA-PVA/GO) nanocomposites.

The conductivity for (PMMA-PVA/GO) nanocomposite against frequency is shown in Figure 8. The figure displays that A.C conductivity behavior increased substantially with the rise in the frequency (f) from $(1\times10^6$ - 3×10^6 Hz). In the conduction band, this is recognized when the charge carriers' number was growing and also due to the charge carriers excitation to upper states. Accordingly, the conductivity was enhanced with the rise of the frequency, also the increasing ratio of GO presented significant enhancing in the result of all the nanocomposite in comparison with PM-PV1, in agreement with the other finding [51][52].
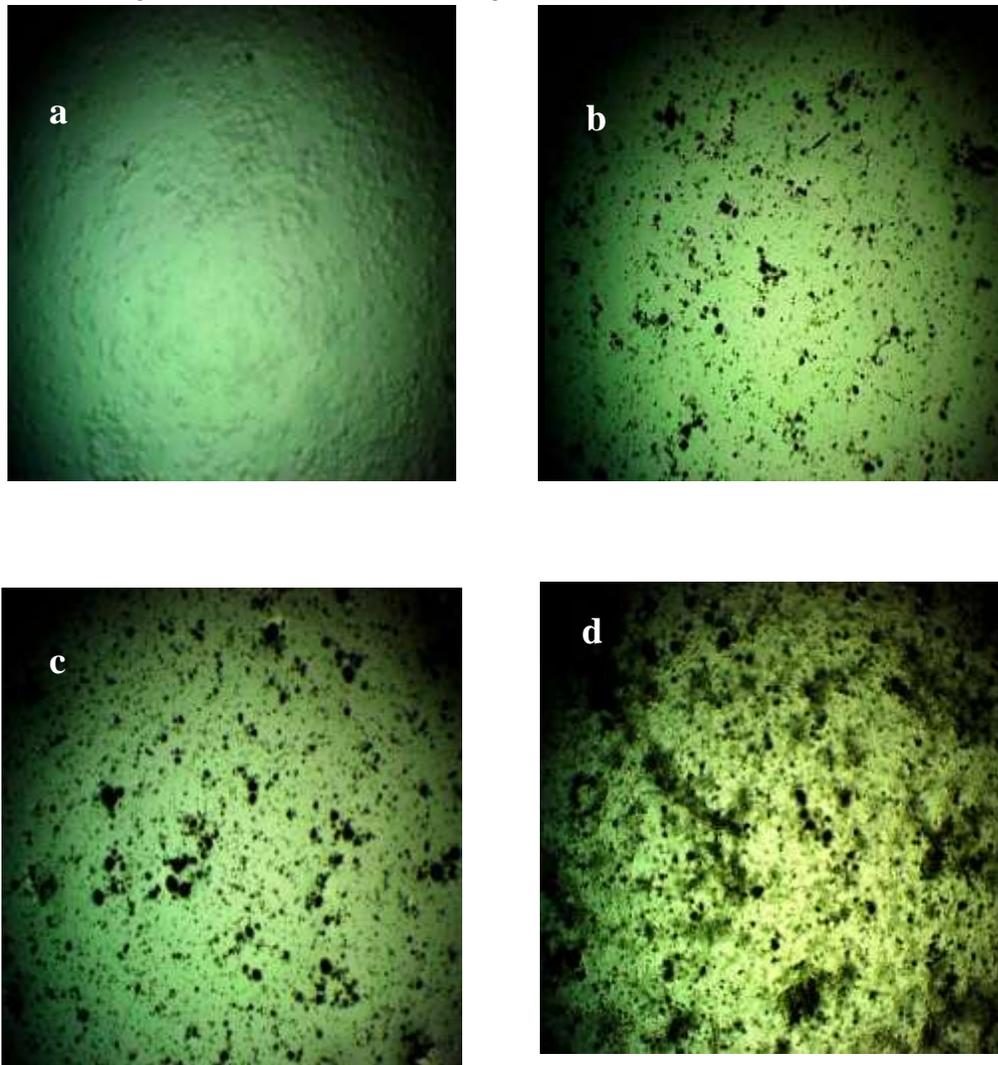
Figure 9: the images of the optical microscopy with a magnification of (40X) of (a) PM-PV1, (b) PM-PVGO2, (c) PM-PVGO3 and (d) PM-PVGO4

The optical images of PMMA-PVA and PMMA-PVA/GO nanocomposites with different concentrations of GONS magnification power at 40 X were revealed in Figure 9. Fine distribution of GO was presented with the blend-polymer nanocomposites.in addition to fine homogeneity of the matrix. Where these images exhibited the successful method that was implied to fabricated of the PMMA-PVA/GO nanocomposites. It clear observed that the distribution of GONs was considerably enhanced with increasing the loading ratio of the GONs, where the sample PM-PVGO4 exhibited the better clearly in figure 9 (d), and this support and described the best results of this sample in agreement with other results. Moreover, the contribution of GONs there is no aggregation or emigration of The GONs or even no influence on the transparency of the films.

5   Conclusions

New PMMA-PVA/ GO nanocomposites were successfully fabricated for the first time in this study and investigate using mixing-sonication- casting methods. Strong interfacial interactions were formed among both polymers and GONS were presented in the FTIR and XR. OM was shown good homogenous and fine dispersion of the GO nanosheets in the nanocomposite matrix. Additionally, both the contribution of GONS and increasing the loading ratio observed considerably improved in most of the electrical properties such as dielectric constant, dielectric loss and A.C conductivity up to 50%, 96.3 % and 96.7 % with the increase of concentrations ratio of GO by 0.09, 0.18 and 0.27 wt. % respectively, in the nanocomposites. These results helped to understand these materials together as it is the first study for these materials together, also it reduced the gap of knowledge and discovered promising materials for new applications.

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