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New Fabricated (PAA-PVA/GO) and (PAAm-PVA/GO) Nanocomposites: Functional Groups and Graphene Nanosheets effect on the Morphology and Mechanical Properties

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Abstract. Graphene reinforcement brings high attraction from researchers as its sole properties improving the mechanical and structural properties of nanomaterials. The research aims to explore the effect of the functional group of the polymers have the same mainstay of the polymer chain but with a different end functional group in addition to the influence of graphene oxide (GO) nanosheets. Poly (acrylic acid) (PAA) and Poly (acrylamide) (PAAm) were the model polymer. These polymers mixed with Poly (vinyl alcohol) (PVA) separately after dissolved in distilled water (DW) then reinforced with GO applying the developed acoustic-sonication-casting method. The applying method was successfully fabricated the new nanocomposites from these mixed materials for the first time with a ratio of 4.5:4.5:1 wt. % of PAA: PVA: GO and PAAm: PVA: GO as nanocomposites, respectively. The new nanocomposites exhibited homogeneous combinations with an acceptable dispersal of GO in the polymers matrix as presented in the visual microscopy (OM). Coupled with the strong interplay between polymers in the matrix and the polymer with GO nanoparticles as nanocomposites, Fourier transform infrared (FTIR) verified the successful preparation of GO. Ultrasound measurements with a multi-frequency channel (30, 40 and 50 kHz) were used to characterize the ultrasonic velocity, absorption coefficient, compressibility and Bulk modulus, etc. Most of the mechanical properties were significantly improved of both new nanocomposites after the contribution of GO in comparison with blended polymers for both nanocomposites up to 74 %, 100 %, 198 and 177% respectively compared to their blended polymers. Interestingly, the PAA-PVA/GO nanocomposites presented a smoother surface and better mechanical results in comparison with PAAm-PVA/GO that showed rough surface and soften behaviour. The results of these new nanocomposites could aid in the development of promising materials for a variety of applications, such as improving oil recovery or developing medical and pharmaceutical applications.

Keywords: PAAm, PAA, GO, PVA, mechanical properties, reinforcement, nanocomposites.

1- Introduction

The functional group is one of the important factors and key that have a direct effect on fabricated nanocomposites that the nanofillers in the matrix and the polymer have a strong interfacial interaction. In addition to achieving a stable and same scattering of nanofiller the



matrix of the polymer. As a result, the properties of polymer nanocomposites can be greatly improved. [1,2]. Whereas, lack in the functional groups could lead to loss of bonding and aggregation that may lead to weak interaction and drawback in the improvement of properties of the new supplies. So, several experiments have been conducted to advance the homogeneity of the diffusion of the filler and interactions to enhance the compatibility of nanofillers in the polymers matrix applying functionalizing the nanofillers, such as graphene [3,4], silica nanoparticles [5].

Several factors are affecting preparation and fabricated polymer-based nanocomposites, such as strong interfacial interaction, good homogeneity and fine dispersion in the matrix. [6–8], etc. That is significant to achieve high performance and enhancements in the properties of the nanocomposites. The functional group of both polymer and nanofillers are so important to achieve this goal. This important factor does not been focused and report till now. Therefore, this study focused on polymers that contain the same backbone but with different end functional groups such as the first one contain an amide group and the second contains an acid. Polyacrylamide (PAAm) and Poly acryl acid (PAA) are polymers that have the same backbone but with different functional groups which are amide and acid, respectively. The former is a water-soluble polymer. This polymer has several advantages of not being similar to monomers and non-toxic. It is commonly used to increase the viscosity of water (i.e. thickening the solution) and it is a chemical compound that can be applied in a broad variety of applications as it is used in the manufacture of soft contact lenses because it has a high water absorption percentage and is in the form of a gel [9]. Moreover, it is used in water and mining treatment, oil recovery, surface grafting polymerization, flocculants, in the field of soft tissues and artificial corneas and in the manufacture of tissues to cover burns [9]. Polyacrylic acid (PAA) each monomer unit contains a carboxylic group. The dissociation of acid groups in this polymer causes it to become a polyelectrolyte in water. Polycarbonate (PAA) contains a carboxylate group of each of the main chain's two carbon atoms when all carboxyl groups split, it has a high negative charge density. [10]. Polyvinyl alcohol (PVA) is a polymer with unique properties, including water solubility, biodegradation, biocompatibility, and toxin-free [11]. PVA has a sufficiently high tensile strength and satisfactory elasticity. PVA is usually annealed by a wide range of low-molecular-weight compounds that often contain groups polarized [12,13], which form hydrogen bonds with the hydroxyl groups of the PVA chain (with or without water), reducing direct hydrogen bonding between large PVA molecules [14,15].

Graphene is a two-dimensional carbon atom network. [16] with carbon particles with unique mechanical properties that are ultimate 130 GPa tensile power and a high Young's modulus of 1 Tpa [17]. It is considered the strongest material ever discovered, flexible membrane and able to modify in the carbon backbone, such as functionalization the graphene [18]. Graphene oxide's surface and structure are rich in broad and diverse functional groups such as hydroxyl, carbonyl, carboxyl, and epoxy groups, making it one of the best nanomaterials available [19]. The presence of these groups causes Graphene oxide nanoparticles to become hydrophilic and capable, resulting in a more uniform distribution in polymer nanocomposites and strong interactions in the matrix with the polymer. [13,20]. Graphene oxide (GO) serves as an important precursor of the graphene-based structure Because of its hydrophilic nature and ability to process a solution, it has also attracted a lot of interest, especially from chemists. In addition, GO is used in nanoelectronic instruments. gas sensors, supercapacitors, and drug delivery systems, [10].

P. Onder et al. (2017) [21] The mechanical properties of graphene oxide - polyacrylamides were investigated before and after swelling in water.” The mechanical behavior of GO-PAAm compounds made from various GO materials was evaluated using the piezo test technique before and after the swelling process. To measure force versus stress, this composite technique was used. The mechanical properties of GO-PAAm compounds, which are highly dependent on the GO content, improved significantly. [21]. Luo, YL, Feng, QS, & Xu, F. (2011) [22] reported the training and characteristics of Copper nanocomposites made from

PVA-PAAm IPN hydrogels and PVA-PAAm IPN hydrogels with acceptable swelling ratios. Where excellent mechanical strength of compression is achieved by adjusting and controlling. This nanocomposite hydrogel is supposed to have a wide range of applications in a drug-delivery system. H.C Schniepp et al., (2006) [23] investigated the process for producing functional graphene single sheets by thermal peeling of graphite oxide. Graphene sheets have already produced notable thermal, mechanical, and electrical properties, combining the best features of both clay and carbon-tube-based nanocomposites in one material. Functional single-layer graphene sheets are expected to provide further benefits, not only for improvements in structural properties for the development of new materials such as polymers, electrically conductive inks, and supercapacitors.

Few investigations reported PAA or PAAm with PVA and these materials have never been reported with graphene or its derivative. Therefore, the research aims to study the influence of polymer function group, reinforcing using graphene nanosheets with a different frequency of the ultrasound velocity on the mechanical properties and structure of new fabricate (PAA-PVA/GO) and (PAAm-PVA/GO) nanocomposites

2- The experimental part

2.1 Materials

Poly (vinyl alcohol)(PVA) with ($160000 \text{ g mol}^{-1}$) molecular weight was complete by Dindori, Nashik, India. Poly (acrylamide) (PAAm) with molecular weight (5-30 million g mol^{-1}) purity $\geq 90\%$. Color: white. Appearance: crystal granular. Poly (acrylic acid)(PAA) with molecular weight ($250000 \text{ g mol}^{-1}$) Appearance white crystal powder was provided from Shenzhen sendi Biotechnology Co.Ltd- China. Sigma-Aldrich Company, UK, produced graphite powder ($\leq 40\mu\text{m}$), hydrochloric acid (35%), potassium permanganate, sulfuric acid (99.8%), hydrogen peroxide, and sodium nitrate.

2.2 Methods

2.2.1. Constructing of Graphene Oxide (GO)

The procedure of preparation GO was described in our previous publication [18].

2.2.2. Synthesis of blended polymer and Nanocomposites

Graphene oxide (GO) was dispersed at 1 wt. In distilled water. % using magnetic stirrer with the assist of sonication bath until get fully dispersion and homogeneous GO-distilled water. In distilled water, PAA, PAAm, and PVA were dissolved separately using a magnetic stirrer. PAA was mixed with PVA and PAAm with PVA with ratio 1:1, to prepare the blended polymer PAA-PVA and PAAm-PVA, respectively. The GO nanosheets were added to the mixture of both polymers to synthesis the new nanocomposites, which were PAA-PVA/ GO and PAAm-PVA/ GO, respectively. With the addition of GO solution to PAA-PVA and PAAm-PVA, the samples were mixed with a magnetic stirrer. 30 minutes and then sonication for 15 minutes, followed by this method repeatedly for two weeks. In order to achieve strong GO homogeneity and dispersion in the polymer matrix. Lastly, these samples were placed in a Petri dish using Methods of casting dish under air to dry and obtain fine samples.

2.3 Characterization

Full details of characterizations used in this study were summarized in Table (1).

Table (1). Summarized the characterizations used.

Device	Model	Details	Manufacturing, Country
Fourier Transforms Infrared (FTIR)	vertex 70	Measurements range between $4000 - 500 \text{ cm}^{-1}$	Bruker Company, Germany
Optical Microscope (OM)	Nikon 73346	40X Magnification	Olympus Company, Japan

Ultrasound Measurement	SV-DH- 7A/SVX-7	Measurements range (30, 40 and 50) KHz	Korea
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3. Theoretical Section

Equation (1) was used to measure the viscosity of the experimental liquids η_s , As in the source [24]:

$$\frac{\eta_s}{\eta_0} = \frac{t_s \rho_s}{t_0 \rho_0} \quad (1)$$

η_0 , t_0 , ρ_0 , t_s , ρ_s , These symbols mean, water viscosity, water flow time, water density, experimental fluid flow time and density respectively. The ultrasonic velocity (V) was calculated using the equation (2) [25]:

$$V = \frac{X}{t} \quad (2)$$

X , t , these symbols mean, the sample thickness and the time of wave recording across the sample, respectively. The value of the absorption coefficient (α) is calculated from Lambert Birkma's Law in equation (3).

$$A = A_0 \exp(-\alpha X) \quad (3)$$

A_0 , A , These symbols are used in the above equation the initial amplitude of the ultrasound, and the amplitude of the final after absorption wave, respectively. Compressibility (B) was calculated using the Laplace equation (4) [26]:

$$B = (\rho V^2)^{-1} \quad (4)$$

Equation (5) was applied to calculate the value of the modulus of elasticity (k) [27].

$$k = B^{-1} = \rho V^2 \quad (5)$$

Where D denotes the amplitude of an ultrasonic wave's relaxation as determined by the equation (6) [28]:

$$D = \alpha / f^2 \quad (6)$$

Where α means in the equation the absorption coefficient. The relaxation time for mechanical properties (τ) was calculated from the equation (7) [26]:

$$\tau = \frac{4\eta_s}{3\rho V^2} \quad (7)$$

The measured basic acoustic impedance (Z) of a medium applying equation (8)[29]:

$$z = \rho V \quad (8)$$

4. Results and discussion

FT-IR spectra of the PAA-PVA and PAAM-PVA blended polymers and the PAA-PVA/ GO and PAAM-PVA/ GO nanocomposites were logged in the RT region ($4000-1000 \text{ cm}^{-1}$) as shown in Figure (1). The results for the molecular vibration pattern for PAA-PVA showed that the broadband peak at 3292 , and 2940 , 1706 , 1212 and 1082 cm^{-1} associated with the expansion oscillations of the hydroxyl group (O-H) stretch, methylene (C-H₂) stretch, carboxyl acid (C=O) stretch, the hydroxyl group at (O-H) bend and carbon dioxide (C-O) stretch [30]. The FTIR spectra of PAA-PVA/GO showed a shift in the most broadband peaks after the contribution of GO nanosheets in agreement with another finding [31].

The PAAM-PVA spectrum exhibited a broadband peak at 3285 , 2940 , 1715 , 1644 , 1258 and 1087 cm^{-1} that associated with the expansion oscillations of the hydroxyl (O-H) group, methylene (C-H), carboxyl acid (C=O) stretch, amide group, hydroxyl group, (O-H) and epoxy group (C-O-C). The results of the molecular vibration pattern of PAAM-PVA/GO showed the same peaks of their polymer with shifting in the position after the contribution of GO nanosheets in agreement with other researches finding [32,33].

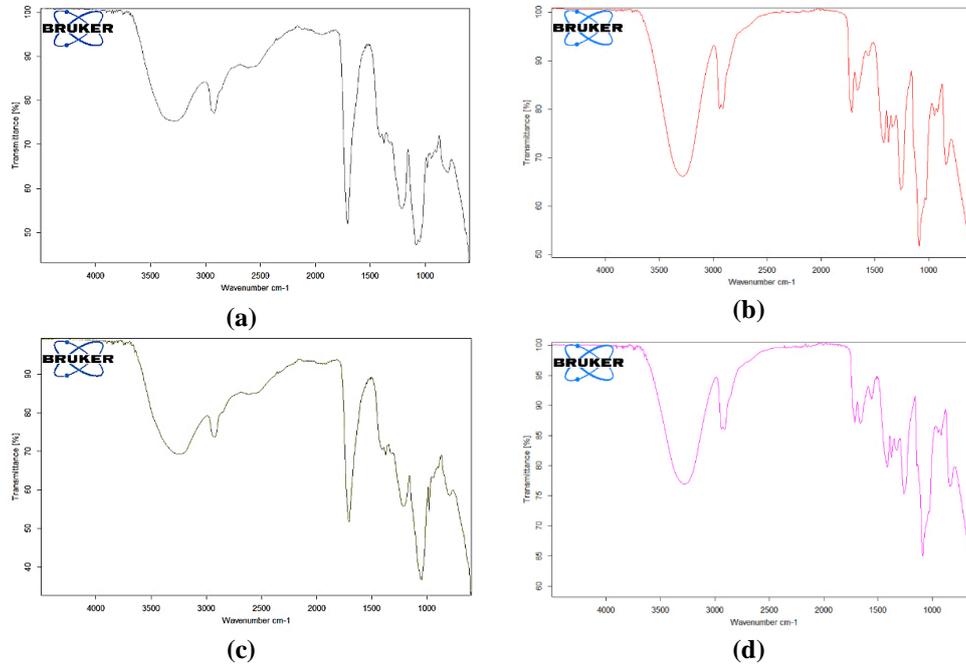
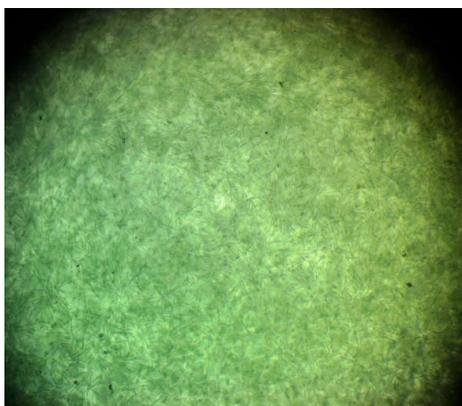


Figure (1). FTIR Spectra of a) PAA-PVA, b) PAAm-PVA, c) PAA-PVA/GO and d) PAAm-PVA/GO nanocomposites.

Figure (2) shows the OM images of PAA-PVA and PAAm-PVA blended polymer films and, PAA-PVA/ GO and PAAm-PVA/ GO nanocomposites. These images showed the homogeneity between the blended polymer as shown in Figure (3 a and b) of PAA-PVA and PAAm-PVA, respectively. In addition, it is clear exhibited the fine distribution of GO reinforced the blended polymers. Where Figure (3 c and d) shows the PAA-PVA/ GO and PAAm-PVA/ GO nanocomposites [34]. These images exhibited the successful preparation of nanocomposite films without any aggregation of nanomaterials.



(a)



(b)

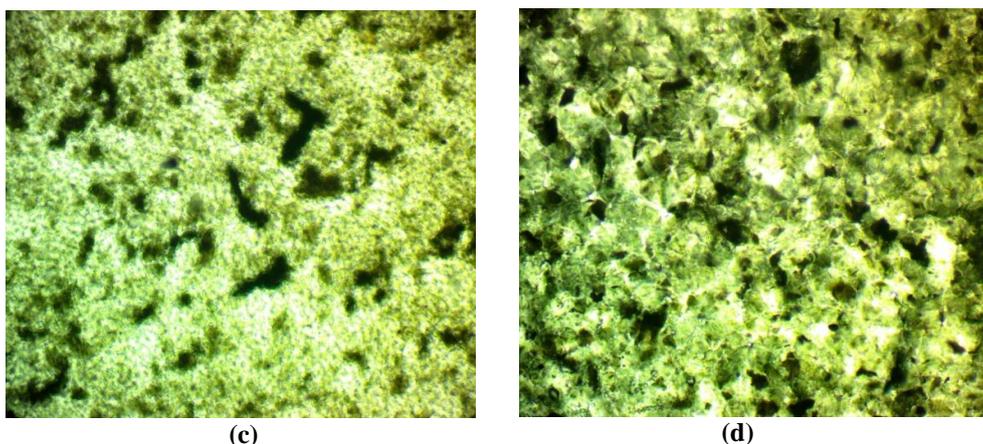


Figure (2). The OM pictures with magnification 100X of a) PAA-PVA, b) PAAm-PVA, c) PAA-PVA/GO and d) PAAm-PVA/GO nanocomposites.

The density of all samples was measured before and after adding graphene oxide as shown in Table (2). This may be due to the solubility of high molecular weight polymers in distilled water, which causes the mass of the solution to increase and the polymer particles to swell.

Where it was noticed that the density of PAA/PVA was less than that of PAAm/PVA, and after adding graphene oxide to each solution, the density values increase as shown in Table (2), it can be seen that the density increased after the contribution of GO, as it is notice that PAA/PVA/GO is less than PAAm/PVA/GO. Density is characterized as mass per unit volume and increasing the density linearly by applying a different weight of polymer to a fixed volume of the solvent resulted in a linear increase in density. Table (2) summarized the density of the samples.

Table (2). The density of the samples.

Compounds	Density g.ml^{-1}
PAA-PVA	1.98
PAA-PVA/GO	2.012
PAAm-PVA	2.069
PAAm-PVA/GO	2.108

The solution flow time was measured for all samples before and after the addition of GO. The results listed in Table (3) demonstrated the significant difference in the flow time values between the type of polymer i.e. as the molecules become larger, the number of molecules in the polymeric solution increases due to an increase in the friction forces between the molecules (large molecules), which is obstructed during the flow within the capillary viscosity. It can also be observed from Table (3) that the flow time increased with the contribution of GO up to 13 % and 17 % of PAA-PVA/ GO and PAAm- PVA/ GO, respectively compared with their blended polymers. These results were exhibited due to the effect of nanosheets additives that bigger the density and the friction forces between the polymer particles and the tube walls on the one hand and between the polymer particles themselves on the other hand. These findings result in a significant increase in the viscosity as shown in Figure (3), where the PAA-PVA blended polymer exhibited higher viscosity than PAAm-PVA. Whereas, the addition of GO presented notable improvement in the viscosity of the PAA-PVA/ GO, which was also revealed the best results of the viscosity than the PAAm-PVA/ GO as shown in Figure (4) and Table (3) summarize the flow time samples.

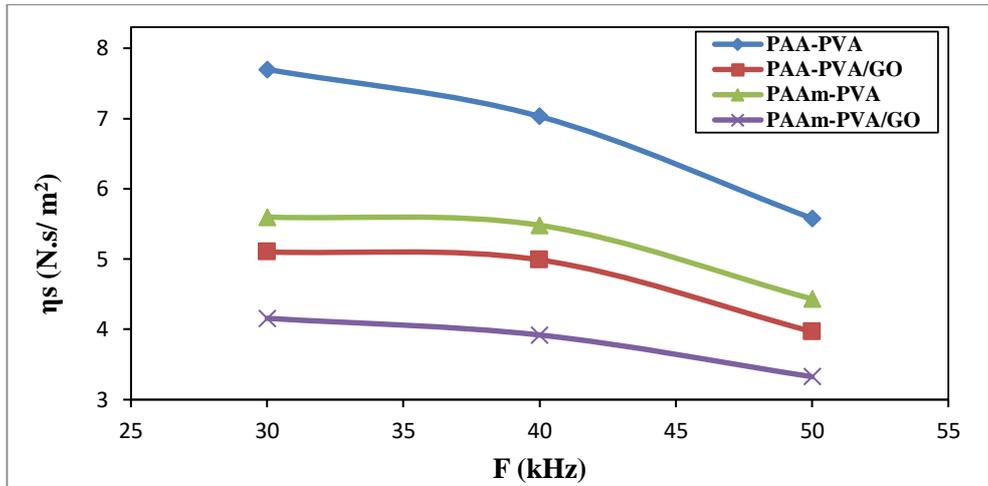


Figure (3). The viscosity with increasing the frequency of the blended polymers and nanocomposites.

Table (3). The flow time of the blended polymers and nanocomposites.

Compounds	Solution flow time, Sec
PAA-PVA	69:36
PAA-PVA/GO	78:10
PAAm-PVA	1403:85
PAAm-PVA/GO	1633:75

Figure (4) shows the relationship between the ultrasound velocities with the frequency of the PAA-PVA, PAA-PVA/GO-PAAm-PVA and PAAm-PVA/GO) samples. The ultrasound velocity was determined using equation (2). This figure shows that the ultrasound speeds of each sample were increased with increasing the frequency of the ultrasound velocity. The solute particles are dispersed through the solvent without bonding, and this leads to the particles having freedom of movement within the solution and then allowing the ultrasound waves to pass without changes (12). The ultrasound velocity increased after adding GO as exhibited in Figure (5). The reason for this fact is the large size of the polymeric chains that meet with each other and the GO nanosheet increasing the interfacial interactions between the polymers as presented clearly in the FTIR results. Therefore, the average transmissibility of ultrasound increased and thus the ultrasound velocity increased after GO was added. Where it was noticed that the ultrasound velocity of PAAm-PVA was higher than the ultrasound velocity of PAA-PVA, Additionally, the incorporation of GO observed significant improvement in the value of the ultrasound velocity of PAAm-PVA/GO than PAA-PVA/GO. This improvement was up to 66% and 74 % respectively, compared with their blended polymers. These results supported the density finding and similar behavior with the researcher's finding [35].

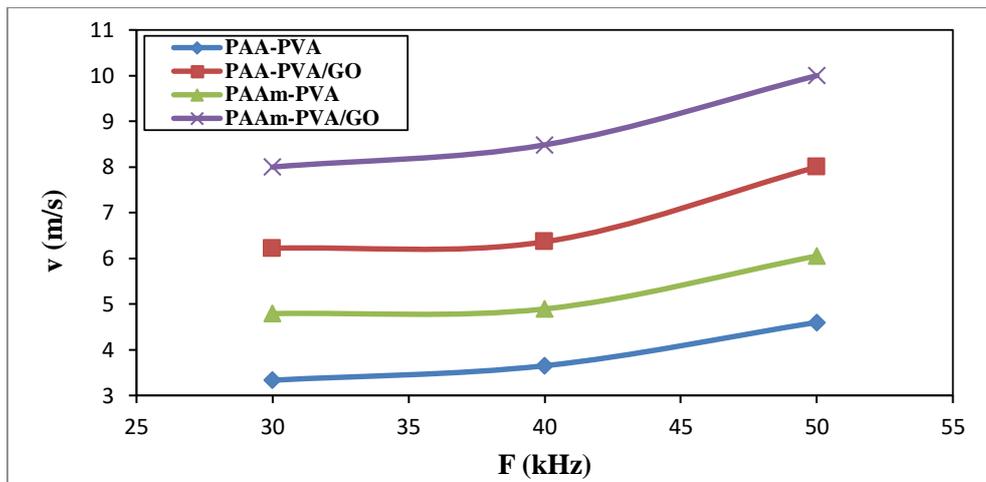


Figure (4). The Ultrasound velocity with the frequency of the blended polymers and nanocomposites.

The ultrasound absorption coefficient values of the blended polymers and nanocomposites were found both before and after the addition of nanosheets. The results in Figure (5) showed an increase in the absorption coefficient of the samples included polymer with amide group in comparison with samples included polymer with the acid group, where the former presented a higher absorption coefficient of the ultrasound. This is according to the type of polymer because the absorption coefficient depends mainly on the polymer and also depends on the structural relaxation that occurs between the polymer molecules. Friction and heat exchange between the particles and the surrounding medium is responsible for the attenuation. The absorption coefficient of PAAm-PVA was higher than the absorption coefficient of PAA-PVA, and after the contribution of GO nanosheets, an excellent increase was observed for both nanocomposites PAA-PVA/GO, PAAm-PVA/GO up to 100 % and 89 % respectively compared with their blended polymers. However, it showed through the results that PAAm-PVA/GO was higher than PAA-PVA/GO nanocomposites.

Because of the dispersion of the particles, the ultrasound wave travels forward. The addition of GO improves the absorption coefficient by increasing its value. Since the ultrasound propagates in the form of compression and dissociation in a medium, this location increases the viscosity of the solution, which means that there is more flexibility for these polymer chains in the solution as a result of the addition of GO, and because the ultrasound propagates in the form of compression and decomposition in the medium, there is variation in the intensity range, which means that there was more attenuation of the GO ultrasound energy. These results are similar to the findings of the researchers [36].

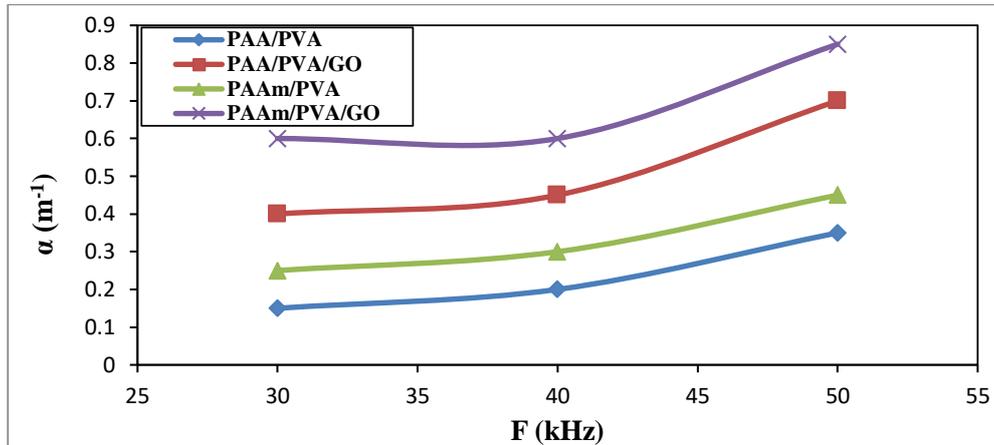


Figure (5). The ultrasound absorption coefficient with increasing the frequency of the blended polymers and nanocomposites.

Figure (6) shows that the relaxation time with the increasing frequency of ultrasound velocity of the blended polymer and nanocomposites. The results of the PAA-PVA blended polymers exhibited higher than the relaxation time of the PAAm-PVA blended polymers films. Whereas, the incorporation of GO nanosheets observed an increase of the nanocomposites result compared with their polymers blended, whereas the PAA-PVA/GO revealed better results than PAAm-PVA/GO nanocomposites. Because of disruptions and the effects of ultrasound, internal friction between the fluid layers increased as the length of the polymeric chains grew longer. As a consequence, the amount of time it takes to return a molecule to its original state increases. The behavior of relaxation time is similar to that of shear viscosity that shows the samples with the acid group showed swelling and more space for a particle to move. These results are similar to those of the researcher (14).

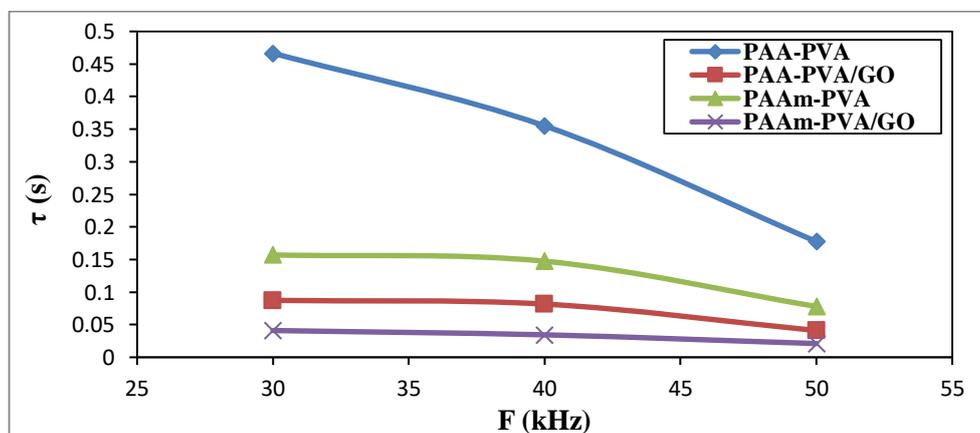


Figure (6). The Relaxation time with increasing the frequency) of the blended polymers and nanocomposites.

Figure (7) shows that the compressibility of the blended polymer and nanocomposites. The results presented a decreased behaviour with increasing the frequency of the samples. The samples included amide group exhibited better results than another sample, it was presented a reduction in compressibility, whereas the samples with the acid group presented more ability to compress. The contribution of GO nanosheets was significantly reduced the PAA-PVA/GO and PAAm-PVA/GO nanocomposite's ability to compress up to 198 % and 178 % respectively, compared with their blended polymer samples. The pressure behavior has the

same velocity as ultrasound but in the reverse direction. The slight decrease in thermal pressure is because the polymer particles ionize in dilute solutions. In the solution, these ions are surrounded by a layer of solvent molecules that are tightly bound and directed towards the ions. The electrostatic field of the ions affects the direction of the solvent particles around the ions, which affects the internal pressure and reduces the compressibility of the solution, and the solutions become difficult to compress in addition to reinforcement by GO, it was filled the space between the molecular of polymer in the matrix. This is consistent with the researcher's findings [37].

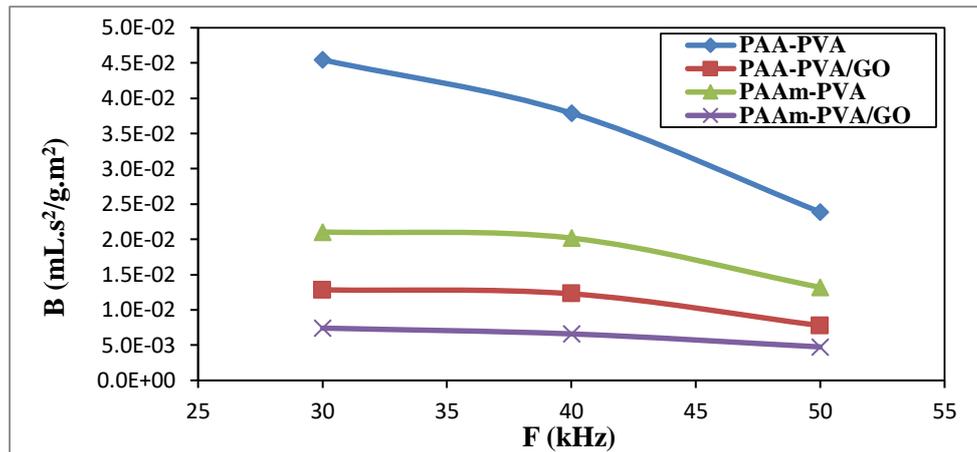


Figure (7). The compressibility with increasing the frequency of the blended polymers and nanocomposites.

Figure (8) shows that the elastic modulus with increasing the frequency of the blended polymer and nanocomposites. The result of the PAAm-PVA is higher than that of PAA-PVA that was significantly improved after the reinforcement by GO, where both nanocomposites show notable improvement up to 177 % and 128 % respectively compared to their blended polymers. Meanwhile, PAAm-PVA/GO nanocomposites illustrated significant improvement compared with PAA-/PVA/GO nanocomposites. Because the modulus of elasticity is inversely related to the strong effect of graphene oxide that has 1 TPa. This led to the strong reinforcement of nanocomposites (16).

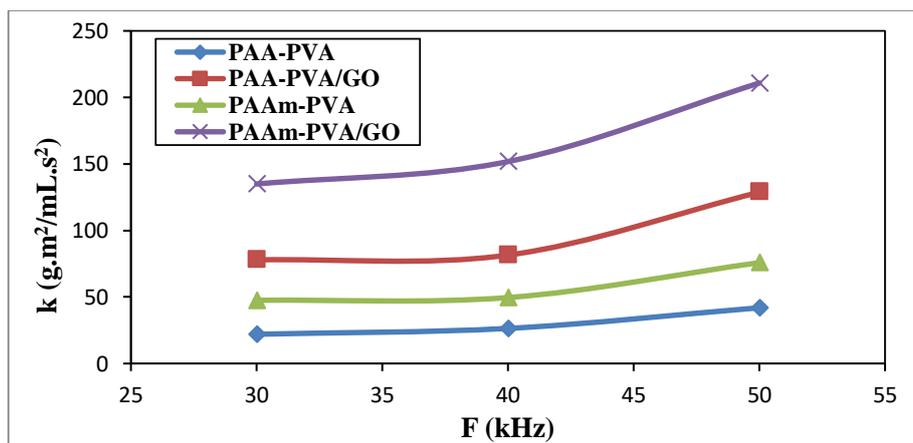


Figure (8). The bulk modulus with increasing the frequency of the blended polymer and nanocomposites.

Fig (9) shows the same behavior of the impedance acoustic with increasing the frequency of the blended polymer and nanocomposites. The specified acoustic impedance values were increased with increasing the frequency of the ultrasound velocity in all samples. Where. The results from Figure (10) show that the PAAm-PVA with amide group had a specific acoustic impedance higher than PAA-PVA, and after adding GO, the results improved for all the nanocomposites, whereas the best results exhibited for the PAAm-PVA/GO. The acoustic impedance is mainly dependent on velocity and density as presented in equation (8). This behavior is similar to what the researcher presented (12).

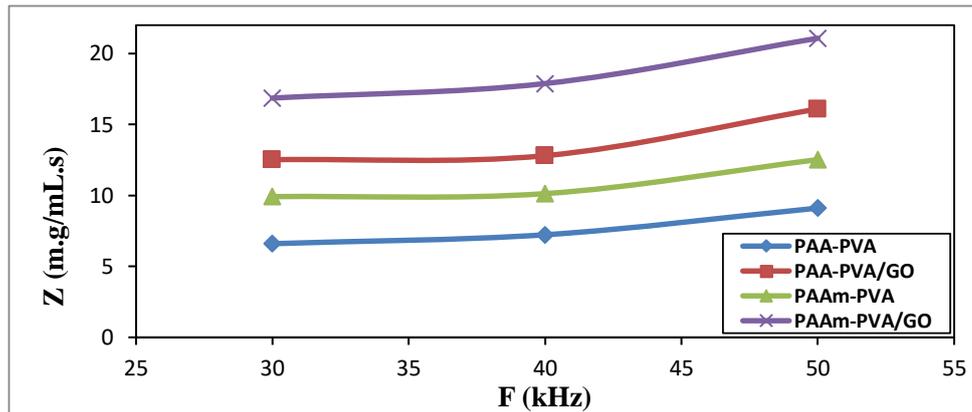


Figure (9). The specific acoustic resistance with increasing the frequency of the blended polymers and nanocomposites.

Figure (10) shows that the relaxation amplitude increases with increasing frequency for each condition before and after the addition of GO. This is due to the displacement of the excited particles until they become small because the moment of inertia of the larger polymer molecules is reduced [38]. The relaxation amplitude displayed the same behavior as the absorption coefficient, then at the constant frequency, with an increase in the absorption coefficient, the relaxation amplitude increased. The results showed that PAAm/PVA had a higher relaxation amplitude than PAA/PVA, and after adding GO, the results were significantly improved in comparison to their blended polymers, whereas with PAAm/PVA/GO exposed the best relaxation amplitude than PAA/PVA/GO, as shown in Figure (10).

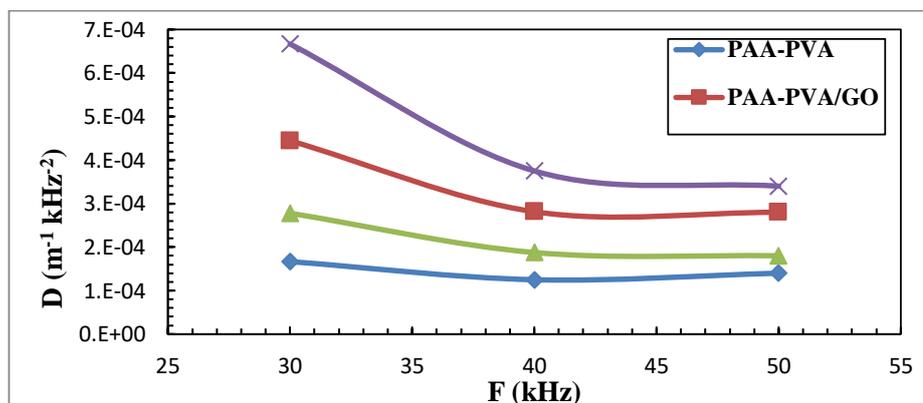


Figure (10). The relaxation capacity with increasing the frequency of the blended polymers and nanocomposites.

Generally, these resulted exhibited the influence of the polymer functional group an important factor that could affect the properties of nanocomposites and fabricated significant interfacial interaction with other polymers as blended or nanomaterials as nanocomposites. Moreover, the nanomaterial is an important key to bring significant upgrading of the mechanical properties due to the reinforcement of polymers by the significant interfacial interaction between polymers and GO nanosheets that resulted in improvement of both nanocomposites compared with their blended polymers.

5. Conclusions

The method was successfully fabricated new nanocomposites with homogeneous and fine dispersion as presented by optical microscopy images that provided improvements in the good morphology properties. The FTIR test showed the difference between the functional group of each component that has the same backbone. Also, it shows how the polymer and GO nanosheets have a good interfacial interaction. The results showed the important effect of functional group that exhibited the sample with amid group has the better results than polymer with the acid group. Moreover, As compared to their polymers, both nanocomposites showed a significant improvement in mechanical properties thanks to GO. Whereas the nanocomposites with amid group displayed the best mechanical properties compared to all materials.

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