UKRAINIAN JOURNAL OF MECHANICAL ENGINEERING AND MATERIALS SCIENCE
Vol. 7, No. 1–2, 2021

Taraneh Javanbakht

Department of Chemistry and Biochemistry, Department of Physics, Concordia University, Richard J. Renaud Science Complex, 7141 Sherbrooke Street West, Montreal, Quebec, Canada H4B 1R6
E-mail: taraneh.javanbakht@concordia.ca, ORCID 0000-0002-2658-330X

INVESTIGATION OF RHEOLOGICAL PROPERTIES OF GRAPHENE OXIDE AND ITS NANOCOMPOSITE WITH POLYVINYL ALCOHOL

Received: March 17, 2021 / Revised: April 28, 2021 / Accepted: April 30, 2021

https://doi.org/10.23939/ujmems2021. 01-02.023

Abstract. This paper focuses on the rheological properties of graphene oxide (GO) and its nanocomposite with polyvinyl alcohol (PVA). The purpose of this paper is to compare the mechanical properties of these materials. GO is a nanomaterial that has been widely studied in engineering. The comparison of its rheological properties with those of its nanocomposite with PVA has been required. Rheology is an appropriate method for the comparative investigation of the mechancial behavior of these materials. The challenge has been to compare the rheological parameters of these materials regarding their mechanical properties. This investigation shows that GO and GO-PVA do not exhibit the same viscosity change at low shear rates. While GO shows a significant decrease of viscosity, GO-PVA shows a steady state behavior at these shear rates. However, both GO and GO-PVA show a constant viscosity at high shear rates. These materials show the same behavior concerning the change of their viscosity versus shear strain or time. The changes of torque versus shear strain and time for GO and GO-PVA are representes as increasing curves and lines, respectively. The increase of shear stress versus shear rate or shear strain coincide for both materials. However, as expected, the first change represents an increasing line and the second one a curve. This original investigation shows the difference between the mechanical behaviour of GO and GO-PVA concerning the change of their viscosity at low shear rates and their similarity at high shear rates. Moreover, this study gives new results concernig the change of other parameters as described above. The unique physicochemical properties of GO have made it an important candidate in engineering and materials science. This investigation can lead to a better understanding of the rheological properties of GO and GO-PVA for the improvement of these applications.

Keywords: rheology, GO, GO-PVA, viscosity, shear rate, shear stress, shear strain, mechanical engineering.

Introduction

Graphene oxide (GO) is an oxidized product of graphene that has a similar composition. GO and other carbon-bases nanomaterials, such as carbon nanotubes and graphete nanoplateletes, have been investigated in composition with diverse polymers, previously [1–4] Adding nanostructures into the polymers is an appropriate technique for the preparation of nanocompsoites [5–8]. The properties of nanocomposites with lower loadings than for commonly used polymer composites can be enhaced with micron-scale fillers used such as glass or carbon fibers [9]. GO nanoribbons have oxygenated graphene structure in which lower van der Waals interactions exist in comparison with those of graphene sheets [10], [11]. The two-dimensional structure of GO consists of covalently bonded carbon atoms in which several oxygen functional groups such as hydroxyl, epoxy, or carbonyl groups are found on their planes and edges. The presence of these functional groups makes GO nanoribbons hydrophilic molecules with high

dispersibility in water [3]. There is an important advantage of this structural property of GO. These functional groups faciliate the hydrogen bonding interaction of this nanomaterial with polymers [7], [12–14]. The epoxy groups can be modified when the ring-opening reactions occur in desired conditions. These reactions can be carried out with a nucleophilic attack at by the amine group [15]. Another modification such as the epoxide ring-opening reaction can be carried out by the addition of octadecylamine to GO [16]. The epoxy group of GO can react to stabilize solid-phase dispersions of chemically modified graphenes. It has been reported that the covalent attachment of propyltriethoxysilane to GO platelets can do this kind of modification [17].

The expanded interlayer spacing of GO can change with humidity, based on the intercalation of water molecules [18]. Thermal shocking can be used for the exfoliation of this nanomaterial [19]. Chemical reduction can yield a product with similar structure to that of pristine graphene [20]. The oxidation of graphite using strong oxidants such as $KMnO_4$ or $KClO_3$ in the presence of an acid is an apporpriate method for the preparation of GO [21]. These reactions can yield similar levels of oxidation [15], [22].

Nanocomposites can be prepared with GO when its non-covalent dispersion is used for its preparation with polymers [23], [24]. Some other techniques such as non-covalent *in situ* polymerization [25–27], making covalent bonds between matrix and filler [28], [29], grafting of polymers to GO platelets that are reduced [30], or other methods such as solid-state shear pulverization [31], assembly of polymer composites [14], backfilling of GO with polymers [32] and freeze-drying of GO with polymers [33] can also be applied. The improvement of the thermal and electrical properties of polymers can be achieved with the incorporation of GO nanoribbons to them [13], [14], [34–36].

Polyvinyl alcohol (PVA) is a polymer that is hydrated when dissolved in water [37]. This hydrophilic polymer has been widely studied for the preparation of drug delivery systems, as well as wound dressing, and medical prosthetics due to its appropriate biocompatibility [38][39][40][41] It has been shown that small molecules or ions can diffuse through the solutions of this polymer. This phenomenon is not affected by the presence of large molecules [38]. It is worth noting that the molecular-level dispersion of GO nanoribbons can improve the mechanical properties of its nanocomposite [42]. The imrpovement of the dispersion of GO nanoribbons in PVA can be achieved due to the presence of the same functional groups on the surface of GO and the polymer.

The physicochemical properties of GO and PVA were investigated, previously [43], [44]. The antibacterial effects of GO nanoribbons could be related to its physicochemical properties. Moreover, the surface modification of PVA could modify its physicochemical properties. Althogh these studies were required for a better understanding of these properties in relation with the structural characterizations of these materials, their rheological properties were also needed to be determined for yielding important results for further investigations. A recent study has revealed the rheological properties of PVA in comparison with polyethylene glycol (PEG). This study showed that the rheological properties of PVA and PEG were similar to each other at low shear rates, but at high shear rates, they behaved differently. Moreover, it was shown that the increase in the shear strain values required more increase in the shear stress on PVA than that on PEG [45].

The current research work presents the rheological properties of GO and GO-PVA in a comparative investigation. To the author's knowledge, this is the first comparative study of these materials including the anlaysis of their parameters.

Experimental approach

Materials and methods.

GO and PVA (MW 89.000–98.000) were both purchased from Sigma Aldrich. The concentration of the GO solution was 2 mg/mL. The hydrogel solution of PVA was prepared as described in the previous work [10]. Briefly, PVA (0.4 g) was dissolved in deionized water (10 mL). Then, the solution was prepared with mixing and heating at 90 °C for 2 h. The solution was divided in two parts each containing

0.2 g of PVA. One mL of the GO solution was added to the PVA solution containing 0.2 g of polymer. The concentration of GO in the nanocomposite solution was 1 % (w/w) [10].

The rheological properties of the solutions of GO and GO-PVA were analyzed with an Anton Paar MCR-302 rheometer. All the measurements in triplicate were performed at room temperature [46], [47].

The QtiPlot software was used for the determination of mean values, standard deviations and statistical significance of data. The graphs were plotted with this software [47], [48].

Results and discussion

Figure 1 shows the viscosity variations of GO and GO-PVA versus shear rate. The variation range of shear rate was between 0 s^{-1} and 1000 s^{-1}. Fig. 2 shows the viscosity variations of GO and GO-PVA versus shear strain.

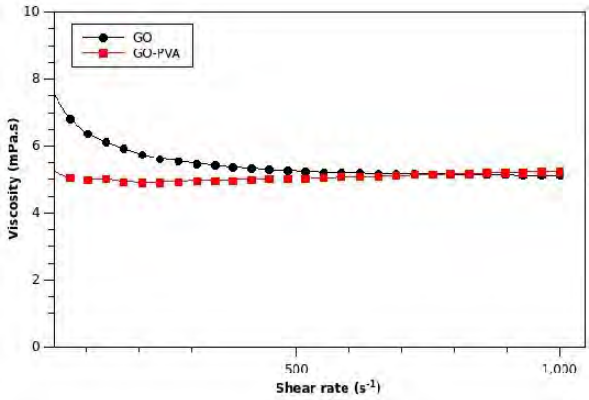

Fig. 1. Viscosity of GO and GO-PVA versus shear rate

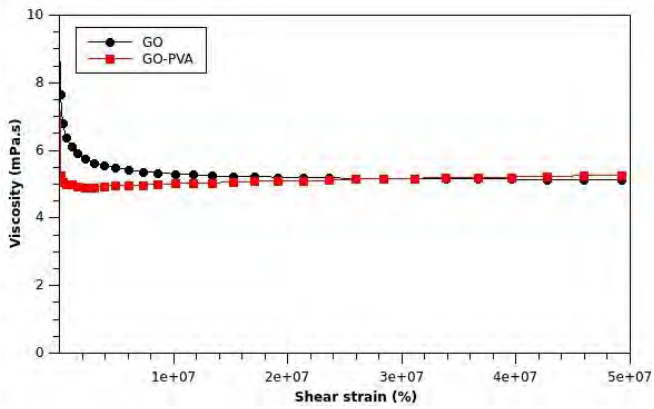

Fig. 2. Viscosity of GO and GO-PVA versus shear strain

As shown in Fig. 1, the viscosity of GO nanoribbons decreased with shear rate and became constant at high shear rate values. However, GO-PVA showed a different behavior as its viscosity was almost constant at low and high shear rate values. This can be explained with the presence of PVA in the nanocomposite as the molecules of the polymer make a restriction in the structure of GO to flow with the increase of shear rate.

As shown in Fig. 2, the same behaviors of GO and GO-PVA as presented in Fig. 1 were observed for their viscosity values versus shear strain. In other words, although GO-PVA showed a steady state behavior, GO showed the decrease of viscosity and constant viscosity at low and high shear strain values, respectively. This can be explained with the presence of PVA as the polymer hinders the deformation of GO in the nanocomposite samples.

Figure 3 shows the viscosity variations of GO and GO-PVA versus time.

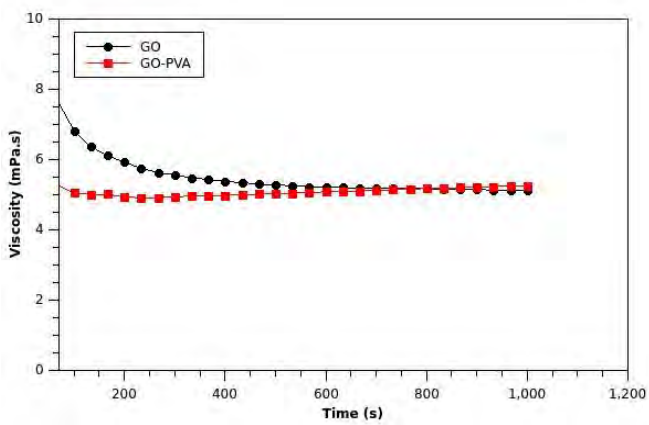

Fig. 3. Viscosity of GO and GO-PVA versus time

The changes of viscosity versus time in Fig. 3 showed that the decrease of viscosity versus time was occurred for GO nanoribbons at low duration and no change was observed after 600 s. Almost a steady state was observed for the viscosity of GO-PVA. This result was expected as the increase of shear rate and shear strain with time showed almost no change for the viscosity of the nanocomposite.

Table 1 shows the viscosity values of GO and GO-PVA versus time.

Table 1

Viscosity values of GO and GO-PVA versus time

Materials/Viscosity (mPa.s)	Time (s)		
	200	600	1000
GO	5.93 ± 0.10	5.22 ± 0.03	5.12 ± 0.02
GO-PVA	4.93 ± 0.10	5.08 ± 0.03	5.25 ± 0.02

As shown in Table 1, the viscosity decrease of GO between 600 s and 200 s was more than the decrease observed between 1000 s and 600 s. The viscosity of GO-PVA did not decrease very significantly in the first time span but it increased a bit in the second time span.

Figure 4 shows the changes of torque versus shear strain. The torque values increased for both GO and GO-PVA. Figure 5 shows the changes of torque versus time.

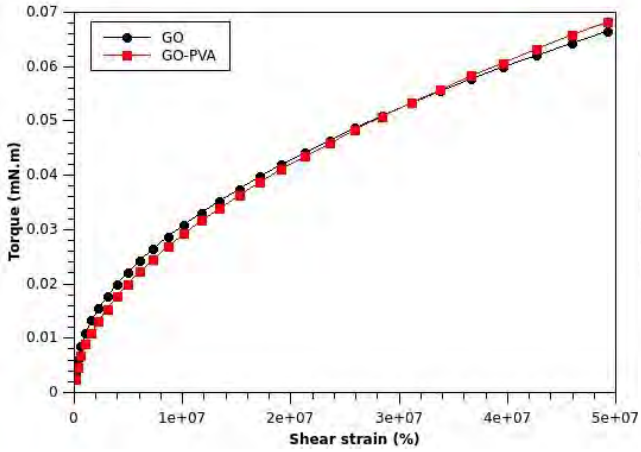

Fig. 4. Torque versus shear strain

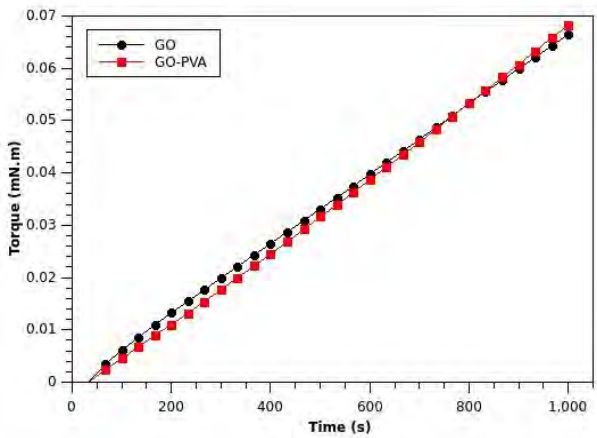

Fig. 5. Torque versus shear time

As shown in Fig. 4, the slope of the torque increase was high at low shear strain and it decreased with the increase of this second. This was an expected result as less values of the torque change were needed to be applied on GO and GO-PVA with the increase of their deformation.

As shown in Fig. 5, the slope of the torque increase was constant with time for both GO and GO-PVA as a linear change of torque versus time was observed for these materials. This revealed that no change in the torque increase was applied over time.

The results shown in Fig. 4 and 5 were consistent as a constant increase of torque was observed with time for GO and GO-PVA and this increase was more when their deformations were low and it decreased with the increase of their deformations.

Fig. 6 shows the shear stress changes of GO and GO-PVA versus shear rate. Linear changes were observed for the shear stress applied on both materials as it increased with shear rate. Fig. 7 shows the shear stress changes of GO and GO-PVA versus shear strain. Non-linear changes were observed for the shear stress applied on both materials as it increased with shear strain.

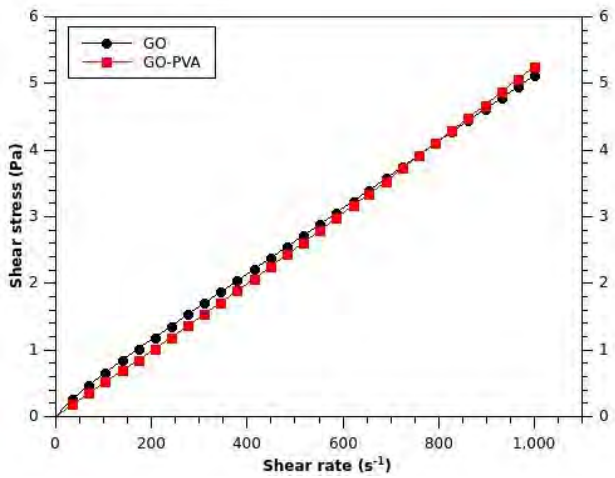

Fig. 6. Shear stress of GO and GO-PVA versus shear rate

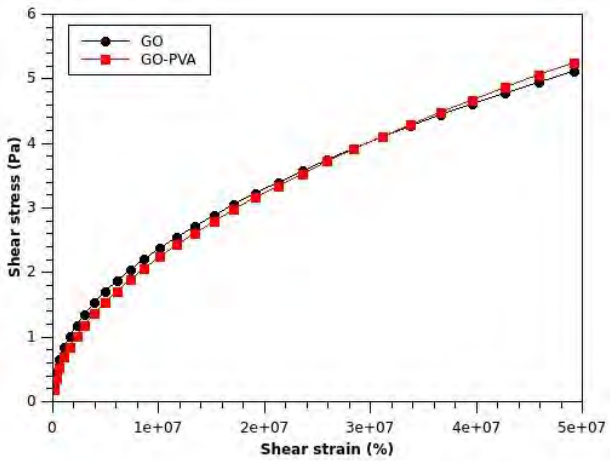

Fig. 7. Shear stress of GO and GO-PVA versus shear strain

As shown in Fig. 6, almost the same slope was observed for the increase of shear stress with shear rate for GO and GO-PVA. This indicated that both materials had nearly the same constant rate of shear stress as it changed with shear rate.

As shown in Fig. 7, almost the same slopes were observed for the increase of shear stress with shear strain for GO and GO-PVA at different shear strains. This indicated that both materials had nearly the same increase rate as the shear stress values changed with shear strain.

The observations of the shear stress change with shear rate and shear strain were required to determine how these parameters changed for GO and GO-PVA. The comparisons of the results shown in Fig. 1 and 6 indicated that although shear stress increased with shear rate for both GO and GO-PVA, the viscosity of the first decreased whereas that of the second was constant at low shear rates. This revealed the difference in the rheological behavior of these materials with shear rate. The difference in this behavior was confirmed with the viscosity decrease of GO and almost no change in the viscosity of GO-PVA with shear strain or time as shown in Fig. 2 and 3.

Table 2 shows the shear stress values of GO and GO-PVA versus shear strain.

Table 2

Shear stress values of GO and GO-PVA versus shear strain

Materials/Shear stress (Pa)	Shear strain (%)		
	$1*10^6$	$2*10^7$	$4*10^7$
GO	0.84 ± 0.02	3.40 ± 0.02	4.62 ± 0.03
GO-PVA	0.69 ± 0.03	3.35 ± 0.02	4.67 ± 0.03

As shown in Table 2, a little difference was observed between the shear stress values of GO and GO-PVA at low shear strain. However, no difference was observed for the shear stress applied on these materials with the increase of their deformation up to $4*10^7$.

An important issue in engineering and materials science is the improvement of the properties of materials that are applied in these fields. The properties of some of the materials that have shown diverse applications were investigated, previously [49][50][51][52][53][54]. The rheological properties of these materials have not been investigated, yet. These materials and their nanocomposites with polymers can be investigated for the improvement of their mechanical properties. It was shown that the non-Newtonian behavior of PVA could be maintained when it was mixed with GO nanoribbons [10]. Some other nanocomposites and blends of PVA have also been investigated. A study on the rheological properties of nanocomposites of PVA and cellulose nanocrystals showed that they had limited molecular weight dependence [55]. Another investigation showed that the viscosity of PVA could increase slowly as

the aging of its blends with PEG could occur much faster than those of pure polymers [56]. However, the viscoelastic behaviour of the blend could remain stable upon storage [57]. It is worth noting that long polymers can interconnect several particles. In fact, these polymers can act as cross-links and enhance the viscosity of products [58].

It has been shown that embedding the modified nanoparticles into the blend polymer could effect the optical absorbance of the product [59]. The presence of PEG forces PVA for phase separation, which can help the formation of gel [60]. Crosslinking has a positive impact in the preparation of the blends of PVA with other polymers. In fact, the rheological properties of these blends can reveal their viscoelastic behavior and stability [61], [62]. Adding PEG to PVA can make the blend more appropriate for biomedical applications as it can counterfact the reduction of the water content in it [63].

As GO nanoribbons have an amphiphilic structure, they can be used for compatibilizing immiscible polymer blends. This unique structural characteristic of GO makes possible the preparation improvement of compatibilizers for industrial applications [64]. A study on the compatibilization of GO and polymers showed serious phase separation and declined tensile strength with a reversed mixing sequence [65]. Moreover, the rheological properties of the blends of GO and polymers can be modulated at low frequency and became constant at high frequency [66]. Some processing routes for the preparation of high-quality graphene-based materials in polymer nanocomposites, such as melt compounding [66], [67], solution blending [68], [69], in situ polymerization [70], [71], latex mixing [72], [73], and electropolymerization [74–76] were applied, previously. It is required to investigate the rehological properties of these materials with GO nanoribbons. The results obtained in the current paper can lead to further investigations for a better understanding of the impact of each preparation method on the rheological beahvior of these materials.

Conclusions

In this paper, the comparative study of rheological properties of GO and GO-PVA were presented. The results showed that the viscosity of GO nanoribbons decreased with shear rate and it became constant at high shear rate values, but GO-PVA showed a different behavior as its viscosity was almost constant at low and high shear rates. The same results were obtained for the viscosity change with shear strain or time. These results indicated that the presence of the polymer in the nanocomposite could hinder the viscosity change of GO nanoribbons. As the torque values increased with shear strain and time and the shear stress values increased with shear rate and shear strain almost in the same manner for both materials, this indicated that the differences in the rheological behavior of GO and its nanocomposite with PVA versus shear rate, shear strain or time were not due to the difference in the torque or shear stress applied on GO and GO-PVA. The results of tis investigation can lead to the improvement of the rheological properties of these materials for their applications in engineering and materials science.

Acknowledgments

The author gratefully thanks Professor Ingo Salzmann for the provision of GO and PVA.

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