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Comparative Rheological Investigation of Nanocomposites of Surface Charged Superparamagnetic Iron Oxide Nanoparticles with Polyethylene Glycol

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Abstract: This paper focuses on a new investigation of the rheological properties of the nanocomposites of surface charged superparamagnetic iron oxide nanoparticles with polyethylene glycol. Both studied nanocomposites showed the steady-state behavior at 20 °C and 40 °C. Moreover, the increase of viscosity versus shear strain, shear rate or time for the nanocomposites was small at 60 °C. The effect of the coating of nanoparticles with the polymer was comparable for different nanocomposites. The data presented in this paper can provide the required information for the preparation of assemblies of nanocomposites with polymers.

Keywords: Rheological properties, nanocomposites, surface charged SPIONs, PEG, mechanical properties.

1. Introduction

Rheological investigation of nanomaterials is an important issue as these materials with various mechanical properties can be applied in diverse fields of science and engineering. Moreover, the surface functionalization of these materials can modify these properties such as viscosity, shear rate, etc. The viscous nature and solid-like behavior of these materials have made them important candidates at different levels with low filler loading and high concentration [1-3]. The rheological properties of these materials or their composites can determine their processing performance for their preparation. It is worth noting that their rheological properties can be modified according to their weight change when a few amount of these materials is increased. In each polymer composite, one or several nanomaterials are mixed with one or several polymers. Computer modeling and simulation are two methods for the investigation of the rheological properties of these materials. In these methods,

the mechanisms at the molecular level are explored for improving the dispersion of nanoparticles in matrices. In addition, the information on the effects of nanoparticles on the chain conformation and glass transition temperature of the samples is searched [4-5].

The rheological properties of a polymer matrix and the presence of branched structures in its structure are affected by its molecular weight [6-8]. These behaviors also depend on the values of shear rate. The stiffening of polymer chains and agglomeration as well as the increase in the viscosity of nanocomposites can happen because of the increase of the nanomaterials loading in the polymeric matrix at low shear rate values due to the confinement of the polymer chains with the embedded nanofiller [9-12]. At high shear, the differences between nanocomposites and corresponding matrices become less significant due to several phenomena such as the shear thinning behavior, the wall slip phenomenon and the unaffected material viscosity after changing the nanomaterial content in the samples. It has been explored that the nanocomposites with high molar mass and low melt flow index could show a decrease in their viscosity values in the high shear rate region. Moreover, the nanocomposites that contain a small amount of nanomaterials can have lower viscosity than that of the unfilled matrix due to the higher viscosity of the polymeric matrix at high shear stress. During recent years, several nanofillers with different chemical nature, shape and morphology have been investigated among which are the nanoparticles such as metals, carbon-based fillers and ceramics [13-18].

Polymer nanocomposites containing nanosized fillers having large surface areas are considered as better materials than other composites with microsized fillers. To maximize these enhancements, fillers should be well-dispersed in nanocomposites [19]. There are several techniques for the improvement of the quality of filler dispersions in these nanocomposites such as intercalation of polymers from solution, *in situ* intercalation, or melt intercalation [20-22]. The quality of filler dispersions can also be improved with the use of compatibilizers [23-25], nanofiller surface treatments [26-28] or the application of an electric field to clay nanocomposites [29-30].

The comparative analysis of the rheological properties of the nanocomposites of different surface charged superparamagnetic iron oxide nanoparticles (SPIONs) coated with polyethylene glycol (PEG) has not been reported, yet. The results of this article can be used for the preparation improvement and applications of these nanocomposites in science and engineering.

2. Materials and methods

PEG (MW= 8000) was purchased from Sigma Aldrich. SPIONs were synthesized as described previously [31]. For the preparation of bare SPIONs, an aqueous solution of ferrous chloride (5 mL, 0.045 M) and ferric chloride (0.0375 M) was added to diethyleneglycol (250 mL). Sodium hydroxide was added to the mixture, then the mixture, with the final concentration of 0.375 M, was heated at 170 °C during 15 minutes. After maintaining its temperature at the same temperature during an hour, it was cooled to 60 °C. The collection of SPIONs was performed using a neodymium magnet, then the nanoparticles were washed with a nitric acid solution (1 M) [31]. For the synthesis of negatively charged SPIONs, 3-(triethoxysilyl)propylsuccinic anhydride (TEPSA) (14.2 mL, 50 mmol) were added to nanoparticles. The solvent was DMF (100 mM of iron in 100 mL). After the addition of water (8.6 mL) and TMAOH (5 mL, 1M) at room temperature, the solution was heated at 100 °C during 24 hours. After the addition of acetone/ether (50/50), SPIONs were precipitated and collected with a magnet, washed with acetone and dispersed in water. Then, excess of additives was removed by filtration using a membrane with a cut-off of 30 kDa. [31] For the preparation of positively charged SPIONs, N-[3-(trimethoxysilyl)propyl] ethylenediamine (TPED) was grafted onto SPIONs with the addition of TPED (25 mmol, 5.4 mL) to a suspension of nanoparticles (100 mL, concentration of iron: 25 mM) at 50 °C. The mixture was stirred for 2 hours, then cooled at room temperature. For the filtration of suspension, a membrane with a cut-off of 30 kDa was used, then SPIONs was centrifuged at 16 500 g for 45 minutes [31].

The nanocomposites of positively charged SPIONs-PEG and negatively charged SPIONs-PEG were prepared as explained in the previous work [31]. After mixing PEG (4.8 g) with deionized water during 15 minutes at room temperature, four portions of the polymer mixture were prepared: there was no SPIONs in one portion, the three other portions were considered for the preparation of nanocomposites of the polymer with these nanoparticles, each of them having 1.2 g of the polymer, which was dissolved in water (5 mL). The solutions of nanocomposites (1%) were prepared by adding 12 mg of bare, positively charged or negatively charged SPIONs separately to each of the three portions of the PEG solution. The samples were separately mixed during 15 minutes at room temperature [31].

The samples of each nanocomposite were prepared and analyzed in triplicate. An Anton Paar MCR-302 rheometer was used for the rheological investigation of nanocomposites. The measurements in triplicate were performed at 20 °C, 40 °C and 60 °C. The analysis of parameters such as mean values, standard deviations and statistical significance was performed with the QtiPlot software [32-33].

3. Results and Discussion

Fig. 1 shows the viscosity variations of the nanocomposites of positively charged SPIONs-PEG and negatively charged SPIONs-PEG versus shear rate.

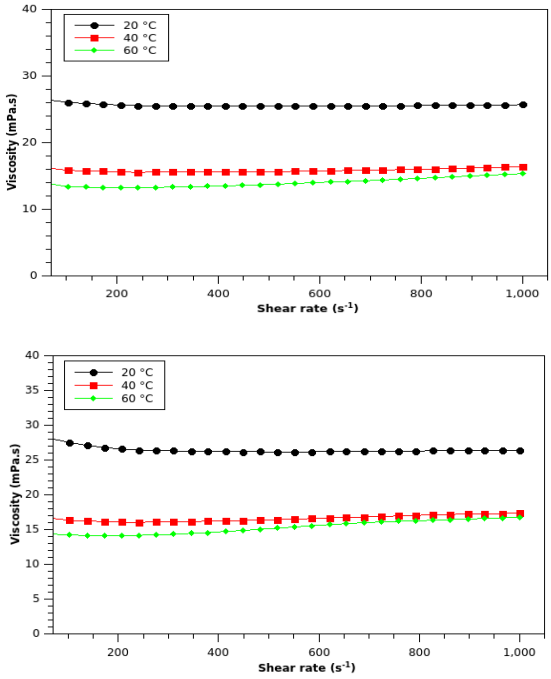


Figure 1: Viscosity (mPa·s) of the nanocomposites of a) positively charged SPIONs-PEG and b) negatively charged SPIONs-PEG versus shear rate

The viscosity of the nanocomposites was constant with shear rate at 20 °C and 40 °C, but it had a small increase at 60 °C. As expected, for all the samples the drop in their viscosity values was observed when the temperature increased.

Fig. 2 shows the viscosity variations of the nanocomposites of positively charged SPIONs-PEG and negatively charged SPIONs-PEG versus shear strain at 20 °C, 40 °C and 60 °C. As shown in this figure, the similarity in the behavior or each type of nanocomposite with the same type of nanocomposite in *Fig. 1* was observed. In other words, both nanocomposites showed a steady-state behavior at 20 °C and 40 °C, but their viscosity increased a bit versus shear strain at 60 °C.

Fig. 3 shows the viscosity variations of the nanocomposites of positively charged SPIONs-PEG and negatively charged SPIONS-PEG versus time at 20

°C, 40 °C and 60 °C. The small increase of viscosity versus time at 60 °C and the steady-state behavior of the nanocomposites was observed similar to the results presented in the previous figures.

The changes of torque versus shear strain for both nanocomposites at 20 °C, 40 °C and 60 °C are shown in *Fig. 4*. The torque values increased with the increase of the shear strain of the samples. As shown in this figure, the torque did not increase with the same slope at different temperatures, as it was higher at 20 °C, but it decreased when the temperature increased to 40 °C and 60 °C. In other words, less torque values were observed for both nanocomposites when the temperature increased.

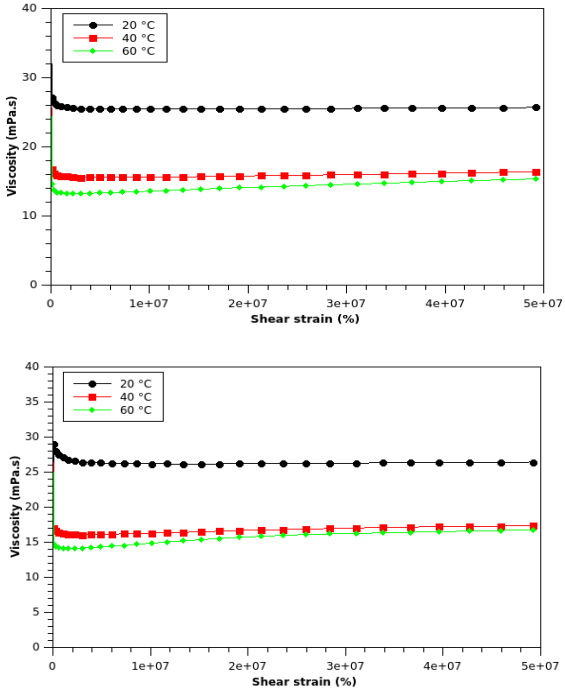


Figure 2: Viscosity of the nanocomposites of a) positively charged SPIONs-PEG and b) negatively charged SPIONs-PEG versus shear strain

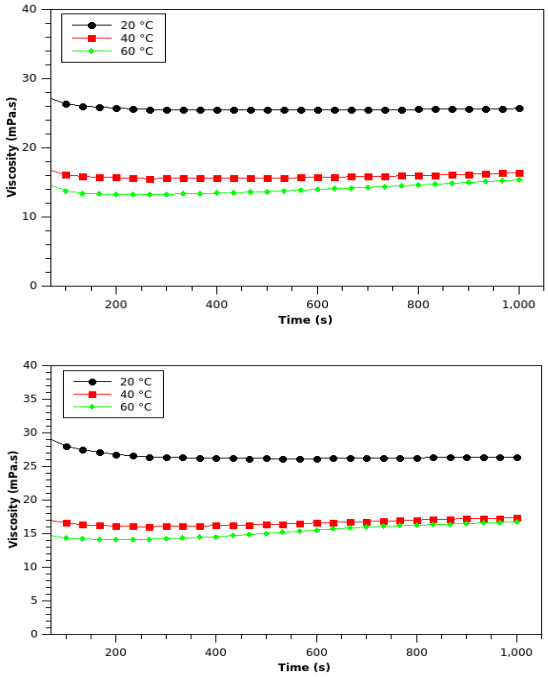


Figure 3: Viscosity of the nanocomposites of a) positively charged SPIONs-PEG and b) negatively charged SPIONs-PEG versus time

As observed in the previous figures, the data for 40 °C and 60 °C were close to each other. Therefore, we can conclude that the rheological properties of both nanocomposites changed significantly from 20 °C to 40 °C and changed a bit with the increase of temperature.

The changes of torque versus time at 20 °C, 40 °C and 60 °C are shown in *Fig. 5* for both nanocomposites. The torque increased with time with a constant slope for each nanocomposite. A linear change of torque versus time was observed at each temperature. In other words, the increase of torque was constant with time for the nanocomposites. Therefore, no change in the torque increase was applied over time on each nanocomposite. So, the constant increase of torque versus time was expected.

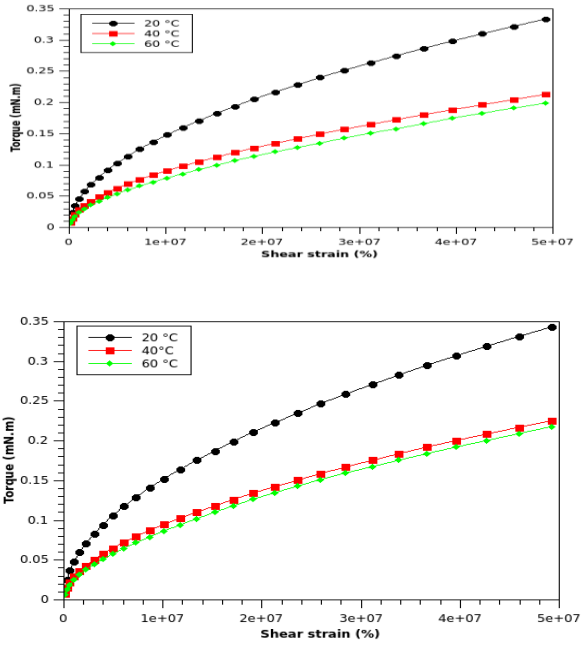
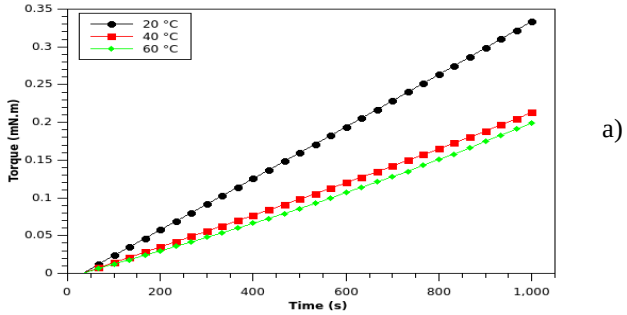
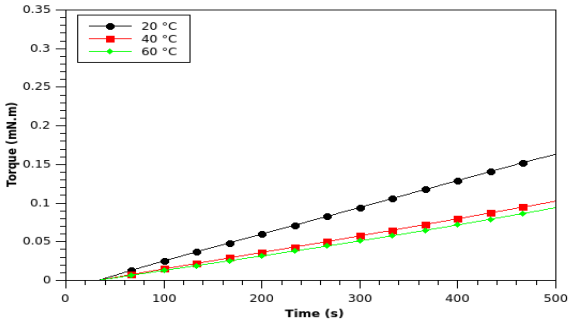


Figure 4: Torque versus shear strain for the nanocomposites of a) positively charged SPIONs-PEG and b) negatively charged SPIONs-PEG



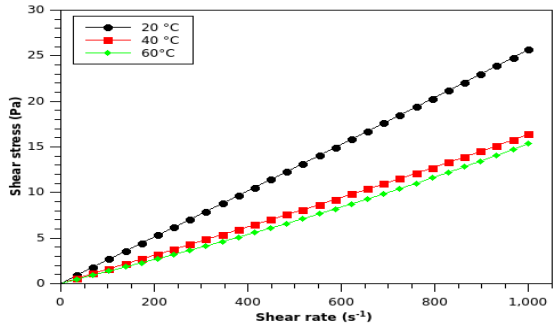


b)

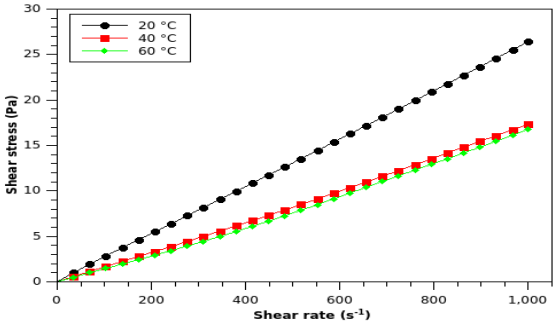
Figure 5: Torque versus time for the nanocomposites of a) positively charged SPIONs-PEG and b) negatively charged SPIONs-PEG

Fig. 6 shows the shear stress values versus shear rate at 20 °C, 40 °C and 60 °C for the nanocomposites of positively charged SPIONs-PEG and negatively charged SPIONs-PEG.

As shown in *Fig. 6*, the changes of the shear stress applied on the samples were linear and the slope of the increase of shear stress with shear rate was the same for each nanocomposite at 40 °C and 60 °C, which indicated the same constant rate of shear stress at these temperatures when it changed with shear rate. Moreover, the slope was higher at 20 °C, which showed that the change of the shear stress applied on each nanocomposite of SPIONs with PEG versus shear rate was higher at a lower temperature.

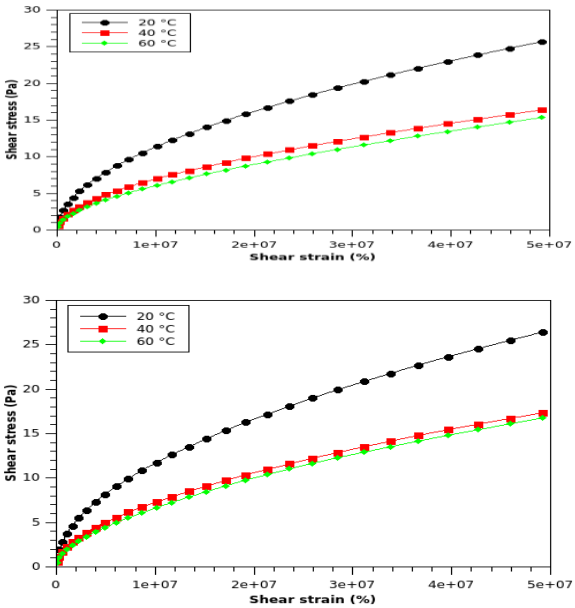


a)



b)

Figure 6: Shear stress of the nanocomposites of a) positively charged SPIONs-PEG and b) negatively charged SPIONs-PEG versus shear rate



a)

b)

Figure 7: Shear stress of the nanocomposites of a) positively charged SPIONs-PEG and b) negatively charged SPIONs-PEG versus shear strain

Fig. 7 shows the shear stress applied on the nanocomposites of positively charged SPIONs-PEG and negatively charged SPIONs-PEG versus shear strain at 20 °C, 40 °C and 60 °C.

For both nanocomposites non-linear changes were observed for the shear

stress. Moreover, the shear stress values increased with the increase of the shear strain. Almost the same slopes were observed for the samples for the increase of shear stress with shear strain at 40 °C and 60 °C, which indicated that they had nearly the same increase rate of the shear stress versus shear strain, but the slope observed in the figure was higher at 20 °C, which showed that a higher change of the shear stress of each nanocomposite versus shear strain at a lower temperature.

In this paper, the variations of the shear stress with shear rate and shear strain were investigated for explaining how these parameters changed for the nanocomposites of positively surface charged SPIONs-PEG and negatively surface charged SPIONs-PEG. As observed, although shear stress increased with shear rate for both nanocomposites, their viscosity values versus shear rate, shear strain or time did not show a significant change at different temperatures.

The molecular structure of surface charged SPIONs have been investigated previously with amine and carboxyl groups on the surface of positively charged and negatively charged SPIONs, respectively [34]. In the current research work, the similarity in the results obtained for the samples showed that the difference in their surface charge did not have a significant impact on their rheological properties. This rheological investigation was performed for providing the information required for the determination of the properties of these nanocomposites and their further improvement in future studies.

Several studies have been performed on nanomaterials [35-39], biomaterials [40-44] and construction materials [45-46] with diverse applications in science and engineering. Some of these materials have shown non-Newtonian behavior maintainable with their preparation in polymeric matrices [47-48]. The optimization of the properties of these materials can provide important information for the improvement of their mechanical properties for further applications. The Technique for Order of Preference by Similarity to Ideal Solution (TOPSIS) is an appropriate method that has been widely applied for the optimization and prediction of various characteristics [49-54]. Some investigations on the nanocomposites of iron oxide nanoparticles with polymers have shown that these materials would be appropriate for the function improvement of electrical devices [55-56]. The present study can provide new insight to the investigation of their properties for the manufacture and optimization of these devices.

4. Conclusion

This paper aimed to investigate the rheological properties of surface-charged SPIONs that have found their diverse applications in science and engineering during recent years. The samples of two types of nanocomposites, positively

surface charged SPIONs-PEG and negatively surface charged SPIONs-PEG, were investigated. The results in this study showed that the rheological properties of the nanocomposites did not depend on their surface charge but depended on temperature, because they changed with the temperature increase. The steady-state behavior of both nanocomposites was observed at 20 °C and 40 °C. Moreover, a small increase of viscosity versus shear strain, shear rate or time for the samples was observed at 60 °C. The shear stress increased with shear rate and shear strain in all the measurements with higher slopes at 20 °C, which decreased with the increase of temperature. The results of this investigation can be used for the preparation improvement of these nanocomposites as well as the correlation of their physical and mechanical properties.

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