EFFECTS OF SAMPLE PREPARATION ON THE THERMAL CONDUCTIVITY OF POLYMERIC NANOCOMPOSITES

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Abstract. *This paper comprises an experimental investigation of the thermal conductivity of nanocomposites manufactured from an unsaturated polyester resin loaded with alumina (*$\mathrm{Al_2O_3}$*), tenorite (*CuO*) and hematite (*$\mathrm{Fe_2O_3}$*) nanoparticles. The main objective of this investigation is to determine the effects that different approaches for fabricating nanocomposite samples can have on their effective thermal conductivity. Some manufacturing characteristics that have been studied include: different mixing and homogenizing alternatives, sample surface finishing, sample property change with aging after curing, number of samples required for a reasonable statistical significance, and molding preparation. Results presented in this paper can help achieving a better preparation of nanocomposite samples.*

Keywords: *Nanocomposites, Thermal conductivity, Polymers*

1. INTRODUCTION

Polymeric materials have desirable qualities for the industry, like improved corrosion resistance and light weight. Despite these remarkable characteristics, some of polymer properties, like low thermal conductivity, may be a limiting factor to certain applications. By adding fibers or particles to a polymer, some properties may be enhanced. Changes in macroscopical properties of compounds loaded with nanofillers (nanofibers, nanoparticles and other) have been continuously investigated, as it has been shown that enhancements in properties can be obtained with little increase in weight. Choi and Eastman (1995) observed that the addition of a small fraction of nanoparticles to a solution can lead to a noticeable change in the overall thermal conductivity. With the inclusion of nanoparticles of higher thermal conductivity than the matrix, the macroscopically observed thermal conductivity of a solution (liquid or solid) is significantly higher than the predicted value if particles of a larger size scale were used.

Several studies, both of experimental and theoretical nature, have been conducted with the attempt of investigating this phenomenon, most of them are oriented towards thermal intensification of liquids (Wang *et al.*, 1999; Eastman *et al.*, 2001, 2004; Zhu *et al.*, 2007), leading to the so-called nanofluids. Although most previous studies are nanofluid oriented, the same thermal intensification can occur in solids. Some studies investigate the thermal conductivity of different polymeric matrix composites (Kuriger and Alam, 2002; Putnam *et al.*, 2003; Kumluta *et al.*, 2003; Sim *et al.*, 2005; Fu *et al.*, 2010). The unpredicted enhancement of properties of liquids and solids may be attributed to the high aspect ratio of nanofillers. Due to nanoparticle dimensions (< 100 nm), large fractions of atoms may reside on its surface and interfacial interaction of the filler with polymer matrix is strengthened, giving greater importance to interfacial properties (Rong *et al.*, 2006; Kim *et al.*, 2007; Hanemann and Szabó, 2010). However, this high surface area also leads to higher attraction between the particles, resulting in agglomeration.

The effective properties of nanocomposites are as dependent on the properties of the components as on the morphologies of the dispersed phase and the degree of homogenization. If nanoparticles exists in the matrix as macro- or micro-sized agglomerates, the effect produced by these fillers will be lower than the possible effect if a good dispersion was achieved. A good homogenization depends on dispersion and distribution of the filler and it is essential to obtain the best properties in the manufactured nanocomposites (Kim *et al.*, 2007). In this context, the goal of this study is to develop an effective and inexpensive fabrication method for nanocomposites and experimentally analyze variations in the effective thermal conductivity of the samples fabricated. Different homogenizing methods, sample surface finishing and variation of thermal conductivity with temperature are compared for an unsaturated polyester resin separately filled with three different metal oxides.

2. MATERIALS AND METHODOLOGY

The polymer used in this study was Polylite 10316-10 (provided by Reichhold), an unsaturated polyester resin with low viscosity and fast curing, commonly used in the production of fiber-reinforced plastics or non-reinforced filled products for various applications and end-markets. The resin is diluted in 44% styrene and this system is pre-accelerated by the manufacturer. The initiator used was methyl ethyl ketone peroxide (1.5 phr). The resin properties, provided by the manufacturer, are presented in table 1.

Table 1. Properties of Unsaturated Polyester Resin

Property	Value
Viscosity at 25°C μ (cP)	250 to 350
Density ρ (kg/m^3)	1090
Heat Distortion Temperature HDT (°C)	85
Modulus of Elasticity E (GPa)	3.3
Flexural Strength (MPa)	45
Tensile Strength (MPa)	40
Maximum Elongation (%)	1

In order to obtain the different composite systems, the nanoparticles employed as fillers were Al_2O_3 (alumina), CuO (tenorite) and Fe_2O_3 (hematite), provided by NanoAmor (www.nanoamor.com). The average particles size and other information are presented in table 2. Except for the thermal conductivity, obtained from (Ren *et al.*, 2005) for Al_2O_3 and CuO and from (Molgaard and Smeltzer, 1971) for Fe_2O_3, all of the presented properties were obtained from the manufacturer.

Table 2. Thermophysical properties of nanoparticles.

Property	Al_2O_3	CuO	Fe_2O_3
thermal conductivity (W m^{-1} K^{-1})	41.1	32.9	6.4
true density (kg/m^3)	3500 to 3900	6300 to 6490	5240
morphology	nearly spherical	nearly spherical	spherical
particle size (nm)	30 - 40	30 - 50	20 - 60
specific surface area (10^3 m^2/kg)	35	13.1	20 - 60
purity	99.99%	>99%	98%

2.1 Fabrication of nanocomposites

The samples were manufactured by adding different amounts of nanoparticles (from 0% to 10% in volume of the total mixture) to the liquid resin. Since some metal oxide particles can uptake considerable amounts of water vapor from atmospheric air, the nanoparticles must be dried before being added to the resin. Drying was performed at 120°C for 24 hours. Mixtures containing from 1% to 10% in volume fraction of each of the used metal oxides were obtained. Also, samples of pure polyester resin with no filler were made as references. The volume fraction (ϕ) was calculated based on true densities data provided by the manufacturers. Homogenization by manual mixing, magnetic stirring, mechanical stirring and ball milling was tried. Ball milling was tried with stainless steel grinding jar and balls.

Samples were manufactured using simple molding. The mold is composed of a central metal frame between two glass plates. Frames were built from tin and aluminum plates of different thicknesses (6 mm, 12 mm and 15 mm) that were machined with the sample holes (50 mm in diameter) and a flow channel for introducing the liquid resin, as displayed in figure 1. Parting wax was applied to all internal surfaces of the mold. After mixing, the initiator was added and the mixture was poured into the mold.

After mold releasing, the sample's surfaces must be carefully sanded, since they should be flat-parallel. Otherwise, the thermal contact resistance with the surfaces of the conductivity meter will be increased, which will lead to underestimated values for the thermal conductivity of the samples. Batches with 4 samples of each mixture were made. Also, a batch containing 10 samples of pure polyester was made in spite of investigating if the average value and the straight deviation would change significantly with variation in number of samples tested.

2.2 Characterization

The experiments were conducted using a direct thermal conductivity measurement device (LaserComp, Fox-50) showed in figure 2. The measurement method conforms to the guarded heat flow meter technique, modified to take into account the thermal contact resistance. Design of the instrument follows ASTM standards C518-04 and E1530-06 and the device specifications can be seen in table 3.

There are two possible test modes, one-thickness and two-thickness. In one-thickness mode, a sample with flat-

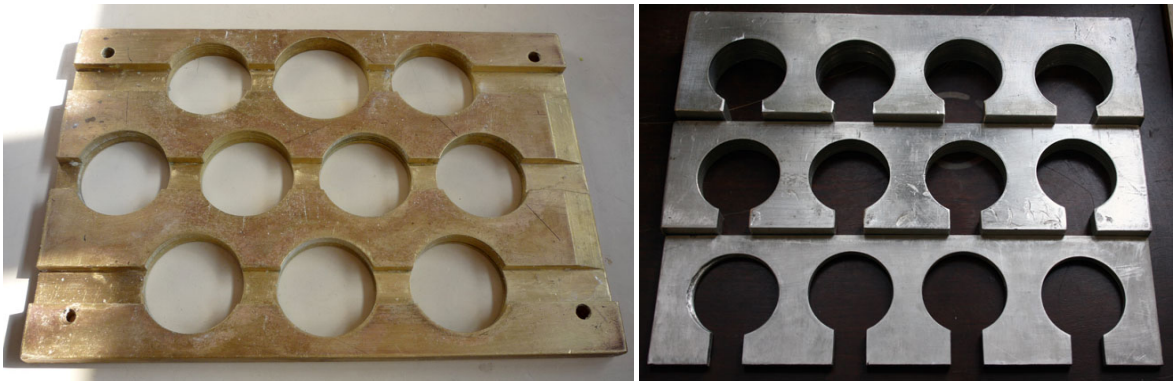

Figure 1. Frames used in molding the samples.

Table 3. LaserComp Fox-50 specifications

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Description	Value
Thermal conductivity range ($W\,m^{-1}\,K^{-1}$)	0.1 - 10
Precision (two-thickness)	3%
Maximum temperature of hot plate	110°C
Minimum temperature of cold plate	-10°C
Temperature control stability	+/- 0.02°C
Thickness measurement accuracy	+/- 0.025 mm
Maximum sample size	63.4 mm
Minimum sample size	50 mm
Maximum sample thickness	25 mm
Actual metering area	25x25 mm

Figure 2. Thermal conductivity measuring device (LaserComp Fox-50).

parallel surfaces is required and the thermal contact resistance is assumed to be the same as the value in the calibration file. Samples were approximately 12 mm thick. In contrast, in two-thickness mode, two samples of the same material and with the same surface finish must be used, so the contact resistance can be calculated by the instrument, once it is assumed to be the same for both samples. Thicknesses of thin samples were about 6 mm while thick samples had 12 mm or 15 mm.

To run a test, the test mode must be chosen and desired pairs of temperature set points should be entered. Next, a calibration file to be used for calculations must be selected. It is recommended that the selected calibration curve is based on a material that has a thermal conductivity closer to the measured sample. For batches with low concentrations of particles the chosen calibration curve was based on Perspex, while Polycarbonate was selected for higher filler loadings. Thermal conductivity values (in $\mathrm{W\,m^{-1}K^{-1}}$) of the two materials selected as calibrations are displayed in table 4.

Table 4. Perspex and polycarbonate calibration curves

T (°C)	Perspex	Polycarbonate
-10	0.184800	0.211300
20	0.188500	0.223000
40	0.190900	0.230000
60	0.193300	0.237000
70	0.194550	-
80	-	0.245000
100	-	0.253000
110	-	0.256400

3. RESULTS AND DISCUSSION

It was noticed that some mixtures with high volume fraction of nanoparticles had not completed the polymerization even after 24 hours of being poured into the mold. This problem occurred with mixtures containing 7.5% and 10% of $\mathrm{Fe_2O_3}$ and some samples with 10% of CuO. These mixtures were then submitted to a post-cure at 60 °C for 3 hours. This procedure not only has completed the curing process of the mixtures, but also has made the mold releasing much easier.

Results of thermal conductivity of pure polyester resin samples and samples loaded with $\mathrm{Al_2O_3}$, CuO and $\mathrm{Fe_2O_3}$ were experimentally obtained from Fox50 and are presented in the following tables. Table 5 presents values obtained for pure resin samples. It is worth mentioning that many of the manufactured samples were tested in two occasions: a few days after the polymerization and again, some months after. It was not observed any considerable variations in the obtained results. Average results and straight deviation of different batch sizes for three different setpoints (0, 25 and 50 °C) are demonstrated in table 5. It was not observed any significant variation in these values for batches containing less samples. Furthermore, the obtained results presented a dispersion of less than 1%, while LaserComp guarantees a precision of 3%. Based on that, the subsequent batches were composed of 4 samples each, though 3 samples of the same mixture (in good conditions, naturally) are enough for a reasonable estimation of the effective thermal conductivity of a material with this equipment.

Table 5. Thermal conductivity measurements (Average±St. Dev.) of pure polyester batches

	k ($\mathrm{W\,m^{-1}K^{-1}}$)		
T (°C)	10 Samples	6 Samples	3 Samples
0	0.1550±0.0013	0.1550±0.0017	0.1551±0.0010
25	0.1574±0.0011	0.1575±0.0014	0.1575±0.0008
50	0.1578±0.0008	0.1579±0.0008	0.1579±0.0006

It is important to mention that higher concentrations of particles lead to difficulties in homogenization. Polyester/alumina mixtures containing 5%, 10% and 20% in mass (approximately 1.5%, 3.1% and 6.9% volume fraction, respectively) were mixed by a magnetic stirrer for 4 hours and the thermal conductivities of these fabricated samples are showed in table 6. Naturally, for higher concentrations of nanoparticles the thermal conductivity is increased, but it is also observed a significant increase in straight deviation of the results, which can be attributed to a poor homogenization due to higher concentrations of fillers and probably larger agglomerates.

Figure 3 presents the enhancement of thermal conductivity for alumina loaded samples containing from 0% to 10% in volume fraction of nanoparticles. As it was observed an increase in straight deviation, mixing time was also increased for higher loaded blends and mixtures containing ϕ = 10% were stirred for 6 hours. As a consequence, dispersion of the results of samples containing ϕ = 10% is lower than that of samples with ϕ = 6.9%, which were stirred for 4 hours. It is also noticed an enhancement of approximately 70% in the overall thermal conductivity of the polyester resin for samples containing 10% of $\mathrm{Al_2O_3}$.

Table 6. Thermal conductivity measurements (Average±St. Dev.) of alumina loaded nanocomposites mixed by magnetic stirrer for 4 hours

	k (W m^{-1}K^{-1})		
T (°C)	1.5%	3.1%	6.9%
0	0.1682±0.0006	0.1816±0.0007	0.2179±0.004
25	0.1696±0.0006	0.1837±0.0007	0.2202±0.0040
50	0.1724±0.0014	0.1854±0.0008	0.2223±0.0043

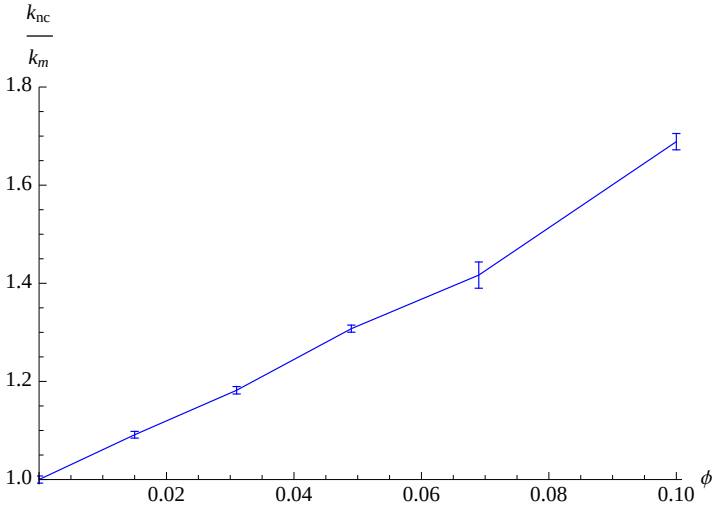

Figure 3. Enhancement in thermal conductivity obtained at 25°C for samples composed by different fractions of polyester and Al_2O_3, mixed by magnetic stirrer

Different batches containing alumina or hematite nanoparticles were prepared by manual mixing, ball milling, magnetic stirring and mechanical stirring. Figure 4 shows samples loaded with approximately 1.5% volume fraction of alumina mixed by different methods. Samples manufactured by manual mixing showed large amounts of visible agglomerates even with low particle loadings, while samples homogenized by the planetary ball mill were clearly contaminated by steel.

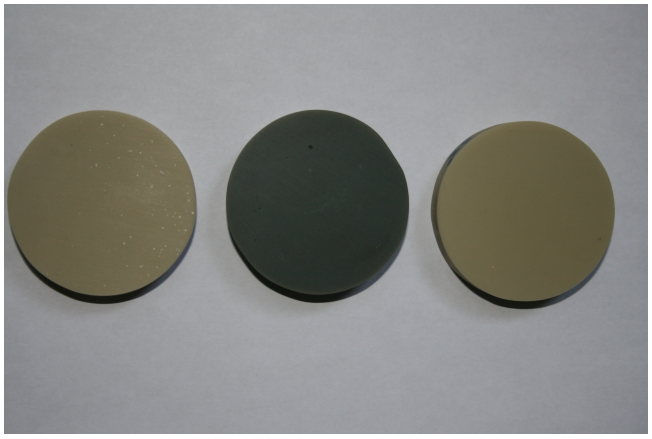

Figure 4. Samples containing alumina nanoparticles ($\phi = 1.5\%$) manually mixed, using the planetary ball mill and by magnetic stirring, respectively.

Batches containing 5 phr of alumina nanoparticles were manufactured by manual mixing and ball milling. Also, batches with 5% and 15% in mass of Al_2O_3 ($\phi_m = 5\%$ and $\phi_m = 15\%$) were prepared by manual mixing and magnetic stirring. Finally, samples loaded with Fe_2O_3 ($\phi = 1\%$) were homogenized by magnetic stirring, ball milling and mechanical stirring. Table 7 compares the results obtained for these mixing methods.

Results of milled mixtures are higher than the manually mixed ones, however, this effect can be attributed to steel contamination, as can be observed in alumina samples in figure 4. Despite the fact of contamination, table 7 also shows

Table 7. Thermal conductivity measurements (Average±St. Dev.) of polyester/Al_2O_3 and polyester/Fe_2O_3 mixed by different devices

			k (W m^{-1}K^{-1})			
Material	ϕ (%)	T (°C)	Manually	Ball Mill	Magnetic Stirrer	Mechanical Stirrer
Al_2O_3	1.4	0	0.1622±0.0016	0.1692±0.0006	-	-
		25	0.1642±0.0011	0.1707±0.0002	-	-
		50	0.1667±0.0010	0.1718±0.0004	-	-
	1.5	0	0.1670±0.0017	-	0.1682±0.0006	-
		25	0.1691±0.0013	-	0.1696±0.0006	-
		50	0.1703±0.0014	-	0.1724±0.0008	-
	4.9	0	0.1962±0.0025	-	0.2015±0.0006	-
		25	0.1975±0.0024	-	0.2032±0.0004	-
		50	0.2016±0.0027	-	0.2052±0.0019	-
Fe_2O_3	1	0	-	0.1623±0.0009	0.1614±0.0019	0.1525±0.0001
		25	-	0.1647±0.0010	0.1639±0.0015	0.1542±0.0000
		50	-	0.1654±0.0010	0.1651±0.0010	0.1539±0.0004

that straight deviation of samples homogenized by the planetary ball mill is clearly lower than that of manually mixed alumina loaded samples and stirred hematite loaded ones, which suggests that a better homogenization was achieved. This would be easier observed for higher particle loadings, when variations in results would be evidenced due to the difficulty in homogenizing high filled mixtures. Yet, greater concentrations would lead to more abrasive mixtures and it could intensify steel contamination of the samples. Therefore, the ball mill was not used for higher loaded mixtures, once the available grinding jar and balls were stainless steel made.

It was observed that Fe_2O_3 nanoparticles were affected by the magnetic field of the stirrer, with great deposition of particles at the bottom of the glass. Hence, magnetic stirring seemed to be the best available way for homogenizing mixtures containing non-magnetic nanoparticles but, for batches containing Fe_2O_3 the ball mill and a mechanical paddle mixer were employed. Because of the possibility of steel contamination the ball mill was used only for 1% loading, just for comparison purposes, while other batches with 1%, 2.5%, 5%, 7.5% and 10% were homogenized by the mechanical mixer for 2 hours at 1000 RPM.

Figure 5 shows the enhancement obtained at 25°C for mixtures containing different fractions of Fe_2O_3 mixed by the mechanical mixer. As one can see, there is a high variation between the results of samples obtained from the same mixtures. During the fabrication process, it was very difficult to homogenize the mixtures and there was deposition of particles each time the different blends were poured into the mold. Also, it was observed the presence of bubbles in some samples and the thermal conductivity may have been underestimated, especially for samples containing ϕ = 1%, which presented lower results than pure polyester samples. In addition, it should be mentioned that this tendency of deposition continued while the blends were already in the mold and the distribution of the filler was visually affected by that.

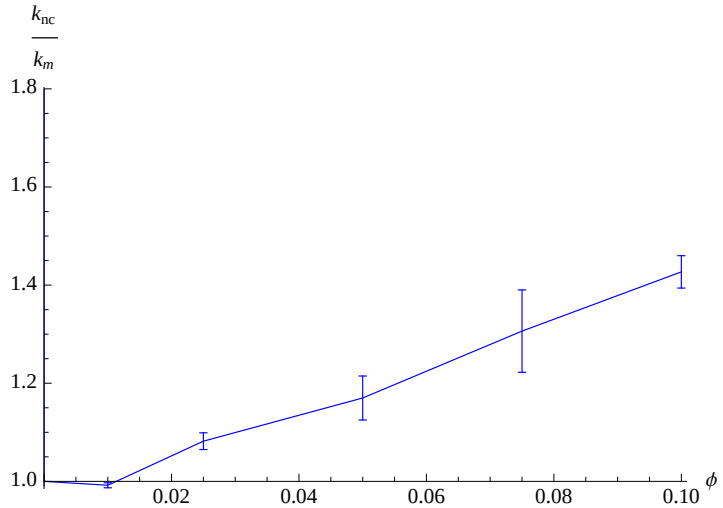

Figure 5. Enhancement in thermal conductivity obtained at 25°C for samples composed by different fractions of polyester and Fe_2O_3 mixed by mechanical mixer for 2 hours

Samples containing CuO were also manufactured. These mixtures were homogenized only by magnetic stirrer, initially for 2 hours, but mixing time was increased with concentration and mixtures containing 10% of CuO particles were mixed for 7 hours. Like all of the other samples, these samples were sanded until polished surfaces were obtained, but they were tested with coarse and polished surfaces. Table 8 shows a comparison between the average measurements obtained at 25 °C for the same pure polyester and CuO loaded samples sanded with grit sizes of 100 and 600. As showed by this table, the effective thermal conductivity measurements are slightly higher (approximately 1%) for samples sanded with finer sandpapers, which can be attributed to the higher thermal contact resistance between coarser sample's surfaces and the surfaces of the equipment.

Table 8. Comparison of average values of thermal conductivity at 25°C of samples of pure polyester and containing different loadings of CuO nanoparticles sanded with grit sizes of 100 and 600

	k (W m^{-1}K^{-1})					
Sand grit	0%	1%	2.5%	5%	7.5%	10%
100	0.1570	0.1659	0.1808	0.2090	0.2389	0.2668
600	0.1574	0.1671	0.1816	0.2113	0.2406	0.2702

Enhancement in thermal conductivity resulted of polished samples containing CuO measured at 25 °C can be seen in figure 6. Dispersion of the values obtained for the thermal conductivity of CuO loaded samples are very little for $\phi < 5\%$ and is increased for higher concentrations, as can be observed in figure 6. It is also noticed an enhancement in the overall thermal conductivity of the polyester resin of approximately 75 % for samples containing 10 % of CuO, while Al_2O_3 loaded samples achieved an enhancement of approximately 70 % and Fe_2O_3 loaded samples achieved an increment of less than 50%, as showed in figure 5.

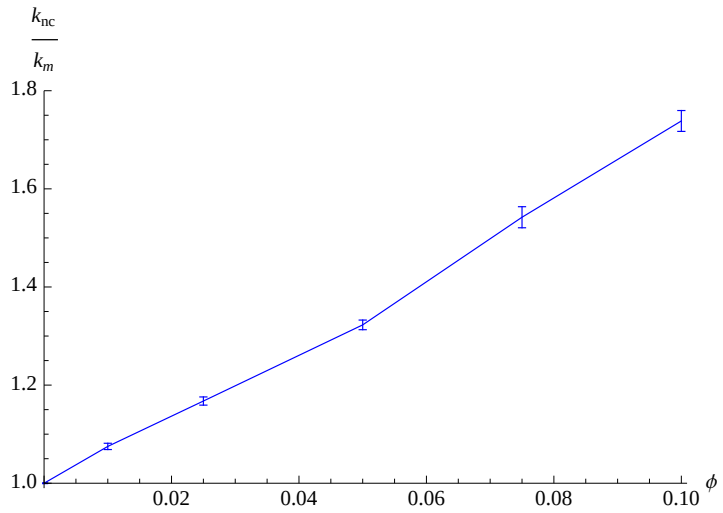

Figure 6. Enhancement in thermal conductivity obtained at 25°C for polished samples composed by different fractions of polyester and CuO mixed by magnetic stirrer

As it was expected, polyester/Fe_2O_3 samples presented lower values than samples loaded with CuO and Al_2O_3, due to its lower thermal conductivity and difficulties in homogenization. Alumina loaded samples should present the higher augmentation in thermal conductivity. Nevertheless, results obtained with the addition of tenorite nanoparticles to the polyester resin were higher than those obtained with the addition of alumina. This can be attributed to a better homogenization of blends containing CuO.

4. CONCLUSIONS

This paper presented an experimental investigation of heat conduction in nanocomposites and the effects that different approaches of manufacturing can have on the effective thermal conductivity. Polymer samples containing different concentrations of alumina, hematite and tenorite were manufactured and experimentally analyzed using a thermal conductivity measuring device. As expected, the results showed that the thermal conductivity increases with nanoparticle concentration.

Samples were tested at 0, 25 and 50°C a few days after the polymerization and some months after. It was noticed a minor variation with the temperature and no considerable variations of the results were observed with sample aging.

Also, tests were performed with polished and coarse samples' surfaces and it was observed that the effective thermal conductivity measured for polished samples is approximately 1% higher than coarse samples, because of the higher thermal contact resistance between coarser sample's surfaces and the surfaces of the conductivity meter.

Moreover, it was observed that CuO loaded samples presented the higher enhancement of thermal conductivity though the thermal conductivity of CuO is lower than that of $\mathrm{Al_2O_3}$. This can be attributed to a better homogenization. Although good results were obtained with the magnetic stirrer, for blends containing $\phi > 5\%$ the straight deviation increases even with an increase on the mixing time, which suggests that a more efficient mixing method should be employed for higher particle loadings.

Besides presenting the lower enhancement in thermal conductivity of the polyester resin, it should be mentioned that $\mathrm{Fe_2O_3}$ loaded mixtures were very unstable and the particles were not suspended in the solution during the fabrication process and after, during the polymerization. Thus results obtained with the addition of $\mathrm{Fe_2O_3}$ to the polyester resin had great dispersions.

The planetary ball mill was used and it has been noticed that alumina loaded samples were contaminated by stainless steel because of the high abrasive power of these particles. $\mathrm{Fe_2O_3}$ loaded mixture was also milled and there was no visible contamination, yet it could not be discarded. Further investigation with mixtures homogenized by the planetary ball mill shall be carried out with grinding jar and balls made of a more wear resistant material, so alumina loaded loaded blends and higher loaded mixtures may be milled with no risk of contamination.

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