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# Investigation of Carbon Black/ Polyester Micro-composites: An Insight into Nano-size Interfacial Interactions

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#### PAPER INFO

ABSTRACT

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Keywords: Interfacial Interactions Cooperative Rearranging Region Nano-micro Composites Mechanical Properties Interphase In the micro/nanomaterial reinforced composites, interfacial interactions at the interface of filler/polymer lead to the formation of a third layer called interphase as the secondary reinforcing mechanism. The interphase may be formed due to local adsorption of polymer chains at the interface, mechanical interlocking, and interdiffusion of polymer chains. Since the interactions govern the load transfer at the filler/polymer interface, they play a key role in the mechanical response of reinforced composites. However, there exist only a few well-established and validated studies in the description of the interfacial interactions presented in thermosetting composites. This research aims at the understanding of correlations amongst the mechanical properties of thermosetting polyester composites reinforced with 0-15 wt. % of carbon black (CB) focusing on the nano-size cooperative rearranging region (CRR). To estimate the length of CRR, thermal analysis of the variations in the specific heat capacity or the relaxation strength within the glass transition temperature (Tg) range was measured using a thermodynamic model. A nano-size CRR of 10 nm on average was estimated and correlated to the enhanced impact and toughness behavior of the specimens. The results suggested the presence of softer interphase based on the T<sub>g</sub> values influenced by the CBs agglomeration level and cross-linking density, which in turn governs the mechanical response of the composites. The methodology introduced in this study can be used in the explanation of changes in mechanical and physical properties of reinforced composites with a focus on the underlying role of nano-size interfacial interactions.

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#### NOMENCLATURE

$\Delta C_p$	Calorimetric relaxation strength (J/K.g)	$V_{\alpha}$	The volume of the CRR (nm <sup>3</sup> )	
CI	Crystalline index	K <sub>B</sub>	The Boltzman constant (1.380649×10 <sup><math>-23</math></sup> J/K)	
CRR	The cooperative rearranging region (nm)	Greek Symbols		
$C_v$	Specific heat capacity (J/K.g)	ρ	Density (g/cm <sup>3</sup> )	
P.I	Peak intensity	θ	Half of XRD characteristic peak (9)	
Tg	Glass transition temperature (°C)	δ	The characteristic length of the glass transition (nm)	

#### **1. INTRODUCTION**

The interfacial interactions govern the polymer properties at or near the interface between the filler and polymer resulting in the creation of an interfacial zone or "interphase" [1, 2]. The modified interfacial chains control communication between fillers and polymer chains away from the filler surface and have chemical, physical, microstructural, and mechanical properties other than those of the composite constituents [3].

Interfacial interactions may be governed by the thermodynamic compatibility and the chemical affinity of the reinforcement and polymer, the dispersion/distribution quality of fillers, crystallization characteristics of the matrix, and the processing technique used. The latter is reported to govern overall performance of thermosetting parts filled with

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Please cite this article as: M. Karevan, Investigation of Carbon Black/ Polyester Micro-composites: An Insight into Nano-size Interfacial Interactions, *International Journal of Engineering, Transactions C: Aspects*, Vol. 35, No. 06, (2022) 1135-1143 micro/nano-reinforcements [4, 5]. It has been reported that the higher the strength of the interfacial interactions, the greater the level of fillers dispersion [6]. The higher degree of dispersion means the existence of greater reinforcing sites, stronger filler/polymer adhesion and thus more enhanced mechanical properties of composites. The latter may be also resulted from hindering deformation and shearing of polymer chains around the filler.

The main challenge is that weak interfacial interaction cannot be avoided due to the tendency of fillers to agglomerate leading to a significant decrease in the mechanical and electrical properties compared to the initial expectations and predicting elastic models that fail due to wrong assumptions regarding the filler/polymer bonding [7, 8]. Although surface modifications or applying high shear force during the dispersion state in the fabrication of micro/nanocomposites have been widely used, a useful tool to better understand the formation and changes in the extent of the interfacial interactions is still needed. This is in particular of higher importance when considering effect particles agglomeration/dispersion and the need for advanced methods in the qualification or quantification of the interactions [9, 10]. For instance, characterization techniques such as the atomic force microscopy and nano-indentation not only can be considered as rather expensive and time-consuming method and high level of skills, but also these methods cannot estimate the representative thickness of interphase through the composite bulk [11].

Methods such as Raman spectroscopy, FTIR spectroscopy and X-ray photoelectron spectroscopy (XPS) have been also reported as those useful in the characterization of the interphase [12, 13]. However, such methods normally are not able to give quantitative data about the size/geometry of the interphase. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) provide qualitative information, too [14]. The presence of agglomeration has been widely reported to unfavorably influence the overall mechanical response of composites, too [15, 16]. The latter is believed to directly affect the strength of interfacial interaction due mainly to the attractive nature of particles when their hydrophobicity differs from that of the parent matrix. Although numerous studies have reported the effect of carbon-based fillers on the mechanical properties of polymeric composites [17, 18], less research has been conducted on the correlations among the mechanical behavior and the interfacial interaction at the filler/polymer and in particular in the case of thermosetting composites [19, 20].

This research emphasizes the characterization of mechanical performance including the tensile response, impact resistance and toughness and thermal behavior of polyester composites reinforced CB followed by the quantification of the interphase represented by the cooperative rearranging region (CRR). The CRR was estimated using a thermodynamic model through variations of the specific heat capacity or the calorimetric relaxation strength ( $\Delta C_p$ ) of the composites over the glass transition temperature (Tg) [21]. The findings were further correlated with the microstructure of the composites to better understand the role of CRR on the mechanical response of the fabricated CB reinforced composite parts. The hypothesis is that the interfacial interactions govern the degree of interfacial bonding and thus the Tg and mechanical performance of the composites [22]. The current study could open up a novel methodology more specifically in the case of thermosetting matrices to quantify the interfacial polymer chains of modified properties linked to the bulk behavior of micro/nano reinforced polymeric composites.

# **2. EXPERIMENTAL**

**2. 1. Material** For fabrication of CB/Polyester composites, industrial-grade CB was used as the reinforcement purchased from Doodeh Sanati Pars Co. (Iran). The CB used is a black powder with particle size estimated from the scanning electron microscopy (SEM) method to be  $\sim 5 \ \mu m$  and a density of  $\sim 1.7 \ g/cm^3$ . An industrial-grade polyester thermoset resin with clear appearance was provided by a local supplier and used as the base polymer matrix. The resin components of hardener and catalyst were used as described by the manufacturer. The curing time of the resin used appeared to be around 20-30 min depending on the catalyst ratio.

2. 2. Fabrication of CB/Polyester Microcomposites Composites of CB/Polyester reinforced with 0 to 10 wt. % of CB were prepared using



Figure 1. Schematic of CB/polyester composites fabrication route from the mixing to mold casting step

direct mixing of CB within the resin utilizing a high shear mixer at the rpm of 3000 for 15 minutes. The CB particles initially were dispersed within isopropyl alcohol and then vacuum filtered. The CB particles were collected and vacuum dried at 80 °C and 8h to ensure removal of the residual IPA and water. To fabricate the polyester based microcomposites, a given wt. % of CB from 0 to 15 wt. % was added into the resin part of the polyester and mixed using a high shear mixer at the rpm of 3000 for 30 minutes. Then, the two-phase compound was mixed with the catalyst and hardener phases as prescribed by the maufacturer. The mixture of resin/hardener/catalyst and CB was then cast into cavities of silicon molds using a mold release agent following the respective standards as required for mechanical behavior characterization with the curing process at the ambient condition. Figure 1 represents the fabrication route used in this study from step 1 (mixing) to 6 (mold casting) as described in details earlier. The same fabricated samples were used for the Xray diffraction (XRD), scanning electron microscopy (SEM) and differential scanning calorimetry (DSC) analysis.

# 2. 3. Characterization of CB/Polyester Microcomposites

2. 3. 1. Mechanical Response of CB/Polyester composites Tensile response of the composites including the Young' modulus, tensile strength and strain at break of composites was determined per the ASTM D638 test method using a universal tensile testing machine (Sanaf Co., Iran). The testing was run using typeV specimens with 3.2 mm thickness with a gage length of 10 mm. Three tensile test specimens were used in the case of each composite system and at least average of two specimens were reported. Measurements were conducted at the deformation rate (stroke speed) of 2.54 mm/min at room temperature. The apparatus used applies tensile loading on the specimen fixed within the grippers measuring force (Newton) versus displacement (mm) until the specimens break. The Izod impact resistance of the specimens was measured per ASTM D256 to understand the effect of the addition of CB particles on the impact performance of the specimens. A homemade loadcell was utilized to conduct testing of the energy absorption of the composites based on the elastic stored energy concerning the initial potential energy of a dropped weight.

2. 3. 2. Thermal Analysis of Micro-composites The thermal properties of the composites including the  $T_g$  and calorimetric relaxation strength were characterized using DSC (Sanaf Co., Iran) utilizing a temperature ramp-up to 200 °C with the rate of 10 °C/min to determine the effect of the addition of fillers on the thermal response and the interfacial characteristics around the reinforcement phase. To provide the DSC test conditions, small material masses, ranging between 10 and 15 mg, were cut from each composite specimen and analyzed using aluminum pans.

2.3.3. Microstructural and Morphological Studies The SEM method was used to assess the morphology of the fractured surface of the fabricated composites including the dispersion quality and microstructural properties of the fracture surface of the samples. The surface of the CB reinforced composites was goldsputtered before the SEM analysis to ensure the absence of charging effects on the non-conductive fractured surface. The XRD technique was performed to examine the structure of CB reinforced composites and the dispersion quality of the CB within the polyester composites. The XRD analysis was performed using an XRD machine with a Cu K $\alpha$  radiation source ( $\lambda$ = 1.5406 Å) generating X-rays on the incident beam monochromator setting. The specimens were scanned in the  $2\theta$  range from 5° to 70° with a 0.05° step size. The Xray equipment was operated at 45 KV and 40 mA.

# **3. RESULTS AND DISCUSSION**

3. 1. Mechanical Response of CB/Polyester Micro-Figure 2 depicts the changes in the composites tensile Young's modulus and tensile strength of the composites as a function of the filler content as previously reported in our studies elsewhere. It can be understood from the results that generally the tensile strength of the parts decreases with the addition of fillers whilst the modulus increases up to an optimized value of the filler content (Figures 2a and 1b, respectively). It is hypothesized that the decrease in the strength might be attributed to the presence of a weaker (softer) interfacial CB/polymer zone so far called interphase and agglomerated regions that will be discussed in the current study. Other factors are thought to be contributing to the decreased tensile strength such as the lower possibility of crosslinking among the polyester monomers and, thus, lowered crosslink density [23]. Moreover, it is believed that the presence of defects including the bubble formed during the processing stage might be a key factor that unfavorably influences the tensile strength of the composites. The findings are in good agreement with results reported elsewhere [24]. Further studies were performed to better support the variations in the tensile response of the composites as described in next sections. The impact resistance of the CB/polyester composites with the addition of filler content is represented in Figure 3. It is realized that with an increase in the CB content, an overall initial improvement in the impact resistance of samples is obtained. The finding could be correlated to the disrupted degree of cross-links formed at the vicinity of the CB particles leading to the presence of weak interphase compared to the stiffness of the pure matrix away from the interfacial surface [25].



Figure 2. (a) Tensile strength and (b) modulus of the composites as a function of the CB filler content



Figure 3. Izod impact resistance of the CB/polyester vs. the filler content

The observed decrease in the impact resistance followed by addition of CB content is further correlated to the formation of competing effects. The inherent stiffness of CB particles normally is believed to decrease the impact behavior of composites. However, as described in the next sections, the generation of softer interphase as a result of poor interfacial interactions and altered degree of cross-links at the vicinity of the fillers contribute to the enhanced impact behavior of the fabricated composites. The findings were further supported by the measured energy absorption of the composites in the drop weight experiments. An overall enhancement in the absorbed energy is observed upon addition of the CB content.

The increase in toughness properties of the composites is related to the presence of softer interphase and the inherent properties of carbon-based materials in absorbing energy (Table 1) [26]. However, with further addition of the CB beyond 10 wt. % a drop in the toughness of the specimens was noticed. The observed finding could be ascribed to the extensive level of CB agglomeration as described earlier leading to the presence of stress concentration and shear slippage amongst CB fillers due to shortage of polymer chain and, thus, the decrease in wetting ability of polyester [27].

2. Glass Transition Temperature 3. and **Interphase Quality** The variations in the Tg of the CB/polyester composites are shown in Table 2. It is sown that the Tg values decrease with an increase in the CB wt. % followed by an increase when the filler content exceeds 10 wt.%. The presence of the weak interphase can be supported by the Tg decrease due mainly to the weaker interfacial interactions or disrupted cross-link density around the surface of the CB [28, 29]. An increase in Tg upon the addition of the greater amount of CB concentration can be correlated to the existence of lower specific surface to volume area of fillers exposed to the polymer matrix leaving a greater amount of polymer network intact due to agglomeration of fillers. It has been shown that the changes in the Tg of polymer composites are strongly related to the extent of the interfacial zone which in turn governs the length of the CRR [30, 31]. To understand the links in the range of the interphase that in the context of polymer composites filled with rigid particles could be correlated to the CRR, the thickness of the CRR was evaluated [3, 21]. It was further released that the changes in the Tg values could be associated with the changes in the crystalline phase as described in the next parts.

**3. 3. CRR Estimation as a Measure of Relaxation Strength** The changes in the calorimetric heat capacity of polymeric composites near Tg can be considered as a

TABLE 1. The absorbed energy of the CB reinforced polyester as a function of CB wt.%

Absorbed Energy (J)	Composite system CB wt.%
12.2	0
12.6	1
12.5	3
15	5
15.1	10
12.6	15

TABLE 2.  $T_g$ ,  $C_p$  and calorimetric relaxation strength of the CB/polyester v.s. CB wt.%

T <sub>g</sub> (°C	ΔT <sub>g</sub> (°C)	T <sub>g</sub> (°C) Liquid	T <sub>g</sub> (°C) Solid	$\frac{\Delta Cp}{(\frac{J}{g.°C})}$	Cp Final $(\frac{J}{g.\circ C})$	Cp Onset $\left(\frac{J}{g.\circ C}\right)$	CB wt. %
65.8	26.4	74.0	47.6	0.054	0.375	0.321	0
62.3	17.8	70.5	52.7	0.03	0.307	0.277	1
61.2	13.2	70.2	57.0	0.002	0.154	0.152	3
61.9	23.6	71.5	47.9	0.046	0.375	0.329	5
65.5	16.1	70.6	54.5	0.027	0.331	0.304	10
65.7	14.9	72.2	57.3	0.006	0.214	0.208	15

key factor in understanding the presence and quantity of the interphase. This effect arises from the fact that upon heating a polymeric specimen, the extent of chains mobility increases over the T<sub>g</sub> transition zone. This results in a transitional behavior of reinforced polymers from solid-like to liquid-like state where the C<sub>p</sub> of the specimens increases. The immobilized chains therefore exhibit greater C<sub>p</sub> values than those restricted at the interface. Table 2 further shows the C<sub>p</sub> values together with the calorimetric relaxation strength  $\Delta C_p$  observed with the addition of the interphase.

The opposite case is also true when the weak interfacial interactions or polymers with the lowered degree of crosslink accommodate a greater region of mobile chains that can easier slide over each other leading to a decrease in the Tg. It has been previously reported that in carbon-based reinforced thermosetting composites, the ratio of hardener/ resin is adversely altered resulting in a lower cross-link density at the interface of the filler/polymer. So far, it was already shown that an addition of CB decreased the Tg values that could be correlated with the extent of the generated CRR. The CRRs have been explained as the cooperative dynamics that predict the length of the immobilized region so far called the interphase and are highly correlated to the specific heat capacity (Cp) before and after  $T_{\rm g}$  as mentioned earlier. In the current study, a model first introduced by Donth [21] was used to estimate the CRRs length using the changes in the heat capacity from solid to liquid behavior of CB/polyester around the T<sub>g</sub> of the fabricated parts on the bases of the DSC thermograms obtained over the heating scan of the specimens (Equations (1) and (2)):

$$V_{\alpha} = \frac{\Delta\left(\frac{1}{c_{\nu}}\right)}{\rho(\delta T)^2} K_B T^2 \tag{1}$$

$$\delta = \sqrt[3]{V_{\alpha}} \tag{2}$$

where  $V_{\alpha}$  is the volume of the cooperative region,  $C_v$  is the specific heat capacity,  $\rho$  is the density of the specimen,  $K_B$  is the Boltzman constant,  $\delta$  is temperature fluctuation, and  $\delta$  is the characteristic length of the glass transition. Table 3 displays the changes in the nano-size CRR namely the representative of the interphase thickness with the addition of the CB content. It is illustrated that the CRR length increases at 1 wt. % of CB concerning that in the case of pure polymer. Nevertheless, the length of CRR starts decreasing upon by addition of 15 wt. % of fillers. As previously shown through the changes in Tg values, when the CB loading grows, the presence of agglomeration results in suppressed filler/polymer interaction as the effective CB/polyester interface decreases. The latter is clearly confirmed by the decrease in the CRR length (Table 3). The CRR predicted in the pure matrix is related to the presence of softer interphase or/and weaker bonding around the harder region regardless of the presence of any fillers. The latter can be related the loose packing perfection of the polymer chains under the unfavourable effect of the weak interactions. Further decrease followed by the addition CB wt. % is correlated to the presence of agglomeration contributing to lower surface area of fillers. As expected, with an addition of CB wt.% beyond 3 wt.%, the increased specific surface of the fillers overcomes the adverse effect of the agglomeration phase. The observed results are in good agreement with the changes in Tg confirming weaker CB/polymer adhesion and interfacial interaction against imperfect interfacial bonding or destroyed cross links.

It is noted that the lower CRR corresponds to an overall lower effect of fillers surface interaction from the interfacial polymer to regions away from this surface [32]. The results further confirm the improvement in the tensile modulus is associated with the inherent properties of CBs, which overcomes the effect of softer interphase than polyester [33]. In opposite, the enhanced absorbed energy as well as the impact resistance of the specimens upon an increase in the CB content could be thought of the growing amount of soft interphase as expected.

It should be noted that the picture of interfacial interactions in the case of thermosetting polymers is more complicated due to the presence of a threedimensional network of polymer chains from the high degree of cross-link. However, the changes in the density of cross-links due to the nano/microscale effect of the

**TABLE 3.** CRR thickness in nm vs. the CB wt.% inCB/polyester composites

CRR (δ) (nm)	<b>CB</b> wt. %
9.2	0
11.1	1
8.4	3
9.3	5
10.7	10
8.83	15

interfacial interactions should be taken into account as a key factor altering the cross-links density through the polymer bulk. Another important observation is that the use of thermosetting polymers should fall within the measurement of T<sub>g</sub> where the resolution and sensitivity of the thermal device could capture the changes in the C<sub>p</sub>. Moreover, whilst the presence of harder interphase may result in the decrease in impact resistance and toughness, this may on the opposite side lead to an enhanced tensile response of the specimens. Therefore, when the application of multi-functional composites comes into account, the optimized values of fillers fraction need to be considered when overall improvement in mechanical properties is desired. The interrelation amongst the structure, filler/polymer adhesion and the filler wt. % with the specific emphasis on the role of interfacial interactions is thus an elemental factor governing the overall properties of reinforced micro/nanocomposites.

**3. 4. XRD Microstructural Analysis** The XRD patterns of the CB reinforced composites are represented in Figure 4. The results indicate two main halos associated with the amorphous hill and the crystalline rigid phase. The first peaks appear around  $\sim 20^{\circ}$  and the second peaks are observed  $\sim 38^{\circ}$  to 41° as characteristic peaks of the polyester resin. The results, however, show that with the addition of CB into the polyester resin, the

second peaks disappear at the loading above 10 wt. % reflecting the decrease in the amorphas shoulder as supported with the  $T_g$  values observed at higher loadings of CB. The results further confirm there a slight change in the first characteristic angle associated with the first halo with respect that observed in the case of pure polyester [24, 34].

It is suggested that by the addition of filler into the polyester matrix, a rigid interphase forms around the fillers leading to the composites with greater solid-like behavior. The findings are in good agreement with the CRR and  $T_g$  values where at the higher CB content greater interactions are present at the interfacial surface of CB/polymer. As understood from the results reported, a little increase in 20 values of the first halo by the addition of filler concerning that in the case of pure polyester indicates lower d-spacing between the galleries of the crystalline phase showing a more compact (denser) structure of the composites according to the Bragg's law [35]. This finding may explain an increase in the elastic modulus of the parts as an underlying synergistic effect as well as the reinforcing function of the CB particles.

Figure 4 further indicates the characteristic peak of CB occurring  $\sim 29.5^{\circ}$  that is in good agreement with that reported elsewhere in the literature. The summary of the XRD structural analysis is reported in Table 4. It is



Figure 4. XRD diffraction traces of the CB/polyester composites as a function of the CB wt%

<b>TABLE 1.</b> THE Characteristic beaks, of standing beak intensity of the CD remoted betweeter composites against	TABLE 4. XRD characteristic	peaks, crystalline index and	peak intensity of the CB reinforced	polvester composites against
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Composite system	Polyester					СВ			
CB wt. %	20ºª First Halo	2θºª Second Halo	P.I <sup>b</sup> (a.u) First peak Amorph	P.I <sup>b</sup> (a.u) Second peak Amorph	2θº <sup>a</sup> Crystal peak	P.I <sup>b</sup> (a.u) Crystal peak	CI <sup>c</sup> (%)	20°a	P.I <sup>b</sup> (a.u)
0	20.4	38.3	290	40	20.5	493	59	-	-
1	21.3	41.6	223	26	20.6	395	61	29.4	124
10	20.9	41.0	32	32	21.2	74	53	29.2	955
15	20.9	39.7	87	30	20.9	221	65	29.1	680

<sup>a</sup> 2-theta (degree) <sup>b</sup> Peak intensity <sup>c</sup> Crystalline Index <sup>e</sup> Crystallite size

shown that when the CB filler is incorporated into the polyester, the peak intensity related to the first halo decreases followed by a second increase confirming the presence of the greater amount of rigid phase (so far linked to the crystalline phase). According to Table 4, it is understood that the peak intensity associated with the presence of CB fillers experience an initial increase compared with the neat polyester followed by the decrease in the peak intensity of CB reinforcement. The results are thought off a better dispersion state of the CB within the polyester matrix at the filler loading of 10 wt. %. The latter supports the increase in the CRR values when the greater surface area of the CB particles contributes to more significant immobilized polymer chains around the fillers due to stronger interfacial interactions. This further confirms the increase in Young's modulus, the absorbed energy quality and the impact resistance of the fabricated part at the filler loadings of 10 wt.%. However, the decrease in the absorbed energy of the specimens at the filler content above wt.% could be correlated with the presence of greater fractions of rigid phase resulting in a more brittle nature of the parts as expected. The crystalline index (CI%) of the composites as proposed by Segal et al. [36] as an empirical approach in the determination of the crystallinity is calculated by Equation 3 and reparented in Table 4.

$$CI\% = \frac{P.I_{crystal}}{P.I_{crystal} + P.I_{amorph}}$$
(3)

in which P.I<sub>amorph</sub> and P.I<sub>crystal</sub> are the peak intensities associated with the amorphous and crystalline phases, respectively. An overall increase is observed in the crystallinity of the fabricated composites with the addition of the fillers as explained earlier (Table 4). However, the decrease in the amount of the crystalline phase at the filler loading of 10 wt.% might be ascribed to the presence of the agglomeration phase or decreased degree of crosslinking density. It is noted that the level of interfacial interaction is also correlated to the number of crystalline rigid phases not the number of crystals as the latter acts mainly as the sites immobilizing the polymer chains movement [3, 37].

**3. 4. SEM Microstructural Study of CB/Polyester Composites** Figure 5 represents the SEM images of the fracture surface of pure polyester and composites reinforced with 15 wt.% of CB particles. Figure 5a indicates that there exist complex and greater crack marks on the fractured surface of the 15 wt. % CB reinforced specimen compared to that observed in the case of pure polyester parts. This finding supports the extended number of parallel crack marks when the CB loading increases as previously shown by the decrease in the impact resistance and absorbed energy quality of the composites filled with higher loadings of CB.



**Figure 5.** Representative SEM images of (a) pure polyester and (b) CB/polyester composite fracture surface filled with 15 wt.% of CB

The SEM images further indicates he presence of both the dispersed and agglomerated phase of CB particles that contributes to the observed CCR values as well as the mechanical response of the fabricated composites. However, minimal presence of agglomeration is represented confirming the extended values of the relaxation strength released by the thermal analysis. The SEM results further suggest multiple stress concentration sites leading to the observed river crack traces in the reinforced composites.

Although the fractured surface of the neat polyester exhibits a greater plastic deformation, as displayed by the roughness and undulation marks, which in turn shows the greater capability of the pure specimens in the energy absorption, the inherent properties of carbon-based materials provide the reinforced parts with improved toughness against the undesired local stress concentration zones within the composite bulk [34].

#### 4. CONCLUSIONS

To better understand the role of filler/polymer interactions and their correlation with mechanical properties including tensile behavior, impact resistance and energy absorption, composites of thermosetting polyester reinforced with 0 to 15 wt. % of CB were fabricated using shear mixing method followed by silicon mold casting of the CB/resin/hardener composites. The results indicated that the extent of interfacial interactions estimated to be ~10 nm highly governs the thermal and mechanical properties of the composites. It was revealed that the weaker strength of interaction as a result of suppressed cross-links leads to lower Tg values and calorimetric  $\Delta$ Cp contributing to a smaller length of nano-size CRR. The improved mechanical properties

including the impact resistance and energy-absorbing behavior were linked to the weaker interfacial bonding and, thus, the presence of softer interphase as the damping phase. The results further suggested the adverse effect of the agglomeration phase on the decrease in the tensile strength and the lower amount of generated interphase at higher fillers ratio up to 10 wt.% of CBs as shown by the changes in the Tg values of the composites.

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#### Persian Abstract

# چکیدہ

در کامپوزیت های تقویت شده میکرو/نانومواد، بر همکنش های سطحی در سطح مشترک پرکننده/پلیمر منجر به تشکیل لایه سومی به نام فازمیانی به عنوان مکانیزم تقویت کننده ثانویه می شود. فاز میانی ممکن است بهدلیل لایه نشینی سطحی موضعی زنجیره های پلیمری در سطح مشترک، و به هم پیوستگی مکانیکی و انتشار میان زنجیره های پلیمری تشکیل شود. از آنجایی که برهمکنش بر انتقال بار در رابط پرکننده/پلیمر حاکم هستند، نقش کلیدی در پاسخ مکانیکی کامپوزیت های تقویت شده دارند. با این حال، تنها مطالعه به خوبی پایه گذاری شده و معتبر در توصیف برهمکنش های سطحی ارائه شده در رابطه با کامپوزیت های ترموست وجود دارد. هدف این تحقیق درک ارتباط بین خواص مکانیکی کامپوزیتهای پلیاستر ترموست تقویت شده با ۲ ا ۱۵ درصد وزنی کربن سیاه با تمرکز بر منطقه بازآرایی مقید با اندازه نانو (CRR) است. برای تخمین طول خواص مکانیکی کامپوزیتهای پلیاستر ترموست تقویت شده با ۲ تا ۱۵ درصد وزنی کربن سیاه با تمرکز بر منطقه بازآرایی مقید با اندازه نانو (CRR) است. برای تخمین طول اندازه گیری شد. یک RRT با اندازه ۱۰ نانومتر به طور متوسط تخمین زده شد که با تاثیر اثر افزایشی در رفتال شیهای با استفاده از یک مدل ترمودینامیکی اندازه گیری شد. یک RRT با اندازه ۱۰ نانومتر به طور متوسط تخمین زده شد که با تاثیر اثر افزایشی در رفتان را می پذیری و چقرمگی نمونه ها رتبط بود. نتایچ حاکی از وجود می کند. روش معرفی شده در این مطالعه را می توان در تبیین تغییرات خواص مکانیکی و فیزیکی کامپوزیت های متقویت شده با تمرکز بر نقش اساسی برهمکنش ها در اندازه می کند. روش معرفی شده در این مطالعه را می توان در تبیین تغییرات خواص مکانیکی و فیزیکی کامپوزیت های تقویت شده با تمرکز بر نقش اساسی برهمکنش ها در اندازه دانو استفاده کرد.