ORIGINAL CONTRIBUTION



# The reinforcing and characteristics of interphase as the polymer chains adsorbed on the nanoparticles in polymer nanocomposites

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Abstract In this study, the adsorbed polymer chains on the nanoparticle surface are assumed as an interphase layer, which increases the reinforcing efficiency of nanoparticles in polymer nanocomposites. The interphase is taken into account to enhance the effective volume fraction of nanoparticles, which is determined by the Maxwell model for Young's modulus of composites. Also, the modulus and strength of interphase are calculated by proper models. The interphase properties are estimated and discussed for various samples containing different nanoparticles. The present methodology gives fine agreement between the experimental results of mechanical properties and the calculations. Also, the acceptable results for interphase properties are obtained which confirm the validity of the presented models. The small nanoparticles and thick interphase significantly increase the nanocomposite reinforcement. Additionally, Young's modulus of nanoparticles causes negligible effect on the modulus of nanocomposites in spite of the interphase properties such as thickness and interfacial area.

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## Introduction

Today, polymer nanocomposites have involved much interest in academic and industrial communities, due to the unexpected properties compared to neat polymers and conventional composites. Only adding 2–5 wt% of nano-platelets like nanoclay or graphene, nano-spheres such as silica (SiO<sub>2</sub>) or nanotubes such as carbon nanotube (CNT) to a polymer matrix produces significant improvements in mechanical, chemical, thermal, optical, and electrical properties [1–7]. Additionally, some advantages such as low weight, easy fabrication, and low cost recommend the application of nanocomposites in different fields. However, the reinforcing effects of nanoparticles depend on different parameters such as the nature of matrix and nanoparticles, dispersion quality of nanoparticles, and the interfacial/interphase properties such as area and interaction/adhesion [8–12].

Recent researches have revealed the effects of both enthalpic and entropic interactions on the dispersion/distribution of nanoparticles which control the performance of nanocomposites [13]. The thermodynamically stable dispersion of nanoparticles improves when the gyration radius of linear polymer is higher than the nanoparticle radius [14]. However, the dispersion of layered nanoparticles is controlled by surface modification and processing conditions. Also, an efficient stress transfer from the polymer matrix to nanoparticles depends on the interfacial adhesion. The conformation and viscoelasticity of the polymer chains adsorbed on the nanoparticles are unlike from others. The confined polymer segments exhibit different relaxation behavior, which can be directly revealed by the glass transition temperature ( $T_g$ ) of polymer. For example, the  $T_g$  of poly(methyl methacrylate) (PMMA) is increased by approximately 30 °C with addition of 0.05 wt% of graphene [15]. This occurrence has been also reported for other nanocomposites [15].

However, the conventional models for micro-composites cannot answer the requirements for polymer nanocomposites, because they cannot assume the size effect of nanoparticles, high interfacial area, and the strong interfacial interaction, while these factors significantly manage the performance of nanocomposites. Such properties attributed to nanoparticles cause a difference between experimental data and predictions of conventional models in polymer nanocomposites.

In this regard, many authors have considered the formation of an interphase with considerable thickness and strength in polymer nanocomposites [12, 16–18]. Also, many new or developed models were suggested in the literature to give a simple technique to determine the properties of interphase in binary and ternary polymer nanocomposites [19–21]. However, the researches continue and the challenges remain to calculate the exact and valid characteristics of interphase by very simple and correct models. Understanding and modeling the properties of polymer nanocomposites have attracted much attention in recent years, due to the guiding potential for development of high-quality materials for industrial requests.

In the present work, the interphase characteristics in polymer nanocomposites are measured by the experimental data of mechanical properties. The interphase is assumed as the adsorbed polymer chains on the nanoparticle surface which increase the effective volume fraction of nanoparticles. The thickness of interphase is determined assuming the effective volume fraction of nanoparticles in the Maxwell model for Young's modulus. Also, the modulus and strength of interphase are calculated by Ji and Pukanszky models. The interphase properties are measured and evaluated for various samples filling with different nanoparticles such as graphene oxide, nanoclay, CNT, and SiO<sub>2</sub>.

#### **Modeling approaches**

It was suggested that the layer of adsorbed polymer chains on the exfoliated/dispersed nanoparticle surface as interphase has a thickness which is smaller than the gyration radius of polymer macromolecules ( $R_g$ ) [22]. In this regard, the volume fraction of nanoparticles ( $\varphi_f$ ) is increased giving an effective level as:

$$\varphi_{eff} = \varphi_f (1 + t_i A_c \rho_f) \tag{1}$$

where " $t_i$ " is the thickness of interphase and " $A_c$ " and " $\rho_f$ " are the specific surface area and density of nanofiller, respectively.

So, the reinforcement inclusions consist of inorganic nanoparticles and the adsorbed polymer chains. " $\varphi_{eff}$ " can be presented as:

$$\varphi_{eff} = \varphi_f (1+Y) \tag{2}$$

where "Y" is defined as an interphase parameter by:

$$Y = t_{\rm i} A_{\rm c} \rho_{\rm f} \tag{3}$$

The value of "Y" can be estimated from the models for mechanical properties of composites such as Young's modulus.

For a composite containing dispersed particle, Young's modulus can be calculated by the Maxwell model [23] as:

$$E = E_{\rm m} \left[ \frac{1 + 2\varphi_f(a-1) / (a+2)}{1 - \varphi_f(a-1) / (a+2)} \right]$$
(4)

$$a = E_{\rm f} / E_{\rm m} \tag{5}$$

where " $E_{\rm m}$ " and " $E_{\rm f}$ " are the Young's modulus of matrix and filler, respectively. However, this model underpredicts the modulus in different polymer nanocomposites, due to the disregarding of interphase (it will be shown in the next section). The value of "Y" and thus, the thickness of interphase can be measured by assuming the interphase in the Maxwell model using the " $\varphi_{eff}$ ." To calculate the " $t_i$ " by "Y" value, " $A_c$ " must be determined. " $A_c$ " can be expressed for nanocomposites containing plate-like particles such as nanoclay and graphene with "t" thickness and both length and width of "l" (l > t) as:

$$A_{\rm c} = \frac{A}{m} = \frac{A}{\rho_{\rm f} V} = \frac{2l^2 + 4lt}{\rho_{\rm f} l^2 t} = \frac{2}{\rho_{\rm f} t} + \frac{4}{\rho_{\rm f} l} \cong \frac{2}{\rho_{\rm f} t}$$
(6)

where "A", "m," and "V" are surface area, mass, and volume of nanoparticles, respectively. Also, the specific surface area for cylindrical (fibrous) particles can be given by:

$$A_{\rm c} = \frac{A}{m} = \frac{2\pi Rl}{\rho_{\rm f} V} = \frac{2\pi Rl}{\rho_{\rm f} \pi R^2 l} = \frac{2}{\rho_{\rm f} R}$$
(7)

where "R" is the radius of nanoparticles. Similarly, " $A_c$ " can be defined for nanocomposites comprising spherical particles as:

$$A_{\rm c} = \frac{A}{m} = \frac{A}{\rho_{\rm f} V} = \frac{4\pi R^2}{\rho_{\rm f} \frac{4}{3}\pi R^3} = \frac{3}{\rho_{\rm f} R}$$
(8)

By replacing the " $A_c$ " from above equations into Eq. 3, the "Y" parameters for nanocomposites consisting of layered (1), cylindrical (2), and spherical (3) nanoparticles are expressed as:

$$Y_1 = \frac{2t_i}{t} \tag{9}$$

$$Y_2 = \frac{2t_i}{R} \tag{10}$$

$$Y_3 = \frac{3t_i}{R} \tag{11}$$

which demonstrate that the "*Y*" parameters only depend on the interphase thickness and particle size. Accordingly, it is possible to calculate the " $t_i$ " by assuming the interphase in nanocomposites through " $\varphi_{eff}$ ."

Ji et al. [24] also proposed a developed model taking into account the matrix, nanofiller, and interphase for Young's modulus of nanocomposites. The Ji model for nanocomposites containing different nanoparticles is expressed as:

$$E = E_{\rm m} \left[ (1-\alpha) + \frac{\alpha - \beta}{(1-\alpha) + \frac{\alpha(k-1)}{\ln(k)}} + \frac{\beta}{(1-\alpha) + \frac{(\alpha - \beta)(k+1)}{2} + \beta \frac{E_{\rm f}}{E_{\rm m}}} \right]^{-1}$$
(12)

$$\alpha_1 = \sqrt{\left(2\frac{t_i}{t} + 1\right)\varphi_f} \tag{13}$$

$$\alpha_2 = \sqrt{\left(\frac{t_i}{R} + 1\right)^2 \varphi_f} \tag{14}$$

$$\alpha_3 = \sqrt{\left(\frac{t_i}{R} + 1\right)^3 \varphi_f} \tag{15}$$

$$\beta = \sqrt{\varphi_f} \tag{16}$$

$$k = \frac{E_{\rm i}}{E_{\rm m}} \tag{17}$$

where " $E_i$ " is the modulus of interphase. This model has been successfully applied in different studies on the polymer nano-composites [24].

Pukanszky [25] also suggested a model to define the composition dependence of tensile strength in composites as:

$$\sigma_{\rm R} = \frac{1 - \varphi_f}{1 + 2.5\varphi_f} \exp\left(B\varphi_f\right) \tag{18}$$

where " $\sigma_R$ " is relative strength as  $\sigma_c/\sigma_m$ , where " $\sigma_c$ " and " $\sigma_m$ " are the strength of composite and matrix, respectively. Also, a "*B*" parameter which displays the quantitative level of polymer-filler interfacial adhesion is expressed as:

$$B = (1 + A_{\rm c}\rho_{\rm f}t_{\rm i})\ln\left(\frac{\sigma_{\rm i}}{\sigma_{\rm m}}\right) \tag{19}$$

where " $\sigma_i$ " is the strength of interphase. The Pukanszky model has suggested many acceptable fittings with the experimental data of dissimilar nanocomposites in the literature [26, 27]. The Pukanszky model can be restructured to:

$$\ln(\sigma_{Reduced}) = \ln\left(\sigma_{R} \frac{1 + 2.5\varphi_{f}}{1 - \varphi_{f}}\right) = B\varphi_{f}$$
(20)

which can present "*B*" from a linear connection between the experimental results of  $\ln(\sigma_{\text{Reduced}})$  and " $\varphi_{f}$ ." Substituting of "*A*<sub>c</sub>" from Eqs. 9–11 into Eq. 19 results in the following equations for "*B*" as:

$$B_1 = \left(1 + 2\frac{t_i}{t}\right) \ln\left(\frac{\sigma_i}{\sigma_m}\right) \tag{21}$$

$$B_2 = \left(1 + 2\frac{t_i}{R}\right) \ln\left(\frac{\sigma_i}{\sigma_m}\right) \tag{22}$$

$$B_3 = \left(1 + 3\frac{t_i}{R}\right) \ln\left(\frac{\sigma_i}{\sigma_m}\right) \tag{23}$$

which can calculate the " $\sigma_i$ " by the values of "B", "R," or "t", " $\sigma_m$ ," and " $t_i$ ."

### **Results and discussion**

Figure 1 illustrates the calculations of modulus by the Maxwell model (Y = 0) for four samples from valid literature. It is observed that this model underpredicts the modulus in all reported samples. Therefore, the micromechanical models for composites without considering the interphase are insufficient to describe the mechanical properties of nanocomposites. It can be concluded that the interphase including the polymer chains adsorbed on the nanoparticles plays a main role in the reinforcement of nanocomposites.

Figure 2 shows a good agreement between the experimental data and the predictions of the Maxwell model assuming a proper level for the "Y" interphase parameter in " $\varphi_{eff}$ ." This evidence demonstrates that the assumption of interphase between polymer and nanoparticles is necessary for calculation of modulus in nanocomposites, i.e., the interphase additionally reinforces the nanocomposites beside the nanoparticles. The best fitting of the model is commonly obtained at low nanofiller concentrations, because high filler contents cause many aggregations/agglomerations, which reduce the surface area of nanoparticles and weaken the modulus. As a result, the suggested model is suitable for low nanofiller contents.

Figure 3 illustrates the effects of " $E_f$ " and "Y" parameter on Young's modulus of nanocomposites by the developed Maxwell model (assuming " $\varphi_{eff}$ ") at  $\varphi_f = 0.03$  and  $E_m = 2$  GPa. The " $E_f$ " causes an insignificant effect on the modulus, but the modulus is considerably attributed to the level of "Y." As a result, the modulus significantly depends on the properties of interphase such as thickness and surface area rather than the nanoparticles and matrix properties.

However, it was reported that some undesirable phenomena such as aggregation/agglomeration of nanoparticles and poor compatibility between the components deteriorate the interphase properties and introduce a low modulus for nanocomposites [28–31]. Therefore, the material and processing



Fig. 1 The predictions of the Maxwell model for a PLLA/GO, b PBT/o-clay, c epoxy/MWCNT, and d PP/CaCO<sub>3</sub> samples

parameters should be tuned to induce a good interphase in polymer nanocomposites, which generally improves the mechanical properties. Using some techniques including application of a compatibilizer or coupling agent as well as treatment of nanofiller may produce high interfacial adhesion/ interaction at polymer-nanoparticle interface which promote the dispersion of nanoparticles and interfacial adhesion in nanocomposites [32, 33]. The studied samples as well as their calculated properties are expressed in Table 1. The characteristics of the samples are derived from the original references. The values of "Y" which show the best prediction for the modulus of the samples are also presented in Table 1. As illustrated, different levels of "Y" are calculated by the Maxwell model demonstrating the various interphase properties in the reported samples. The "Y" depends on the characteristics of interphase such as " $t_i$ " and " $A_c$ " (see Eq. 3). Obviously, the poor compatibility between the polymer and nanoparticles and the weak dispersion or aggregation/ agglomeration of nanoparticles leads to the low level of interphase properties which cause a low "Y."

The best "*Y*" is obtained for a poly(L-lactic acid) (PLLA)/ GO sample as 12, while the smallest one is observed for polypropylene (PP)/SiO<sub>2</sub> and PP/CaCO<sub>3</sub> samples as 3. It indicates that a good interphase, which includes a large interfacial area and strong interaction/adhesion at interface, is formed in a PLLA/GO sample. But, a poor interphase with low extents of interfacial adhesion/interaction can be found in  $PP/SiO_2$ and  $PP/CaCO_3$  samples. The evidence and information in the original work of the studied samples confirm these results by morphological images and mechanical properties.

Figure 4 displays the dependence of the "Y" parameter on nanoparticle and interphase size for nanocomposites containing cylindrical (fibrous) inclusions (Eq. 10). A very low level of "Y" as about zero is calculated by large nanoparticles (R>20 nm) and thin interphase ( $t_i < 12$  nm). However, "Y" increases by decreasing the size of nanoparticles and formation of a thick interphase. The highest level of "Y" is obtained as nine at R = 5 nm and  $t_i = 25$  nm. Moreover, both levels of "R" and " $t_i$ " must be passable to achieve a high "Y", i.e., a small "R" and a high " $t_i$ " should be simultaneously obtained to provide a great "Y." As a result, incorporating small nanoparticles in the polymer matrix and providing a strong interaction between polymer chains and nanoparticles to introduce high "t<sub>i</sub>" suggest a high level of "Y" and thus, considerable Young's modulus in nanocomposites. Nevertheless, the nanoparticles commonly aggregate/agglomerate in the polymer matrix, due



Fig. 2 The predictions of the developed Maxwell model for a PLLA/GO, b PBT/o-clay, c epoxy/MWCNT, and d PP/CaCO<sub>3</sub> samples

to high surface energy which increases the "*R*." Also, less interfacial interaction between the constituents results in fewer number of adsorbed chains on the nanoparticle surface which produce a thin interphase. As indicated, some compatibilizing techniques which mean the modification of surface chemistry of nanoparticles can enhance the interfacial/interphase properties [38–40]. From the values of "Y" and the thickness or radius of nanoparticles, it is possible to calculate the values of " $t_i$ " in the samples. Table 1 shows the values of " $t_i$ ," where the thickest and the thinnest interphase are observed in PLLA/GO



Fig. 3 a 3D and b contour plots for dependence of modulus on " $E_f$ " and "Y" by the developed Maxwell model at  $\varphi_f = 0.03$  and  $E_m = 2$  GPa

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Samples [ref.]	E <sub>m</sub> (GPa)	$\sigma_{\rm m}$ (MPa)	$E_{\rm f}({ m GPa})$	<i>R</i> , <i>t</i> (nm)	Y	<i>t</i> <sub>i</sub> (nm)	E <sub>i</sub> (GPa)	В	$\sigma_{\rm i}$ (MPa)
PLLA <sup>a</sup> /GO <sup>b</sup> [22]	1.56	41.4	1000	7.7	12	46.2	62.4	17.02	153.3
PBT <sup>c</sup> /o-clay <sup>d</sup> [34]	2.14	55.2	178	2	5.5	5.5	85	4.83	116.1
epoxy/MWCNT <sup>e</sup> [35]	1.9	45	1000	15	5.5	41.25	11	7.66	146.2
PP <sup>f</sup> /SiO <sub>2</sub> [36]	0.95	35.5	80	7.5	3	7.5	3.4	5.85	153.2
PP/CaCO <sub>3</sub> [37]	1.75	31.8	26	22	3	22	4.4	2.5	59.4

 Table 1
 The samples and the calculated properties

<sup>a</sup> Poly(L-lactic acid)

<sup>b</sup> Graphene oxide

<sup>c</sup> Poly(butylene terephthalate)

<sup>d</sup> Organoclay (Cloisite 30B)

<sup>e</sup> Multiwall carbon nanotubes

<sup>f</sup> Polypropylene

and poly(butylene terephthalate) (PBT)/o-clay samples, respectively. The values of " $t_i$ " should be smaller than the " $R_g$ " in nanocomposites which is observed in Table 1 for all " $t_i$ " levels, because " $R_g$ " commonly reaches to around 40–50 nm.

Applying the calculated values of " $t_i$ " and the characteristics of the samples, the " $E_i$ " level can be calculated by the Ji model for all samples. Figure 5 depicts the calculations of the Ji model and the experimental data for PBT/o-clay and epoxy/ MWCNT samples. As observed, the Ji model can suggest correct values for Young's modulus of samples comparable with the experimental data.

The calculated " $E_i$ " values by applying the experimental data to the Ji model are shown in Table 1. The " $E_i$ " varies from 3.4 to 85 GPa for the reported samples which point to the different properties of interphase in the samples. As known, " $E_i$ " should be obtained between " $E_m$ " and " $E_f$ " levels in nanocomposites. All the results pass this condition, which confirms the validity of the suggested approach for interphase properties in different nanocomposites. Additionally, a high

level of " $E_i$ " is achieved in the samples, which show a great "Y" value. As a result, the "Y" parameter indicates the general properties of interphase such as thickness, modulus, strength, and volume fraction. Also, a large "Y" reveals the significant reinforcement of nanocomposite by interphase.

The experimental tensile strength of the samples is also utilized into the Pukanszky model (Eq. 20) to present the interphase strength by the material and interphase parameters. Figure 6 exhibits the comparison between the experimental and the theoretical data of ln ( $\sigma_{\text{Reduced}}$ ) for PBT/o-clay and PP/SiO<sub>2</sub> samples. It is observed that a straight line with a "B" slope accurately follows the experimental data of ln( $\sigma_{\text{Reduced}}$ ) which expresses that the Pukanszky model is valid for the reported samples.

The calculated values of the "B" interfacial parameter are presented in Table 1. The "B" is calculated from 2.5 to 17.02, which shows the different levels of interface/interphase properties in the samples. The literature reports commonly reported the "B" from negative levels to about 20 for different nanocomposites [26, 27]. The values of "B" are also comparable



Fig. 4 "Y" as a function of "R" and "t<sub>i</sub>" according to Eq. 10 for cylindrical (fibrous) nanofillers: a 3D and b contour plots



Fig. 5 The predictions of the Ji model by suitable interphase properties for a PBT/o-clay and b epoxy/MWCNT samples

with the "Y" parameter, because both these parameters show the interface/interphase properties. As shown in Table 1, the high levels of "B" and "Y" are obtained for the PLLA/GO sample, whereas the PP/CaCO<sub>3</sub> represents the small values of "B" and "Y." According to Eqs. 21–23, the values of " $\sigma_i$ " can be calculated by "B", " $t_i$ ," and material properties. The calculated results of " $\sigma_i$ " are depicted in Table 1 for all studied samples. The maximum and minimum values of " $\sigma_i$ " are calculated for PLLA/GO and PP/CaCO<sub>3</sub> samples, respectively. These results are expected based on the calculated values of "B" and "Y" parameters for these samples. The " $\sigma_i$ " values in nanocomposites should be smaller than " $\sigma_f$ " and higher than " $\sigma_m$ ." This condition is satisfied by the suggested model demonstrating its validity for prediction of interphase properties.

To confirm the effect of aggregation/agglomeration of nanoparticles on the predictions, the morphological and mechanical properties of the  $PP/SiO_2$  sample containing 1 wt% poly(propylene-g-maleic anhydride) copolymer from [41] are studied. Figure 7 depicts the transmission electron microscopy (TEM) images of samples at different filler percentages. Large agglomerates of  $SiO_2$  are shown in a PP matrix, while the diameter of neat  $SiO_2$  nanoparticles was reported as 12 nm. In addition, the size of agglomerates increases by filler concentration confirming the mentioned remarks.

Figure 8 also shows the experimental data of the modulus and the predictions of the Ji model at  $t_i = E_i = 0$ . It is found that the predictions cannot agree with the experimental results even in absence of interphase regions. In other words, the Ji model overpredicts the modulus in this nanocomposite, because the aggregation/agglomeration of nanoparticles significantly deteriorates the interfacial area between polymer and nanoparticles, which removes the interphase regions and weakens the modulus. As a result, the Ji model can properly show that the aggregation/agglomeration of nanoparticles significantly decreases the interphase properties and tensile modulus. Moreover, a greater deviation between experimental results and predictions is observed at high filler percentages revealing the strong extent of agglomeration in this condition.



Fig. 6 The fitting of the Pukanszky model (Eq. 20) to experimental data of ln ( $\sigma_{\text{Reduced}}$ ) for a PBT/o-clay and b PP/SiO<sub>2</sub>



Fig. 7 TEM images of PP/SiO<sub>2</sub> sample containing 1 wt% poly(propylene-g-maleic anhydride) copolymer [41] at different filler concentrations

## Conclusions

The adsorbed polymer chains on the nanoparticle surface as interphase were assumed by the "*Y*" parameter which enhances



**Fig. 8** The experimental results and the predictions of modulus by the Ji model at  $t_i = E_i = 0$  for PP/SiO<sub>2</sub> sample containing 1 wt% poly(propylene-g-maleic anhydride) copolymer from [41]

the effective volume fraction of nanoparticles. Also, the different models such as Maxwell, Ji, and Pukanszky for mechanical properties were applied to determine the interphase characteristics. Additionally, the interphase properties were measured for various samples and the results were discussed.

All the models revealed a good agreement between the experimental data and the predictions. Also, much acceptable results for interphase properties were obtained which prove the validity of the suggested methodology for different nanocomposites. It was concluded that the interphase plays a main role in the reinforcement of nanocomposites and disregarding it suggests incorrect predictions for reinforcement. Moreover, different levels of interphase properties were obtained exhibiting the dissimilar levels of interfacial area/adhesion/ interaction in the studied samples.

The small nanoparticles and thick interphase significantly improved the nanocomposite modulus. However, the poor compatibility between the polymer and nanoparticles and the weak dispersion or aggregation/agglomeration of nanoparticles induces a poor interphase (low "Y") which weakens the nanocomposite. Also, it was found that the interphase properties cause significant effect on Young's modulus of nanocomposites, while the role of nanoparticles modulus is negligible. As a result, it is important to pay attention to the interphase role in polymer nanocomposites to obtain a high reinforcement in polymer nanocomposites by nanoparticles.

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**Conflicts of interest** The authors declare that they have no conflict of interest.

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