

## Waste PET and nano-silica effect on the dynamic-mechanical properties of polyester resin-based composites.

### Efecto del PET de desecho y la nano-sílice en las propiedades dinámico-mecánicas de los compuestos a base de resina poliéster.

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#### Abstract

The increasing production of plastic waste demands the implementation of strategies for its reuse, to reduce its accumulation, and mitigate its environmental impact. In this context, a promising strategy is the use of this waste as reinforcement in composite materials, which offers both economic and performance benefits. Moreover, in recent years, the use of nano-reinforcements has gained notoriety because their high surface area allows them to greatly enhance the performance of polymers. For this reason, this work evaluates the effect of adding waste PET (flakes and fibers), and nano-silica (with sizes of 7 and 200 nm) on the dynamic-mechanical properties of polyester resin-based composites. The results showed that short PET fibers produced a greater increase in the glass transition temperature than the long fibers, which was attributed to a superior interfacial interaction with the matrix compared to long fibers or PET flakes. Meanwhile, nano-silica also increased the polyester resin glass transition temperature, an effect associated with the restricted mobility of the polymer chains imposed by the nanoparticles.

*Keywords: Polyester resin, nano-silica, waste PET fibers, waste PET flakes, dynamic-mechanical properties.*

#### Resumen

La creciente generación de residuos plásticos exige la implementación de estrategias para su reutilización, con el fin de reducir su acumulación y mitigar su impacto ambiental. En este contexto, una estrategia prometedora es el uso de dichos residuos como refuerzos en materiales compuestos. Por otro lado, en los últimos años ha ganado relevancia el empleo de refuerzos nanométricos debido a que gracias a su alta área superficial pueden mejorar en gran medida las propiedades de los polímeros. Por ello, este trabajo evalúa el efecto de la adición de PET de desecho (en hojuela y fibra) y de nano-sílice (de 7 y 200 nm) en las propiedades dinámico-mecánicas de la resina poliéster. Los resultados mostraron que las fibras cortas de PET provocaron un mayor incremento en la transición vítrea que las fibras largas, lo que se atribuye a una interacción interfacial superior con la matriz en comparación con las fibras largas o las hojuelas de PET. Por su parte, la nano-sílice también incrementó la temperatura de transición vítrea de la resina poliéster, un efecto asociado a la restricción en la movilidad de las cadenas poliméricas impuesta por las nanopartículas.

*Palabras claves: Resina poliéster, nano-sílice, fibras de PET de desecho, hojuelas de PET de desecho, propiedades dinámico-mecánicas.*

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## 1. Introduction

Unsaturated polyester resins (UPR) are synthetic polymers created by the polycondensation reaction of propylene glycol with phthalic and maleic anhydrides. These resins are valued for their versatility, cost-effectiveness, and good mechanical performance, making them useful in composites, construction, consumer products, and automotive applications (Manikumar, 2024; Kirshanov, 2022; Patel, 2023). However, they are relatively brittle and have limited impact resistance. Hence, when used in more demanding applications, their mechanical and thermal performance is modified by adding reinforcements, such as glass fibres, mineral fillers, and carbon fibres (Yadav, 2024).

In this context, nano-reinforcements, such as nano-silica, stand out due to their ability to significantly improve the mechanical, thermal, dielectric, and rheological properties of polyester resin (Ali, 2024; Chu, 2025; Gañán, 2023; Sapuan, 2020).

In composites made with UPR and nano-silica (10 nm) extracted from sugarcane bagasse ash, the addition of 0.5 wt.% nano-silica produced the best results, achieving a 30.45% increase in tensile strength (up to 50 MPa), a 33.9% increase in flexural strength (up to 3.0 MPa), a 17.8 % increase in compressive strength (up to 141.4 MPa), and a slight improvement of 10 % for impact strength, compared with neat UPR. This enhancement in mechanical properties was attributed to the presence of a favourable interface between polyester resin and nano-silica, the densification of the composites, and crack path deflection, along with crack bridging toughening mechanisms that occurred because of the addition of nano-silica (Engidaw, 2024). Furthermore, Kaka et al. reinforced polyester resin with 1%, 5%, 10%, and 20% nano-silica (50-100 nm) by weight. Experimentally, the addition of 5% nano-silica caused a 64% increase in tensile strength (up to 11.5 MPa) and a decrease of tensile strain of 27% compared to neat UPR; on the other hand, when adding 10 % nano-silica, the tensile strength increased by 104%, while the tensile strain decreased by about 98%, showing the composite became more fragile. The best performance in composites with 5 % nano-silica was due to the good dispersion of the nanoparticles, which permitted an effective load transfer between the matrix and the reinforcement, giving high strength with moderate loss of ductility to the composites; when increasing the nano-silica content to 10%, the excessive nanoparticle content limits chain mobility and creates stress concentration, causing a very high strength with a severe loss of strain (Kaka, 2021).

Moreover, the amount of plastic waste is increasing (more than 150 million tonnes are produced each year), and most of it ends up in landfills or pollutes ecosystems. This continuous growth poses a severe environmental and health concern. Furthermore, microplastics-related issues reported in recent years exacerbate the severity of this problem (Haba, 2025; Nuruzzaman, 2025; Ochigüe, 2025). To address this crisis, some countries have introduced regulations to limit the use of single-use plastics. Additionally, mechanical, chemical, and thermal recycling processes have been adopted and promoted worldwide. Nevertheless, the need for further research to

identify alternative approaches for effectively resolving this matter remains (Başalp, 2020; Khalid, 2021; Ochigüe, 2025).

In that framework, the employment of plastic waste as reinforcement or filler in composites is a viable and increasingly researched approach that can assist in the reintegration of the waste into the production chain, as well as the mitigation of the environmental issues it generates. This approach offers economic and material performance benefits in a variety of sectors. (Jiang, 2024; Nuruzzaman, 2025; Yusuf, 2024). Speaking of UPR (Unsaturated Polyester Resin), waste and recycled PET stand out among other plastic wastes usable for its reinforcement. This is because both the polyester resin and polyethylene terephthalate share an ester chemical structure, leading to an expected good interfacial interaction between them (Dehas, 2016). Haseebuddin et al. evaluated the effect of adding waste PET flakes (with approximate dimensions of 4x6x0.7 mm) as reinforcement in UPR. The optimal concentration of reinforcement varied depending on the desired mechanical property. A concentration of 10 wt. % PET flakes produced a maximum tensile strength of 10.5 MPa, while 5 wt.% PET flakes yielded the highest compressive strength at 74.1 MPa. Furthermore, the highest shore D hardness (90) was achieved with 15 wt.% PET flakes. Nevertheless, the compressive and tensile strength decreased as the PET flakes content increased over 10%. This reduction was due to poor interfacial bonding between the matrix and the reinforcement (Haseebuddin, 2021)

However, the impact of waste PET aggregates and nano-silica on the rheological and thermal behaviour of polyester resin, however, has not received much attention; for this reason, this study addresses the subject.

## 2. Materials and methods

### 2.1. Materials

Aged orthophthalic polyester resin (donated by Concretos Poliméricos S.A. de C.V., Mexico) was employed as the matrix of the composites studied. Reinforcements consisted of short waste PET fibers (with a length ranging from 5 to 8 mm and an average diameter of 10  $\mu$ m), long waste PET fibers (with a length of 15 mm and an average diameter of 10  $\mu$ m), waste PET flakes manually cut from bottles for carbonated drinks (with an approximate size of 3x2x0.7 mm), as well as nano-silica particles supplied by Sigma Aldrich with an average particle size of 7nm and 200 nm (Code S5505, specific surface 175-225 m<sup>2</sup>/g, and pH of 3.7-4.3). Moreover, the curing of these composites was initiated by adding 1.2 wt.% of methyl ethyl ketone peroxide (MEKP).

### 2.2. Sample preparation and compositions

Five kinds of specimens were prepared for their testing with Dynamical Mechanical Analysis. Their compositions are listed in Table 1.

Table 1: Weight percentages used for the reinforcement of the test samples used for dynamic mechanical analysis

The preparation procedure for the samples is shown in Figure 1.

It should be noted that while PET flakes were utilized at weight percentages of 1 and 6, the concentration of PET fibers was limited to 5 wt.% because mixtures with higher loading percentages were too voluminous to fit the mold properly.

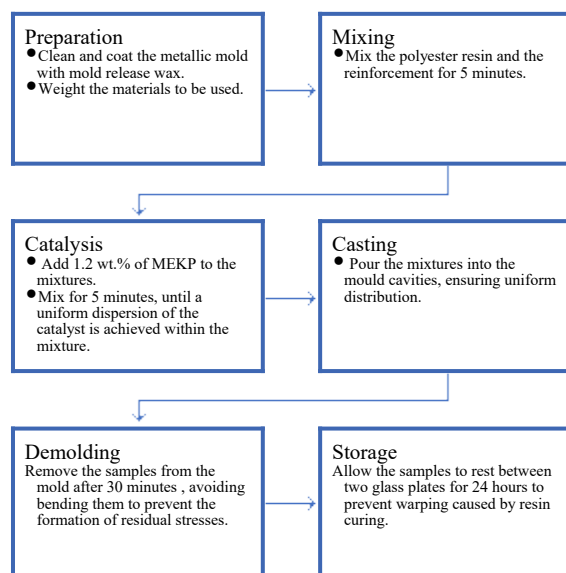


Figure 1: Flowchart of the sample preparation procedure for DMA.

### 3. Experimental testing

#### 3.1. Dynamic Mechanical Analysis

The rheological behavior of the polymer concretes was analyzed by dynamic mechanical analysis, performed in accordance with the ISO 6721 standard. The tests were conducted on TA Instruments DMA 800 machine (Figure 2).



Figure 2: TA Dynamic Mechanical Analyzer

The Dynamic Mechanical Analyzer is equipped with adjustable clamps, an oscillating arm (cantilever), and an enclosed furnace. The tests were conducted according to the parameters shown in Table 2. It should be noted that the same compliance value was used for all tests.

Table 2: Conditions used for the Dynamic Mechanical Analysis

Parameter	Value
Heating rate, °C/min	5

Reinforcement	Weight percentage (%)
Short PET fibers	1 and 5
Long PET fibers	1 and 5
Waste PET flakes	1 and 6
Nano-silica (with particle size of 7 nm)	0.3 and 0.9
<b>Nano-silica (with a particle size of 200 nm)</b>	<b>0.3 and 0.9</b>

Method used	Amplitude
Amplitude value, $\mu\text{m}$	30
Compliance, $\mu\text{m/N}$	0.773

### 4. Results and discussion

#### 4.1. Polyester resin with waste PET fibers and waste PET flakes.

Figure 3 and Table 3 show the effect of PET flakes and PET fibers on the rheological and thermal behavior of polyester resin.

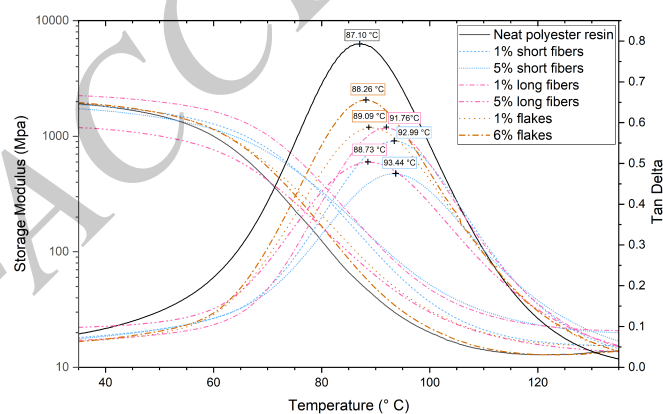


Figure 3: Storage Modulus and Tan  $\delta$  curves of polyester resin and waste PET composites

The glass transition temperature ( $T_g$ ) of all composites containing PET was higher than that of the neat polyester resin, which has a  $T_g$  of 87.10 °C. The  $T_g$  values showed a trend where composites with PET flakes had the lowest values (with a maximum  $T_g$  of 89.09°C), followed by those containing long fibers (with a maximum  $T_g$  of 91.76°C), and finally, those with short fibers (with a maximum  $T_g$  of 93.44 °C). This might be because of two reasons: (1) PET and polyester resin share chemical similarities (both have polar ester groups in their molecular structures), which encourages good interfacial interactions between them, mainly through polar-polar (ester-ester) affinity, and (2) the size and roughness of PET flakes and PET fibers plays a role in how effectively they interact with the UPR matrix. For PET flakes,

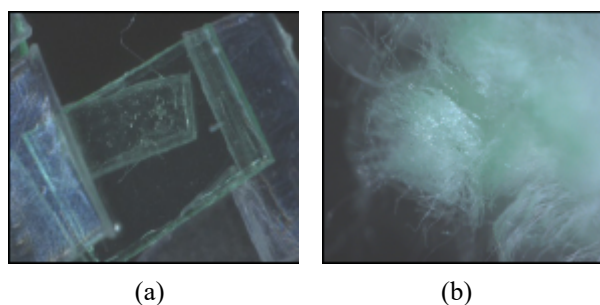


Figure 4: Optical micrographs of (a) PET flakes and (b) PET fibers.

their rigid and particulate nature, coupled with their larger size and smooth surface, allows them to hinder the movement of polyester resin polymer chains to a small extent, which causes a slight increase in the  $T_g$  of the composites. On the other hand, PET fibers have a smaller size and a rougher surface than PET flakes (as can be seen in the optical microscopies shown in Figure 4), which allows them to interact to a greater extent with the UPR and hinder the mobility of their molecular chains to a higher degree, consequently increasing  $T_g$  to a greater extent (Dehas, 2016; Dehas, 2024; Lendvai, 2024).

Table 3:  $T_g$ ,  $\tan \delta$  and  $E'$  modulus of polyester resin composites with waste PET fibers or waste PET flakes

Sample	$T_g$ (°C)	$\tan \delta$	Storage Modulus ( $\epsilon'$ )
Neat polyester resin	87.10	0.79	1913
1% short fibers	92.99	0.554	1871
5% short fibers	93.44	0.474	1727
1% long fibers	91.76	0.586	2243
5% long fibers	88.73	0.502	1186
1% flakes	89.09	0.587	1912
6% flakes	88.26	0.655	1948

Additionally, the higher  $T_g$  observed in composites with short PET fibers is hypothesized to result from the good distribution within the UPR, facilitated by their shorter fiber length, which leads to stronger interfacial bonding than that achieved with longer fiber reinforcement (Dehas, 2016; Dehas, 2024; Lendvai, 2024).

In the case of  $\tan \delta$ , its value for all composites ranges from 0.48 to 0.66, which is lower than the 0.79 seen in polyester resin. The behavior of the composites is the opposite of that observed for  $T_g$ : the lower  $\tan \delta$  values are found for the composites with short and long fibers, and higher values for those containing flakes. This further supports the idea that there is good compatibility between UPR and PET, which mainly stems from both having polar ester groups in their chemical structure. In addition, composites with short PET fibers had the lowest  $\tan \delta$  of all composites, indicating that short fibers have a greater interface with the matrix compared to the other reinforcements (Dehas, 2016). Pothan (2003) notes that an increase in interfacial bonding in composite materials, along with the restriction of polymer chain movement, causes a decrease in  $\tan \delta$  values. Furthermore, as damping at the interfaces increases, the bond between the composite's components worsens.

The lesser adhesion between the polyester resin and the PET flakes can be supported by the ease with which the flakes could be separated from the surface of the prepared samples, as shown in Figure 5.

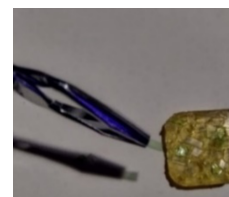


Figure 5: Detachment of a flake in the polyester resin composite with 6% PET flakes.

Regarding the storage modulus ( $E'$ ), the values fluctuate depending on the type and amount of reinforcement used. In composites with 1% fibers, a higher modulus is obtained when long fibers are used instead of short fibers. However, in composites with 5% fibers, the opposite occurs, with values being higher for short fibers. This may be because long fibers tend to agglomerate more easily than short fibers when used in high quantities. For samples with PET flakes, no significant difference in storage modulus was observed when the content of flakes varied.

#### 4.2. Polyester resin with nano-silica.

Figure 6 and Table 4 show the  $\tan \delta$  and storage modulus ( $E'$ ) curves for polyester resin with nano-silica.

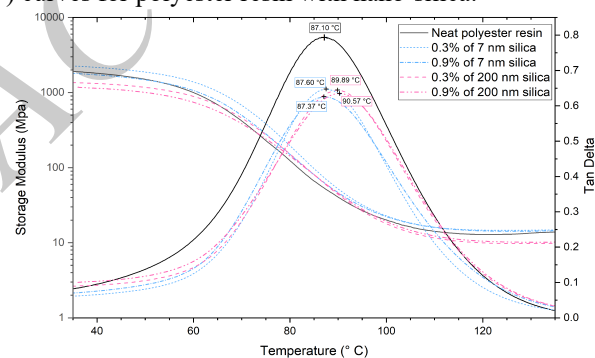


Figure 6 Storage Modulus and  $\tan \delta$  curves of polyester resin and nano-silica composites

In composites with nano-silica particles of 7 nm or 200 nm, the increase in  $T_g$  of UPR was greater than that seen with PET reinforcement, achieving a maximum  $T_g$  of 90.57 °C. This can be widely attributed to the strong interfacial interaction it has with UPR, mainly because of (1) the formation of hydrogen bonds between the silanol groups on the nano-silica surface and the ester carbonyl groups in the polyester resin and, (2) the high surface area-to-volume ratio of the nano-silica particles that enables them to interact more extensively with the matrix compared to reinforcements of larger size (Anggaravidya, 2012). Moreover, the composites with 200 nm silica exhibit a higher value than the composites with 7 nm silica in terms of  $T_g$ . This was due to the 200 nm silica restricting the mobility of the polyester polymer chains to a greater extent than the 7 nm silica, resulting in a greater increase in the glass transition temperature. In this context, Panwar (2017) reports that the presence of nanoparticles in a polymer matrix can hinder the mobility of the polymer chains, which becomes more noticeable as the percentage of nanoparticles used increases.

Table 4: Tg, Tan  $\delta$  and E' modulus of polyester resin composites with nano-silica

Samples	Tg (°C)	Tan $\delta$	Storage Modulus (E')
Neat polyester resin	87.10	0.79	1913
0.3 wt. % of 7 nm silica	87.60	0.646	2249
0.9 wt. % of 7 nm silica	87.37	0.622	1793
0.3 wt. % of 200 nm silica	89.89	0.644	1351
0.9 wt. % of 200 nm silica	90.57	0.634	1175

On the other hand, it was evident that using 0.9 wt.% if nano-silica caused a reduction of Tan  $\delta$  compared to the value for the neat polyester resin. That indicates even when using nano-silica at that percentage, there's still good interaction between it and the polyester resin. Panwar (2017) emphasizes that the inclusion of nano-reinforcement always leads to a decrease in Tan  $\delta$ .

In the case of the storage modulus (E'), the composites with 200 nm silica show higher values than those with 7 nm silica. In other words, greater stiffness is achieved with bigger nanoparticles, as they tend to agglomerate. Furthermore, the addition of 0.9 wt.% of nano-silica (of either 7 nm or 200 nm) results in a decrease in the storage modulus because a higher content of nanoparticles in the polyester matrix increases the likelihood that they will agglomerate.

## 5. Conclusions

Little information exists regarding the effect of adding waste PET (flakes and fibers) and nano-silica on the rheological properties of polyester resin, for this reason, this study centers on this topic.

Experimental results show that the addition of both reinforcements considered in this study can significantly alter the properties of polyester resin. In the case of waste PET, size and surface roughness proved to be critical factors, with short PET fibers providing the most significant increase in glass transition temperature. This is attributed to their rough surface and small size, which promote superior interaction with the resin matrix compared to the poor bonding observed with PET flakes.

Similarly, the inclusion of nano-silica also increased the Tg, but it also caused a decrease in Tan  $\delta$  because it limits the movement of polyester resin polymer chains. Moreover, the lower storage modules seen in composites with 200 nm indicate that they agglomerate to a greater extent than 7 nm silica particles when added to polyester resin.

Future work will focus on the alkaline surface modification of waste PET fibers and flakes using NaOH. The primary aim is to investigate the effects of this treatment on the rheological properties on plastic waste and polyester based composites.

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