Investigating the Effect of Pressing Temperature on the Thermal, Mechanical, and Morphological Properties of Nanocomposites Made from Recycled Polyethylene, Nanosilica, and Wood Flour

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This study investigated the effect of pressurized temperature on the thermal, mechanical, and morphological properties of nanocomposites made from recycled polyethylene with the weight ratio of 50%. Nanosilica was applied at 3 levels (0, 4, 8 %) and wood flour had a weight ratio of 50%. High-density polyethylene (HDPE) went through multiple procedures. It was found that by increasing the nanosilica content, the tensile and flexural strength properties, the residual ash content, and the thermal stability increased along with a reduction in the tensile and flexural modulus and impact resistance. As the temperature increased, the tensile and flexural strength and modulus and the impact resistance decreased. Scanning electron microscopy (SEM) images revealed that samples with 8% nanosilica showed different polymerization than the wood flour particles.

Keywords: Beech wood flour; Recycled polyethylene; Tensile strength and modulus; Nanosilica

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INTRODUCTION

In recent decades, the multilayer plastic wood industry has grown significantly due to its unique characteristics, such as resistance to weathering and low maintenance requirements compared to wood (Shahi *et al*. 2012). Heat-treated polymers are used to incorporate wood into a polymer matrix in which the lignocellulose materials serve as reinforcements. Every day, a significant amount of plastic waste pollutes the environment. The recycling of this plastic waste is of great economic and environmental importance. In general, polyethylene, polypropylene, polyethylene terephthalate, polystyrene, and polyvinyl chloride (PVC) are the main components of plastics in solid urban waste (Najafi 2013). One way to recycle plastics is to use them to make multi-purpose wood-plastic composites, and many studies have examined the construction of a multi-purpose wood-plastic recycled polymer material that focuses more on polyethylene, polypropylene, and PVC (Najafi 2013). Polyethylene is one polymer that can be used to manufacture wood-plastic composites due to its suitable melting point (Klyosov 2007).

Adding natural fibers and particles as a reinforcer to the thermoplastic polymers has been shown to improve mechanical properties, such as stiffness (Fu *et al*. 2002). Natural fibers are advantageous to use due to their low density, renewability, high recyclability, degradability, and most importantly, low cost (Ku *et al*. 2011). The distribution of the lignocellulosic material, the quantity and type of additives, the quality of the joint, the characteristics of the components, the type of equipment, and the conditions of the process affect the multilayer properties of plastic wood (Godard *et al*. 2009). The largest issue when combining natural fibers with plastics is the incompatibility between the polymer hydrophobicity and the hydrophilicity of the fibers, which can cause poor adhesion and reduce the matrix's ability to transfer stress to the fibers (Selke and Wichman 2004). The adhesion can be enhanced by the addition of a compatibilizing agent. Maleic anhydride is one of the adaptive agents that can be used in conjunction with polyethylene (Harper and Wolcott 2004). There have been recent innovations in nanoparticle technology to strengthen multi-structures. Nanosilica is a promising nanoparticle, due to its spherical and porous structure, high contact surface area, and mineral nature, which can improve the polymeric properties of polymers like polystyrene (Xanthos 2010). Nanopolymers, such as nanosilica, have many industrial applications (Wu *et al*. 2005). Nanosilica is a promising material due to its hardness and high strength. In addition, silica is a relatively inexpensive material that is chemically inert, heat-resistant, and environmentally friendly (de Dios and Díaz-García 2010). Compared to pure polymers, polymer-silica nanostructures have different physical and mechanical properties, including higher strength characteristics (Du *et al*. 2012). Researchers believe that silica nanoparticles can improve the strength, hardness, modulus, crystallinity, and melt viscosity properties in polypropylene (Guyard *et al*. 2006). Much research has been done on the application of silica particles in the form of mixtures with various polymers (Chung *et al*. 2002; Zhang *et al*. 2003; Xu *et al*. 2008; Zhang *et al*. 2008), but few of them are related to wood-plastic materials. Researchers have found that silica particles can improve the strength, hardness, modulus, crystallization, viscosity, creep resistance, and intrinsic adhesion of polyethylene, polypropylene, and thermoplastic elastomers due to the surface properties of nanosilica particles (Zhang *et al*. 2003; Rong *et al*. 2004; Guyard *et al*. 2006; Parvinzadeh *et al*. 2010). Various devices have been used to make plastic-wood multilayers (Bledzki *et al*. 2005), one of which is injection molding, of which there has been little research on the effect of pressurized cylinder temperature on thermal and mechanical properties. Therefore, this study is aimed to investigate the effect of the injection pressure temperature and the nanosilica content on the mechanical, thermal, refractory, and morphological behavior of composites comprising wood flour and recycled polyethylene.

EXPERIMENTAL

Materials

Lignocellulose

Beech wood flour was used as a powder enhancer in the polymer matrix. The beech wood flour was meshed to uniformly measure the size of the particles and reach the desired size with a laboratory sieve. The wood flour was passed through a 60-mesh sieve followed by an 80-mesh sieve. The sieved flour was oven-dried to reach 1% moisture content.

Polyethylene

As the disposable container retailers in Iran are usually unaware of the components of waste containers and the possible presence of impurities in their constituents, municipal solid waste is not systematically separated. Therefore, in an effort to avoid error and increase accuracy, it was decided to prepare matrix materials (recycled polyethylene) in the laboratory instead of collecting used milk bottles. The high-density polyethylene

(HDPE) was supplied by the Shazand Petrochemical Company (Arak, Iran). The product name for the polymer was HD52518. It had a melt index of 18 g/ 10 min and a density of 959 g/cm^3.

Plastic recycling

To simulate the effect of recycling, the HDPE was subjected to two stages of thermomechanical degradation using a twin-screw extruder (Brabender, Duisburg, Germany). After each extrusion, the output materials were shredded and were converted to granules. The HDPE molten flow index was measured according to the ASTM standard D1238-98 (1998) using a melt index machine (MI-4; Gottfer, Buchen, Germany).

Compatibilizer

Polyethylene-g-maleic anhydride (PE-g-MA, *i.e.* MAPE) produced by the Krangin Company (Karaj, Iran) was used in order to make the wood flour and the polymer matrix compatible. The MAPE had a melt flow index of 7 g/10 min and a density of 0.965 g/cm^3.

Nanosilica

The nanosilica powder was obtained from Degussa Goldhandel GmbH (Frankfurt, Germany). The specifications of the nanosilica are provided in Table 1.

Table 1. The Nanosilica Profile

Property	**Specification**
Silicon Oxide	SiO_2
Purity	99+ %
Average Particle Size (APS) (nm)	11 to 13 nm
Specific Surface Area (SSA)	200 m^2/g
Bulk Density	< 0.10 g/cm^3
Color	White
Moisture	< 2%
Weight Loss on Ignition	30%
Certificate of Analysis	**Specification**
SiO_2	< 99%
Ti	< 120 ppm
Ca	< 70 ppm
Na	< 50 ppm
Fe	< 20 ppm
*Note: According to the producer information	

Methods

Mixing materials

The recycled HDPE, beech wood flour, MAPE, and nanosilica were combined in an extruder (COLLIN Lab & Pilot Solutions, Maitenbeth, Germany). The compositions of each treatment are shown in Table 2. After the mixing process, the samples were ground to prepare granules using a pilot-scale grinder (WG-LS 200; WIESER, Bayern, Germany).

Table 2. Weight Percentage of the Components from Different Compositions of the Multilayer Plastic Wood Treatments

Pressure Temperature (°C)	Nanosilica (%) *	MAPE (%) **	rHDPE (%)	Wood Flour (%)
170	0	3	50	50
	4	3	50	50
	8	3	50	50
190	0	3	50	50
	4	3	50	50
	8	3	50	50
*Based on the total weight of several structures ** Based on the total weight of the polymer				

Preparation of samples

To prepare the samples, the granules obtained from each compound were dried for 24 h in an oven at 80 °C. Then, a pilot-scale injection molding machine (MPC-40; Aslanian Machine Company, Tehran, Iran) developed the test specimens at cylinder temperatures of 170 °C and 190 °C for the tensile, flexural, and impact tests. Prior to the mechanical tests, the samples were conditioned for two weeks at 20 °C ± 2 °C and 65% ± 5% relative humidity. The ASTM D638-10 (2010) for the tensile (dimensions of the standard dumbbell (dog-bone) samples were 145 x 10 x 4 mm (length x width x thickness)), ASTM D790-10 (2010) for flexural (dimensions of the specimens were 105 x 13 x 5 mm (length x width x thickness)), and ASTM D256-10 (2010) for impact (dimensions of the specimens were 60 x 12 x 6 mm (length x width x thickness)), were used respectively. For each treatment level, four replications were measured for each property and average values were reported.

X-ray diffraction test

The distribution of the nanoparticles in the polymer matrix was investigated using X-ray diffractograms from a Philips diffractometer (Amsterdam, Netherlands). The diffractometer was used in the range of angles 2θ=20° to 80°. The current of the device was 30 mA, its voltage was 40 kV, its step was 0.02 °C, and its speed was 0.3 °C/s. In this device, a cobalt atomic beam with a wavelength of λ=1.79 °A was used as a detector.

Scanning electron microscopy (SEM)

An AIS2100scanning electron microscope made by the Seron Technology Company (Gyeonggi-do, Korea) was used to assess the samples.

Thermogravimetric Analysis (TGA) test

The TGA measurements were performed using a thermal analyzer (TGA Q50; PL-150, Agilent Technologies, Santa Clara, CA, USA), a 7 mg sample was measured within a temperature range of 25 to 700 °C with a heat rate of 15 °C/min.

Statistical analysis

The results from the mechanical tests were analyzed by a completely randomized statistical design under the factorial test, using SPSS software (IBM, Armonk, USA). The Duncan multi-domain test was used at the 95% confidence level to compare the means.

RESULTS AND DISCUSSION

Melted Flow Index

The melt flow index indicates the degree of polymer fluidity. Changes in the melt flow index indicate changes in the polymer structure. The results showed that the HDPE molten flow index decreased from 18 g/10 min for the virgin polymer to 17.75 (0.06) g/10 min after the material was recycled twice. The increase in viscosity during the recycling process is probably due to branching or cross-linking. In general, the destruction mechanism in the HDPE specimens includes branching, cross-linking, and main chain failure (Zahavich *et al*. 1997; Pinheiro *et al*. 2004). Similar results have been reported in HDPE by other researchers (Mendes *et al*. 2011).

The effect of nanosilica and press temperature on the mechanical properties of polymers

The results from the analysis of variance (ANOVA) tests of the independent and interaction effect of nanosilica and the press temperature on the modulus and tensile strength, the modulus and flexural strength, and the shock-free impact resistance, are shown in Table 3. According to Table 3, the effect of nanosilica on the tensile strength, flexural strength, and impact resistance is significant, but not significant on other studied features. In addition, the effect of the press temperature was significant on just tensile strength and not in other features. In contrast, the interaction of the nanosilica and the pressing temperature on any of the resistive properties was not significant.

Table 3. ANOVA Test Results for the Studied Factors and the F-value

	Tension (MPa)		Flexural (MPa)		Impact Strength (KJ/m^2)
	Strength	Modulus	Strength	Modulus	
The Effect of Nanosilica	57.143**	3.121 ns	40.360**	2.816 ns	61.084**
The Effect of Pressure Temperature	83.419**	0.010 ns	0.720 ns	1.862 ns	0.268 ns
The Effect of Nanosilica and Press Temperature	1.892 ns	2.537 ns	0.0988 ns	3.321 ns	0.079 ns
*Meaningful at the level of confidence 95%; **Meaningful at the level of confidence 99% ns Meaningless					

Figures 1 to 5 show the effects of nanosilica and the press temperature on the mechanical properties of the nanostructures. After investigating the effect of nanosilica on the mechanical properties of recycled polyethylene nanoparticles and beech wood flour, several results were obtained. First, the tensile and flexural strength increased linearly with higher addition levels of nanosilica. This increase in strength can be attributed to the fact that nanosilica increases the tension between flour and the matrix by withstanding stress (Ramos *et al*. 2005). In addition, silica nanoparticles act as a nucleating agent in crystal growth, so as the nanoparticles enter, the number of crystals increases (Tian *et al*. 2006). The tensile and flexural modulus decreased as the nanosilica content increased. This is likely because the high surface energy of the silica nanoparticles causes them to absorb each other due to the presence of the hydroxyl hydrophilic groups and the silica gravitational forces. Therefore, the reduction in the tensile and flexural modulus due to the increased nanosilica content is attributed to the accumulation of nanoparticles, their non-uniform distribution, and the phenomenon of particle chlorination in the multilayer bed (Yang and Gu 2007).

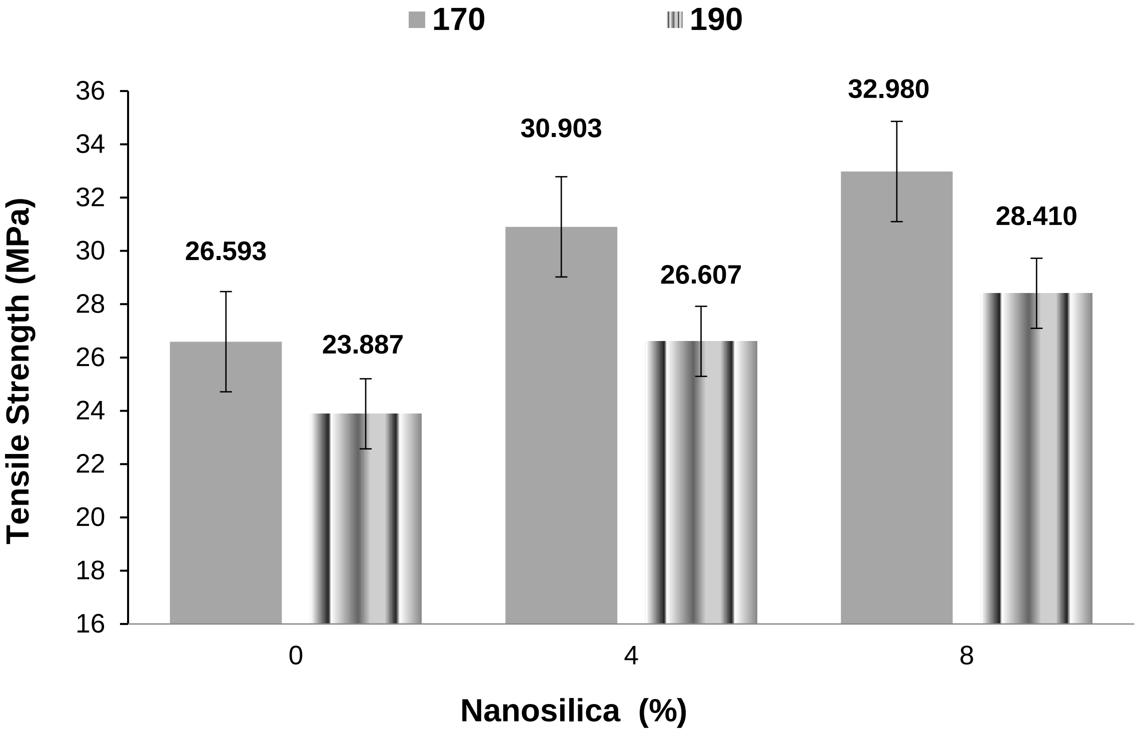

Fig. 1. The interaction of nanosilica and pressing temperature on the tensile strength

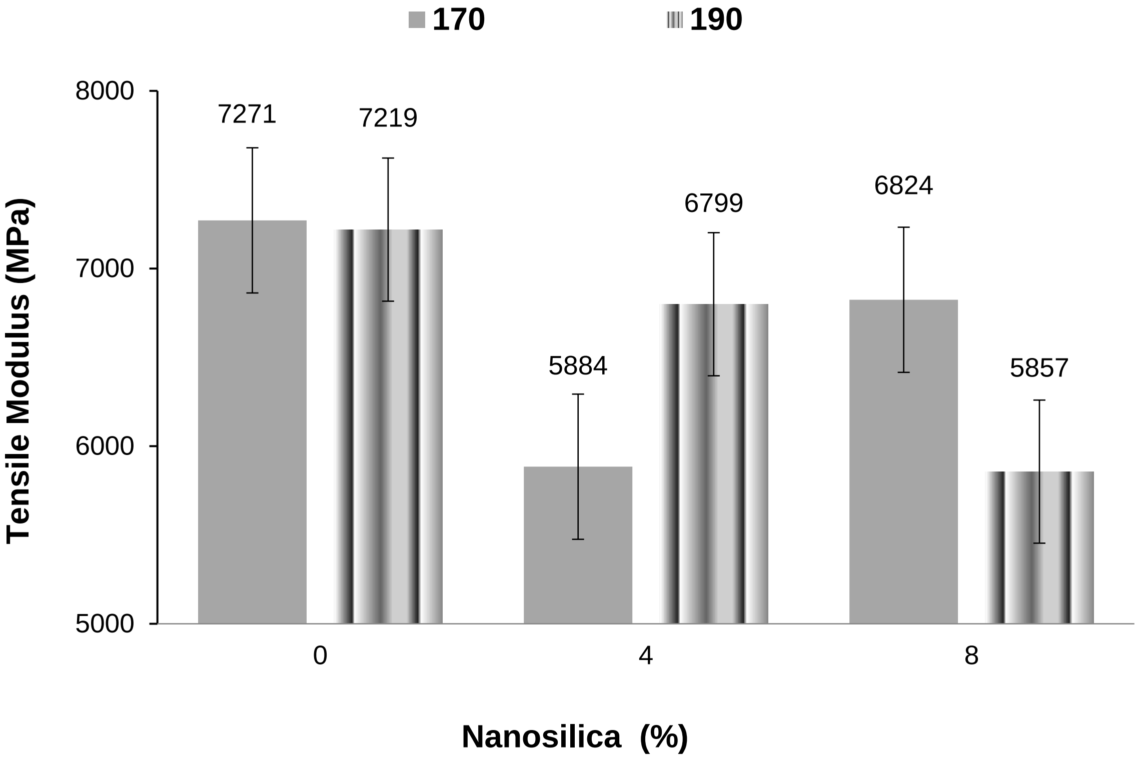

Fig. 2. The interaction of nanosilica and pressing temperature on the tensile modulus

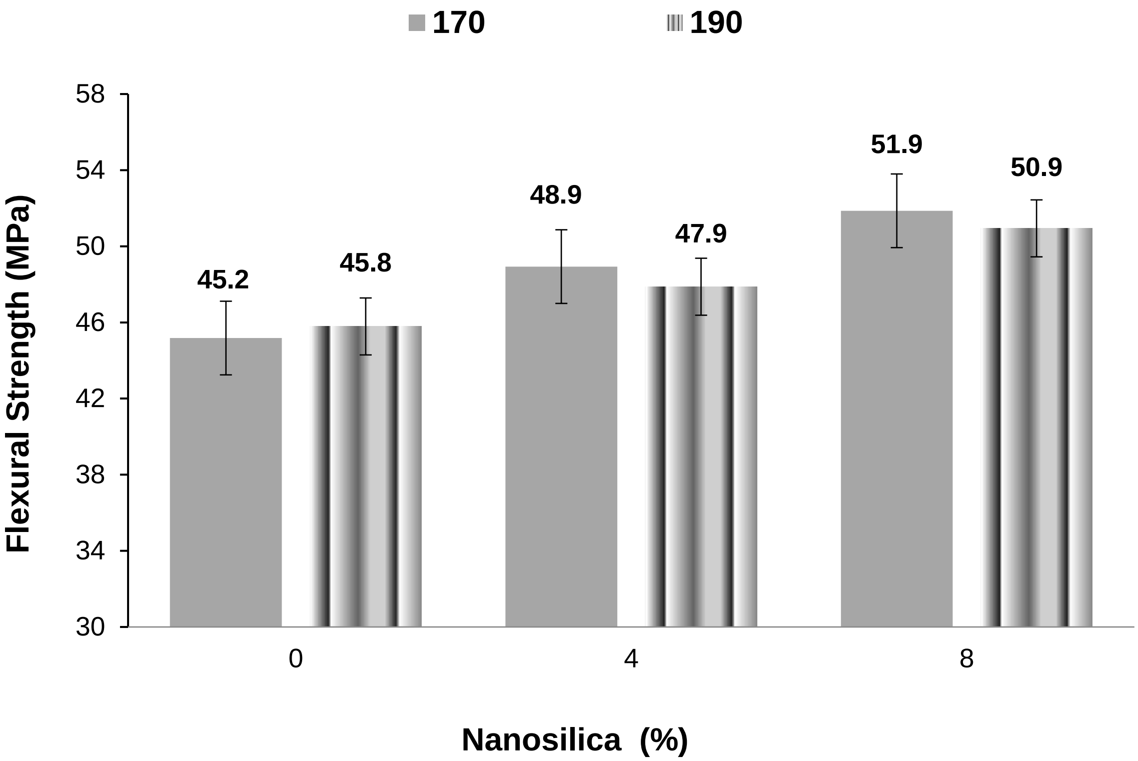

Fig. 3. The interaction of nanosilica and pressing temperature on the flexural strength

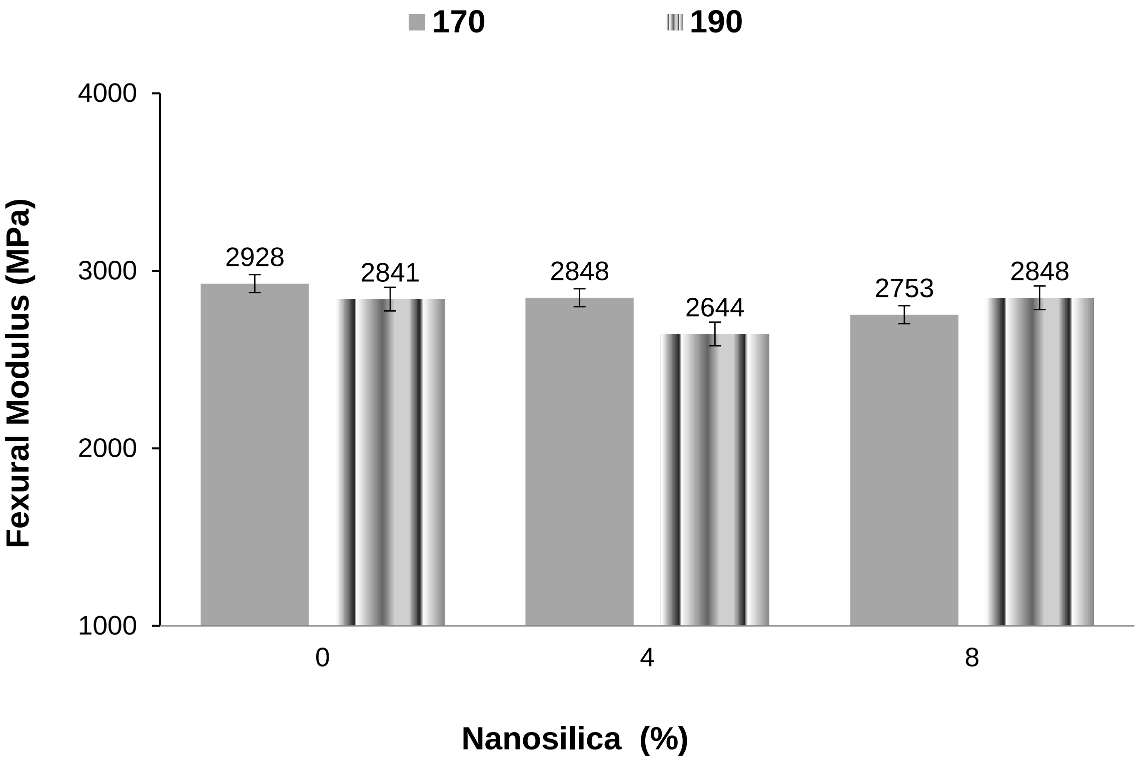

Fig. 4. The interaction of nanosilica and pressing temperature on the flexural modulus

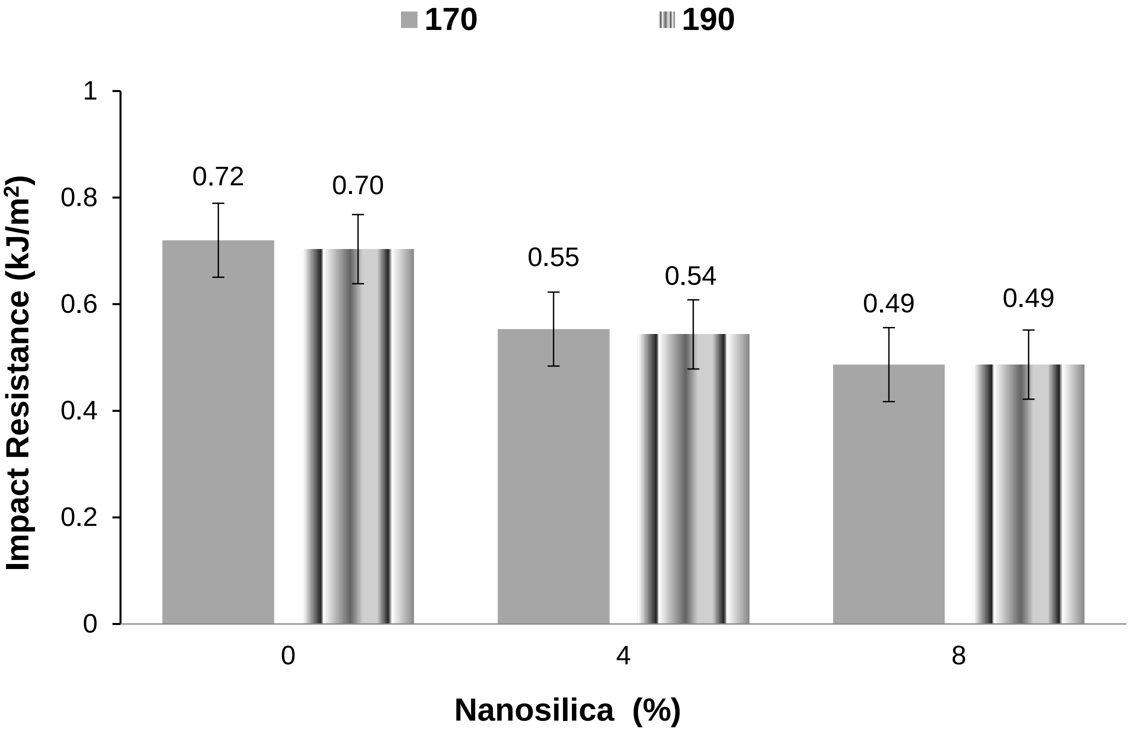

Fig. 5. The interaction of nanosilica and pressing temperature on the impact resistance

Another factor in the reduction of strength properties is the reduction in the polymerizing properties of the polymers due to the use of high amounts of nanosilica, which can cause improper bonding of the lignocellulose and the matrix fibers (Deka and Maji 2013).

It was observed that when the nanosilica content was increased, the impact resistance of the multi-dimensional plastic wood bearing decreased. This is likely because hard fillers generally reduce the impact strength. In hard and brittle polymers, cracks grow easily, so the particles act as a crack initiator and reduce the impact resistance. The reduction in the impact resistance was predictable due to the addition of the nanosilica, as its presence makes multi-structures more brittle and reduces their impact resistance. The presence of nanomaterials in the polymer matrix reduces the mobility of the chains, reduces the possibility of wasting their energy, increases the energy absorbed by the polymer, and creates stress points. The stress points can be the starting point for failure (Han *et al*. 2008). In most cases, the impact resistance is determined by the polymer extruding from the filler surface and the amount of fine cracking. Hard and flexible polymers can be made into chunks by adding hard particles from which the polymer has been removed. When this occurs, the micro-crack energy is increased, and a large volume fraction of the sample is impacted. While this can increase the toughness of the samples, it consumed more energy during preparation (Hemmasi *et al*. 2013).

A higher nanomaterial content will cause reduction in impact strength (Martin-Gallego *et al*. 2011). This relationship can be impacted by the accumulation of nanomaterials, which can lead to the creation of multiple concentrations and stresses on the background material. In fact, this leads to the development of large cracks and a reduced impact strength. Therefore, the addition of high percentages of nanosilica did not allow the absorption of energy into the matrix, which led to the crushing of the samples with higher amounts of the nanographs (Rafiee *et al*. 2009). The stresses in the joint between the nanosilica and the ground material can cause the separation of the reinforcing phase from the background phase. This can cause the formation of cavities that will lead to multiple cracks (Rafiee *et al*. 2009).

At 170 °C, the heat transfer and the adhesion improved. However, at 190 °C, the adhesion characteristics and the quality of the polymer surface decreased and the tensile and the flexural resistance decreased. Increasing the flexural modulus defines the change in the form of compound material under the load, which is a positive factor in engineering structures that have to endure a large load without changing shape.

Table 2 represents that the tensile strength of the polystructures constructed at 170 °C was approximately 14% higher than the tensile strength of the structures constructed at 190 °C. At 170 °C, the heat transfer and the adhesion improved; However, at 190 °C, the adhesion characteristics and the quality of the polymer surface decreased and the tensile and the flexural resistance decreased. Increasing the flexural modulus defines the change in the form of compound material under the load, which is a positive factor in engineering structures that have to endure a large load without changing shape.

The highest resistance, tensile modulus, flexural modulus, and flexural resistance were obtained in the composite formed at 170 °C. The increased resistance can be attributed to the uniform dispersion and distribution of the filler inside the polymer field, which increased the interaction between the filler and the polymer matrix. As expected, the mechanical properties improved due to the uniform distribution of the filler in the polymer field (Andrews *et al*. 2002; Jose *et al*. 2007).

Between the flour and the polymer, a strong adhesion is required to improve the mechanical properties of the composites. The final composite properties depend on the construction conditions such as mixing at the proper temperature to achieve the optimal dispersion of the composite properties (Bledzki *et al*. 2005). The reduced mechanical properties at 190 °C can be due to the formation of acidic chemicals such as acetic acid and formic acid, which cause hemicellulosic decomposition (Garrote *et al*. 2001). The decomposition of the long-chain cellulosic acids to short-chains acids causes cellulose dissolution. In addition, the carbon-carbon and carbon-oxygen connections are lost at the polymer level as the temperature increases. This causes the isolation of the lignin, hemicellulose, and cellulose copolymer system (Çolak *et al*. 2007). The shortening of the cellulose chain affects the resistance properties (Rowell 2012).

The TGA Analysis

The TGA is a suitable tool to determine the sustainability of the composite in temperature conditions and the process of thermal decomposition. The effect of the nanosilica content on the weight loss of the polyethylene/flour composite at the 50% level, during the heating time (from 25 °C to 700 °C), is shown in Table 4. This table shows the temperature of degradation in different percentage weight loss and residual weight at 600 °C for nanoparticles of plastic wood. The addition of the nanosilica also sped up the first stage of destruction and produced more coal.

Table 4. Temperature Required to Reach Different Percentages of Weight Loss for the Nanosilica Composite Samples

		% Weight Loss of Sample							
Temperature	**Sample**	**5%**	**10%**	**20%**	**40%**	**60%**	**80%**	**90%**	**The Remaining Weight at 600 °C**
170 °C	**0% Nanosilica**	287	319	354	445	473	489	550	8.7
	4% Nanosilica	288	315	355	458	479	492	511	8.9
	8% Nanosilica	289	319	362	462	481	494	668	10.5
190 °C	**0% Nanosilica**	289	319	359	460	479	491	504	8.4
	4% Nanosilica	289	319	360	461	480	492	505	8.4
	8% Nanosilica	292	322	363	462	480	494	-	11.9

The thermal stability of the composites is one of the important parameters for the processing and application of these materials. The production of some composites requires mixing fibers and a high temperature matrix. Therefore, the thermal degradation of the lignocellulosic materials causes adverse effects on the composite properties.

Of the main chemical components, hemicellulose has the lowest stability against heat, and its decomposition starts from 225 °C. After hemicellulose, lignin and cellulose begin to decompose (Enayati *et al*. 2009; Rowell 2012). The initial decomposition of lignin and cellulose begin at approximately 250 °C and 275 °C, respectively. However, because lignin has a higher resistance to heat, it degrades slower than cellulose with a maximum thermal destruction at 500 °C (Yang and Gu 2007; Enayati *et al*. 2009; Rowell 2012). The degradation behavior of lignin is due to the destruction of some relatively weak bonds of alkyl binders attached to aromatic lignin rings at low temperatures. Meanwhile, stronger links in the aromatic circles of lignin are destroyed at higher temperatures, which leads to its high resistance properties (Enayati *et al*. 2009).

Table 4 shows that 5% of the weight loss for the 0%, 4 %, and 8% nanosilica samples formed at 170 °C occurred at 287 °C, 288°C, and 289 °C, respectively. The samples with a higher nanosilica content required higher temperatures to achieve the same weight loss of samples with lower amounts of nanosilica. The ash content increased in the samples with higher nanosilica content. The amount of ash remaining at 600 °C in the 0%, 4%, and 8% nanosilica samples formed at 170 °C, was 8.7%, 8.9%, and 11.9%, respectively. The amount of ash remaining at 600 °C in the 0%, 4%, and 8% nanosilica samples formed at 190 °C, respectively, was 8.4%, 8.4%, and 10.4%.

Morphology

Structural study via X-ray diffraction test

Figures 6 and 7 show the scattering intensity at an angle of 2θ for nanosilica powder and the 4% and 8% nanosilica composites formed at 170 and 190 °C. The scattering peak for the nanoparticles shows an unformed silica particle equal to 2θ=22.07°. The X-ray diffraction analysis was used to evaluate the nanosilica in the polymer field in order to measure the crystalline proportion of nanosilica.

In the nanocomposite samples, the crystallinity was lower in the samples with higher nanosilica content (Figs. 6 and 7). The intensity of the other crystal peaks was also lower. The decrease in the crystal peak intensity can be attributed to the partial crystallization of the amorphous part in the composite materials due to the addition of the amorphous silica nanoparticles and wood flour.

The lower peak intensity indicates that the silica particles will become oxidized at a higher nanoparticle content. This can be attributed to the formation of hydrogen bonds among silica particles, due to the increased distance between the layers and their proximity. Deka and Maji (2013) examined the effect of nanosilica on the properties of wood flour and polymer composites and found that the peak intensity decreased as the silica content increased. This showed that nano-silica particles have been injected into plastic wood.

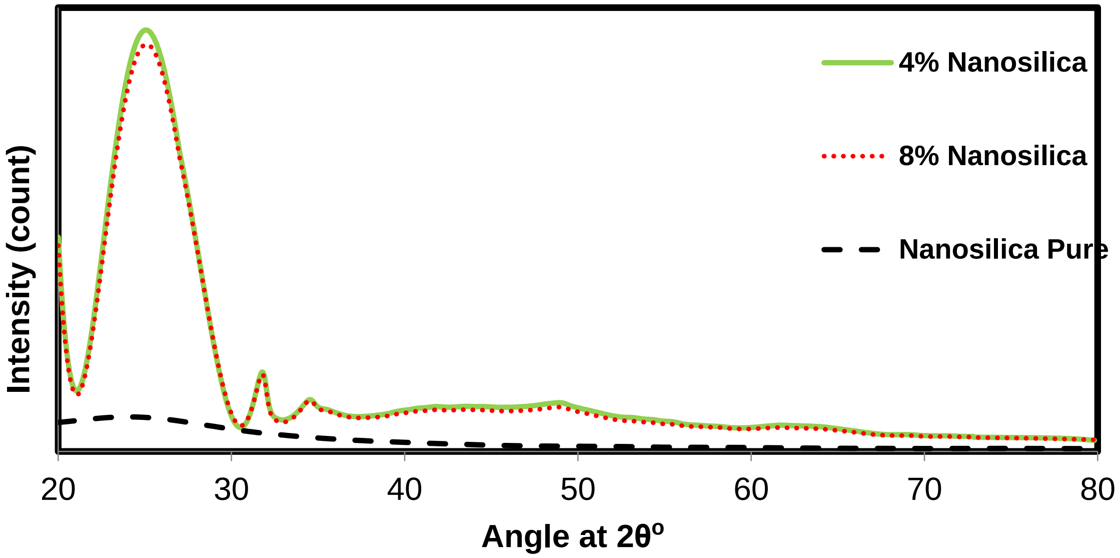

Fig. 6. The multiprupose X-ray nanoscale spectrum containing the 4% nanosilica, 8% nanosilica, and pure nanosilica samples at 170 °C

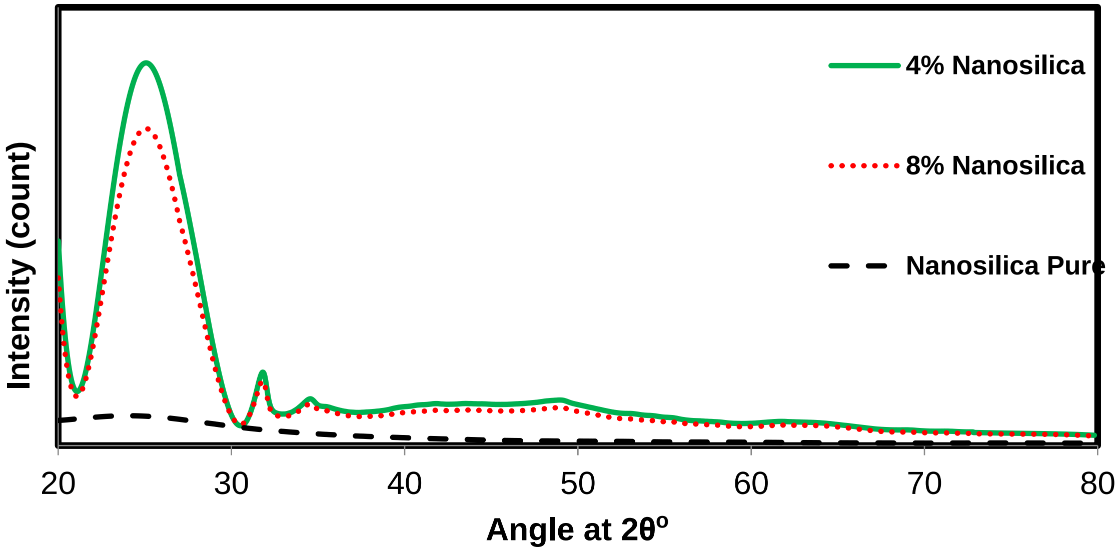

Fig. 7. The multipurpose X-ray nanoscale spectrum containing the 4% nanosilica, 8% nanosilica, and pure nanosilica samples at 190 °C

Morphology Analysis *via* SEM

The distribution and compatibilizer between the fillers and the matrices were observed using images obtained from the SEM analysis. Figures 8a, 8b, and 8c show the failure levels of the 170 °C nanocomposite samples at 0%, 4%, and 8% nanosilica content, respectively. Figures 8d, 8e, and 8f show the failure levels of the 190 °C nanocomposite samples at 0% 4%, and 8% nanosilica content, respectively. As can be seen in Fig. 8a and Fig. 8d, when the 0% nanosilica sample was broken, the wood flour particles were safely removed from their place in the background polymer. There are visible gaps around the wood flour particles, which indicates poor interaction within the flour surface. As the nanosilica content was increased to 8%, a strong bond was formed between the wood flour particles and the polymer. In the common surface area, there was greater bonding and fewer surface gaps. The operation of the wood flour particles by polymer is much better and failure of wood flour particles along with polymer failure has occurred.

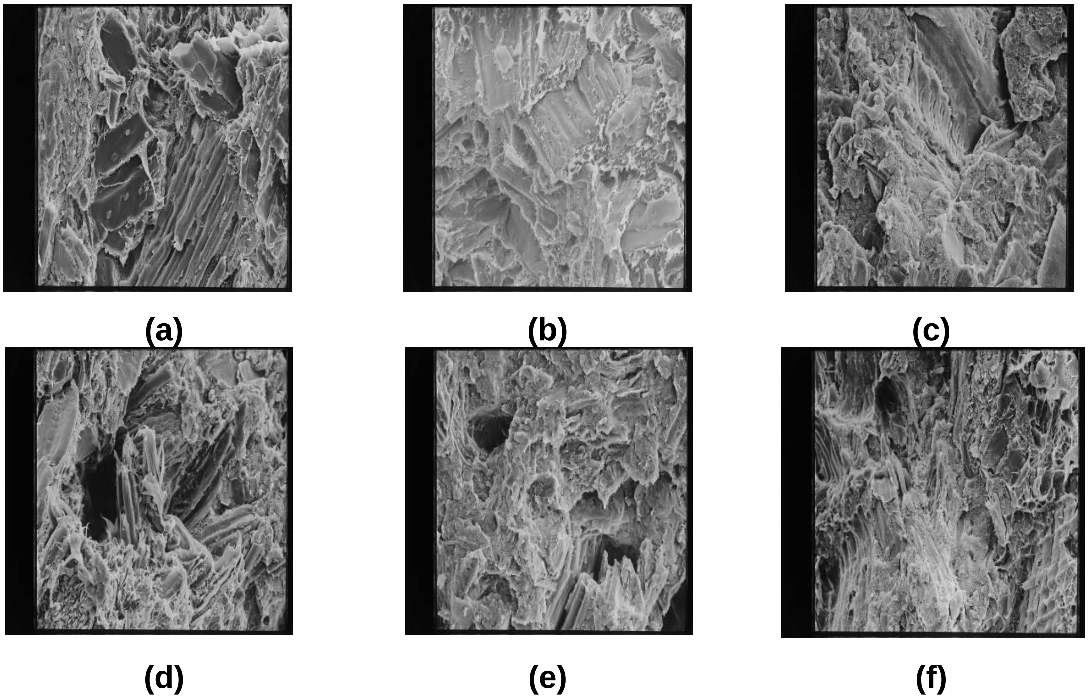

Fig. 8. The SEM images of a) 0% nanosilica and 170 °C, b) 4% nanosilica and 170 °C, c) 8% nanosilica and 170 °C, d) 0% nanosilica and 190 °C, e) 4% nanosilica and 190 °C, and f) 8% nanosilica and 190 °C

CONCLUSIONS

1. As the nanosilica content in the composite samples increased, the tensile and flexural strength increased significantly, while the tensile and flexural modulus decreased at insignificant levels.
2. As the nanosilica content increased, the resistance to immovable impact decreased.
3. As the temperature increased, the impact resistance for the tensile and flexural strength and modulus decreased.

4. With the increase in the nanosilica content, the residual ash content and the thermal stability increased. However, temperature had a negative affect on the residual ash content.
5. The SEM image analysis revealed that the samples with a nanosilica content of 8% had much better polymerization of the wood flour particles and the failure of the wood flour particles occurred with the polymer failure.

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