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ORIGINAL RESEARCH

Biodegradable composites of recycled thermoplastic starch and sawdust: the effect of cellulose nanofibers, nanoclay and temperature

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Abstract
Studies were performed on the effects of small amounts of cellulose nanofibers and nanoclay particles on mechanical, physical and morphological properties of biodegradable composites of recycled thermoplastic starch biopolymer and regular mixed industrial sawdust of Iranian wood species including rush, walnut and hornbeam. To this aim, nanoparticles at 0, 3, and 5 wt% were added to the prepared biodegradable composites. Mechanical, thermal, water absorption and thickness swelling tests were performed according to their corresponding standards. In addition, to investigate the effects of working temperature on derived characteristics of the resulting composites, the selected mechanical tests were performed at various temperatures ranging from 23 to 80 °C. For validation of the results obtained from the physical tests, scanning electron microscopy was also utilized to examine the morphologies of the nanocomposites. The results showed that adding nanoparticles improved tensile modulus by 50%, tensile strength by 110%, flexural modulus by 115%, and flexural strength by 18%. Adding 5 wt% nanocellulose fibers showed better results than 3 wt% addition, while in the case of nanoclay, the trend was reverse. No significant change was observed for impact strength. Glass transition temperature was increased from 90 to 123 °C depending on the amount of nanoparticles. Water absorption and thickness swelling were reduced by around 20%. It was also observed that at elevated temperatures nanoparticles led to greater stability of the composite structure. From the results of this study it can be concluded that cellulose nanofibers and nanoclay particles can be successfully used for improving the mechanical and physical characteristics and performance of the biodegradable WPCs made of thermoplastic starch and industrial sawdust. Furthermore, since the effect of adding each of these nanoparticles on mechanical and physical performance of WPCs is different, the results of this study can be used to decide on selection of the type and amount of nanoparticles for fabrication of a product for a desired application.

Keywords Biodegradable nanocomposite · Cellulose nanofiber · Nanoclay · Thermoplastic starch · Sawdust

Introduction

Despite plenty of undeniable benefits that advances are made in synthetic polymer technology in improving human life, abundant use of non-degradable plastic materials and high volume of wastes have led to many environmental problems for human and wildlife. With the depletion of oil resources in near future, these problems will double because the main primary raw materials of these products will also be scarce. Hence, the interest of researchers in design and use of biodegradable polymers, based on sustainable materials, has greatly increased [1–6].

Among the natural biodegradable polymers, starch is a good choice for producing biodegradable thermoplastics. Starch is a multi-purpose and inexpensive polymer with

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great features which make it suitable to use in various industries [7–9]. Because of low mechanical strength and high moisture absorption, pure starch cannot provide desired properties for intended applications such as packing [10, 11]. Wood plastic composites (WPCs) are one of solutions, if we use thermoplastic starch as matrix polymer and wood fibers or woodflour as primary filler.

In recent decades, understanding the behavior and interaction of materials within the nanoscale range has been one of the research priorities. Due to their exceptional properties, small amounts of these nanoparticles can improve the properties and performance of WPCs desirably [12–15]. Cellulose nanofibers (CNFs) are one of the thinnest and finest fibers in nature. Interesting features such as biodegradability, good mechanical properties and low density distinguish them from other nano-enhancers [16]. By adding CNF to thermoplastic starch, improvements in mechanical properties as well as reduced water absorption of the obtained composites has been reported [17].

Nanoclays (NCs) are abundant and easily available minerals. It is reported that the addition of a small amount of nanoclay particles would improve the mechanical properties and dimensional stability of designated composites [18].

In this research, the effects of nanocellulose fibers (NCFs) and nanoclay (NC) particles on mechanical and physical properties of WPCs made of biodegradable thermoplastic starch polymer, prepared from recycled materials and industrial sawdust, are investigated. In addition, the effect of working temperature which is an important parameter affecting the mechanical performance of these composites and limiting their usage in different applications such as automobile interior trims, floorboards and ceilings or packaging was also investigated. Finally, for morphological study, scanning electron microscopy (SEM) was performed. The innovations of this study are the use of recycled materials and also mechanical tests which were conducted at controlled temperatures (working temperatures).

Experimental

Materials

In this research, corn starch with a density 1.5 g/cm^3 and a molar mass of 105–108 g/mol, the product of Alvand Conversational Industries Co. Hamedan, Iran, was used. Glycerol with a density of 1.267 g/cm^3 and a molar mass of 92.09 g/mol, the product of Dr Mojalali Chemical Industry Complex, Iran, was used as a plasticizer. These two ingredients were mixed to make a thermoplastic biodegradable polymer. Industrial sawdust, most of which is a mixture of beech, horn beam and walnut species was used as the main filler. NCF with an average diameter of 35 nm produced by freeze dryer method was obtained from Nano Novin Polymer Co. Iran. NC in the form of montmorillonite with a density of 0.5–0.7 g/cm^3 and particle size of 1–2 nm was supplied from Novin Pasargad Chemical Co., Iran. Maleic anhydride-grafted polypropylene (MAPP) as one of the commonly used coupling agents with a melting flow index of 64 g/10 min was used in order to improve adhesion.

Composite preparation

Sawdust and corn starch, which were passed through a 60-mesh sieve, were placed in an oven at 60 °C for 24 h, separately. The corn starch was then processed with 20 wt% glycerol (as a plasticizer) in an internal mixer machine (Haake model HBI system 90, NY, USA) for 3 min at 10 rpm and 60 °C. By cooling down, the obtained polymer was grinded in an industrial grinder (Wiser, A-8992, Germany) in order to simulate recycling condition. Then, the resultant thermoplastic polymer was mixed with specified amounts of NCF, NC, coupling agent and sawdust in an internal mixer at 100 °C for 3 min at 10 rpm and then for 7 min at 80 rpm to produce composite samples coded A-E. After this process, a hot pressing machine (Toyoseiki, Mini Test Press, Tokyo, Japan) was utilized to fabricate sheets of each of A-E composite sample at 35 MPa and 150 °C. This pressure was required to produce uniform and porous composite sheets. Dimensions of the sheets were 120 mm × 120 mm × 1 mm and 120 mm × 120 mm × 1.5 mm to prepare samples suitable for designated tests. Table 1 shows the composition (wt%) of the materials in each sample type.

Methods

Mechanical tests including flexural, tensile and fracture tests were performed according to ASTM D790, ASTM D638, and ASTM D256 standard test methods, respectively. Bending tests were carried out using a GOTECH AL-7000 M Universal Testing Machine. Loading rate was 1 mm/min. In order to maintain thermal condition, a thermal chamber, which was designed and implemented, was used. Tests were

Table 1 Composition of nanocomposite samples

Specimen code	Polymer wt%	Sawdust wt%	Coupling agent wt%	Nanoclay wt%	Nanocellulose wt%
A	49.5	49.5	1	0	0
B	48	48	1	3	0
C	47	47	1	5	0
D	48	48	1	0	3
E	47	47	1	0	5

performed at 23 (room temperature), 40, 60 and 80 °C, and each test was repeated 3 times. Flexural modulus and flexural strength of each specimen (A-E) were calculated. Dimension of bending specimens was 50.0 mm × 12.7 mm × 1.5 mm.

Water absorption and thickness swelling tests were also performed according to ASTMD7031-04, and dynamic mechanical thermal analysis (DMTA) tests were conducted according to ASTM E1356. Square specimens with the dimension of 20 mm × 20 mm × 1.5 mm were used for each type of designated WPCs. Samples were first dried in an oven and their initial weight and dimensions were determined. Then they were immersed in distilled water and their weight and thickness were measured after 72 and 1000 h. The obtained data were used to calculate water absorption using Eq. 1. Each test was repeated 5 times.

$$WA_t = \left[\frac{W_t - W_0}{W_0}\right] \times 100 \tag{1}$$

In this equation, W_0 is the dry weight of specimen (g) before immersion in water, W_t is the weight of specimen after 72 or 1000 h immersion in water (g) and WA_t is the percentage of absorbed water after 72 or 1000 h (%). Percentage of thickness swelling was calculated using Eq. 2; in this equation T_0 is the dry thickness of specimen (mm), T_t is the thickness of specimen (mm) after immersion over specified time and TS_t is the percentage of thickness swelling (%) over specified immersion time:

$$TS_t = \left[\frac{T_t - T_0}{T_0}\right] \times 100 \tag{2}$$

Each test was repeated at least 3 times, and statistical analyses were conducted using SPSS software (IBM, Armonk, USA, ver. 11.5).

Results and discussion

Mechanical tests

Bending properties including flexural modulus and strength of composite samples (A-E) are shown in Figs. 1 and 2, respectively.

Figure 1 shows that at room temperature (23 °C) CNF can improve the flexural properties better than NC and as CNF content increases, the flexural modulus of WPCs improves further. By adding more NC particles has not led to more improved results. With increasing the test temperature, the flexural characteristics of the composites show a sharp decline and it can be seen that at higher temperatures, 5% NC content leads to better flexural modulus.

According to Fig. 2, it can be seen that at room temperature (23 °C), 5% CNF leads to better results for bending

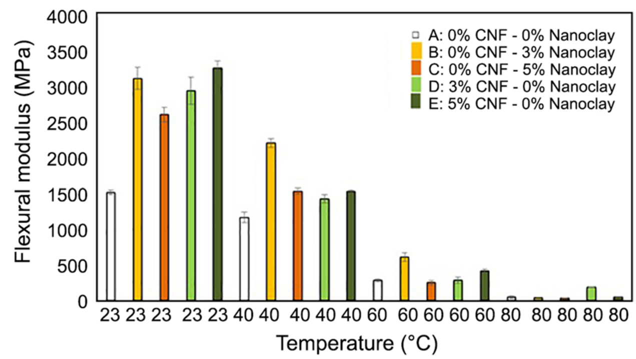

Fig. 1 Flexural modulus of WPCs versus test temperature

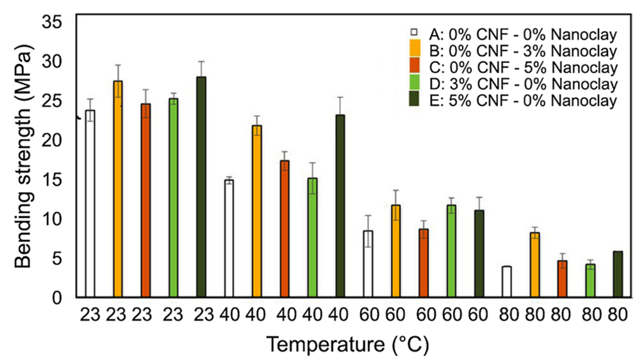

Fig. 2 Bending strength of designated WPCs versus test temperature

strength. As the temperature rises, this condition remains valid until the temperature reaches 60 °C, in which the effect of NC particles is dominant. It may be concluded that for the flexural properties of the WPCs, 3% NC shows better results than 5% NC.

Tensile tests were performed using a Universal Testing Machine (GOTECH model AL-7000 M) at temperatures of 23, 40, 60, and 80 °C, and at a loading rate of 1 mm/min, moreover, each test was repeated 3 times. In Figs. 3 and 4 tensile modulus and strength of the composites are shown.

In Fig. 3 it can be seen that the E specimen containing 5% cellulose nanofibers has superior tensile modulus at room temperature.

With increasing temperature, tensile modulus declines considerably, and although adding nanoparticles leads to better tensile properties, 3% nanoclay addition has resulted in more stable tensile modulus at higher temperatures. According to Fig. 4, it is immediately obvious that adding 3% NC or 5% CNF considerably improves tensile strength of the WPCs, which is nearly twice of the tensile strength of the WPCs without any nanoparticles (specimen A). The addition of 5% nanoclay or 3% CNF has not led to a large increase in tensile strength.

Charpy impact tests were performed according to ASTM D256 standard test method. To this aim, a Karl

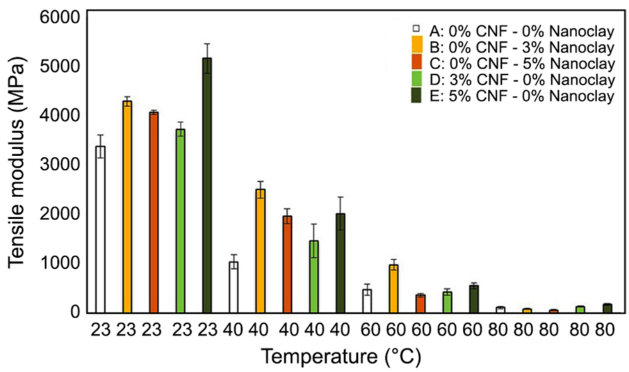

Fig. 3 Tensile modulus of designated composites at various test temperatures

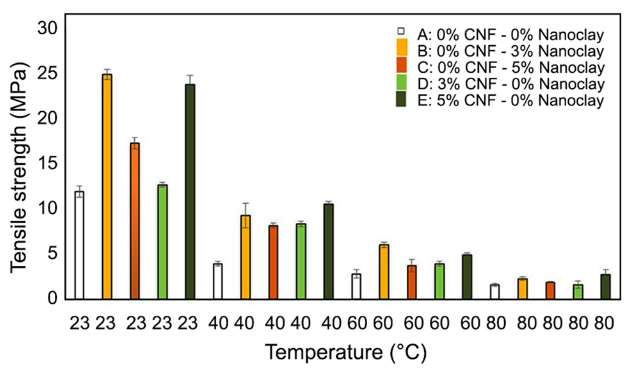

Fig. 4 Tensile strength of designated WPCs at various temperatures

Frank Model CMBH Charpy impact test device was used. Center-notched rectangular specimens were used and fracture test was repeated 3 times for each WPC specimen of 63.5 mm × 12.7 mm × 1.5 mm dimension.

Summary of mechanical tests results at room temperature (23 °C) is presented in Table 2. In this table, the percentage of material property improvement after adding specific nanoparticle (for WPCs type B-E) is also mentioned.

In this table, $P_{x/A}$ ($= [P_x - P_A] \times 100/P_A$) is percentage change of material property relative to the WPC type "A" which reflects the effect of adding nanoparticle on the specific material property. The results in Table 2 show that adding a small amount of CNF or NC improves the mechanical properties of WPCs considerably. While adding 5% CNF leads to better improvement in mechanical properties than by adding 3% CNF, which this may show better mechanical improvement than by the addition of 5% NC. It is also notable that 5% CNF improves all mechanical properties than by adding 3% NC, except for tensile strength which is improved by the addition of 3% NC. Regarding impact strength (fracture properties), which showed no improvement after adding NC or CNF particles.

Key factors affecting these results are the strong interaction between the matrix and nanoclay silicate layers, the formation of hydrogen bonds, and structural factors such as volume ratio. Usually with the increase in nanoclay content the new structures are combined and clay masses in nanocomposite are formed which causes lump formation which may cause weaker mechanical properties. One of the main reasons for adding lignocellulosic material to wood-plastic composites is because the mechanical properties of cellulose fibers (especially CNF) is much better than that of composite materials, and thus improves the mechanical properties.

Dynamic mechanical thermal analysis

Triton Technology Model Tritec DMTA 2000 (made in UK) device was used to evaluate loss factor, tanδ, and glass transition temperature, T_g, according to ASTM E1356 standard test method. This device was used in flexural mode. Tests were performed in the temperature ranging from − 100 to 150 °C, heating rate was 4 °C/min and load frequency was 1 Hz and fixed. Rectangular specimens

Table 2 Mechanical characteristics of WPCs at 23 °C and effect of the addition of nanoparticles

WPC type/comparison of properties: $P_{X/A}$	Flexural modulus (MPa)	Flexural strength (MPa)	Tensile modulus (MPa)	Tensile strength (MPa)	Impact strength (J/m)
A: No nano	1528.0	23.5	3367.0	11.9	10.8
B: 3 wt% NC	2958.8	27.4	4281.9	24.8	10.5
C: 5 wt% NC	3275.3	24.6	4064.2	17.3	10.1
D: 3 wt% CNF	3133.1	25.2	3722.0	12.7	10.5
E: 5 wt% CNF	2619.6	28.0	5154.3	23.7	10.2
$P_{B/A}$	+ 105.1%	+ 15.5%	+ 27.2%	+ 108.7%	≈ 0%
$P_{C/A}$	+ 71.5%	+ 3.4%	+ 20.7%	+ 45%	≈ 0%
$P_{D/A}$	+ 93.6%	+ 6.1%	+ 10.5%	+ 6.4%	≈ 0%
$P_{E/A}$	+ 114.4%	+ 17.7%	+ 53.1%	+ 99.2%	≈ 0%

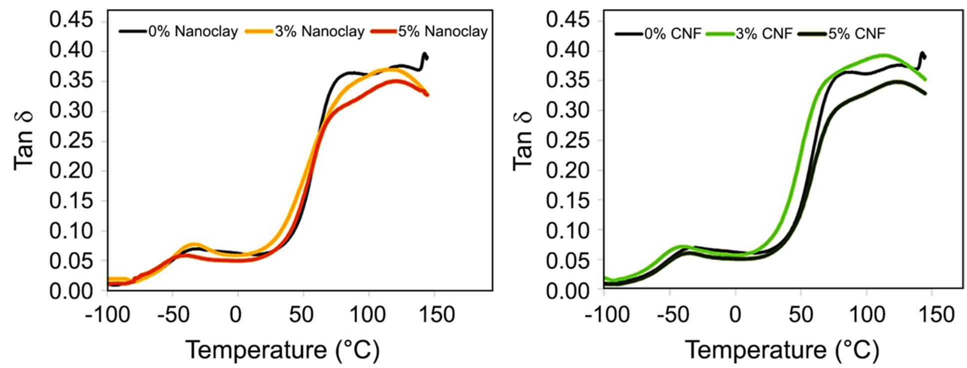

Fig. 5 tanδ curves for WPCs with various amounts of nanoparticles

Table 3 Glass transition temperatures for WPCs with various amount of nanoparticles

Sample type/nanoparticle content	$T_{g\alpha}$ (°C) Low glass transition temp	$T_{g\beta}$ (°C) High glass transition temp
A: No nano	−37	89
B: 3 wt% NC	−38	112
C: 5 wt% NC	−36	119
D: 3 wt% CNF	−39	113
E: 5 wt% CNF	−38	123

with dimension 30 mm × 10 mm × 1 mm were used. The tanδ curves for WPC type A-E are shown in Fig. 5.

According to Fig. 5, it is immediately obvious that the shape of tanδ curves for WPCs with various amounts of nanoparticles is almost identical, two fairly flat peaks can be observed in all tanδ curves, which represent low and high glass transition temperatures $T_{g\alpha}$ and $T_{g\beta}$, accordingly. The glass transition temperatures for WPCs with various amounts of nanoparticles are presented in Table 3.

The first low glass transition temperature is related to glycerol-rich region and the second high glass transition temperature is for thermoplastic starch-rich region. As can be seen in Table 3, with addition and increase of nanoparticles into WPCs, there is no significant change in low glass transition temperature ($T_{g\alpha}$), this is due to fixed and unchangeable nature of glycerol in the structure of nanocomposites. On the other hand, it was observed that with adding and increasing the amounts of nanoparticles into WPCs, high glass transition temperature ($T_{g\beta}$) is increased considerably and CNF addition has better improvement. Strong interactions between NCF and starch limit the mobility of starch molecules and also starch chains. This limitation makes the WPCs more rigid, and consequently, $T_{g\beta}$ increases. The broad width of the peaks indicates the heterogeneity of thermoplastic starch structures and nanoparticles dispersed into the composites.

Table 4 Summary of physical test results and effect of nanoparticles added

WPC type/ $P_{X/A}$	Water absorption (%)	Thickness swelling after 72 h (%)	Thickness swelling after 1000 h (%)
A: No nano	106.8	98.5	126.6
B: 3 wt% NC	88.0	80.6	101.2
C: 5 wt% NC	81.9	78.2	91.4
D: 3 wt% CNF	104.9	96.5	120.5
E: 5 wt% CNF	100.3	89.5	114.2
$P_{B/A}$	−18.8	−17.9	−25.4
$P_{C/A}$	−24.9	−20.3	−35.2
$P_{D/A}$	−1.9	−2.0	−6.1
$P_{E/A}$	−6.5	−9.0	−12.4

Water absorption and thickness swelling tests

Since the thickness of specimens after 1000 h immersion in water was not measurable accurately due to partial degradation and insufficient strength, results of thickness swelling tests are only reported for 72 h. The summary of physical and morphological properties of designated WPCs is presented in Table 4.

According to the results of Table 4, it is observed that adding 5% NC results in better resistance to water absorption and thickness swelling than adding 3% NC, also the results show that 5% CNF reduces water absorption and thickness swelling more than adding 3% CNF. So the addition of more nanoparticles results in better physical performance of the designated WPCs. It is also worth mentioning that the effect of adding NC is far better than adding CNF in terms of physical performance.

Scanning electron microscopy observations

For more observations in this study, the microstructure of WPCs was investigated on fracture surfaces using scanning electron microscopy (SEM). Appropriate fracture surfaces were created at liquid nitrogen temperature. By observing and examining the images obtained from the scanning electron microscope, the distribution and compatibility between the fillers (industrial sawdust and added nanoparticles) and matrix (thermoplastic starch) could be investigated.

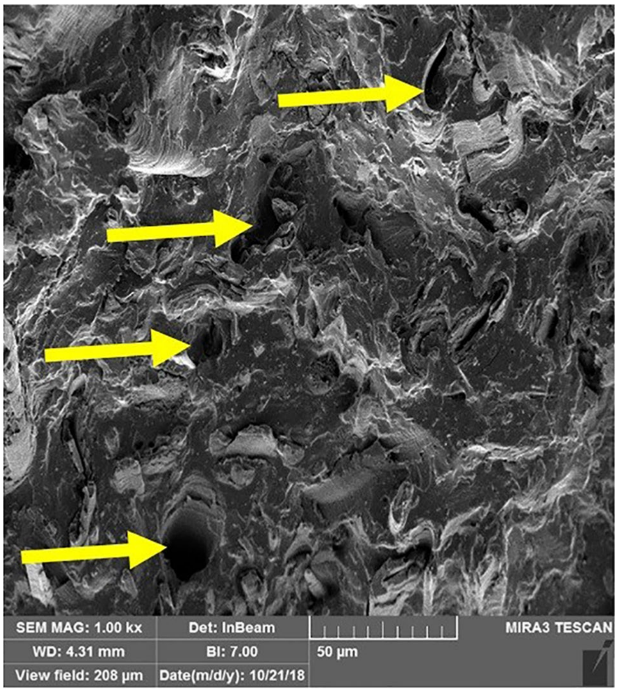

Fig. 6 Fracture surface of WPC sample A with no nanoparticle content

In Fig. 6, the SEM fracture surface of WPC without nanoparticle addition (type A) is presented. Yellow arrows which show the vacant spaces (cavities) imply poor binding surfaces between the fibers and the matrix in absence of any nanoparticle addition. It means that by applying tension or pressure the fibers are separated from the matrix surface due to weak connections between them. In Fig. 7, yellow arrows show the condition of bonds or cavities and yellow circles show NC particles dispersion into the composite. Figure 7a shows that the adhesion between the fibers and polymeric phase has been improved by adding 3% nanoclay, in this figure the decrease in cavities implies this improvement. Figure 7b is a SEM image of WPC with 5% NC, this image shows that the adhesion between the matrix and fibers is weakened compared to previous image (Fig. 7a) and in some areas, cavities and cracks are nucleated at fiber separation spots. According to the tensile and flexural test results, the increase in NC content from 3 to 5 wt% reduced these mechanical properties, an acceptable reason for this behavior can be interpreted as absorption of coupling agent by NC particles [19]. By increasing nanoclay amounts, coupling agent absorption increases, so the coupling agent does not establish a suitable connection between the matrix and lignocellulosic materials, thus the tensile and flexural strength decrease.

In Fig. 8, yellow arrows show the condition of bonds and yellow circles show some CNF particles dispersed into the composite. Figure 8a, b show that by adding 3 wt% and 5 wt% cellulose nanofibers, the strength of the bonds and joint surfaces becomes better, and the transfer of stress from

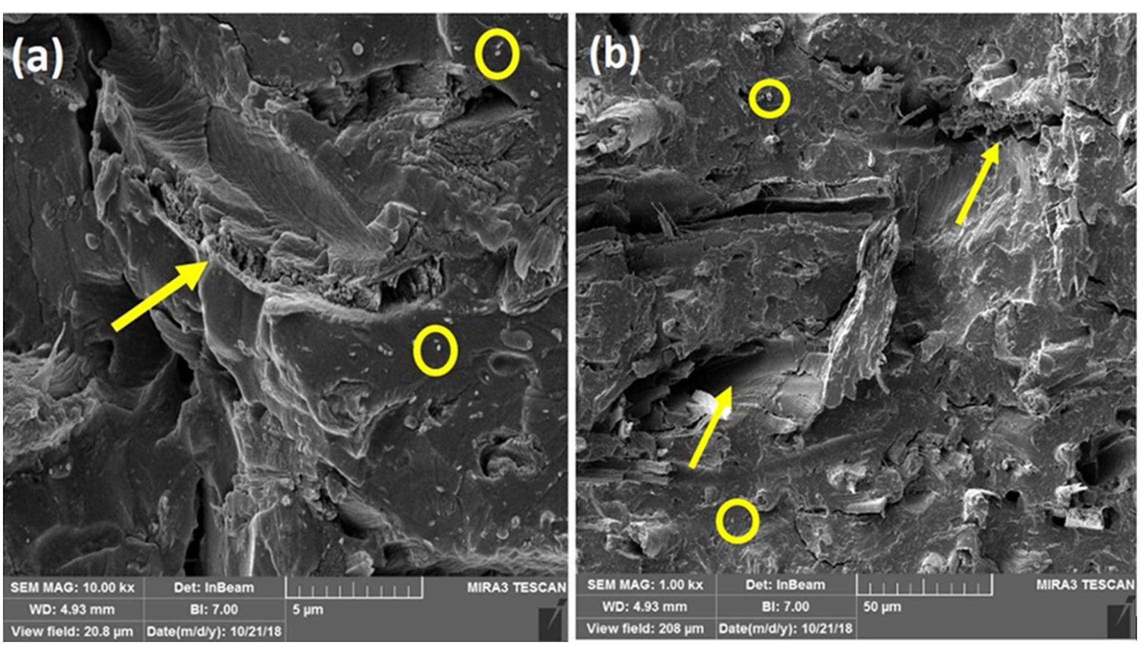

Fig. 7 Fracture surface of **a** WPC sample B with 3 wt% NC and **b** sample C with 5 wt% NC

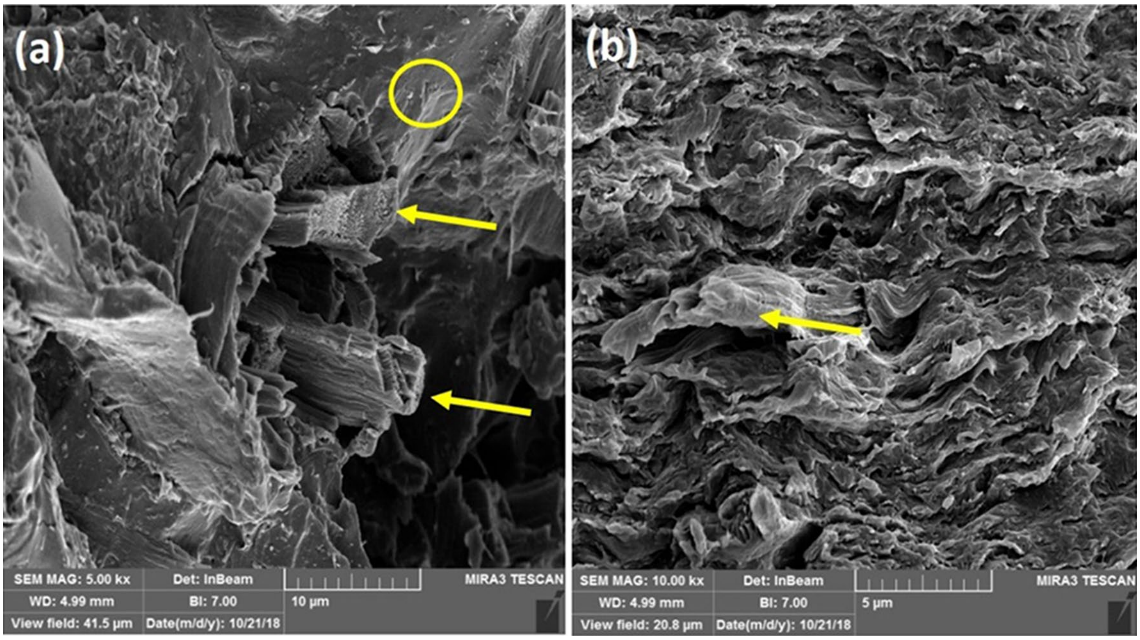

Fig. 8 Fracture surface of **a** WPC sample D with 3 wt% CNF and **b** sample E with 5 wt% CNF

the matrix to fibers become more effective, leading to better mechanical properties.

According to the SEM images presented in this study, the lowest adhesion strength between the fillers (industrial sawdust) and matrix (thermoplastic starch) was observed for WPCs without nanoparticle. In these WPCs, the fracture surfaces showed weak adhesion and relatively large heterogeneity, and the presence of cavities and holes also demonstrated this issue.

Conclusion

The effect of adding small amounts of cellulose nanofibers (CNF) and nanoclay (NC) particles into the WPC consisting of thermoplastic starch and industrial sawdust was investigated. To this aim, comprehensive mechanical and physical tests were carried out to obtain mechanical properties and study morphological behavior of the designated WPCs. Also examine the performance of these biodegradable nano WPCs against the temperature of the environment, mechanical tests were performed at various temperatures ranging from 23 to 80 °C. The followings can be concluded from the results and observations of this research:

- Adding small amounts of CNF and NC particles to the starch-sawdust biocomposite, increases tensile strength, tensile modulus, flexural strength and bending modulus, but has no considerable effect on impact strength (fracture toughness).
- The addition of CNF and NC particles into the starch-sawdust biocomposite improves physical and morphological characteristics and performance, such as water absorption and thickness swelling.
- As temperature increases, mechanical properties of all WPC types decrease considerably, but at elevated environment temperatures, WPCs containing NC particles have structure more stabilized than WPCs containing CNF particles.
- In terms of mechanical characteristics, adding 5 wt% CNF is more effective than 3 wt% CNF, but, in contrary, 3 wt% NC shows better results compared to 5 wt% NC addition.
- Addition of nanoparticles leads to higher glass transition temperatures ($T_{g\beta}$), also 5 wt% nanoparticle addition results in higher $T_{g\beta}$ than 3 wt% nanoparticle addition, furthermore CNF particles lead to higher $T_{g\beta}$ than NC particles for both 3 wt% and 5 wt% additions.

From the results of this study, it can be concluded that cellulose nanofibers and nanoclay particles can be successfully used for improving mechanical and physical characteristics and performance of biodegradable WPCs made of starch thermoplastic and industrial sawdust. Furthermore since the effect of type and amount of nanoparticles on mechanical and physical performance of WPCs is different, so the results of this study can be used for different specific applications with desired properties.

The raw processed data required to reproduce these findings cannot be shared at this time as the data also form part of an ongoing study.

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