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RESEARCH PAPER

Investigation of the Thermal and Mechanical Properties of Organic Waste Reinforced Polyester Composites

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Abstract

The aim of the research is to investigate the usability of composites produced using the organic waste from hazelnut and corn processing in the Black Sea Region in constructional applications with regard to mechanical and thermal conduction. Polymer composite test samples were produced to which hazelnut shells, corn stalk, nettle stalk (excl. fibres) and nettle fibres had been added in varying (5, 10, 15, 20%) weights to the polyester matrix. Thermal conductivity coefficients were determined in the context of the thermal properties of composite samples. In addition, mechanical properties were determined by compression strength and a three-point bending test. The physical properties of the wastes were determined via XRD, glass transition temperature, one of the thermal features of the composites that is found via DSC and, lastly, the thermal conductivity and specific heat capacity are determined via a heat flow meter. The effect of organic waste amounts and their chemical and physical features on composites' mechanical and thermal properties are discussed in this study. The results of the study show that nettle stalk-reinforced composites have a low density and more advantageous features than the other composites tested in terms of their thermal conductivities.

Keywords Composite · Mechanical properties organic wastes · Polyester · Thermal conductivity

Abbreviation

ASTM	American Society for Testing and Materials
EN	European Standards
DSC	Differential scanning calorimetry
MPa	Megapascal
XRD	X-ray diffraction
CrI	Crystallinity index
Q	Heat flow
C_p	Specific heat capacity

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1 Introduction

Increased environmental awareness and social interest, energy and raw material consumption from unsustainable fossil sources, and new environmental regulations have resulted in an increased demand for environmentally friendly materials. At the same time, social sensitivity has developed to point that the reduction of the environmental cost of conventional synthetic fibre-reinforced composite materials, among many other such materials, is now considered a social responsibility (Donnell et al. 2004; Low et al. 2007). This demand has created new requirements such as the desire or need for environmentally friendly materials, products, and processes. Recently, considerable attention has become focussed on "green" composites that consist of natural or synthetic resins reinforced with natural fibres and wastes (Bledzki and Gassan 1999). A portion of the filling and reinforcing materials used in this study are from natural materials and wastes, some examples of these natural materials being organic wastes such as sisal, jute, hemp, and kapok fibres (Mohanty et al. 2001; Mishra et al. 2002; Mwaikambo and Ansell 2002; Hepworth et al. 2000).

In their study, Cerqueiraa et al. (2011) considered the mechanical properties of sugar cane bagasse fibre/PP composites, demonstrating that composites had improved tensile, bending and impact strengths compared to their pure polymer precursors. Teja et al. (2016) investigated the effects of the addition of silicon carbide to natural sisal fibres on the mechanical and thermal properties of the composites produced; polyester was used as a matrix. Experimental results showed that tensile strength, impact strength, thermal conductivity, thermal stability, and thermal propagation ability increased, whereas specific heat capacity first increased and then decreased. Venkatram et al. (2016) evaluated the sisal-nanoclay composites' mechanical properties, namely its tensile, bending and impact strengths, showing that the addition of nanoclay into sisal fibre/GP may improve its properties and that it can be used as an alternative to glass fibre-reinforced polymer composites. In their study with banana-jute/epoxy hybrid composites, Devireddy and Biswas (2016) investigated the effects of the fibre content on density, specific heat, thermal conductivity, thermal expansion, thermal stability, and water absorption, confirming results reported in the general literature accumulated by experimental, numerical simulation, and analytical methods. The measured properties of hybrid composites were found to be suitable for use in automobiles and building components to reduce energy consumption. Dong et al. (2015) characterized the thermal properties of untreated and treated linen threads with differential scanning calorimeter and thermal gravimetric analysis, showing that the thermal stability of linen threads were increased and the glass permeation temperature (T_g) decreased; further, a significant endothermic peak was observed with untreated linen threads, while a significant exothermic peak was observed for a different heat range for treated linen threads. Hurtado et al. (2016) evaluated the physical properties of cellulose insulation, and environmental factors affecting these properties and possible future innovations in their review essay. Although these materials appear to offer good thermal properties, they are not being used as widely as traditional insulation materials due to lack of expertise on application and knowledge on properties. In their study examining the hygrothermal behaviours of bio-based construction materials, Aslı et al. (2019) investigated the heat and mass transfer of various materials in a porous medium under both dry and wet conditions, using a mathematical model to define mass transfer that used the material properties obtained from the characterization section as input parameters. The results showed conformity between the theoretical and measured data. Mohapatra et al. (2017) determined the thermal conductivity of a pine wood powder/epoxy composite experimentally forced convection apparatus. The addition of pine wood powder resulted in a decrease in thermal conductivity; an increasing amount of powder was found to decrease thermal conductivity further. In the study, a polyester matrix material with good mechanical properties and low cost was used. In the choice of matrix material, it is important that the thermal conductivity is lower (Gu et al. 2012) than epoxy resin (0.202 W/mK).

In this study, industrially unusable organic wastes (hazelnut shell, corn stalk, nettle fibre and nettle stalk) have been used in the production of composites with a polyester matrix as the filling material, on which three-point bending and impact tests have been carried out according to ASTM E-399 and EN 826 standards. The mechanical and physical features of the composites are explained according to density, bending stress, bending modules and compression strength, and their thermal features defined via DSC analysis and by measuring their thermal conductivity coefficients.

The purpose of this study is to determine a suitable material for structural applications from these composites by determining the mechanical and thermal properties of composites produced from various wastes (nutshell, nettle fibre, nettle stalk particulates) that, to the best of our knowledge, have not previously been studied.

2 Materials and Methods

2.1 Material

Ground nutshell powder was used to fabricate the nutshell-polyester composites. The nutshell, as obtained from hazelnut cracking factories, was ground in local mills. The corn stalk residues were collected from the fields and, after drying, the inner and outer parts were separated from each other and powdered using a chopper, and then subsequently used for composite production. Stinging nettle fibre was obtained from the stinging nettle (*Urtica dioica* L.) naturally grown in the Black Sea region. A mechanical method was used to obtain the fibres. The fibres, cut to about 0.5 mm in size with an industrial chopper, were used in the production of the associated composites. After the fibres of the stinging nettle were separated, the stalks were crushed and powdered and used for composite production. The chemical compositions of the organic wastes used to form the various composites are summarized in Table 1.

The polymer matrix material (UN1866) used to create the composites was provided by Poliya. Cobalt and MEK-P (methyl ethyl ketone peroxide) were used to harden the polymer matrix material. The rate of accelerator and hardener over polymer mass is 0.1% for 6% Cobalt and 1% for MEK-P. The mechanical properties of the pure polymer are also reported in Table 1 (Poliya 2017).

In order to be able to correctly analyse the organic wastes pulverized with the chopper, the wastes were sieved with a 425 µm sieve and then with a 600 µm sieve. The powders obtained with sizes between 425 600 µm were then used to form the various composites studied.

Table 1 Mechanical and physical properties of solid Polyester

Water absorp. 20 °C (%)	Bending Strength (MPa)	Bending Modulus (MPa)	Elong. Bending (%)	Tensile strength (MPa)	Tensile modulus (MPa)	Elong. Tensile (%)	Impact Strength (kJ/m^2)	Density gr/cm^3 23 °C
0.17–0.19	145	4800	5.5	57	2950	2.6	10	1.1

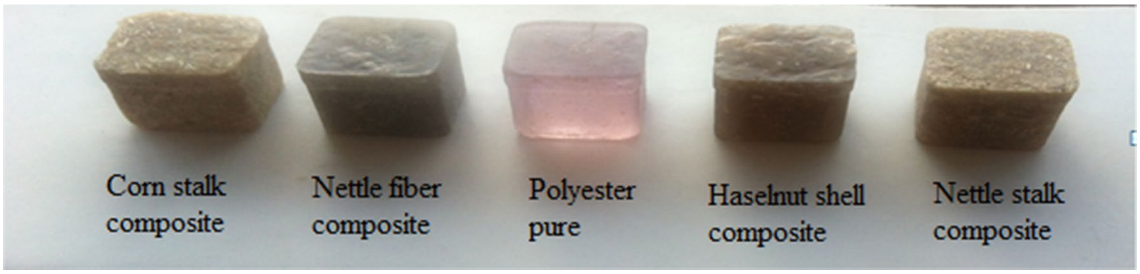

Fig. 1 Composite samples

Table 2 The elemental analysis method of the samples

Element	Method
Alpha cellulose	Wise's chloride meth.
Holocellulose	TAPPI T 203 os-71
Lignin	TAPPI T 222 om-88

To produce the composites, hazelnut shell, corn stalk, nettle stalk and nettle fibres were added to liquid matrix material in proportions of 5%, 10%, 15% and 20% by weight. The liquid mixture so prepared was poured into the moulds to obtain untreated test samples which were then kept in an oven at 80 °C for 24 h to allow for final curing. Samples' surfaces were fixed with a sanding machine to prepare them for testing (Fig. 1).

2.2 Method

2.2.1 Elemental Analysis and Density

Chemical analyses were performed using the methods given in Table 2 in the wood chemistry laboratory of Karadeniz Technical University Forest Industry Department (Tutuş et al. 2017)

Density measurement was determined using a liquid picnometer. Pure acetone was used as the liquid in the measurement method.

2.2.2 X-Ray Diffraction Analysis

X-ray diffraction was performed via an advanced Diffractometer (Europe 600 XRD) using Cu Ka radiation at 40 kV and 30 mA. The scanning interval chosen to be 10–30, step size as 0.02 and scanning speed as 0.4/min. Crystalline percentages of the materials were determined using numeral data obtained and Eq. 1 (Segal et al. 1959)

$$\mathrm{CrI} = (\mathrm{I}_{002} - \mathrm{I}_{am}) \times 100/\mathrm{I}_{002} \quad (1)$$

Here, I_{002}, is the maximum point of the 002 crystalline peak and I_{am}, is the lowest point of amorphous material between the 101 and 002 peaks.

2.2.3 Determination of Heat Conductivity (λ)

Thermal conductivity, as dependency on different sample content, was measured via Lasercomp HFM Fox-50 heat flow meter device measuring under the conditions described by the ASTM C518, EN 12664 and ISO 8301 standards. The working principle of this device is given in Fig. 2.

When performing the test via the HFM device, a sample is placed between two plates held at different temperatures. These plates are kept at a constant temperature by way of the Peltier effect. Thermal flow along the sample and temperatures are measured via the heat flow meter and thermocouples located on the upper and lower plates of the device. When the HFM as at thermal equilibrium and has a uniform temperature gradient, the thermal conductivity can be determined. The thermal conductivity measured by the HFM device is determined by the Fourier thermal conductivity equation below.

$$q = -kA\frac{\Delta T}{\Delta x} \quad (2)$$

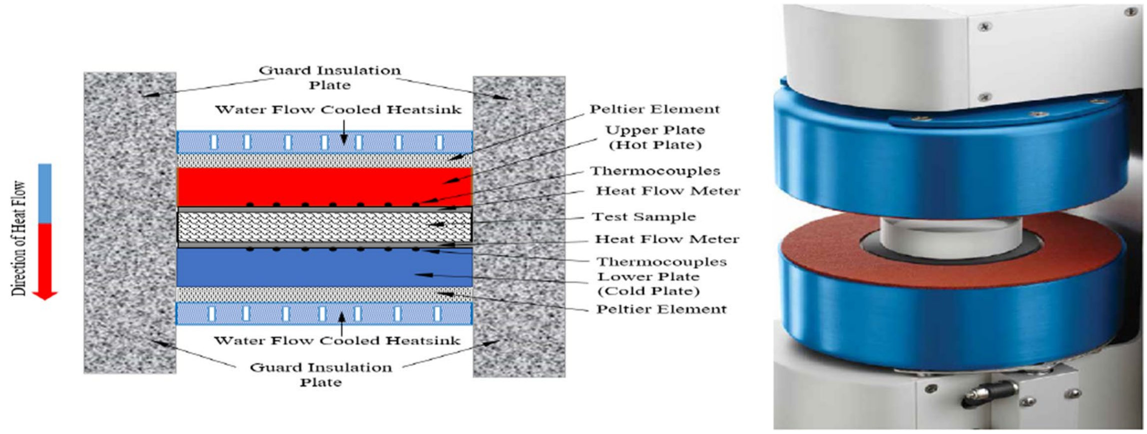

Fig. 2 Schematic diagram of heat flow meter

Here, q (W/m^2) is heat flow, k (W/mK) is thermal conductivity, A (m^2) is area, ΔT (K) is temperature difference and x (m) is sample thickness. HFM operates by measuring, recording, and printing using this equation and an appropriately calibrated piece of software, where the device is calibrated using Pyrex-7740, for which the thermal conductivity is accurately known for various temperatures, and also using a Pyrex calibration file. In addition, the reliability of the results and repeatability of the measurements have been checked.

2.2.4 Determination of Compressive Stress at 10% Relative Deformation

Compression strength of the samples are determined in accordance with the requirements of EN 826 (ASTM 2013). According to this standard, sample test specimens of various dimensions may be chosen. The test specimens are cut into cubes of dimensions of 30 mm × 30 mm × 30 mm. In order to ensure close contact between the equipment plates and the test specimen surface, the test specimen is preloaded with a pressure of 250 ± 10 Pa (Fig. 3).

The test specimen compression period, i.e., the rate at which the force increases, is dependent on the test specimen height because it is compressed at a constant rate of $0.1 \times d$ (where d is the thickness of test specimen expressed in mm) per min with a tolerance of ± 25%.

The compression test is continued until the test specimen reaches a 10% relative deformation. Thus, compressive stress (σ_{10}) is determined at a 10% relative deformation.

2.2.5 Determination of Bending Strength

Bending strength (σ_f) was measured for all the composite types. For each type, a three-point bending test was carried out according to European standard EN 12089 (ASTM 2013) on three specimens (of dimensions 30 cm × 40 cm × 100 cm), using an loading machine (maximum load 5 kN) at a constant displacement rate of 10 mm/min (Fig. 4).

Fig. 3 Compressive stress test at 10% relative deformation

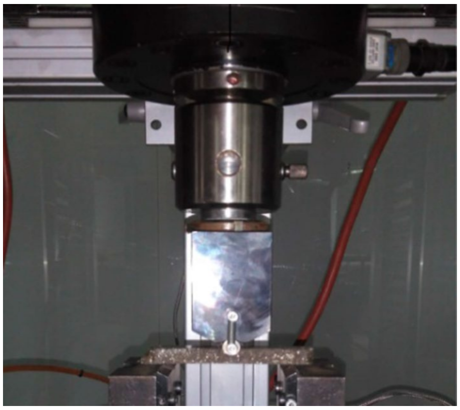

Fig. 4 Bending strength test

3 Results and Discussions

3.1 Elemental Analysis and Density

It is known that the number of elements and physical conditions of a material significantly affect its thermal behaviour (Tian et al. 2014). Elemental analysis and density measurements for the materials used to produce composites are provided in Table 3.

3.2 X-Ray Diffraction Analysis of Nettle Fibre

The XRD pattern of a typical crystal cage of cellulose within corn stalk and nettle stalk is presented in Fig. 5a, b (given as image files in additional material 6 and 7). XDR data forhazelnut shell and nettle fibre are presented in additional files 8 and 9. The hazelnut shell, nettle fibre, corn stalk, and nettle stalk crystallinity in dices were found to be, respectively, 66%, 63%, 54.6%, and 40%. In previous studies, the same method determined Guerreroa sisal's crystalline index to be 60% (Benítez-Guerreroa et al. 2017) and Jonoobi's kenaf's crystalline index to be 41% (Jonoobi et al. 2010).

In the study, it was confirmed that the degree of crystallinity of the material and thermal conductivity have a linear relationship. The results obtained reflect typical findings in the literature. Thermal conductivity is often lower in amorphous structures whose interactions are often characterized by the van der Waals forces of randomly oriented chain structures than by well-arranged polymer crystals (Kittel 2005). It was found that the relative degree of crystallinity had an indirect effect on in-plane thermal propagation. The increase in crystallite size is indicative of greater thermal propagation on a larger plane, which is referred to as the crystallite size effect. Size effect is well known when considering micro/nano scale thermo physical properties (Peng and Wu 2010).

3.3 DSC Analysis

DSC analysis for pure polyester and composites with 10% reinforcement are shown in Fig. 6, from which it can be seen that the glass transition heat of the pure polyester is lower than that of the composite.

DSC analyses of the composites are provided in Table 4. In general, when the proportion of reinforcement material added to the matrix increases, glass transition temperature also increases. These results are corroborated by those in the literature (Garcia–Garcia et al. 2018).

These increases are, respectively, 16% and 23% in polyester/hazelnut shell composites, 17% and 48% in polyester/cornstalk composites, 24% and 53% in polyester/nettle fibre composites in proportions of 0%/20%. The glass transition temperature increased by 10% in polyester nettle stalk composites; however, a partial decrease was found to occur at higher proportions. The change rates in these composites are 13% and 8%. Similarly, the literature suggests that the glass transition temperature decreases as reinforcement material is added to the matrix (Tajvidi et al. 2006). In this study, as the amount of nettle stalk in the composite increased over 10%, it was observed that the glass transition temperature decreased.

Low-crystalline cellulosic materials contain high levels of moisture. The amount of water contained in the reinforcing material causes the formation of areas without a polymer matrix in the composite, thus shifting T_g to lower temperature (Wypych 2012). In addition, an increase in the amount of reinforcing material causes agglomeration and prevents diffusion of cellulose molecules into the chains of the polymer matrix, which similarly reduces T_g (Borysiak 2013).

Table 3 Results of elemental analysis for organic waste

Material	Holocellul (%)	α-cellulose (%)	Hem.cell (%)	Lignin (%)	Density (g/cm^3)
HazelnutShell	54.1	32.68	21.42	35.84	1.24
Corn stalk	67.50	44.49	23.1	20.2	0.72
Nettle fibres	76.46	55.74	20.72	14.69	1.17
Nettle stalks	74.79	36.62	25.21	25.21	0.58

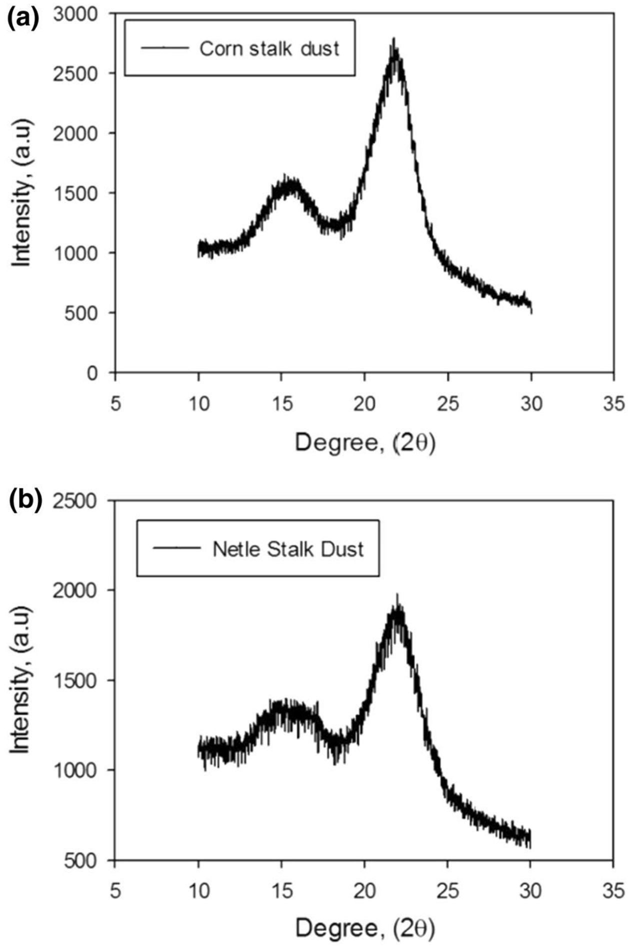

Fig. 5 **a** XRD image of Corn stalk. **b** XRD image of Nettle stalk

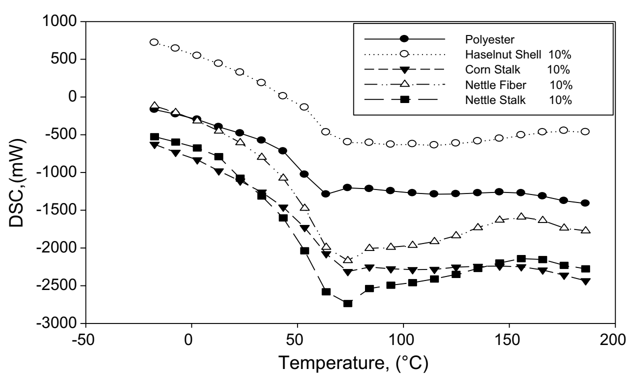

Fig. 6 DCS graphics for pure polyester and composites with 10% reinforcement

3.3.1 Specific Heat Capacity

The specific heat values of composites produced with different organic fillers are given in Fig. 7.

Table 4 DCS analysis of the composites

Reinforc. proporti.	Hazelnut shell	Corn stalk	Nettle fibre	Nettle stalk
0	50.9	50.9	50.9	50.9
10%	58.9	59.4	62.9	57.5
20%	69.1	75.3	78.0	55.5

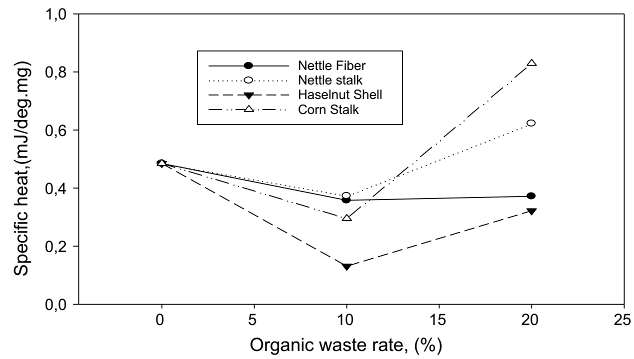

Fig. 7 Specific heat values of composites

In the study, by increasing the amount of organic waste added to the composite from 10 to 20%, the specific heat capacity of the composite increased by 59.3%, 3.5%, 66.7%, 181%, respectively, in the additions of hazelnut, nettle fibre, nettle stalk and corn stalk. The density of nettle fibre having a density similar to the binder polyester and the increase in the amount of additives increased the specific heat value to a limited extent, while nettle stem part which is lower in density increased the specific heat capacity at a higher value. Heat conduction results are not consistent with specific heat increase in corn stalk composites. The reason for the high rise of specific heat is probably due to the internal structure of the composite. In a study conducted by (Bertoncelj et al. 2015) it is stated that the specific heat increases with the temperature when the amount of glass fibre additive increases by weight. Again, in some cases, they associated the low specific heat increase with the inhomogeneity of the structure by scanning electron microscope images.

The specific heat capacity of pure polyester is higher than some composite products. However, higher specific heat capacity was determined than pure polyester in cases where nettle stalk and corn stalk additive amounts were 20%. This is due to the low thermal conductivity of the additive material with the increasing amount of additives. In a similar study using different materials, it was stated that the low specific heat capacity was caused by the high thermal conductivity of the multilayer carbon-nano tube reinforced layers (Korkmaz et al. 2016).

According to the results, the increase in the specific heat capacity with the decrease in the thermal conductivity of the polyester, added with organic waste, is a desirable situation for structural applications because the increase in thermal resistance together with the decrease in thermal conductivity for the composite product increases the usability of the produced composite. With the increase in the specific heat capacity, the heat retention potential of the composite material increases. Thus, the decrease in the temperature difference that may occur and the potential of the produced composite to become a building component increases.

3.4 Unit Mass

The change in composite density with filler is given in Fig. 8. Hazelnut shell composites increased considerably in density, while nettle fibre increased slightly, whereas nettle stalk and corn stalk composites each showed a decreased density. Changes in density with increasing amount of nettle stalk, corn stalk, nettle fibre and hazelnut shell composites are −9%, −7%, 1% and 4.6%, respectively. These values are lower than Lightweight Concrete Masonry densities (ASTM 2011) stated in literature.

3.5 Bending Strength and Modulus

The change in bending strength of organic waste/polyester composites with filler amount is shown in Fig. 9. Due to the increase in amount of filler, the bending strength was found to decrease rapidly up to 5%. The decreases were 68%, 80%, 74% and 63% for nettle stalk, corn stalk, nettle fibre and hazelnut shell composites, respectively. After these ratios, the increased tension of some composites resulted in a final decrease of 58%, 77%, 74%, and 73% in the materials with 20% additives compared to the pure matrix. It is thought that one of the reasons for the limited increase is that the hydroxyl groups of hemicelluloses (Peng and Wu 2010) interact with the matrix and thus increase the mechanical properties of the composite by strengthening the matrix-fill interface strength.

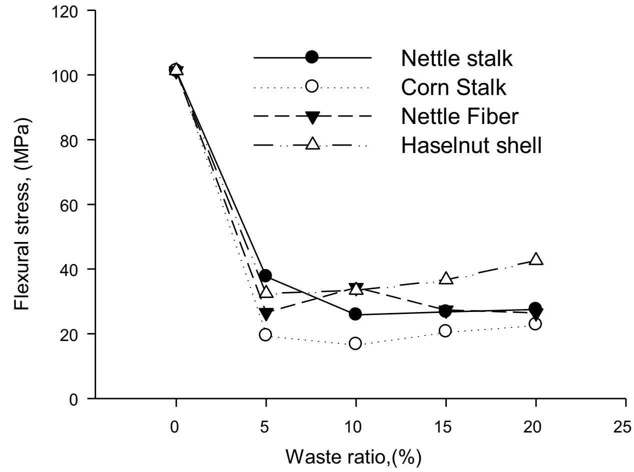

Fig. 9 Flexural stress of organic waste-polyester composites

When the bending strengths of the polyester composites made with four different reinforcement materials were evaluated, partial increases in bending strengths were found to occur due to the increase in the amount of additive in the hazelnut shell composites. Correspondingly, it was found that the decrease in nettle stalk and nettle waste was less than the decrease for the corn wastes. The results obtained in the studies with organic wastes were paralleled by the decrease in bending stress in this study (Dong and Davies 2012).

The bending moduli of the composites are given in Fig. 10. In all the composites created regardless of additive, the bending modulus increased up to a 5% additive rate. The

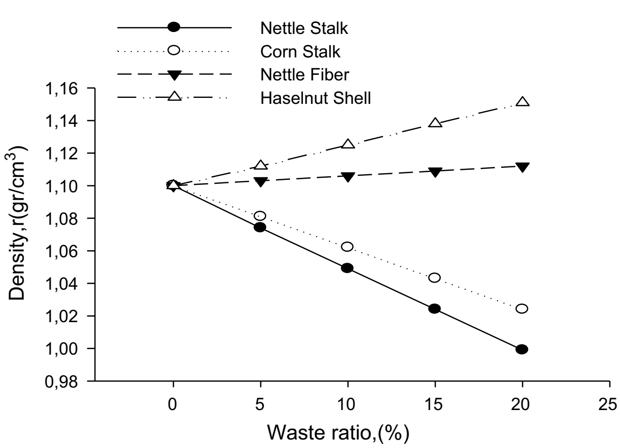

Fig. 8 Change the amount of filler involved with density of the composite

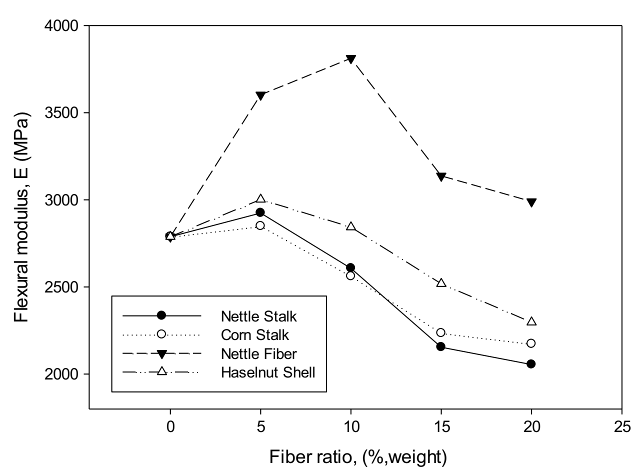

Fig. 10 Flexural modulus of organic waste/polyester composites

increase for the nettle fibre-reinforced composites continued up to 10%. It was then found that the bending elasticity modulus decreased in the composites prepared with reinforcing materials other than nettle fibre. The decreases were 26%, 22%, and 18% in the nettle stalk, corn stalk and hazelnut shell composites, respectively, at the maximum additive proportions. The final increase in the composites prepared with nettle fibre was 7%. These results are corroborated by the decline in the bending moduli of composites with a grain size of over 250 µm reported in the literature (Stark and Berger 1997). Since the size of the wastes used in our study is 425–600 µm, the decrease in bending strength modulus with increasing proportion of additive was consistent with the literature (Kumara et al. 2014). As the nettle fibre is different from other wastes due to its shape, composites with 20% additive saw an increase compared to the pure matrix despite the decrease in the bending modulus.

In studies into the bending modulus of composites, it has been found that the modulus increases for low proportions of additive; on the other hand, increasing the amount of additive increases the surface area and the matrix-reinforcement connection decreases as a result of a decreasing wetting effect of the matrix. As a result, the bending module decreases (Shibata et al. 2005).

3.6 Compression Stress

The compression stress durability is given in Fig. 11 for the four different composites formed using hazelnut shell dust, corn stalk, nettle stalk and nettle fibres. Although it was observed that the compression stresses for each of the four composites decrease rapidly by 5%, where this decrease is slower in composites with nettle stalk reinforcement; therefore, the compression stress of composites with other wastes result in a quicker decrease in compression stress. When maximum reinforcement rates are considered, the individual decreases in compression stress in composites can be determined to be 62% for hazelnut shell dust, 64% for corn stalk, 59% for nettle stalk, and 43% for nettle fibres. Rasat et al. (2011) reported that the composites they produced with agricultural waste and palm oil leaves increased the compressive strength.

In the various studies conducted, it has been reported that the strengths of particle-reinforced composites depend on whether the material is isotropic and on the matrix-interface bond strength (Teng 2010).

3.7 Thermal Conductivity

The graphs of the composites examined in terms of thermal conduction coefficients are given in Fig. 12. A decrease in heat transfer coefficients were observed in the composites produced with nettle stalk and corn stalk, which were 14% and 8% at the maximum additive proportions compared to the pure matrix material. In other composites, the increase in the amount of filler increased the thermal conduction coefficients. Accordingly, at the maximum additive proportions for hazelnut shell and nettle fibre composites, the increase was 22% and 5%, respectively. The results of studies with hazelnut shell and nettle fibre composites are similar to those of cellulose-based composites (Hossain et al. 2014).

This study has showed that although being cellulose-based, the composites formed with different wastes have different thermal conductivities, and that this difference is primarily defined by the chemical and physical properties of the material. In related studies, it was also noted that a good thermal conductivity in a composite or mixture is related to the conductivity of its components (Agarwal et al. 2003; Nan et al. 1997). Moreover, it has been suggested in such studies that physical properties such as fibre length, fibre length–width ratio, and variables such as relative the moduli of the fibre and matrix and thermal

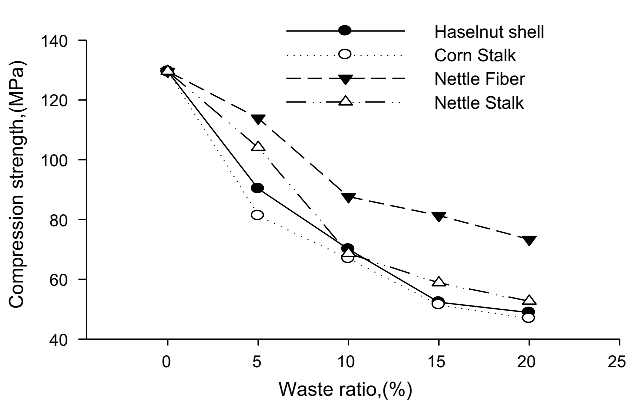

Fig. 11 Compression stresses of hazelnut shell dust, cornstalk, nettle fibre, nettle stalk waste-polyester composites

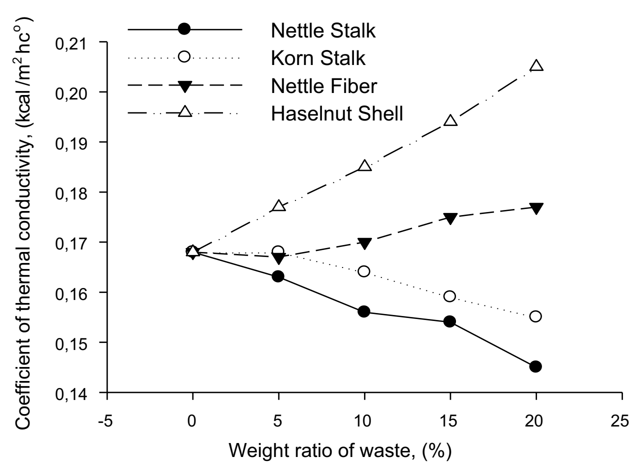

Fig. 12 Thermal conductivity of hazelnut, cornstalk, nettle stalk, and nettle fibre waste-polyester composites

expansion incompatibility can control the properties of a given composite (Hancox 1981; Wakili et al. 2003) determined the thermal conductivity coefficients of six different insulation materials using the heat flow meter (HFM) method.

Such studies have also stated that specific heat capacities for the same insulation materials can be determined via the HFM method. The thermal conductivity coefficients of 32 insulation materials were determined by Abdou et al. (2005) for an average temperature difference of 35 °C, using the heat flow meter method in accordance with the ASTM C518 standard and the ISO 8301 protocol. Measurements were taken for insulating materials at five different (4, 10, 24, 38, 43 °C) temperature sand different densities in the 4–43 °C temperature range. Generally, high-density insulation materials showed low thermal conductivities, and in all cases the conductivity (k) increased with increasing temperature. The authors emphasized that the change of k with temperature is more pronounced at low material densities.

4 Conclusions

According to the results, the aim of producing materials which can be used in construction-related applications by decreasing their thermal conductivity without affecting the mechanical features of the composites below the values mentioned in the appropriate standards, was achieved in this study.

A linear relationship between the densities of waste reinforcements and thermal conductivity coefficients was found. Accordingly, the composites produced with nettle stalk waste, having the lowest density, are appropriate for construction-related applications as a green isolation material with a low thermal conductivity coefficient and that are very lightweight.

Additionally, the use of a waste (nettle), growing naturally under the nut tree and that would otherwise be cleared with pesticides, provides a double benefit as it can be turned into an environmentally efficient constructional material, reduce or negate the need for pesticides, and can also be used as an alternative to fossil oil-based isolation materials.

Finally, the results suggest that the density of 20% nettle stalk-reinforced composites decreased by 9% according to the matrix. Moreover, the thermal conductivity coefficient of these composites indicates a 14% decrease with the same amount of reinforcement. As can be concluded from the various superior properties of nettle stalk composites, nettle stalk composites are appropriate for use in structural applications and can allow for the reuse of wastes and the use of 'green', light and energy saver construction materials.

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