

Determination of mechanical and damping properties of hazelnut shell powder reinforced biocomposites by ultrasonic method

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Abstract

This research was carried out to figure out the effect of chemical treatments of hazelnut shell powders (HSPs) on the elastic properties, ultrasonic wave velocities, and damping properties of bio-based epoxy resin (BER) biocomposites. Natural hazelnut shells (HSs) were chemically treated using sodium hydroxide (NaOH), and acetic anhydride (AA). Then, HSs that were chemically treated with NaOH and AA, and HSs that were not subjected to chemical treatment were ground to obtain HSPs. The treated HSPs (HSP-NaOH and HSP-AA), and untreated HSPs were contributed to the neat BER in varied compositions such as 10–50 wt% to obtain the BER/HSP, BER/HSP-NaOH, and BER/HSP-AA biocomposites. The effect of untreated, and treated HSP ratios on the density, ultrasonic wave velocities, Young's modulus, Bulk modulus, Shear modulus, Poisson ratio, microhardness, and damping characteristics (attenuation coefficient, loss tangent and quality factor) of the novel HSP-based biocomposites, was investigated by the ultrasonic pulse-echo overlap method (PEOM). A significant increase in the density, ultrasonic wave velocities, and elastic modulus values of the biocomposites was seen compared to the neat BER. Based on the obtained elastic modulus values, the most appropriate combination ratio between the neat BER, and HSP-NaOH was determined as 50:50.

KEYWORDS

biocomposite, mechanical properties, non-destructive testing, ultrasonics

1 | INTRODUCTION

In the 21st century, eco-friendly composites made from recyclable sources have become a quite popular research field. Especially, bio-filler reinforced polymer matrix is an interesting area of research. Consequently, many types of research have been conducted to fabricate biocomposites.^{1–3} The bio-fillers used in eco-friendly biocomposites are plentiful,

inexpensive, light, and have high specific modulus.⁴ The lighter biocomposites, which reduce fuel consumption, have been used in aircraft interiors, drug releases, military goods, food packaging systems, etc...^{5,6} Although many advantages of biocomposites, their mechanical properties have not reached the desired level yet due to the insufficient adhesion between the hydrophobic structures of most polymers and the hydrophilic structures of bio-fillers.^{7–9} Lignocellulosic materials can

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integrate effectively with the matrix because of chemical modification, and alteration of hydroxyl groups.¹⁰ Thus, different chemical modification methods have been applied for lignocellulosic materials.^{11,12} It is stated that treatment with sodium hydroxide (*NaOH*) removes non-cellulosic components from the surface.¹³ Therefore, many researchers carried out alkali treatments on different natural bio-fillers. For example, Van de Weyenberg et al.¹⁴ stated that the mechanical properties of the flax fiber-reinforced composite were enhanced by about 30% using alkali treatment. Similarly, Hittini et al.¹⁵ produced polystyrene (PS)/date pit powder (DPP) biocomposites. As a result of the treatment of the DPP with sodium hydroxide, they have determined an improvement of around 4.2%, 190%, and 55% in compressive strength, tensile, and flexural strength, respectively. Arrakhiz et al.¹⁶ treated the coconut shell fibers with *NaOH*. They have figured out that the fiber-matrix adhesion is enhanced. Green epoxy resin (GER)/date stone flour (DSF) biocomposites were produced in the research where DSF fillers were treated with different chemicals such as alkaline, benzoyl chloride, and potassium permanganate.¹⁷ It was reported that the elastic properties of the GER/DSF biocomposites were significantly increased. These research results revealed that the elastic properties of GER/DSF biocomposite were most achieved by treating DSFs with potassium permanganate. Additionally, different acid treatments have been conducted on bio-fillers.^{18–20} For example, Melo et al.²⁰ determined that the functions of lignocelluloses increased with the treatment of bio-fillers with acetic acid (AA). Additionally different acid treatments are conducted on bio-fillers. In the chemical modification process of lignocellulosic substances with acid, the —COOH group of the acid enters the esterification reaction with the —OH groups of cellulose. Such chemical modifications cause the appearance of different reactive groups on the lignocellulosic material surface, facilitating the bonding of the filler with the matrix, that is, “wetting” by the matrix, resulting in composites with better mechanical properties.^{1,21}

Although the exact determination of all elastic coefficients of materials is not possible with destructive testing (DTs), the influence of chemical treatments and biofillers on the elastic properties of biocomposites is still generally driven by DTs. Instead of these DTs, ultrasonic testing (UT) method, which is both more economical and more sensitive, has been developed. UT method has been proven in isotropic material characterization.^{2,22,23} Recently, different biocomposites have been investigated using UT methods.^{2,17,24} For example, Souissi et al.²⁴ determined the Young's modulus of a polypropylene (PP)/olive wood flour (OWF) biocomposites using both of ultrasonic and mechanical methods. Similarly, El-Sabbagh et al.²⁵ mixed the flax chopped fibers with PP at different ratios ranging from 0–50 wt.%. They investigated the effect of flax fiber ratio using ultrasonic longitudinal wave velocity and

attenuation. They revealed an increase in both longitudinal wave velocity and attenuation values as well. On the other hand, the flax/PP fibers nonwoven composites were produced to develop lighter biocomposites by Merotte et al.²⁶ They were carried out the acoustic and tensile test to determine the effect of porosity content in the biocomposites.

To the best of the authors' knowledge, bio-based epoxy resin (BER)/hazelnut shell powder (HSP) biocomposites have not been characterized for their mechanical and damping characteristics using ultrasonic wave velocities. Thus, in the first part of the research, a novel bio-based epoxy resin (BER) was synthesized, and hazelnut shells (HSS) were treated with both sodium hydroxide (*NaOH*) and acetic anhydride (AA). In the second part of the research, BER/HSP, BER/HSP-*NaOH*, and BER/HSP-AA biocomposites were obtained with a contribution in the range of 10–50 wt% of untreated and treated HSPs with the BER. Finally, the effects of the treatment method and proportions of HSPs on the mechanical and damping properties of the BER/HSP biocomposites were determined using ultrasonic wave velocities. Also, SEM and XRD analysis were used for the morphological characterization of the biocomposites obtained.

2 | EXPERIMENTAL SECTION

2.1 | Materials

A novel synthesized bio-based epoxy resin (BER)² was used as the matrix material. Also, the 2,4,6-tris (dimethyl aminomethyl) phenol, and the aromatic *m*-xylene diamine (MXDA) were used as accelerator and hardener, respectively. A basic analytical mill (IKA A11) and a 230-mesh sieve were used for grinding Hazelnut shells (HSS) provided from the nearby local market to obtain hazelnut shell powder (HSP) bio-fillers with particle size <63 μm .

2.2 | Production process of the BER

BER was synthesized by the esterification reaction using bio-based sebacic acid and epichlorohydrin. The detailed information about synthesis and characterization was given in the previous study of authors.²

2.3 | Chemical treatment of hazelnut shells

2.3.1 | Sodium hydroxide (*NaOH*) treatment

Since an alkali concentration ratio of more than over 6% significantly reduces lignocellulose fiber strength,¹²

untreated hazelnut shells particles which were cleaned from waste and crusted into small pieces were kept in 5% NaOH solution for 72 h with shaking at room temperature. Afterward, particles were washed with distilled water, dried, and sieved after grinding. Thus, alkali-treated hazelnut shell powder (HSP-NaOH) was obtained.

2.3.2 | Acetic anhydride treatment

HSP-NaOHs were kept in glacial acetic acid for 1 h, then separated by decanting and soaked in acetic anhydride (AA) containing 2 drops of concentrated H₂SO₄ for 2 min. After, particles were removed, cleaned with water, and dried at 80°C. Finally, the hazelnut shells treated with AA (HSP-AA) were milled and sieved.

2.4 | Preparation of biocomposites

Crushed raw HSs were ground and sieved ($\leq 63 \mu\text{m}$) to obtain untreated filler HSP. Afterward, the HSPs were added to the BER at different ratios (10–50 wt%) and blended by mechanical stirring for 1 h. An ultrasonic bath was applied to this mixture for 1 h at 60°C. After that, 30 wt% epoxy curing agent, and 1 wt% of accelerator are contributed to the mixture as well. This whole mixture was blended again for 15 min. Finally, the samples were obtained by transferring these mixtures in the stainless steel molds which have been prepared based on the ASTM D638-14.²⁷ The obtained the BER/HSP biocomposite samples (*BC-1*, *BC-2*, *BC-3*, *BC-4* and *BC-5*) were cured at 60°C for 1 h. The same procedure was carried out by the addition of HSP-NaOH and HSP-AA into BER at different ratios (10–50 wt. %) to obtain the BER/HSP-NaOH biocomposites (*BC-6*, *BC-7*, *BC-8*, *BC-9* and *BC-10*) and the BER/HSP-AA biocomposites (*BC-11*, *BC-12*, *BC-13*, *BC-14* and *BC-15*).

2.5 | Characterization

2.6 | Morphological and chemical structure analyses

Surface morphologies of the BER and biocomposites (BER/HSPs, BER/HSP-NaOHs, and BER/HSP-AAs) were investigated with Scanning Electron Microscope (SEM) device (Zeiss Evo LS 10, Germany) using ASTM E766-14²⁸ magnification standard. On the other hand,

the X-ray diffraction (XRD) analysis which uses the Cu-K α radiation (power source = 40 kV, $\lambda = 1.5406 \text{ \AA}$,) was used for the XRD analysis of the obtained materials (Bruker D8 Advance, Germany).

2.6.1 | Measurements of densities

A semi-analytical balance (Radwag AS220/C/2, Poland), and a kit of density (Radwag 220, Poland) were used for density measurements of the BER and biocomposites. Density measurements were conducted based on Archimedes' principle using the ASTM D792-20.²⁹ Density measurements were done ten times to increase the accuracy value of measured densities.

2.6.2 | Measurements of ultrasonic wave velocities

Ultrasonic wave velocities in obtained biocomposites were determined based on ASTM E494-20³⁰ by the pulse-echo-overlap method (PEOM).³¹ A pulser/receiver with 35 MHz (5800PR-Panametrics Olympus, USA), an oscilloscope with 60 MHz (GW Instek GDS-2062, Taiwan), a longitudinal wave transducer with 20 MHz (V116-Panametrics Olympus, USA), and a shear wave transducer with 5 MHz (V155-Panametrics Olympus, USA) were used in as part of velocity measurements. The glycerin (BQ Panametrics Olympus, USA) and shear wave (SWC Panametrics Olympus, USA) coupling fluids were applied between transducers and biocomposite samples for longitudinal wave and shear wave velocities measurements, respectively. Equation (1) was used for the calculation of longitudinal and shear wave velocities. Velocity measurement was carried out ten times for each sample to increase the accuracy value of measured velocities.

$$v = \frac{2d}{\Delta t} \quad (1)$$

where v , d , and Δt are the ultrasonic wave velocity, the thickness of the sample, and the time-of-flight between subsequent back wall signals of the oscilloscope, respectively.

2.6.3 | Measurements of elastic properties

The elastic moduli and Poisson's ratios of all materials used in this research were calculated using the Equations (2–6)^{32–34}:

$$L = \rho v_L^2 \quad (2)$$

$$G = \rho v_s^2 \quad (3)$$

$$K = L - \frac{4}{3}G \quad (4)$$

$$\mu = \frac{L - 2G}{2(L - G)} \quad (5)$$

$$E = 2G(1 + \mu) \quad (6)$$

where L , G , K , μ , and E are longitudinal modulus, shear modulus, bulk modulus, Poisson's ratio, and Young's modulus, respectively.

On the other hand, the characteristic acoustic impedance (Z), and ultrasonic micro-hardness (H) values of all materials were calculated using Equation 7 and 8³²⁻³⁴ as well:

$$H = \frac{(1 - 2\mu)E}{6(1 + \mu)} \quad (7)$$

$$Z = \rho v_L \quad (8)$$

2.6.4 | Measurements of damping properties

The attenuation constants (α) of all materials used as a part of this research were calculated using amplitudes of a sinusoidal ultrasonic longitudinal wave obtained with a longitudinal wave transducer having 20 MHz frequency. The attenuation constants of all samples were calculated using Equation (9)^{35,36}:

$$\alpha(Np/m) = \frac{Ln\left(\frac{A_m}{A_n}\right)}{2d(n - m)} \quad (9)$$

where A_m and A_n , are amplitudes of m^{th} and n^{th} back wall reflections ($n > m$) and d is the thickness of the sample in a unit of the meter. α constant is related to the loss tangent ($\tan\delta$) and the relationship between α and $\tan\delta$ is defined with Equation (10)³⁷⁻³⁹:

$$\tan(\delta) = \frac{2\alpha v_L}{\omega} = \frac{\alpha v_L}{\pi f} \quad (10)$$

where f , α and v_L are frequency, attenuation coefficient, ultrasonic longitudinal wave velocity, respectively. The viscous damping that causes attenuation in polymers is related to loss tangent, as well.³⁸

Since the Quality factor (Q) value gives important information about the molecular mobility in the polymer chain in polymeric materials and the strength of the bonds between the matrix and the filler material in polymer composites, Q values of biocomposites were determined performing Equation (11)³⁷:

$$Q = \frac{1}{\tan(\delta)} = \frac{\pi f}{\alpha v_L} \quad (11)$$

3 | RESULTS AND DISCUSSION

3.1 | SEM analysis results

Figure 1 displays the morphology of the synthesized BER, and biocomposites. The surface of neat BER seems slightly rough (Figure 1a), and unlike Bisphenol type epoxy resins, it does not have a structure in the form of overlapping layers, which indicates a brittle structure.⁴⁰ Since the surface can hinder crack propagation, the BER has a slightly rough surface indicating a more ductile structure. The samples with unmodified HSP had a relatively large-sized void (Figure 1b). SEM images prove that treatment carried out on HSP has reduced the size and number of pits, aggregation, which improves interfacial adhesion between treated HSP fillers and the BER matrix. Also, SEM images indicate a better wetting of chemically treated HSP filler particles with neat BER (Figure 1c,d), which lets us obtain biocomposites with better mechanical properties.⁴¹

It can be seen from the SEM images of the composites that the compatibility between the untreated HSP and the matrix is weaker compared to the treated HSPs (Figure 1b). The filler heaps, holes and voids in the structure prove this situation. It is thought that the interfacial adhesion of the filler with the matrix improves with the reduction in size, number of pits and aggregation in the composites after the alkali treatment of HSP (Figure 1c). In the case of modification with AA, this trend continued, and it is determined that the best result is obtained with this modification, since no pits and voids are formed (Figure 1d). The absence of a visible gap in the interfacial region may indicate good adhesion between the BER and the HSP-AA filler.⁴² Regardless of the HSP filler type, the distribution of particles above 30% is uneven in the entire volume of the samples and agglomerate formation was observed in all composites.

3.2 | XRD analysis results

Since determining the isotropic structure of the tested material has crucial importance in the ultrasonic

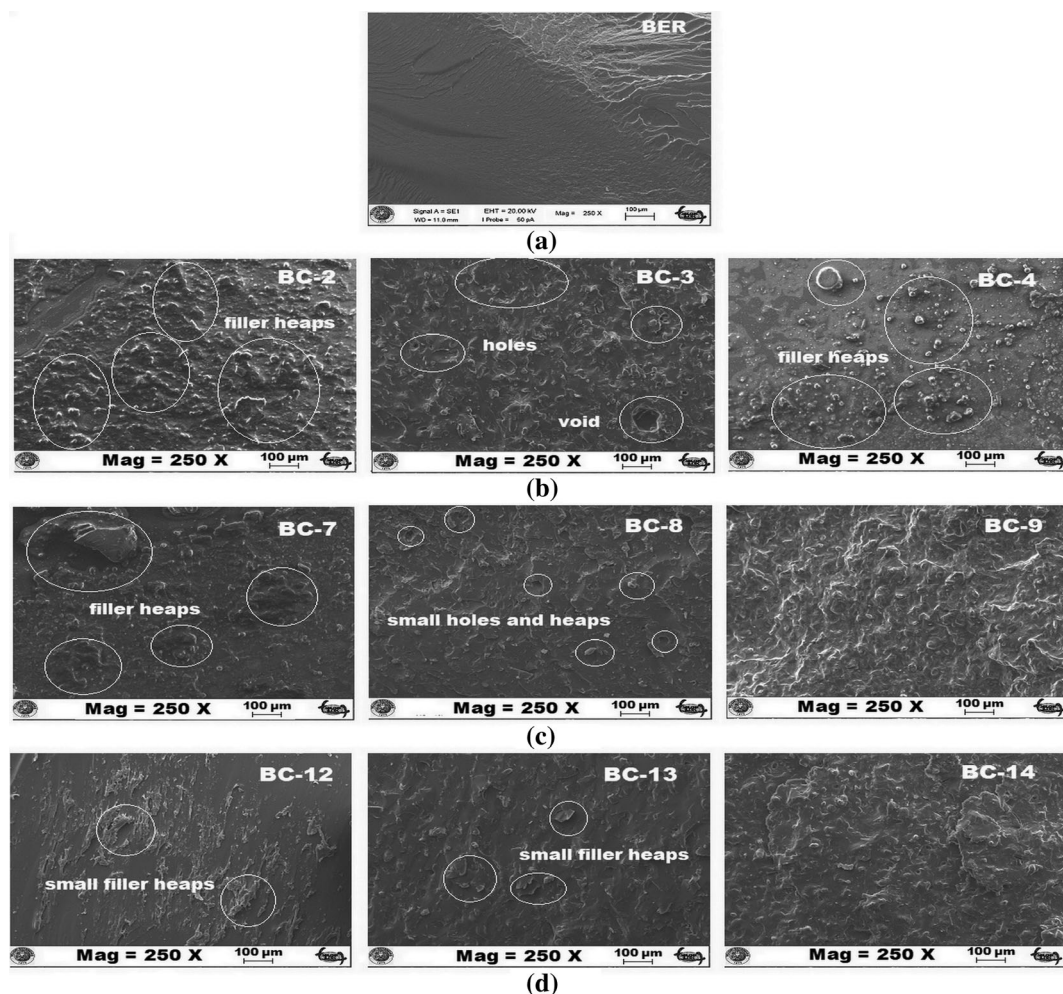
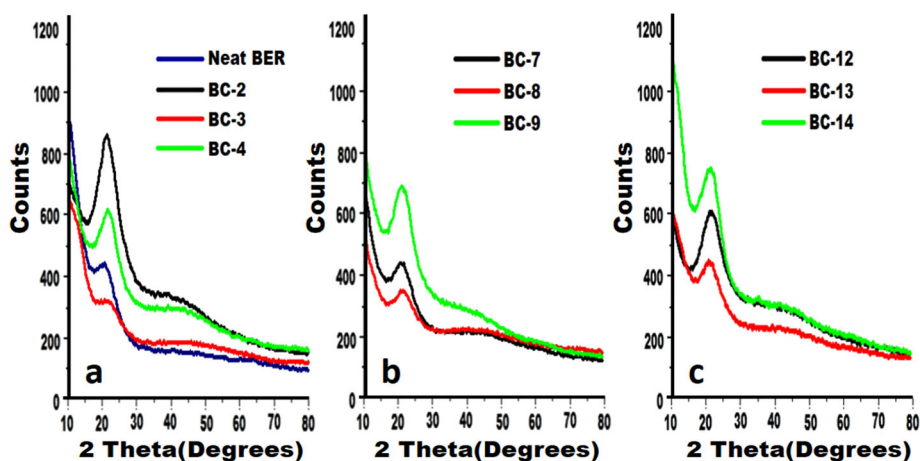


FIGURE 1 SEM images of the neat BER and biocomposites: (a) Neat BER, (b) BER/HSP biocomposite samples (BC-2, BC-3 and BC-4), (c) BER/HSP-NaOH biocomposite samples (BC-7, BC-8 and BC-9), (d) BER/HSP-AA biocomposite samples (BC-12, BC-13 and BC-14).

FIGURE 2 XRD patterns of the neat BER and biocomposites: (a) Neat BER, and BER/HSP biocomposite samples (BC-2, BC-3 and BC-4), (b) BER/HSP-NaOH biocomposite samples (BC-7, BC-8 and BC-9), (c) BER/HSP-AA biocomposite samples (BC-12, BC-13 and BC-14). [Color figure can be viewed at wileyonlinelibrary.com]



method, the isotropic structure of the tested material should be figured out. One of the most effective ways to figure out material's isotropic structure is using XRD analysis. Thus, the XRD analysis of neat BER and obtained biocomposites were analyzed. XRD

patterns of the neat BER and composites are given in Figure 2.

Epoxy resins have an amorphous structure and do not have sharp peaks. Confirming this amorphous structure, a broad and strong peak centered at around

| Samples | Sample ID | ρ (kg/m ³) | v_L (m/s) | v_S (m/s) |
|---------------------|--------------|-----------------------------|----------------|----------------|
| Biomatrix | BER | 1242.96 ± 2.85 | 2507.60 ± 8.78 | 981.00 ± 7.81 |
| BER/HSP | BC-1 | 1262.98 ± 2.88 | 2573.20 ± 4.26 | 1121.37 ± 2.13 |
| | BC-2 | 1275.88 ± 2.96 | 2621.20 ± 5.98 | 1152.80 ± 5.98 |
| | BC-3 | 1281.04 ± 2.58 | 2626.20 ± 1.47 | 1154.40 ± 3.38 |
| | BC-4 | 1291.18 ± 3.95 | 2653.80 ± 3.97 | 1193.20 ± 2.79 |
| | BC-5 | 1295.88 ± 3.19 | 2646.00 ± 3.19 | 1215.20 ± 9.17 |
| BER/HSP-NaOH | BC-6 | 1252.20 ± 3.19 | 2488.00 ± 9.08 | 1082.00 ± 2.53 |
| | BC-7 | 1272.00 ± 1.90 | 2570.40 ± 3.14 | 1107.00 ± 1.67 |
| | BC-8 | 1277.82 ± 1.60 | 2608.20 ± 7.62 | 1141.20 ± 1.94 |
| | BC-9 | 1293.86 ± 2.38 | 2634.20 ± 8.11 | 1149.00 ± 2.61 |
| | BC-10 | 1301.78 ± 3.01 | 3009.40 ± 4.09 | 1374.60 ± 9.20 |
| BER/HSP-AA | BC-11 | 1276.10 ± 7.44 | 2528.60 ± 7.14 | 1108.20 ± 2.23 |
| | BC-12 | 1281.30 ± 2.36 | 2575.20 ± 6.13 | 1116.20 ± 3.82 |
| | BC-13 | 1282.68 ± 2.10 | 2617.00 ± 8.83 | 1172.40 ± 1.62 |
| | BC-14 | 1295.84 ± 3.54 | 2608.80 ± 4.80 | 1188.60 ± 4.54 |
| | BC-15 | 1301.88 ± 2.43 | 2702.80 ± 5.72 | 1227.40 ± 6.47 |

TABLE 1 The density (ρ) and ultrasonic wave velocities (v_L and v_S) values of the BER, and biocomposites.

$2\theta = 22^\circ$, and a weak peak at $2\theta = 42^\circ$ are seen in the XRD curve of the epoxy resin (Figure 2a). It has been reported that a peak at $2\theta = 23.06^\circ$ of type I polymorph cellulose is observed in the XRD spectrum of hazelnut shell powder.⁴³ When the peaks of the BER system are compared with the diffraction peaks of the biocomposites, it is clearly seen that the biocomposites have the same diffraction (XRD) patterns as the matrix. Biocomposites contain dense peaks of cellulose I and BER centered around $22\text{--}23^\circ$. This is also a proof that the original crystal structure of cellulose (cellulose I) is preserved in composites. In addition, peaks at $42\text{--}44^\circ$ also correspond to BER (Figure 2a-c). According to the data of XRD images given in Figure 2a-c, it can be stated that the obtained biocomposites are isotropic materials, which have amorphous structures.

3.3 | Density and ultrasonic wave velocity results

The propagation of ultrasonic waves in polymeric materials not only depends on elastic modulus but also viscoelastic behavior and density as well.⁴⁴ Thus, the variation in density values depending on HSP amounts can help explain the variation in ultrasonic wave velocity and elastic modulus values as well.

The data of Table 1 and Figure 3a presents that the densities of the BER/HSP, BER/HSP-NaOH, and BER/HSP-AA biocomposites are higher than the neat BER. The density value of neat BER is determined as

1242.96 kg/m³. Density values ranged between 1262.98–1295.88, 1252.20–1301.78, and 1276.10–1301.88 kg/m³ for the BER/HSP, BER/HSP-NaOH, and BER/HSP-AA biocomposites, respectively. A remarkable increase was obtained in densities about 4.26%, 4.73%, and 4.74% for BC-5, BC-10, and BC-15 biocomposites, respectively, compared to the neat BER matrix. These results agree well with the density results of related literature.^{2,17}

The interface adhesion between the HSP filler and the neat BER is one of the possible reasons for greater density of biocomposites. The other possible reason is related to the structure of HSP. Because densities of hazelnut shells may differ according to their types. For example, Hebda et al.⁴⁵ tested six varieties of selected hazelnut shells and their bulk densities were found in the range of 368.44–425.07 kg/m³.

Also, Çelik et al.⁴³ have explained that metal content such as Ca, K, Na, Fe, and Si mainly affects the various natural shells' properties. Therefore, the presence of these metals in HSP can be a reason for the increase in the density of biocomposites as well. Chemically modified fillers eliminate micro-voids due to better filler/polymer adhesion, resulting in a slightly higher density of biocomposites.⁴⁶ This behavior in density can also be attributed to the HSP treatment with AA, which eliminates the number of voids in the composites (Figure 1d).

The v_L and v_S values of neat BER were obtained as 2507.60 and 981.00 m/s, respectively. The ultrasonic wave velocities (v_L and v_S) were increased with increase in HSP filler amount (Figure 3b). The higher the HSP ratio, the more v_L and v_S increase. The v_L values of HSP-NaOH

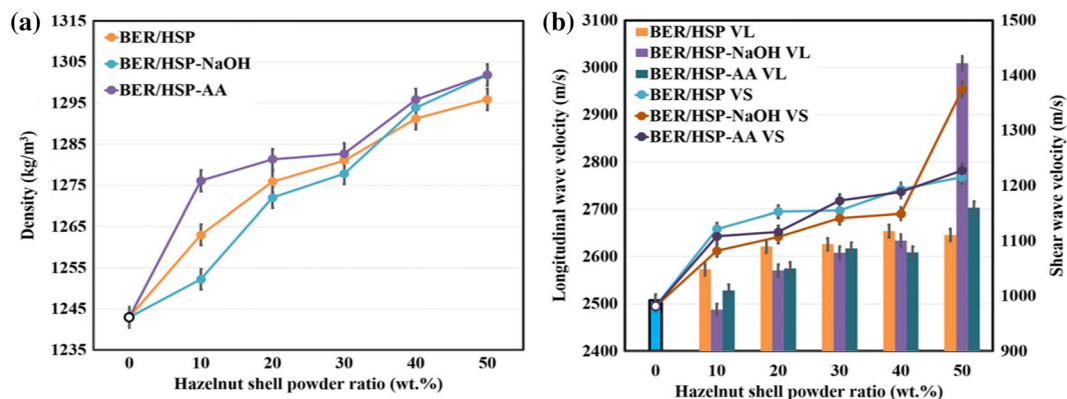


FIGURE 3 Variation in density (ρ), v_L and v_S values of the BER and biocomposites depending on the HSP ratio. [Color figure can be viewed at wileyonlinelibrary.com]

biocomposite samples ranged from 2488.00 to 3009.40 m/s while the v_S values ranged from 1082.00 to 1374.60 m/s. On the other hand, the v_L values of the BER/HSP and BER/HSP-AA biocomposites were determined as 2573.20 to 2653.80 m/s and 2528.60 to 2702.80 m/s, respectively. Also, the v_S values of the BER/HSP and BER/HSP-AA biocomposites were determined as 1121.37 to 1215.20 m/s and 1108.20 to 1227.40 m/s, respectively.

As shown in Table 1 and Figure 3b, the highest v_L and v_S values were measured in the BC-10 biocomposite sample. The v_L and v_S values in BC-10 were increased by approximately 20.01% and 40.12%, respectively whereas the v_S value of the BC-6 was decreased approximately 0.78%.

As a result, the increase in v_L and v_S values in biocomposites by the linear contribution of HSP in neat BER is like the increase in ρ values. This behavior is due to the greater density of materials lead rapid propagation of ultrasonic waves because of smaller chain motions.⁴⁷ Therefore, the significant relationship figured out between uniaxial compressive strength and longitudinal wave velocities of various rocks supports this view.^{37,48} Since the propagation velocity of ultrasonic waves in materials is affected differently by the internal structures of the materials, ultrasonic velocities can be used not only for material densities, but also for the determination of various material properties.⁴⁷ For example, in the research conducted by Haines et al.,⁴⁹ it was stated that there is a direct proportional relationship between the ultrasonic wave velocities and the densities of wood materials. On the other hand, another study conducted by Oral and Ahmetli⁵⁰ involving polyethylene terephthalate char powder reinforced composites revealed a linear correlation between electrical conductivity and ultrasonic longitudinal wave velocity ($R^2 = 0.67$), as well as between ultrasonic-microhardness and ultrasonic shear wave velocity ($R^2 = 0.96$).

3.4 | Elastic moduli of the BER and biocomposite samples

The variation in elastic moduli (L , G , K , E) of the obtained biocomposites as a function of untreated and treated HSP which shows the effect of these HSP fillers, is given in Table 2 and plotted in Figure 4. The findings shown in Table 2 and Figure 4a indicate that except L value of the BC-6 biocomposite sample, L and G values of other biocomposite samples are greater than the L and G values of the neat BER matrix.

The L value for neat the BER was calculated as 7.82 GPa. The L values of the BER/HSP, BER/HSP-NaOH, and BER/HSP-AA biocomposites were determined as 8.36 to 9.09 GPa, 7.75 to 11.79 GPa, and 8.16 to 9.51 GPa, respectively. The G value for neat the BER was calculated as 1.20 GPa. The G values of BER/HSP, BER/HSP-NaOH, and BER/HSP-AA biocomposites ranged between 1.59 and 1.91 GPa, 1.47 and 2.46 GPa, and 1.57 and 1.96 GPa, respectively. Findings of Table 2 and Figure 4a show that except L value of BC-6, the measured elastic modulus values of biocomposites reveal a proportional increase with the HSP weight ratio for both biocomposites. We observed that the L values of BC-10, BC-15, and BC-5 were increased about 50.77%, 21.61%, and 15.98% compared to the neat BER, respectively. Similarly, among the biocomposite samples, the G value increased about 105.00%, 63.33%, and 59.17% for BC-10, BC-15, and BC-5 compared to the neat BER, respectively. One of the remarkable results obtained from Figure 4a is that the highest G values of biocomposites were obtained by adding 50 wt% of the treated and untreated HSP filler to the neat BER.

As shown in Table 2 and Figure 4b, except the BC-6 and BC-11 biocomposites, the K values of all other biocomposites were determined as greater than of the neat BER. The K value of neat BER was obtained as 6.22 GPa.

| Samples | Sample ID | L (GPa) | G (GPa) | K (GPa) | E (GPa) |
|---------------------|--------------|------------------|-----------------|-----------------|-----------------|
| Biomatrix | BER | 7.82 ± 0.05 | 1.20 ± 0.03 | 6.22 ± 0.02 | 3.37 ± 0.06 |
| BER/HSP | BC-1 | 8.36 ± 0.05 | 1.59 ± 0.03 | 6.25 ± 0.03 | 4.39 ± 0.05 |
| | BC-2 | 8.77 ± 0.06 | 1.70 ± 0.04 | 6.51 ± 0.03 | 4.68 ± 0.06 |
| | BC-3 | 8.84 ± 0.05 | 1.71 ± 0.05 | 6.56 ± 0.02 | 4.71 ± 0.06 |
| | BC-4 | 9.09 ± 0.06 | 1.84 ± 0.05 | 6.64 ± 0.04 | 5.05 ± 0.05 |
| | BC-5 | 9.07 ± 0.06 | 1.91 ± 0.04 | 6.52 ± 0.03 | 5.23 ± 0.05 |
| BER/HSP-NaOH | BC-6 | 7.75 ± 0.05 | 1.47 ± 0.04 | 5.80 ± 0.03 | 4.06 ± 0.05 |
| | BC-7 | 8.40 ± 0.06 | 1.56 ± 0.04 | 6.33 ± 0.04 | 4.32 ± 0.06 |
| | BC-8 | 8.69 ± 0.06 | 1.66 ± 0.03 | 6.47 ± 0.03 | 4.60 ± 0.06 |
| | BC-9 | 8.98 ± 0.06 | 1.71 ± 0.05 | 6.70 ± 0.04 | 4.72 ± 0.05 |
| | BC-10 | 11.79 ± 0.05 | 2.46 ± 0.03 | 8.51 ± 0.03 | 6.73 ± 0.06 |
| BER/HSP-AA | BC-11 | 8.16 ± 0.05 | 1.57 ± 0.03 | 6.07 ± 0.04 | 4.33 ± 0.05 |
| | BC-12 | 8.50 ± 0.06 | 1.60 ± 0.04 | 6.37 ± 0.03 | 4.42 ± 0.06 |
| | BC-13 | 8.78 ± 0.06 | 1.76 ± 0.04 | 6.43 ± 0.03 | 4.85 ± 0.06 |
| | BC-14 | 8.82 ± 0.05 | 1.83 ± 0.04 | 6.38 ± 0.03 | 5.01 ± 0.05 |
| | BC-15 | 9.51 ± 0.05 | 1.96 ± 0.03 | 6.90 ± 0.02 | 5.37 ± 0.05 |

TABLE 2 The elastic moduli (L , G , K , E) values of the BER and biocomposites.

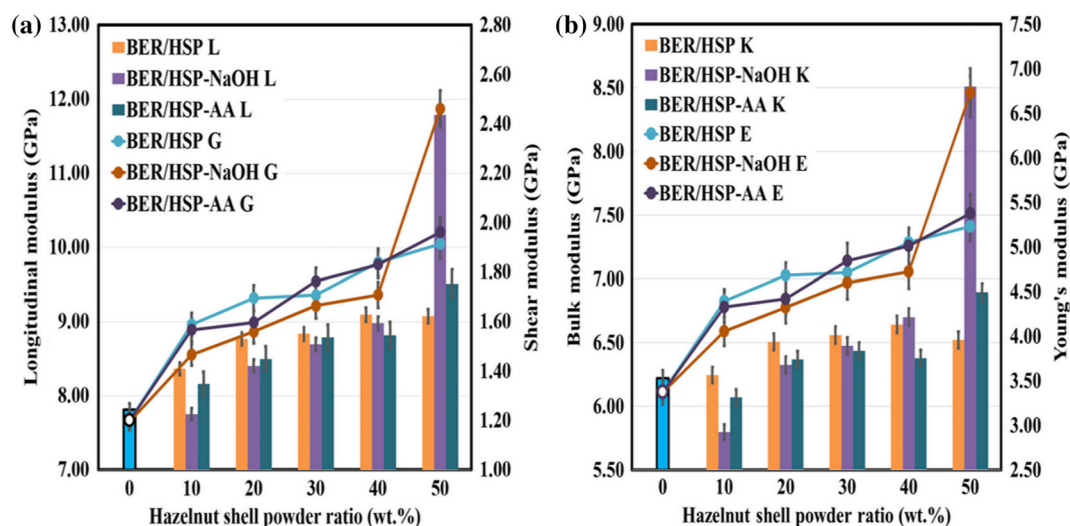


FIGURE 4 Variation in L , G , K and E values of the BER and biocomposites depending on the HSP ratio. [Color figure can be viewed at wileyonlinelibrary.com]

The K values of BER/HSP, BER/HSP-NaOH, and BER/HSP-AA biocomposites ranged between 6.25 and 6.64 GPa, 5.80 and 8.51 GPa, and 6.07 and 6.90 GPa, respectively. The obtained values of the E show a proportional increase with the HSP ratio, for the BER/HSP, BER/HSP-NaOH, and BER/HSP-AA biocomposites. The E value of neat BER was obtained as 3.37 GPa.

The E values of BER/HSP, BER/HSP-NaOH, and BER/HSP-AA biocomposites ranged between 4.39 and 5.23 GPa, 4.06 and 6.73 GPa, and 4.33 and 5.37 GPa,

respectively. An increase of about 36.82%, 10.93%, and 4.82% was seen in the K values of BC-10, BC-15, and BC-5 biocomposites compared to neat BER, respectively. The E value increased about 99.70%, 59.35%, and 55.19% for BC-10, BC-15, and BC-5 compared to the neat BER, respectively. However, the E values of all biocomposites were determined higher than those of neat BER. Among all obtained biocomposites, the highest value of E is found for the BC-10 obtained by treatment of HSP with NaOH. Thus, it can be stated that the most suitable

TABLE 3 The Poisson's ratio (μ), ultrasonic micro-hardness (H), and acoustic impedance (Z) values of the BER, and biocomposites.

| Samples | Sample ID | μ | H (GPa) | Z (MRayl) |
|---------------------|--------------|-------------------|-----------------|-----------------|
| Biomatrix | BER | 0.410 ± 0.010 | 0.14 ± 0.01 | 3.12 ± 0.03 |
| BER/HSP | BC-1 | 0.383 ± 0.011 | 0.24 ± 0.01 | 3.25 ± 0.03 |
| | BC-2 | 0.380 ± 0.010 | 0.26 ± 0.02 | 3.34 ± 0.02 |
| | BC-3 | 0.380 ± 0.012 | 0.26 ± 0.03 | 3.36 ± 0.03 |
| | BC-4 | 0.373 ± 0.010 | 0.29 ± 0.02 | 3.43 ± 0.04 |
| | BC-5 | 0.366 ± 0.012 | 0.32 ± 0.01 | 3.43 ± 0.02 |
| BER/HSP-NaOH | BC-6 | 0.383 ± 0.010 | 0.22 ± 0.02 | 3.12 ± 0.03 |
| | BC-7 | 0.386 ± 0.012 | 0.23 ± 0.01 | 3.27 ± 0.02 |
| | BC-8 | 0.382 ± 0.011 | 0.25 ± 0.01 | 3.33 ± 0.03 |
| | BC-9 | 0.383 ± 0.011 | 0.26 ± 0.02 | 3.41 ± 0.03 |
| | BC-10 | 0.368 ± 0.013 | 0.40 ± 0.02 | 3.92 ± 0.04 |
| BER/HSP-AA | BC-11 | 0.381 ± 0.011 | 0.24 ± 0.02 | 3.23 ± 0.03 |
| | BC-12 | 0.384 ± 0.010 | 0.24 ± 0.02 | 3.30 ± 0.02 |
| | BC-13 | 0.374 ± 0.012 | 0.28 ± 0.01 | 3.36 ± 0.03 |
| | BC-14 | 0.369 ± 0.011 | 0.30 ± 0.01 | 3.38 ± 0.02 |
| | BC-15 | 0.370 ± 0.013 | 0.32 ± 0.02 | 3.52 ± 0.03 |

combination ratio between the neat BER and treated HSP filler with NaOH is 50:50.

Recently, the ultrasonic characterization of elastic modulus of different biocomposites has been succeeded.^{2,17} The elastic modulus values of biocomposites obtained in this research agree well with related literature results.^{3,4} For instance, Rabhi et al.¹⁷ have determined L , G , K , and E values of green epoxy resin (GER)/date stone flour (DSF) biocomposites. They have measured the L , G , K , and E values in the range of 8.12 to 8.89 GPa, 1.39 to 1.45 GPa, 6.26 to 6.96 GPa, and 3.89 to 4.06 GPa, respectively. It has been reported that wheat and corn hulls³ as well as apricot and walnut shell powder⁴ significantly improve Young's modulus of starch-based films. Since E is a material coefficient that expresses the strength of a material, the graph of the function of E relative to HSP shows the effect of these HSP fillers on strength (Figure 4b). The strong bonds formed between the matrix and the additives in polymer composites provide an improvement in all mechanical properties as well as an increase in E values. Because of the strong bonds, the movement of the polymer chains decreases, which gives the materials mechanical strength.⁵¹ Therefore, it can be said that very strong bonds were formed between the HSP treated with NaOH and the BER matrix in the BC-10 biocomposite material obtained. Similarly, Ali et al.⁴² explained the increase in the stiffness values of pomegranate peel/ starch-based films by the strong chemical bonds formed between the pomegranate peel and the starch matrix.

3.5 | Poisson's ratio, ultrasonic micro-hardness, and acoustic impedance

The μ , Z , and H values of neat BER and biocomposites are given in Table 3. Moreover, the variation of these variables (μ , Z , and H) with HSP ratio is plotted in Figure 5, respectively. As seen in Figure 5a, the Poisson's ratio (μ) value for the neat BER was determined as 0.410. The μ values of BER/HSP, BER/HSP-NaOH, and BER/HSP-AA were determined between the ranges of from 0.366 to 0.383, 0.368 to 0.386, and 0.369 to 0.384, respectively.

As seen in Table 3, and Figure 5b, the Z value of neat BER was determined as 3.12 MRayl, while the Z values of BER/HSP, BER/HSP-NaOH, and BER/HSP-AA ranged from 3.25 to 3.43 MRayl, 3.12 to 3.92 MRayl, and 3.23 to 3.52 MRayl, respectively. Acoustic impedance value was determined for epoxy resin as 2.95 MRayl⁵² and for modified BER as 3.37 MRayl.² The H value of neat BER was determined as 0.14 GPa, while the H values of BER/HSP, BER/HSP-NaOH, and BER/HSP-AA ranged from 0.24 to 0.32 GPa, 0.22 to 0.40 GPa, and 0.24 to 0.32 GPa, respectively. Meanwhile, Rabhi et al.¹⁷ have also determined μ , Z , and H values of the GER/DSF biocomposites. They have measured the μ , Z , and H values in the range of 0.395 to 0.411, 3.07 to 3.33 MRayl, 0.078 to 0.097 GPa, respectively. This result indicates a significant difference between the BER/HSP and GER/DSF biocomposites in favor of BER/HSP biocomposites in terms of H values.

A general decrease was observed in Poisson's ratio value of the neat BER with the HSP incorporation

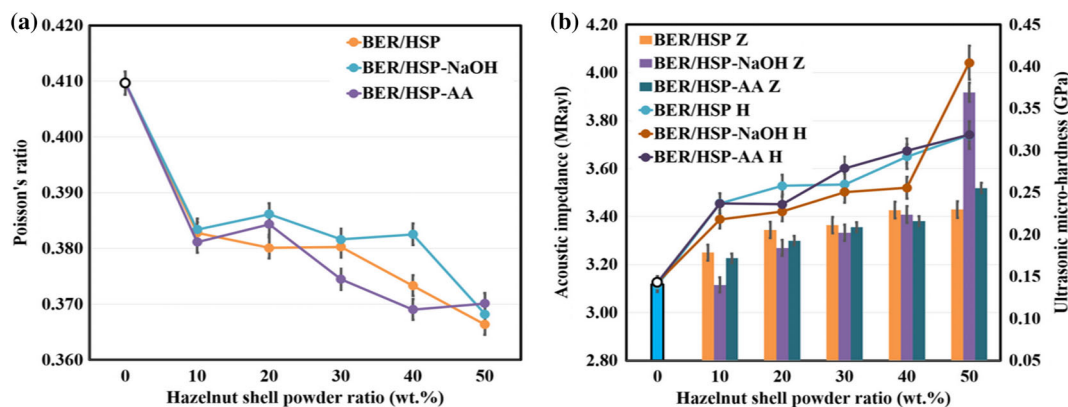


FIGURE 5 Variation in μ , Z and H values of the BER and biocomposites depending on the HSP ratio. [Color figure can be viewed at wileyonlinelibrary.com]

(Figure 5a). A decrease of 10.73%, 10.24%, and 10.00% was determined in the Poisson's ratio value of *BC-5*, *BC-10*, and *BC-14* biocomposites, respectively.

As it can be seen from Table 3 and Figure 5b, a linear increase in Z values with increasing amount of untreated and treated HSP filler in neat BER was observed as well. Most biocomposites have higher Z values compared to the neat BER. Moreover, an increase of approximately 9.94%, 25.64%, and 12.82% was observed in the Z value of *BC-5*, *BC-10*, and *BC-15*, respectively.

Acoustic impedance is defined as the difficulty shown against sound waves traveling in a medium, similar to the resistance shown to electric charges traveling in a conductive medium.⁵³ Acoustic impedance is a very important material property for materials used in acoustic applications. Because the less the sound reflection at the interface between the two environments where the acoustic waves propagate, the higher the ultrasonic wave energy passing to the second medium.

The higher H values of all biocomposites compared to the neat BER is another remarkable result of this research. Because the higher H values also indicate the stronger bonding between matrix and fillers in polymer composites. Also, a linear increase for H value is observed with the increasing amount for both untreated and treated HSP filler in neat BER. A significant increase of about 128.57% was observed in the H value of both *BC-5* and *BC-15*, while a quite significant increase of about 185.71% was observed in the H value of the *BC-10* sample as well.

3.6 | Damping properties of the BER and biocomposites

The ultrasonic wave velocity values (v_L , v_S), the characteristic acoustic impedance (Z), the attenuation coefficient (α), loss tangent ($\tan\delta$), and quality factor (Q) are

the most important acoustical properties for sound applications, such as musical instruments and building interiors. The obtained data related to damping properties (α , $\tan\delta$, and Q) is given in Table 4 and plotted in Figure 6.

According to the data given in Table 4 and Figure 6, the α value of the neat BER was determined as 299.46 Np/m using longitudinal waves of 20 MHz frequency. The α values of the BER/HSP, BER/HSP-NaOH, and BER/HSP-AA were determined in the range of 216.39 to 289.64 Np/m, 241.86 to 267.87 Np/m, and 231.41 to 261.55 Np/m, respectively. As it can be seen in Table 4 and Figure 6, the α values of all biocomposites are lower than of the neat BER. Also, a significant decrease of 27.74%, 22.72%, and 19.23% was observed in the α value of *BC-5*, *BC-11*, and *BC-8*, respectively. Contrary of this behavior of the α , Oral et al.² determined an increase of 6.52% in the α value of biocomposites obtained using modified coconut shell powder at combination ratio of 70:30 compared to the treated epoxy resin. Attenuation is a phenomenon which is defined as exponential reduction in the amplitude, and energy of ultrasonic waves during the propagation of ultrasonic waves through a medium.⁵⁴ The main factors responsible for ultrasonic attenuation are scattering, absorption, refraction, reflection, and divergence. However, the two main factors caused the attenuation in this research are scattering and absorption.

Rayleigh scattering is predicted in the BER/HSP, BER/HSP-NaOH, and BER/HSP-AA biocomposites since average diameter of HSP bio-fillers, and the wavelength value of ultrasonic longitudinal waves used in this research are about 63 μm , and 130 μm , respectively.

Also, the neat BER matrix is predicted to be responsible for ultrasonic energy absorption determined in biocomposites. Considering this prediction, the reason for the decrease in attenuation could be due to the decrease in the amount of energy absorbed by the BER matrix with the

TABLE 4 The attenuation coefficient (α), loss tangent ($\tan\delta$), and quality factor (Q) values of the BER and biocomposites.

| Samples | Sample ID | α (Np/m @ 20 MHz) | $\tan(\delta)$ | Q |
|---------------------|--------------|--------------------------|-------------------|----------------|
| Biomatrix | BER | 299.46 ± 20.14 | 0.01195 ± 0.00108 | 83.67 ± 09.63 |
| BER/HSP | BC-1 | 275.77 ± 19.70 | 0.01129 ± 0.00108 | 88.54 ± 09.45 |
| | BC-2 | 252.76 ± 18.65 | 0.01054 ± 0.00090 | 94.84 ± 10.00 |
| | BC-3 | 289.64 ± 20.04 | 0.01211 ± 0.00100 | 82.60 ± 09.79 |
| | BC-4 | 235.12 ± 18.32 | 0.00993 ± 0.00108 | 100.70 ± 10.05 |
| | BC-5 | 216.39 ± 19.38 | 0.00911 ± 0.00100 | 109.74 ± 09.84 |
| BER/HSP-NaOH | BC-6 | 260.19 ± 20.75 | 0.01030 ± 0.00080 | 97.06 ± 10.06 |
| | BC-7 | 267.87 ± 21.08 | 0.01096 ± 0.00100 | 91.26 ± 09.65 |
| | BC-8 | 241.86 ± 20.64 | 0.01004 ± 0.00108 | 99.60 ± 09.21 |
| | BC-9 | 252.12 ± 20.14 | 0.01057 ± 0.00090 | 94.61 ± 09.45 |
| | BC-10 | 247.77 ± 19.90 | 0.01187 ± 0.00108 | 84.27 ± 09.92 |
| BER/HSP-AA | BC-11 | 231.41 ± 20.24 | 0.00931 ± 0.00100 | 107.38 ± 09.32 |
| | BC-12 | 261.55 ± 19.80 | 0.01072 ± 0.00108 | 93.29 ± 09.68 |
| | BC-13 | 241.63 ± 19.50 | 0.01006 ± 0.00100 | 99.36 ± 10.00 |
| | BC-14 | 251.06 ± 19.20 | 0.01042 ± 0.00090 | 95.93 ± 09.59 |
| | BC-15 | 246.27 ± 20.41 | 0.01059 ± 0.00100 | 94.40 ± 10.12 |

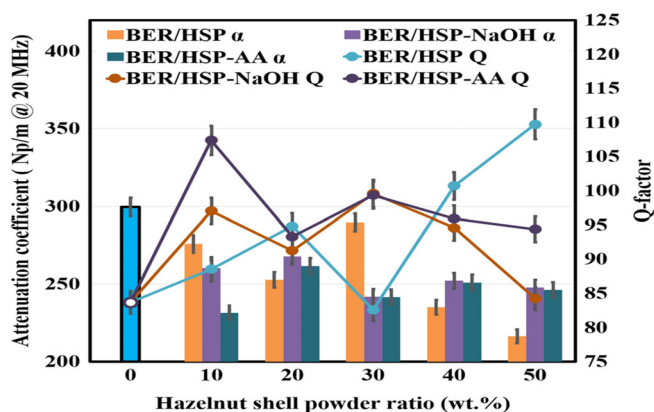


FIGURE 6 Variation in α and Q values of the BER and biocomposites depending on the HSP ratio. [Color figure can be viewed at wileyonlinelibrary.com]

increase in the HSP ratio in the biocomposites. Thus, it can be stated that the attenuation coefficients of the obtained biocomposites depends more on the energy absorbed by the matrix system than the actual Rayleigh scattering.

One of the important data related to damping properties of the neat BER and obtained biocomposites is the $\tan\delta$ value. The $\tan\delta$ value can be measured by both Dynamic Mechanical Analysis (DMA)⁵⁵ and ultrasonic methods.³⁷ In this research, the $\tan\delta$ of neat BER was determined as 0.01195 (Table 4).

Crawford and Escarsega⁵⁶ determined the neat epoxy resin's loss tangent as 0.039 using DMA. Since the lower loss tangent value indicates better mechanical properties,

it can be stated that the BER used in this research have better mechanical properties compared to neat ER. Loss tangent values of all biocomposites except *BC-3* are smaller than of the neat BER (Table 4). Thus, this result proved that except *BC-3*, all biocomposites obtained have better mechanical properties compared to the neat BER. As can be seen from Equation (11), the Q -factor value which is another important damping coefficient, is equal to the inverse of the $\tan\delta$. Since the Q -factor is equal to the inverse of loss tangent, one can state that higher Q -factor means stronger bonds between matrix and filler in a polymer composite⁵⁷ which leads to better mechanical properties.

Figure 6 illustrates the Q -factor values of the neat BER, BER/HSP, BER/HSP-NaOH, and BER/HSP-AA biocomposite samples. The Q -factor value of the neat BER was obtained as 83.67. The Q -factor values of BER/HSP, BER/HSP-NaOH, and BER/HSP-AA ranged from 82.60 to 109.74, 84.27 to 99.60, and 93.29 to 107.38, respectively. Figure 6 revealed that Q -factor values of all biocomposites except *BC-3* are greater than neat BER. This result agrees well with the results of the loss tangent given in Table 4 and approves the result obtained for the *BC-3* sample as well. Higher $\tan\delta$ or lower Q -factor value indicates higher loss energy.³⁷ The research carried out by Ali et al.⁴ confirms this result as well.

Accordingly, it can be stated that the Q -factor values agrees well with α values obtained for biocomposites. A significant increase of 31.16%, 28.34%, and 19.04% was observed in the Q -factor value of *BC-5*, *BC-11*, and *BC-8*,

respectively. However, a decrease of 1.28% has been measured for the BC-3 sample compared to neat BER. The decrease in the Q -factor of the BC-3 sample could be due to increasing happened in the attenuation coefficient due to scattering because of insufficient interaction between the matrix and the filler. The greatest α value obtained for BC-3 among all biocomposites supports this prediction as well.

As a result, small α , small loss tangent, and high Q -factor values are due to the low mobility of molecules in the polymer chain which causes a bigger resistance to movement. The more resistance to the mobility of molecules proves the formation of strong bonds between the matrices and fillers in polymer composites. Also, it is expected to obtain composites with better mechanical strength, greater hardness, and modulus of elasticity by the strong bonds between matrices and fillers.

4 | CONCLUSIONS

The HSs were treated with NaOH, and AA. These HSs chemically treated with NaOH and AA, and untreated HSs were ground to obtain HSPs. Then, untreated HSPs and treated HSPs were added to the novel BER matrix to form the BER/HSP, the BER/HSP-NaOH, and the BER/NaOH-AA biocomposites. The effect of untreated and treated HSP filler on the density, ultrasonic wave velocities, elastic moduli, and damping characteristics of the novel HSP-based biocomposites, was investigated by the ultrasonic wave velocities. SEM and XRD analysis were used for morphological characterization. The general results of the research can be given as follow:

1. XRD images proved the isotropic structures of the BER and biocomposites obtained.
2. Except L value of the BC-6 biocomposite sample, the measured density, ultrasonic wave velocities (v_L and v_S), and elastic modulus values of biocomposites revealed a proportional increase with the HSP weight ratio for all biocomposites.
3. The v_L and v_S values in BC-10 increased by approximately 20.01% and 40.12%, respectively.
4. The E values of BC-10, BC-15, and BC-5 increased about 99.70%, 59.35%, and 55.19% compared to neat BER, respectively. Also, the E values of all biocomposites are greater than the E value of neat BER.
5. A general decrease was observed in Poisson's ratio value of neat BER with the HSP incorporation. Moreover, a significant decrease of 27.74%, 22.72%, and 19.23% was observed in the α value of BC-5, BC-11, and BC-8, respectively.
6. A linear increase in H and Z values with an increasing amount of HSP filler in neat BER was observed.

7. A significant increase of 31.16%, 28.34%, and 19.04% was observed in the Q -factor value of BC-5, BC-11, and BC-8, respectively.
8. Overall, based on the obtained elastic modulus values, the most appropriate combination ratio between the neat BER, and HSP treated with NaOH was determined as 50:50.

To sum up, it was figured out that the density and ultrasonic wave velocities of biocomposites obtained have great importance because they are important clues that prove the improvement in elastic modulus compared to the values of neat BER. The results of this study, encourage the chemical treatment of natural bio-fillers, production of the eco-friendly biocomposites by novel bio-based matrices, and using ultrasonic methods for the characterization of obtained biocomposites.

AUTHOR CONTRIBUTIONS

Imran Oral: Conceptualization (lead); investigation (lead); methodology (lead); writing – review and editing (lead). **Suheyla Kocaman:** Conceptualization (equal); investigation (equal); methodology (equal). **Alaaddin Cerit:** Investigation (supporting); visualization (supporting). **Gulnare Ahmetli:** Conceptualization (lead); supervision (lead); writing – review and editing (supporting).

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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