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Research Article

Ultrasonic Characterization of the Mechanical Behavior of Epoxy/Date Kernel Powder Biocomposites: A Feasibility Study of the Powder Size Effect

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Abstract : This study aims to address the effect of date kernel powder (DKP) as reinforcement obtained from Touggourt oasis, Algeria, on the elastic properties of biocomposites based on two prepared DKPs with grain size 300 μm and 500 μm mixed on epoxy (ER) matrix. The weight percentage of powders with 5%, 10%, and 15% was used to obtain epoxy matrix (ER)/date kernel powder (DKP) biocomposites. The effects of DKP size on the elastic properties of ER/DKP biocomposites, such as ultrasonic wave velocities (longitudinal and shear), longitudinal modulus, shear modulus, bulk modulus, Young's modulus of elasticity, ultrasonic microhardness, Poisson's ratio, and acoustic impedance, were determined using the ultrasonic through-transmission method. In addition, the two biocomposites prepared to analyze the chemical changes in the functional groups and their morphology were studied using X-ray diffraction and optical microscopy, respectively. The results of ultrasonic characterization of the ER/DKP biocomposites showed that there is a significant relationship between the sizes of DKP and elastic constant values. In addition, the experimental results illustrated that the optimum weight percent of DKP reinforcement in neat ER for excellent mechanical behavior of ER/DKP biocomposites is 5% and 10% for 300 μm and 500 μm, respectively.

Keywords : Date kernel powder (DKP), Bio-sourced materials, Ecofriendly materials, Ultrasonic characterization, Elastic constants.

1 Introduction

To reduce the use of synthetic materials in numerous industries, such as composite materials, engineering uses natural fibers as reinforcement in advanced composite materials for specific behavior. Date palm tree (DPT) is a natural fiber. It can be turned into a value added manufacturing process through the production of new eco-friendly composites based on several thermoplastic or thermoset materials replacing synthetic fibers [1]–[3]. It represents abundant lignocellulose and a renewable source in all Afro-Asiatic dry bands from Saharan countries in northern North Africa to the Middle East [4], [5], with approximately two-thirds of the date palm trees cultivated in Arab countries such as the Algerian oasis for exploitation of date fruits [3], [6], with 15941.4 tons of kernel waste in Algeria [7]. The palm date is divided into several parts such as mesh, leaflet, fronds, kernel, petiole, midrib, and rachis. Generally, the reinforcements from the date palm can be extracted from all parts of them as fibers [8]. In addition, particles and powder can be produced by milling the date palm fruit seeds [9]. Furthermore, for example, the kernel derived from date palm fruit is largely available in the Saharan countries in northern Africa [10], and it can be regarded as waste material during the use of whole fruits processing.

The use of date kernel waste is a research topic for many applications such as engineering, including composite materials [11]. Where, Nasser et al. [12] reported that this kind of palm part has constituted a significant alternative to using a potential new renewable energy. In the past few years, several studies have used the parts of date palms as reinforcement in polymeric materials for various applications [5], [13]–[15]. In addition, these natural by-products could be used with or without chemical treatment [5]. Date palm fibers with longitudinal architecture (0°, 45°, and 90°) were used in the study by AlZebdeh et al. [9] to develop a new eco-friendly composite. After chemical treatment of fibers with 1 wt.% of NaOH for 2 h, biocomposite sheet samples were fabricated. The results of the tensile tests (Young's modulus, tensile strength, and elongation break) show that date palm fibers can be used for low-strength applications as reinforcement materials. Short date palm fibers with diameters of 200-400, 400-600, and 600-800 μm and 10 mm of length were used as natural reinforcement on DPF/Epoxy composites by Abdal-Hay et al. [16]. This study investigated the effect of chemical modification of fibers on the tensile and morphological properties of composites.

The authors found that the fibers are amenable to chemical modification, leading to improved adhesion fiber/matrix, tensile strength, and Young's modulus. In another study, date palm fiber in the form of powder was used as reinforcement for polymeric composites by Ibrahem [33]. The effect of a volumetric ratio of up to 25% of powder on the tribological behavior of the studied composites was investigated. The experimental results show the effect of different parameters on the coefficient of friction, rate of wear, and resistance of wear of the proposed materials according to the amount of powder. Cellulosic seed particles of palm date used as reinforcement for unsaturated polyester by Ameh et al. [34]. Composites with particle sizes of 0.5, 2.0, and 2.8 mm and particle loadings of 5–25 wt.% were obtained for traditional destructive characterization, such as tensile strength, water absorption, and hardness. The results indicated that date palm seed particles can be used as reinforcement to enhance the behavior of composites. The authors also, declared that the 15 wt.% and 10 wt.% are the optimum loadings for tensile strength and elastic modulus, respectively, using 0.5 mm size. On the other hand, the better hardness at a particle size of 2 mm at 25 wt%. Masri et al. [35] fabricated and characterized new date palm leaflets/Polystyrene composites, and three sizes of leaflet reinforcements were selected. The authors found that the composites exhibit good thermal insulation and can be recycled after their useful life. Sh. Al-Otaibi et al. [3] investigated the effect of 5%, 10%, and 15% loadings of date palm fiber (DPF) with lengths (8-12 mm and less than 2 mm) on morphological, mechanical, and thermal properties of a composite base on recycled polypropylene (RPP), impact copolymer (ICP), and homopolymer (HPP) reinforced by the treated DPF. Initially, the fiber was treated with 1% NaOH for 1 h at 100°C. Subsequently, the composite samples for morphological, mechanical, and thermal tests were prepared. Among the most important experimental results are that the RPP can be used as an alternative to HPP with DPF treated to improve the tensile strength of the polypropylene (PP) matrix. In addition, the increase in fiber loading increased the modulus and decreased the tensile property. Date palm fiber mesh waste was used as reinforcement on composites in the study of Boumhaout et al. [36]. Mortar/DPF composites for thermal insulation of buildings were examined under thermomechanical tests with a volume percentage of DPF ranging from 0% to 51%. The composites used in the study by Benaniba et al. [37] were formed with DPF and cement. The 7 mm length of fiber was used with 6, 12, 18, 24, and 30% of weight. The results show that the effect of increasing the weight amount of DPF led to a reduction in thermal conductivity, compressive strength, and flexural. Also, the addition of DPF increases the thermomechanical properties of the mortar. Recently, Abd Mohammed [38] studied the hardness properties and wear rates of olive seeds (OS) and date seeds (DS) by destructive investigation. Effect of weight fraction and grain size on the wear and hardness behavior of epoxy resin reinforced with OS and DS. It was found that the weight fraction of powder has a higher effect than particle size on the properties of the studied materials. The effect of date stone flour (DSF) on the mechanical, thermal, and morphological properties of SDF/Polypropylene (PP) composites with ethylene-butyl acrylate-glycidyl methacrylate (EBAGMA) as the compatibility was studied by Hamma et al. [10]. Fixed the amount of compatibility to half of the DSF and varied the loading rate of DSF between 10 and 40 wt%. The results show that the addition of EBAGMA improved the characteristics of the SDP/PP composites, such as the dispersion of SDF and the ductility due to the elastomeric. Debabeche et al. [39] prepared a destructive study that analyzed the effect of three types of treatment (hydrogen peroxide, NaOH, and acetic anhydride) on the surface of palm petiole fiber. We evaluated the Petiole/linear low-density polyethylene (LLDPE) composite with loading 15 wt% of fiber. The mechanical, dynamic mechanical, and morphological properties confirmed the effect of the treatments on the fiber surface. Palm kernels can be used not only to make new materials, but also to clean wastewater with a cheap and simple method. For example, in the research conducted by Ozcelik [40], it was stated that heavy metal ions can be removed from wastewater with an easy and cheap method by adsorbing Cu (II) ions on palm kernels. Many methods have been applied and developed to overcome the behavior of composite materials, including the nondestructive method. From an economical and technical point of view, the use and development of nondestructive test (NDT) methods such as ultrasound measurements for the measurement of elastic constants is important to predict the behavior of materials. Ultrasonic waves have been used for many years both in material characterization and in the characterization of defects in materials [41]. Because this method is accurate, faster, and more sensitive, it is the preferred method compared with destructive methods such as tensile testing [42], [43]. Recently, many studies have been conducted using ultrasonic NDT methods (Table 1).

An important research by Rabhi et al. [18] has been conducted to investigate the effect of the chemical treatment of date stone flour (DSF) on the elastic constants of DSF mixed with green epoxy resin (GER) for biocomposite materials. A nondestructive characterization via the ultrasonic transmission method was applied to the samples. They observed an increase in the elastic constants of treated filler biocomposites compared with pure GER. Also, they were found to be the most suitable treatment of filler via the permanganate chemical treatment compared to other treatments (alkaline and benzoyl chloride).

Table 1: Report of an example of research on the nondestructive NDT characterization of materials.

Material	Parameters	NDT Test	Key findings	Refs
Epoxy/Polyaniline (PANI) composite	weight percentages of 5%,10% and 15% of PANI	Ultrasonic pulse-echo-overlap method (PEOM)	Effects of PANI amount on the mechanical properties of composite	[17]
Epoxy/Date Stone Flour composite	• 30wt% of filler, • Soda(alkaline), potassium permanganate and benzoyl-chloride chemical treatments.	Ultrasonic through-transmission method	Effect of filler chemical treatment on elastics constant of bio-composites	[18]
Epoxy/Polyvinyl alcohol (PVA) nanocomposite	5,10 and 15 layers of PVA nanofiber mats	Ultrasonic wave velocity method	Effect of the number of PVA layers on the elastic properties of nanocomposite	[19]
Modified Epoxy/Andesite Waste composite	• 10-30wt% of andesite waste (aw) filler, • 5wt% of polyaniline to modify the resin.	Ultrasonic pulse-echo method	Examine the effect of filler amount on the ultrasonic and electrical properties of the composites	[20]
Epoxy/Coconut and Epoxy/fique	/	Ultrasonic through-transmission method	Effect of natural fiber on stiffness of composites	[21]
Al/SiC nanocomposites	0,5 and 10 vol.% of nano size SiC particle	Ultrasonic pulse-echo-overlap method (PEOM)	Effect of SiC content on mechanical properties of nanocomposites	[22]
Epoxy/China Poplar Char (CPC) and Epoxy/Pine Cone Chare (PCC) composites	Weight percentage of 10, 20 and 30% of biochars	Ultrasonic pulse-echo-overlap method(PEOM)	Effect of biochars amounts on ultrasonic properties	[23]
Epoxy/Marble Waste Powder (MWP) composite	Weight percentage of 20 of Marble Waste Powder	Ultrasonic pulse-echo method	Effect of marble powder, dosage and coagulant type on elastic properties	[24]
Modified Polystyrenes (MPS)/Pure Polystyrenes (PS) composites	• Molecular weights(350×10^3 and 500×10^3) of (PS), • Molecular weights (230×10^3) of Modified Polystyrenes (MPS), • Weight ratio (%) of 90:10, 20:80 and 70:30 of composites.	Ultrasonic pulse-echo method	Effect of molecular weight and weight ratio (%) on elastic properties	[25]
Epoxy/ Polyethylene Terephthalate (PET) chare powder composites	Weight percentage of 10%, 20% and 30% of chars	Ultrasonic pulse-echo method	Effect of chars powder on the elastic constants	[26]
Epoxy3419/Carbon T700	/	Backwall reflection method (BRM)	Comparison between BRM measured and theoretically calculated ultrasonic travel time for determining the elastic constants	[27]
Polymethyl methacrylate (PMMA)/ Date Stone Flour (DSF) composite and Green Epoxy Resin (GER)/ Date Stone Flour (DSF) composite	• Filler content of 10, 20, and 30wt%, • Potassium permanganate treatment.	Ultrasonic through-transmission method	Examine the effect of matrix and the Potassium permanganate treatment	[28]
Polypropylene (PP)/ Olive Wood Flour (OWF) composite	Weight percentage of 10, 20, and 30wt% of reinforcement	Ultrasonic immersion in water method	Effect of reinforcement weight ratio (%) on elastic properties	[29]
Grafted Marble (M)/Epoxy composite and Granite Powder (G)/Epoxy composite	Weight percentage of 20, 60, and 100wt% of fillers	Ultrasonic pulse-echo method	Effect of fillers weight ratio (%) on elastic properties	[30]
Rigid Polyurethane Foam	Foam density	Ultrasonic wave propagation methods	Effect of density of foam on elastic moduli	[31]
Polymethyl methacrylate (PMMA), Polystyrene (PS), and Polyvinyl-chloride (PVC)	/	Immersion sing-around method and ultrasonic pulse-echo method	To determine of elastic properties of thermoplastic resin	[32]

Table 2: Chemical compositions of the date kernel.([12], [44])

Cellulose (%)	Lignin (%)	Hemicellulose (%)	Ash (%)	Carbon (%)	Oxygen (%)	Hydrogen (%)
32.77	37.03	12.64	1.4	44.1-45.3	47.2-48.3	5.6-6.1

Figure 1: Reinforcement type used: (a) Date kernel (DK), (b) Raw date kernel powder (DKP) 300 μm and (c) Raw date kernel powder (DKP) 500 μm.

Investigations related to the use of natural powders of different sizes, such as date kernel powder (DKP) with epoxy resin (ER) matrix, and to the non-destructive characterization of these composites are very limited and have not been reported in the open scientific literature. The current study predicts the potential of date kernel powder (DKP) incorporation into epoxy resin (ER) to improve its mechanical properties. To this end, the elastic properties of the ER/DKP bio composites based on the size effect of date kernel powder (DKP) will be considered using the ultrasonic wave velocity measurement method.

2 Materials and Methods

2.1 Raw Materials

In this research, the natural reinforcement as kernel of date palm of Deglat Nour (DPDN) cultivar from Oasis agriculture in Touggourt, Algeria (Latitude 33° 06' 18.97'' N and Longitude 6° 03' 28.66'' E) was used as natural reinforcement material of biocomposites. This bio-waste kernel was isolated from date palm fruit collected after full ripeness. Two types of Date Kernel Powder (DKP) were obtained after kernels were cleaned with water to remove impurities and after being naturally dried for two days to reduce water content. The kernels were ground to powder using a mill and sieved to size 300 μm and 500 μm (Figure 1). In this study, we used date palm kernel powder without any treatment. The average values of the chemical composition of the date kernel are shown in Table 2.

2.2 Preparation of the Biocomposites Samples

The matrix used in this investigation was a MEDA-EPOXY INJECT 812 matrix composed of resin and hardener. The preparation of the composite samples took two stages, with the first step manually mixing the powder and matrix for 15-20 min. The second step fabricating the sample composite in the mold, as shown in Figure 2.

Epoxy resin was used in this study because it is commonly used for engineering applications, has good stiffness, better dimensional stability, and is cheaper [45]. The abbreviations and detailed information of the obtained biocomposites are given in Table 3.

2.3 Density Measurement

According to ASTM D792, the density measurements (ρ) with an analytical balance (KERN DBS Germany, readability 10-3mg, and weight capacity 60g) at a room temperature of 25°C were carried out.

Table 3: Contents of epoxy resin ER and date kernel powder (DKP).

Samples' ID	Composition ratio of ER/DKP (wt.%)
ER	100:0
COM305	95:5
COM310	90:10
COM315	85:15
COM505	95:5
COM510	90:10
COM515	85:15

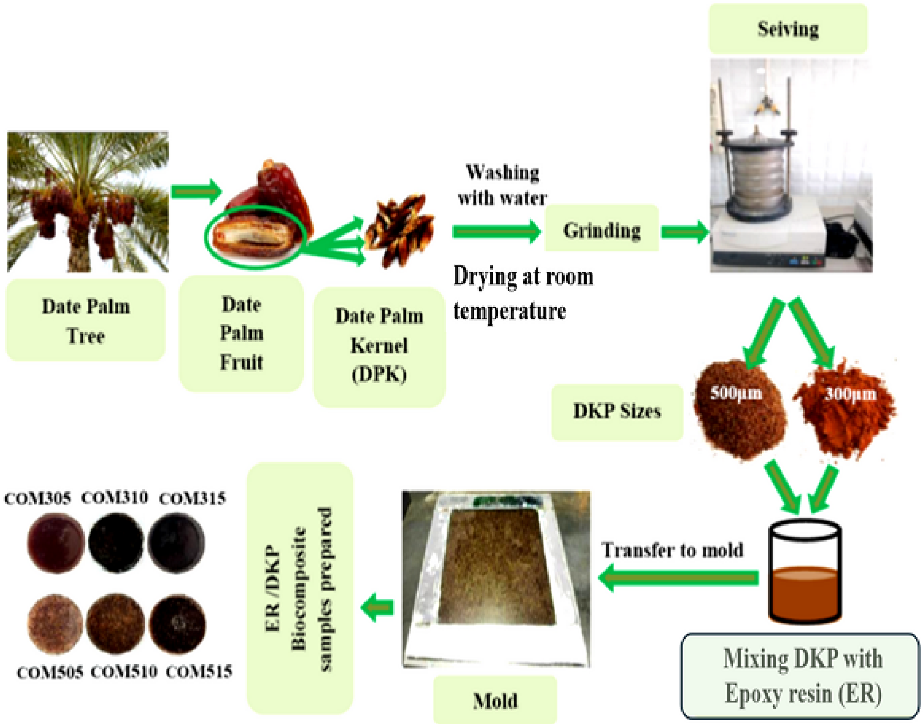

Figure 2: Illustration of the process followed for ER/DKP biocomposites preparation.

2.4 Ultrasonic wave velocity (V_L and V_S) Measurements

Ultrasonic wave velocity measurements were performed using the contact-through transmission method. This method is based on the set of time (t) of flight of the sound through the sample thickness (d). In the first step, the thickness of the specimens was measured. Then, transmitting and receiving transducers type (OLYMPUS) placed on opposite sides of the specimens were used to detect the ultrasonic wave traveling via the thickness of specimens. Ultrasound lubricant was used for coupling through the device of generator (OLYMPUS 35MHz-5800PR) to apply a power supply with a pulse duration of 1 μs. The ultrasonic waves were amplified and transferred to a digital TELEDYNE Locroy oscilloscope with a frequency of 1 GHz. Two longitudinal contact probes with a frequency of 5 MHz and a diameter of 9.5 mm and two shear contact probes with a frequency of 2.25 MHz and a diameter of 12.5 mm. Ultimately, the data were managed on a computer (Figure 3).

The determination of the time of flight (t) led to the calculation of the wave velocities (V) of the transmitted pulse along the sample by applying Equation 1

$$V = \frac{d}{t} \tag{1}$$

where t(s), d(m), and V(m/s) are the time of flight, specimen thickness, and velocity of sound through the specimen, respectively.

2.5 Evaluation of the Elastic Constants

Because the measurement of the density and ultrasonic wave velocities of a material is acceptable for the ultrasonic characterization of isotropic materials, the other elastic constants can be determined depending on these two physical properties [46]. Considering the ER/DKP biocomposite samples as isotropic materials, the elastic properties of the biocomposites are calculated according to the following equations:

$$L = \rho V_L^2 \tag{2}$$

$$G = \rho V_S^2 \tag{3}$$

$$K = L - \frac{4}{3}G \tag{4}$$

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

$$E = 2G(1 + \mu) \tag{6}$$

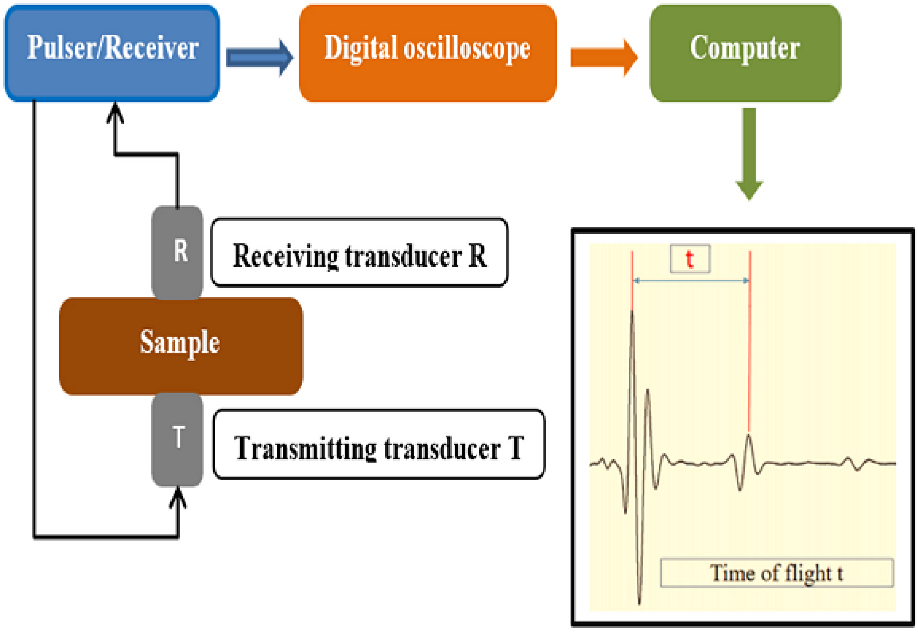

Figure 3: Diagram of the ultrasonic measurement setup used in this study.

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

$$Z = \rho V_L \tag{8}$$

where L, G, K, μ, E, H, and Z are the longitudinal modulus, shear modulus, bulk modulus, Poisson's ratio, Young's modulus of elasticity, ultrasonic microhardness, and acoustic impedance, respectively. It is worth noting that these equations are valid for isotropic materials [47].

2.6 Morphological Measurements

The XRD analysis of the pure ER matrix and ER/DKP biocomposites was conducted using a D2 Phaser Bruker diffractometer, Germany, with operating conditions Cu-kα source (λ= 1.54184A°, power source= 30KV and 10mA) and Bragg angle 2θ (10°-90°), with a step speed of 1°/min. From the obtained XRD curves, the values of the crystalline index of the biocomposites samples were determined using the Ruland-Vonk method [48] and the application of Equation 9.

$$Crystallinity \quad (\%) = \frac{Crystalline \quad area \times 100}{Total \quad area \quad under \quad curve} \tag{9}$$

In addition, the morphological structure of biocomposites was determined using an optical microscope (OM) tool.

3 Results and Discussion

3.1 Density and Wave Velocity

The density values of epoxy resin and ER/DKP composites are reported in Table 4, and it is clear that there is a remarkable change in their values.

Table 4: The density (ρ) and ultrasonic wave velocities (V_L and V_S) of the ER matrix and ER/DKP biocomposites.

Sample ID	Density ρ (kg/m^3)	Longitudinal Wave Velocity V_L (m/s)	Shear Wave Velocity V_S (m/s)
ER	1138.08 (± 7.85)	2200.05 (± 3.75)	1058.27 (± 23.53)
COM305	1151.25 (± 2.58)	1841.25 (± 31.31)	1081.61 (± 16.37)
COM310	1158.10 (± 3.31)	1366.89 (± 6.60)	962.23 (± 48.55)
COM315	1165.14 (± 5.65)	1277.55 (± 67.59)	896.16 (± 21.15)
COM505	1147.02 (± 0.57)	1723.66 (± 14.98)	1080.71 (± 41.00)
COM510	1155.31 (± 1.41)	1995.93 (± 21.13)	1139.26 (± 17.15)
COM515	1151.14 (± 1.82)	1650.47 (± 28.25)	1016.86 (± 32.19)

The obtained experimental density ranged between 1138.08 kg/m^3 and 1165.14 kg/m^3. The density values of biocomposites are higher than those of ER and are increased by approximately 1.16%, 1.76%, 2.38%, 0.79%, 1.51%, and 1.15% for COM305, COM310, COM315, COM505, COM510, and COM515 biocomposites, respectively, compared to the ER matrix. The highest density value was recorded for COM315. This is the reason for better interfacial adhesion between pure ER and DKP [49]. Also, according to the variation in density values given in Figure 4, ρ values of the ER/DKP biocomposites increase with an increase

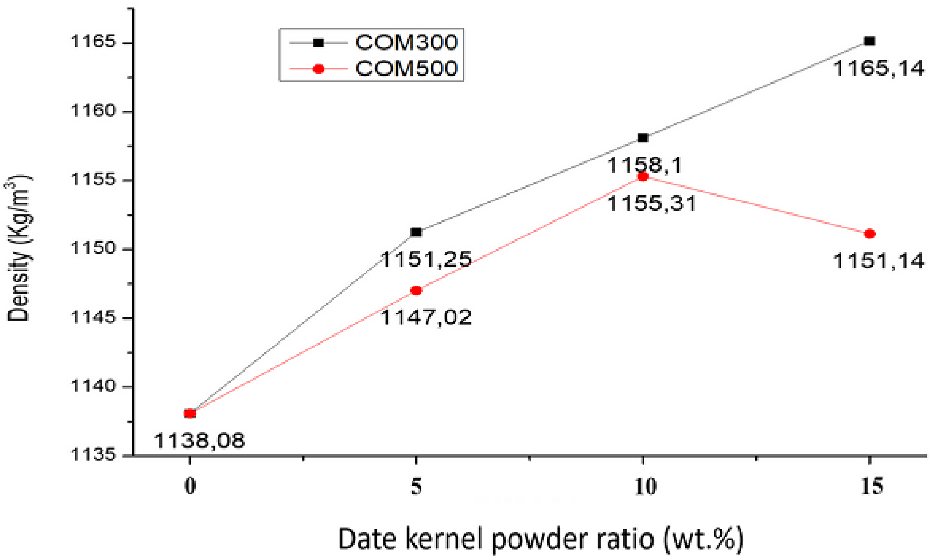

Figure 4: Variation in density (ρ) for ER matrix and ER/DKP biocomposites.

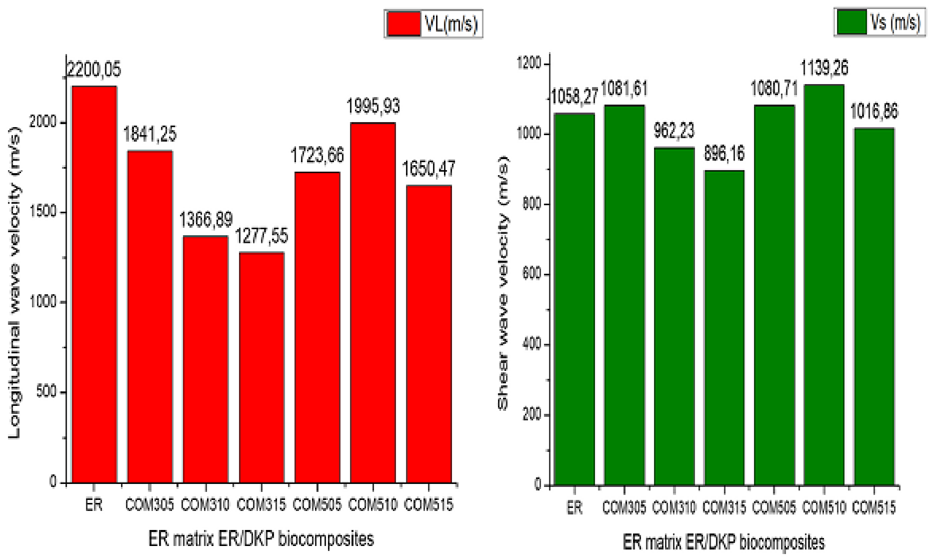

Figure 5: Variation in longitudinal wave velocity (V_L) and shear wave velocity (V_S) for the ER matrix and ER/DKP biocomposites.

in the DKP size 300 μm as compared with the unreinforced ER matrix. However, in the case of powder size 500 μm the density values of the ER/DKP biocomposites increase with an increase in the weight of DKP up to 10 wt% but decrease slightly when the weight of DKP is increased up to 15 wt%. This decrease may be due to an insufficient epoxy matrix to blend with the DKP. As seen also in Table 4 and Figure 4, the density values of the ER/DKP biocomposites with powder size 300 μm are higher than the density values of the ER/DKP biocomposites with powder size 500 μm in the same loading. This can be attributed to the role of reinforcement density and high volume of DKP, where good bond was found in the case of smaller DKP, unlike the presence of voids between the large DKP [50], [51].

The relationship between the ultrasonic wave velocities and the elastic constants of materials provides important information about the elastic and mechanical properties of materials [18].

As shown in Table 4, the values of longitudinal wave velocities V_L of the studied materials ranged from 1277.55 m/s to 2200.05 m/s. Also, V_L values of ER/DKP biocomposites are less than that of neat ER. The velocity values V_L of the COM305, COM310, and COM315 composites decreased by approximately 16.31%, 37.87%, and 41.93%, respectively.

In addition, the COM505, COM510, and COM515 composites decreased by approximately 21.65%, 9.28%, and 24.98%, respectively, compared to the ER matrix. On the other hand, the shear wave velocity values V_S of the ER/DKP biocomposites of the studied materials ranged from 902 m/s to 1139 m/s. In addition, the V_S values of the ER/DKP biocomposites were higher than that of ER, except for the COM310, COM315, and COM515 samples, which had the minimum values. The shear velocity

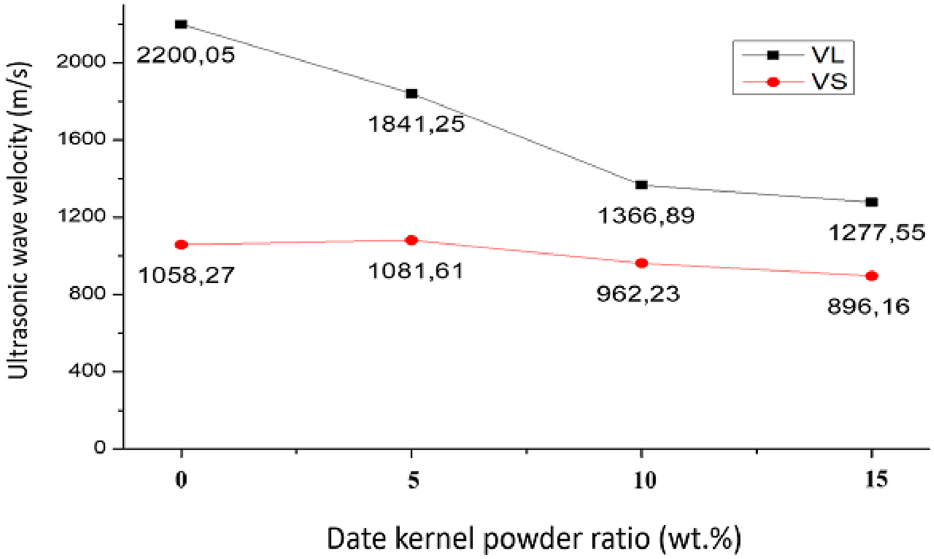

Figure 6: Variation in ultrasonic wave velocities (V_L and V_S) for the ER matrix and ER/DKP biocomposites with a powder size of 300 μm.

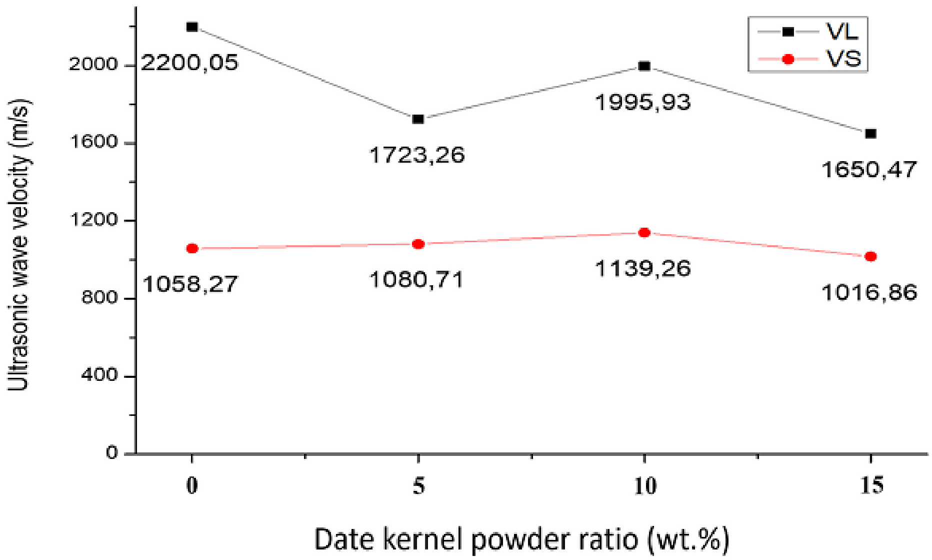

Figure 7: Variation in ultrasonic wave velocities (V_L and V_S) for the ER matrix and ER/DKP biocomposites with a powder size of 500 μm.

values V_S of COM305, COM505, and COM510 increased by approximately 2.21%, 2.12%, and 7.65%, respectively, whereas those of COM310, COM315, and COM515 decreased by approximately 9.08%, 15.32%, and 3.91%, respectively, compared to the ER. The same morphology of evolution of wave velocities V_L and V_S as a function of the loading ratio of powder in the two-configuration biocomposites studied was observed (Figure 5). The highest values for V_L and V_S were seen for COM300 and COM500 biocomposites in the 95:5wt% and 90:10wt%, respectively.

3.2 Elastic Moduli

The experimental values of the elastic moduli (L, G, K, and E) of the ER matrix and ER/DKP obtained by different DKP incorporation contents are reported in Table 4. The variation of these elastic moduli is illustrated in Figure 8. As observed from Table 4, the longitudinal modulus L values range between 3.84 and 9.26 GPa, the shear modulus G values range between 0.95 and 1.50 GPa, the bulk modulus values K range between 2.57 and 7.26 GPa, and the Young's modulus E values range between 2.54 and 4.22 GPa for pure ER and ER/DKP biocomposites. It is consistent with the results of the study [23]. Also, from Table 5, it can be noticed that the values of the elastic moduli (L, G, K, and E) of ER/DKP in the case of powder size 300 μm are lower than pure ER, except for COM305 composite, which have the maximum values, by increasing about 29.77%, 5.18%, 37.12%, and 8.73% for L, G, K, and E, respectively. But on another hand, in the case of powder size 500 μm the values of the elastic

Table 5: Elastic constants of the ER matrix and ER/DKP biocomposites.

Samples ID	Longitudinal Modulus L(GPa)	Shear Modulus G(GPa)	Bulk Modulus K(GPa)	Young's Modulus E(GPa)
ER	5.52(±0.01)	1.28(±0.05)	3.81 (±0.04)	3.45 (±0.07)
COM305	7.86(±0.13)	1.35(±0.04)	6.06(±0.03)	3.78(±0.09)
COM310	4.39(±0.02)	1.08(±0.10)	2.94(±0.07)	2.90(±0.10)
COM315	3.84(±0.20)	0.95(±0.04)	2.57(±0.03)	2.54(±0.08)
COM505	6.91(±0.22)	1.34(±0.10)	5.12(±0.08)	3.70(±0.02)
COM510	9.26(±0.09)	1.50(±0.05)	7.26(±0.07)	4.22(±0.11)
COM515	6.34(±0.10)	1.19(±0.07)	4.75(±0.09)	3.30(±0.07)

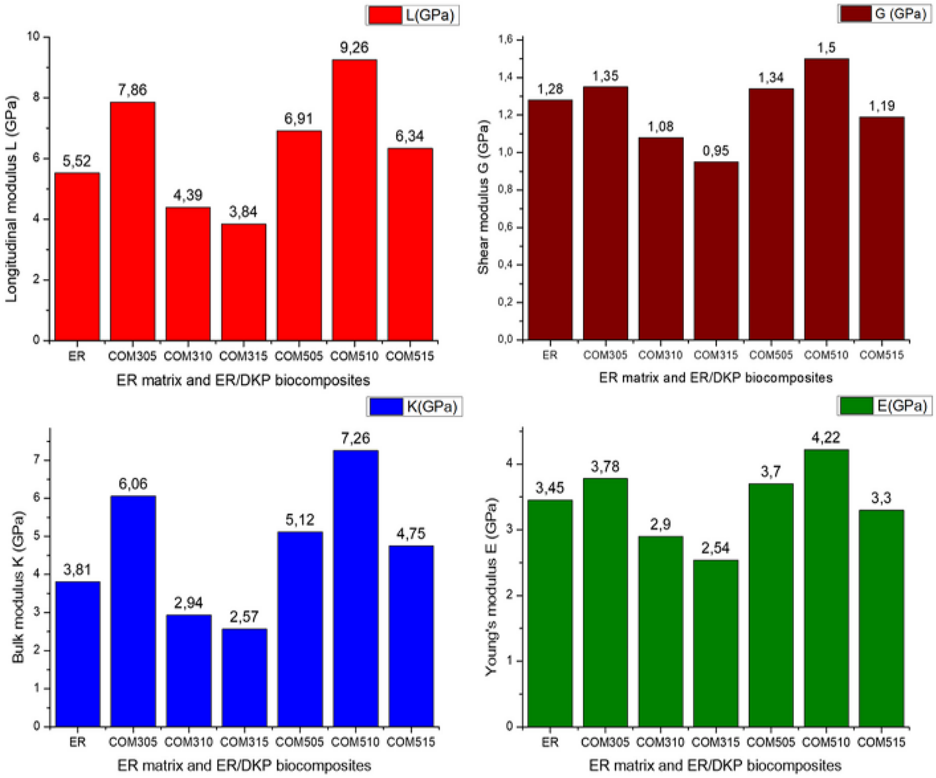

Figure 8: Variation in elastic moduli (L, G, K, and E) for the ER matrix and ER/DKP biocomposites.

moduli (G and E) of ER/DKP are higher than pure ER, except for COM515 composite, which has the minimum value, by decreasing about 7.56% and 4.54% for G and E, respectively.

The value of Young's modulus of elasticity (E) of the ER matrix increased from 3.45 to 3.70 and 4.22 GPa after the incorporation of 5 wt% and 10 wt% of DKP for size 500 μm of powder, respectively, by approximately 6.75% and 18.24% for 5 wt% and 10 wt% of powder, respectively. In addition, these results show that in the case of powder size 500 μm the values of the elastic moduli (L and K) of ER/DKP are higher than those of pure ER. As an example, in the case of powder size 500 μm the value of longitudinal modulus (L) of the ER matrix increased from 5.52 GPa to 6.34 and 6.91GPa for 15wt.% and 5wt.% of powder, respectively, until the maximum value 9.26GPa for 10wt.% of DKP, by about 20.11%, 25.37%, and 12.93% for 5 wt.%, 10 wt.%, and 15 wt.% of powder.

In the case of powder size 500μm, the bulk modulus (K) value of the ER matrix increased from 3.81 GPa to 5.12 and 4.75GPa for 5wt.% and 15wt.% of powder, respectively, until the maximum value 7.26GPa for 10wt.% of DKP, by about 25.58%, 47.52%, and 19.78% for 5 wt.%, 10 wt.%, and 15 wt.% of DKP. These results show the improvement of all elastic moduli (L, G, K, and E) by the incorporation of 5% and 10% of kernel powder with 300 μm and 500μm, respectively, which can be considered as the optimum value of the weight of the reinforcement in these sizes of powder. This result explained that the strong bonding formed between the ER matrix and the reinforcement DKP in these weights, which explained the decrease in the movement of the polymer chains, led to material characteristics strength [49], [52].

Data from Table 5 and Figure 8 show that the values of elastic moduli L, G, K, and E have the same behavior as a function of powder size. We find that in the case of 300 μm powder size, these values decrease when the weight of the powder increases from 5 to 15 wt.%. On the other hand, in the case of 500 μm powder size, the values of elastic constants moduli increase when the powder weight is from 5% to 10% and slightly decrease when the powder weight increases up to 15%. The decrease in these values may be due to insufficient adhesion between the natural powder and epoxy matrix upon further increasing the powder

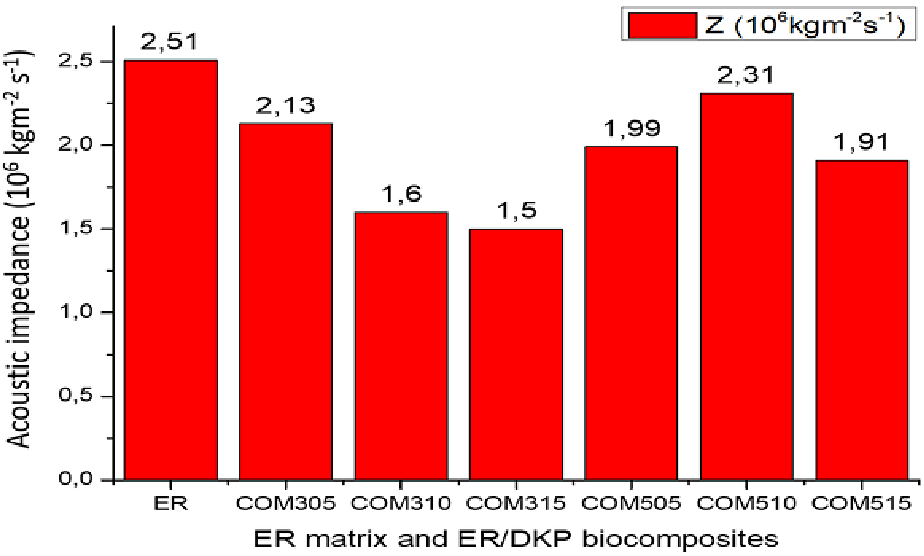

Figure 9: Variation in acoustic impedance Z for the ER matrix and ER/DKP biocomposites.

percentage. This also leads us to predict the optimum and maximum powder loading values.

3.3 Acoustic Impedance, Poisson's Ratio, and Ultrasonic Microhardness

According to Figure 9 and Table 7, a pattern of change in Z values through the change in powder weight was detected. The ultrasonic impedance Z ranged from 1.5 to 2.51 $\times 10^6 kgm^{-2}s^{-1}$(Figure 9).

Table 6: Variation of Z, H, and μ of the pure matrix ER and ER/DKP biocomposites.

Sample ID	Acoustic Impedance Z (10^6 kg·m^{-2}·s^{-1})	Ultrasonic Micro-Hardness H (GPa)	Poisson's Ratio μ
ER	2.51 ($\pm$ 0.02)	0.131 ($\pm$ 0.005)	0.349 ($\pm$ 0.003)
COM305	2.13 ($\pm$ 0.02)	0.094 ($\pm$ 0.003)	0.396 ($\pm$ 0.004)
COM310	1.60 ($\pm$ 0.07)	0.119 ($\pm$ 0.006)	0.335 ($\pm$ 0.001)
COM315	1.50 ($\pm$ 0.03)	0.105 ($\pm$ 0.005)	0.335 ($\pm$ 0.001)
COM505	1.99 ($\pm$ 0.01)	0.107 ($\pm$ 0.001)	0.379 ($\pm$ 0.004)
COM510	2.32 ($\pm$ 0.02)	0.097 ($\pm$ 0.006)	0.403 ($\pm$ 0.004)
COM515	1.91 ($\pm$ 0.01)	0.092 ($\pm$ 0.005)	0.384 ($\pm$ 0.003)

Z decreased from 2.51 to maximum value 2.13 $\times 10^6 kgm^{-2}s^{-1}$, when neat matrix reinforced with 5 wt%, and also decreased to 1.6 and 1.5 $\times 10^6 kgm^{-2}s^{-1}$ when neat matrix reinforced with the 10wt.%, and 15wt.% respectively, in the case of powder size 300μm(Table 7). Also, Z decreased from 2.51 to 1.99 and 1.91 $\times 10^6 kgm^{-2}s^{-1}$ when neat matrix reinforced with 5wt.% and 15wt.% of powder, respectively, in the case of 500 μm of powder, until the case of 10wt.% weight when the value of the Z decreases to 2.32 $\times 10^6 kgm^{-2}s^{-1}$ by about 8.62%.

This parameter is considered an indicator of a material's resistance when wave propagates in it [24]. Therefore, COM305 and COM510 are the most desirable biocomposites and have higher durability against propagation sound waves by their 2.13 and 2.32$\times 10^6 kgm^{-2}s^{-1}$ values of Z, respectively(Figure 9). The obtained results for ultrasonic impedance Z agree with the ultrasonic wave velocities.

The Poisson's ratio μ values were calculated using Eq. (6). μ values ranged from 0.335 to 0.403 (Table 7). These results are consistent with the study of Oral et al. [53], who mentioned that the Poisson's ratio of most materials ranges between 0.0 and 0.5. μ decrease from 0.349 to 0.335 when neat matrix reinforced with the 10wt% and 15wt.% in the case of powder size 300μm, except for the loading weight 5wt.% the value of the Poisson's ratio increases up to a maximum value of 0.396(Figure 10). On another hand, in the case of 500 μm of powder size, μ increased from 0.349 to 0.379 and 0.384 when neat matrix reinforced with 5 wt.% and 15 wt.% of powder, until it reached a maximum value of 0.403 in the case of 10 wt.% weight. Oral et al. [53] declared that in their contribution, the assumption of an inverse relationship between Poisson's ratio and elastic constants is not true every time. As shown in Figure 11, the microhardness H values ranged from 0.092 to 0.119 GPa. The highest values of microhardness were observed for 10 wt.% in the case of 300 μm of powder and 5 wt.% in the case of 500 μm of powder with values of 0.119 GPa and 0.107 GPa, respectively. The increase and decrease in microhardness justify the role of powder on the biocomposite. For example, in the case of powder size 300μm, the optimum weight is 10 wt.% of powder and 5 wt.% of powder in the case of 500 μm. Then, H of ER/DKP biocomposites decreased slightly to 0.092 GPa by increasing DKP weight from 5 wt.% to 15 wt.% in the case of powder size 500 μm.

On the other hand, in the case of powder size 300 μm, the microhardness increased from 0.094 to the maximum value of 0.119 GPa by DKP addition ratio 5 wt.% to 10 wt.% and decreased to 0.105 GPa by increasing DKP weight from 10 wt.% to

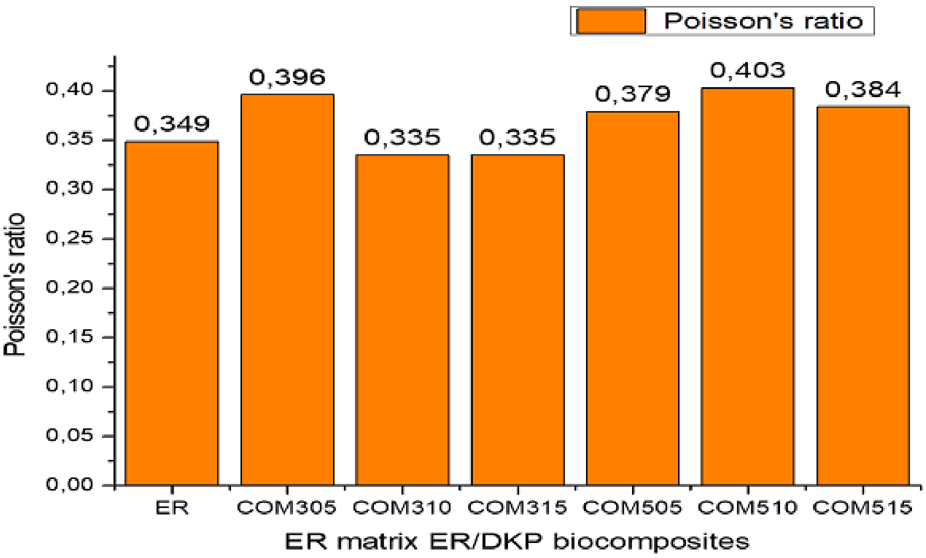

Figure 10: Variation in Poisson's ratio μ for ER matrix and ER/DKP biocomposites.

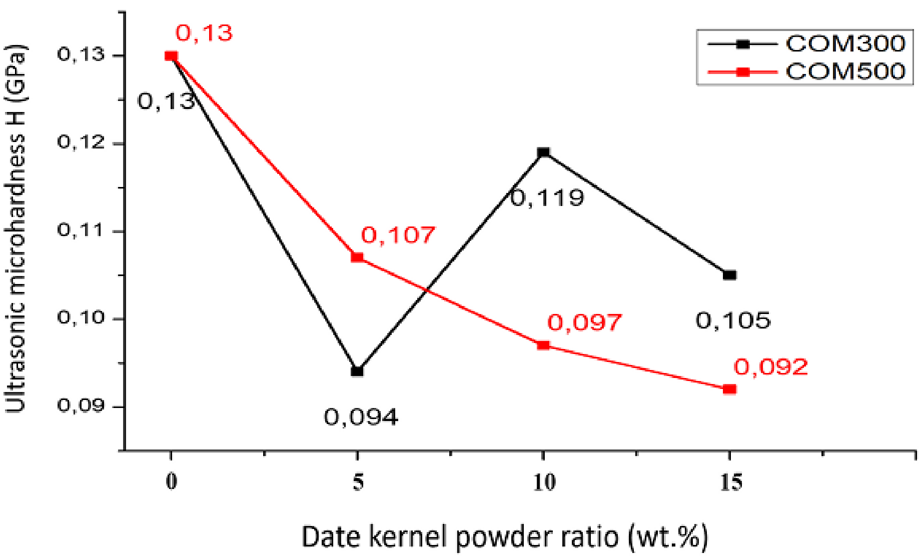

Figure 11: Variation in ultrasonic microhardness H for the ER matrix and ER/DKP biocomposites.

15 wt.% in the case of powder size 300 μm. Table 8 summarizes some ultrasonic investigations on the mechanical behavior of the obtained biocomposite materials.

3.4 XRD Analysis Results for the ER and ER/DKP Biocomposites

Figure 12 represents the XRD patterns of the ER matrix and ER/DKP biocomposites. This shows that the XRD peaks of both the samples of size 300 μm and 500μm were shaped in the same way as the ER matrix. On the other hand, XRD peaks show the amorphous type of the biocomposites and have a higher intensity than the ER matrix, which increased with increasing powder loading in the case of 300 μm. All these results confirmed the good dispersion of the biopowder of date kernel powder in the

Table 7: Comparison of the ultrasonic properties of ER/DKP biocomposites with those of other powder-based composites.

Material	Fiber sizes (μm)	L, G, K, E maximum values	μ and Z values	Treatment condition	Refs
DKP/Epoxy	300 500	7.86,1.35,6.06,3.78 9.26,1.50,7.26,4.22	0.396,2.13 0.403,2.32	fiber not treated	This study
DPS/Epoxy	200	8.89, 1.45, 6.96, 4.06	0.411, 3.33	treated fiber	[18]
Polyaniline/Epoxy	< 63	9.02,1.51,7.00,4.24	0.400,3.26	fiber not treated	[17]
Marble/Epoxy	/	7.37, 1.20, 5.89, 3.14	0.410, 2.78	fiber not treated	[24]
Pine cone char/Epoxy	< 60	9.61, 2.09, 6.83, 5.68	0.374, 3.42	fiber not treated	[23]
Polyaniline/Epoxy	< 63	10.45, 1.98, 8.03, 5.40	0.398, 3.72	modified resin	[20]
Coconut/Epoxy	< 63	10.27, 2.08, 7.83, 5.66	0.393, 3.53	modified resin	[46]

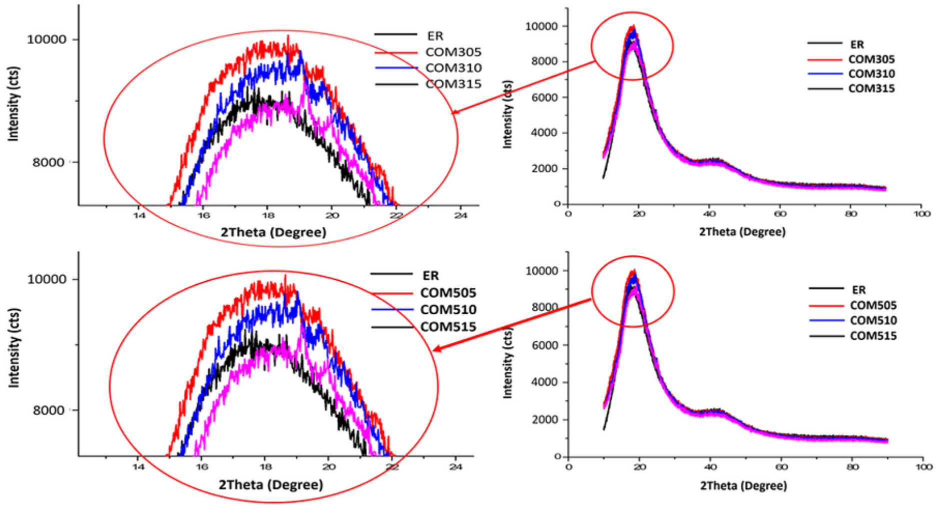

Figure 12: X-ray diffraction curves of the ER matrix and ER/DKP biocomposites.

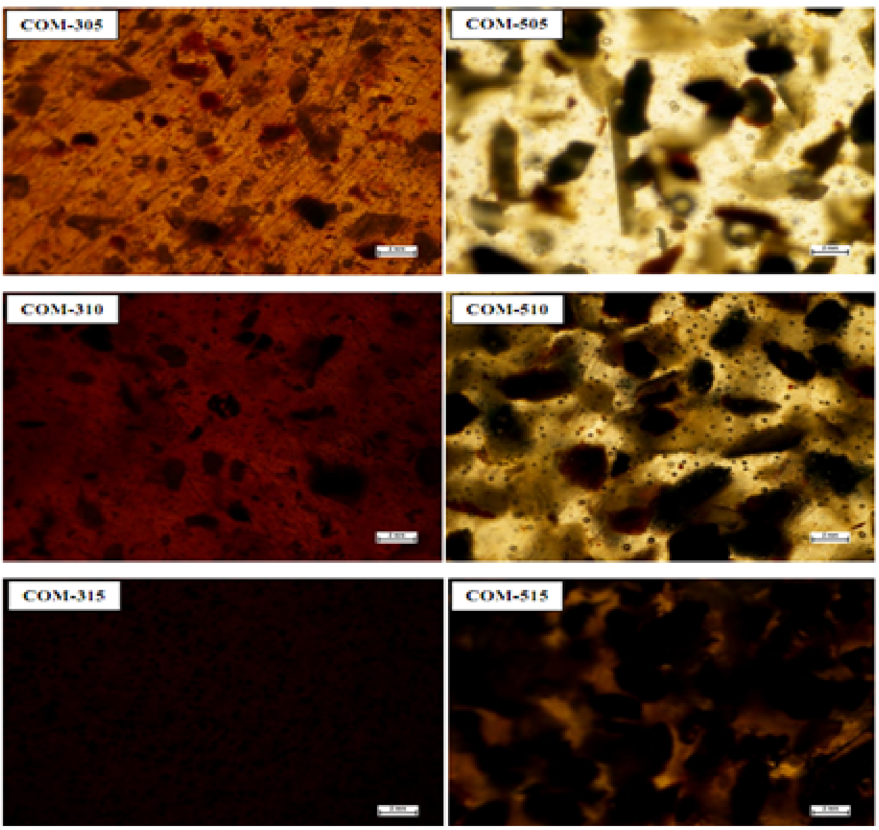

Figure 13: Macroscopic view of the ER/DKP biocomposite samples.

ER matrix [46]. The crystalline index of the biocomposites was measured as 98.10% for the ER matrix, 89.05% for COM305, 80.51% for COM310, 80.38% for COM315, 80.24% for COM505, 78.58% for COM510, and 73.14% for COM515. The same results concerned the increase in the crystalline index in the study of Khosravani et al. [48]. An increase in the crystalline index was observed when the particle size decreased. This high crystallinity index is due to lignin breakage during the grinding process [48], [54].

3.5 Morphological Structure of the ER/DKP Biocomposites

Homogeneity plays an important role in the physical and mechanical properties of filler composites. In addition, the distribution and dispersion of filler in the hydrophilic polymer matrix is a critical issue related to biocomposite manufacturing that needs to be controlled [55], [56]. Therefore, morphological analysis of the ER/DKP biocomposites is essential to explore the effect of DKP loading on adhesion quality.

Figure 13 shows the morphology in terms of size and appearance of the samples prepared with the two powder sizes 300 μm and 500μm. Figure 13 shows the homogenous distribution of powder with varying loading of DKP, where we found a good distribution of the powder due to the increase in powder concentration. Asyraf et al. [56] reported that biocomposite materials reinforced with low fiber loading exhibited more homogenous dispersion with less fiber aggregation than biocomposites reinforced with high fiber loading. On the other hand, some black spots appear in the form of a few agglomerations, especially in biocomposites with higher loading, such as COM315 and COM515. Powder agglomeration is the main cause of weak part formation in which damage initiates and then propagates [55].

4 Conclusions

Within the scope of this study, date kernels, which are among the most abundant agricultural wastes in Algeria, were produced as environmentally friendly biocomposites as an alternative to synthetic composites that pollute nature. The mechanical properties of these biocomposites were determined by ultrasonic through-transmission, which is a non-destructive test method. The experimental results obtained within the scope of this study can be summarized as follows:

- Following a thorough analysis, the different grain sizes of DKP have a crucial effect on the elastic and mechanical properties of the proposed biocomposite.
- The highest ultrasonic values of wave velocities and elastic constants (L, G, K, E, and Z) were observed for the COM305 and COM510 samples. Therefore, it can be stated that the optimum weight percent of DKP reinforcement in neat ER for excellent mechanical behavior of ER/DKP biocomposites is 5% and 10% for 300 μm and 500μm, respectively.
- In the case of comparing the results of elastic moduli of the two studied configurations COM300 and COM500 through the weight rate of DKP, the biocomposite with a powder size of 300 μm and a ratio of 5% is better than of 500 μm biocomposites, but in the case of a ratio of 10% and 15% of DKP, the biocomposites 500 μm are better than of biocomposites 300 μm.
- The highest microhardness values (H) were observed for the COM310 and COM505 samples. Therefore, it can be stated that the optimum weight is 10 wt.% and 5 wt.% for the DKP sizes of 300 μm and 500 μm, respectively.
- The results indicate that the ultrasonic through-transmission method is helpful in determining the elastic properties of ER/DKP biocomposites.

Overall, this study might open new avenues for utilizing the proposed DKP reinforcement for different real-life applications. Similar studies can be conducted using treated DKP having different grain sizes. In addition, the ultrasonic method may be considered the best method for determining the mechanical behaviors of biocomposites.

Authors' Contributions

Fares Mohammed Laid Rekbi: Conceptualization, Writing-original draft, Review, and editing, Performing the ultrasonic test, Analyzing the results. Rafik Halimi: Conceptualization, Writing-original draft, Review, and editing, Performing the ultrasonic test, Analyzing the results. Mabrouka Oustani: Review and editing, Analyzing the results. Imran Oral: Visualization, Writing-original draft, Review, and editing, Analyzing the results. Wahiba Djerir: Review and editing, Analyzing the results. Fethi Remli: Review, and editing, Analyzing the results. Hicham Henna: Editing.

Competing Interests

The authors have no relevant financial or non-financial interests to disclose.

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