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COMPARISON OF THE MECHANICAL PROPERTIES OF LOW-COST BIO FIBER REINFORCED POLYMER COMPOSITES

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In this study, the physical, and mechanical properties of low-cost and biocomposites were evaluated. The walnut shell and date palm frond fibers were thermally treated in an oven at a temperature of *70°C* and then chemically treated with NaOH and distilled water solution, after these treatments, the biofiber materials were thermally treated again at *50°C*. Three types of biocomposite; walnut shell fiber reinforced polymer (WFRP), date palm fiber reinforced polymer (DFRP), and hybrid fiber reinforced polymer (HFRP), whereas the biocomposite sheets consisting of *30%* biofibers and *70%* of unsaturated polyester, the mechanical test specimens were cut by a CNC machine according to ASTM standards. The effect of fiber type was analyzed in terms of the mechanical properties (tensile, compression, and density). The tensile test results showed that the DFRP composite had the best results comparable with WFRP and HFRP composites, the ultimate tensile strength, was increased by *28.6%* and *12.5%* respectively, furthermore, the compression strength of the WFRP composite was increased by *21.5%* and *10.3%* compared with DFRP and HFRP composites respectively. The WFRP composite revealed the lowest value of density $4.60\ g/cm^3$ rather than DFRP and HFRP composites.

Key words: biocomposite, walnut shell, date palm fronds, tensile test, compression test.

1. Introduction

Recently, many researchers focused on natural fibers that contained cellulose as reinforcement for composite materials instead of synthetic fibers. The aim of using biofibers in modern applications is the ability to improve the bond between the surface of the fibers that contain cellulose and the matrix as well as availability in nature, environmentally friendly, and their properties that can be enhanced through various treatments to be used as reinforcement in composite materials [1]. The composite mechanical properties depend on many factors; fiber homogeneity with the matrix, cellulose percentage in the fiber, fiber (length, diameter, and orientation), also the percentage of composite constitutes fiber and matrix [2]. Like the properties, the strength of biocomposites is influenced by several factors which are fiber strength, matrix strength, fiber percentage, adhesion bond between fiber and matrix, and distribution of the fiber [3]. In general, the behavior of the materials is usually expressed in terms of their various mechanical properties like tensile, compression, bending, impact, and shear behavior. These properties are important in identifying the material capability under the maximum applied load and critical circumstances, which are related to the engineering performance. The good adhesion interface between the matrix and fibers allows the matrix to transfer the applied load, shear stresses between the fibers, and the composite material possesses good mechanical properties. Conversely, composites with poor mechanical properties resulted from poor adhesion interface between the matrix and the fibers [4]

Thermoplastics and thermosets improved by being reinforced with biofibers , these biocomposites had good mechanical properties, and chemical resistance which made them suitable for different applications such as aircraft structures and industrial parts due to lightweight to-performance ratio, low cost, and high strength, as well as the capability of recyclability and renewability when compared with synthetic fiber like glass fiber, and biodegradable materials lack of health hazard and nonabrasive nature [4-6]. The new hybrid composite is

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recommended for high performance, low cost, good insulation, building systems, aerospace, and automotive parts[7]. There are several treatments performed on composite materials to ensure better properties the chemical treatment of the fibers is a general procedure to clean the surfaces, reduce the moisture of the fibers, and surface tension, and improve the surface adhesion bond between the matrix and the fiber, leading to good mechanical properties[5].

Recycling environmental waste is very important because every year millions of tons of nuts are produced worldwide[8] also Iraq is the origin of the date palm, and it was the taming center of this harvest. Furthermore, for some years, Iraq was the major producer of dates in the world [9]. To benefit from the natural environmental waste produced by this crop, this study proposed fabricating biocomposite materials from date palm fronds and walnut shell fiber.

The reviewed literature presents an overview of the characterization of biocomposites with different bio fibers with studies of the mechanical properties of biocomposites.

Hamid *et al.* [10] studied the mechanical and physical properties of recycled high-density polyethylene reinforced with a high-loading hybrid of rice husks and sawdust with a thickness of *3 mm*, and found that the properties of biocomposites significantly increased with the addition of antioxidants and fire retardant content. Reza *et al.* [11] used cotton filter waste reinforced corn starch biocomposite with *3.2 mm* thickness and deduced that the biocomposite properties were affected by varying percentages of the cotton waste filter. They concluded that *50%* of the cotton waste filter had a good tensile strength, Young's modulus, toughness, impact strength, bending strength, and thermal conductivity. Also, the SEM presented a good adhesion between fiber and matrix at *50%* of cotton waste. Ramakrishnan *et al.* [12] fabricated flax reinforced with a volume fraction of *40%* with polypropylene thermoplastic by compression molding. The effect of temperature on tensile strength was studied and found to be inversely proportional and the impact of increasing pressure and temperature on minimizing pores and improving the crystallinity of biofibres was demonstrated. Zarna *et al*. [13] compared the bending properties of different cellular structures for utilization in panels made of recycled pine wood with *15* wt.*%* filled PLA/PHA filament to make biocomposite with a thickness of *8 mm* by material extrusion 3D printing method and determined the mechanical properties of this biocomposite.

Yan *et al.* [14] discussed the untreated and treated coir fibres as a reinforcement for epoxy (CFRE) and cementitious (CFRC) composites with a thickness of *5 mm*. They showed that the treated coir fiber in CFRE is clearer and rougher than the untreated one, also that the tensile strength, Young's modulus, and flexural strength increased. In addition to Coir fiber in CFRC composite provided better compressive stress, the facture energy increased by *550%*, and flexural toughness increased by *424%*. Li *et al.* [15] fabricated biocomposite from miscanthus biocarbon (MB), and Poly(3-Hydroxybutyrate-co-3-Hydroxyvalerate) (PHBV) with *0*, *10*, *20*, *30%* of MB. The tensile strength at a speed of *5 mm/min* were *38.3*, *32.2*, *33.1*, and *32.8 MPa* respectively. It concluded that the biocomposite with *30* wt.*%* biocarbon showed a *46%* increase in Young's modulus and a *14%* decrease in tensile strength.

Yadav *et al*. [16] investigated the treatment of natural fiber (sisal, banana, bagasse) with NaOH. When the composite material was made from poly-lactic acid reinforced by natural fibers at *10%* and *20%* percentage of natural fibers. The results indicated that chemical treatment reduced the wear rate and friction coefficient of composites; also concluded that natural fibers are viable options for reinforcing polymers to improve the tribological properties.

Zhang *et al.* [17] compared the properties of polyethylene reinforced with peanut husk, rice husk, and walnut shell with different percentages of fiber content. The fibers were sieved and heat treated for *24* hours at *105°C*. it was observed that the tensile and bending strength of the three composites increased when fiber contents increased. The mechanical strength of the rice husk/polyethylene composite was the best compared with the other composites. Chandramohan, and Kumar [18] developed bio epoxy reinforced by powder coconut shell, walnut shell, and rice husk. Various treatments were performed on these hybrid materials. The results showed that the maximum water absorbed was *4.6 g* for hybrid rice husk and coconut composite comparable with the two hybrid composite types. The tensile strength of hybrid walnut and coconut shells composite is *68.8* and *69.5 MPa* for both with and without moisture respectively comparable with the other two types of biocomposites. Anwara, and Barinem [19] conducted that the tensile strength of ukam fiber-reinforced cashew nut shell resin composite was good for low-cost applications comparable with glass fiber with a tensile strength of *40.5 MPa*. Chegdani, El Mansori [20] fabricated *40%* unidirectional flax fiber as reinforcement with *60%* polypropylene as a matrix to make a UDF/PP composite with a thickness of *4 mm*, then investigated the properties of the biocomposite were *17.6 GPa* for tensile modulus, *109 MPa* for tensile strength, and *1.3%* maximum strain. Miliket *et al*. [21] focused on the mechanical properties of unsaturated polyester resins reinforced by sisal leaves and nacha stems. The characterizations of *3 mm* composite materials were conducted by Taguchi and ANOVA methods to find the maximum value of 16 experiments which were *220.12 MPa* tensile stress, and *308.55 MPa* compression stress.

Wambua *et al.* [4] compared the mechanical properties of natural fibers like sisal, kenaf, hemp, coir, and jute that reinforced polypropylene composites. It was found that hemp composites have the highest value of mechanical properties. Also, it suggested that natural fibers can replace glass fiber in many fields that do not want a high value of load-bearing capabilities. Rong *et al*. [22] improved the strength of sisal fiber by alkali treatment and also enhanced the sisal fiber adhesion with an epoxy matrix which led to improve mechanical properties. Sreenivasan *et al*. [5] concluded that the interfacial bond between the biofiber (short sansevieria cylindrica), and the matrix (polyester) was enhanced by the proper choice of chemical treatment which led to improve mechanical properties of biocomposites. Ayrilmis *et al.* [23] fabricated from polypropylene and reinforced with walnut shell flour with different percentages, the thickness of the composite sample was *3.2* mm. It was found that the bending and tensile strength increased with an increase in the percentage of walnut shell flour. The composite sample with *40%* walnut shell flour, *57%* polypropylene and *3%* MAPP showed an outdoor application due to its stable dimensions. Abdellah *et al*. [7] prepared a composite from polyester reinforced by date palm fiber and sheep wool with a different fiber content of *3.2 mm* thick, the date palm fiber was treated with NaOH. The results showed the best mechanical properties of date palm fiber and sheep wool hybrid reinforced polymer at *20%* fiber content due to the good adhesion bond between fiber-matrix. El-Shekeil *et al*. [3] conducted the effect of various percentages of date palm frond fiber on nitrile rubber with additional materials such as zinc oxide, aromatic oil, etc. At *40* wt. *%* of fiber, the modulus of elasticity and tear resistance increased, and the compression strength improved at the same fiber percentage.

Balogun *et al*.[24] used African walnut shell and jute fiber to reinforce polypropylene with varied percentages of walnut shell and jute fiber. Walnut shell heat treated and chemically treated by NaOH, the composite samples with *5 mm* thick. Taguchi method showed the optimum experiment of tensile and flexural properties at the composite sample with *4%* wt. of walnut shell and *25%* wt. of jute fiber which led to use it for lightweight automobile applications. Zaman and Beg [25] fabricated polypropylene reinforced by coir fiber that was already chemically treated which showed better mechanical properties than untreated fibers. The best properties such as tensile strength and impact strength were achieved at *30* wt. *%* of coir fiber.

In this paper, walnut shell and date palm fibers were treated by chemical and heating processes to improve the adhesion bond with the unsaturated polyester resin then fabricate three types of biocomposite materials, after that cutting the biocomposites according to ASTM standards dimensions to perform mechanical properties tests; tensile, compression, and density tests.

2. Experimental procedure

In this section, it will be discussed the experimental methods used to manufacture three biocomposite materials, and the bio fibers treatments, then obtain the characterization of each material.

2.1. Materials and methods

In this paper, three composite materials were manufactured from two different biomaterials which are walnut shells, and date palm fronds, also the chemical and heat treatments were carried out, block diagram in Fig.1 shows the harvesting and treatment of bio fibers. Walnut shell and date palm fibers were used as substantial materials to fabricate biocomposites based on unsaturated polyester as the matrix, and their properties were tested and compared in this paper.

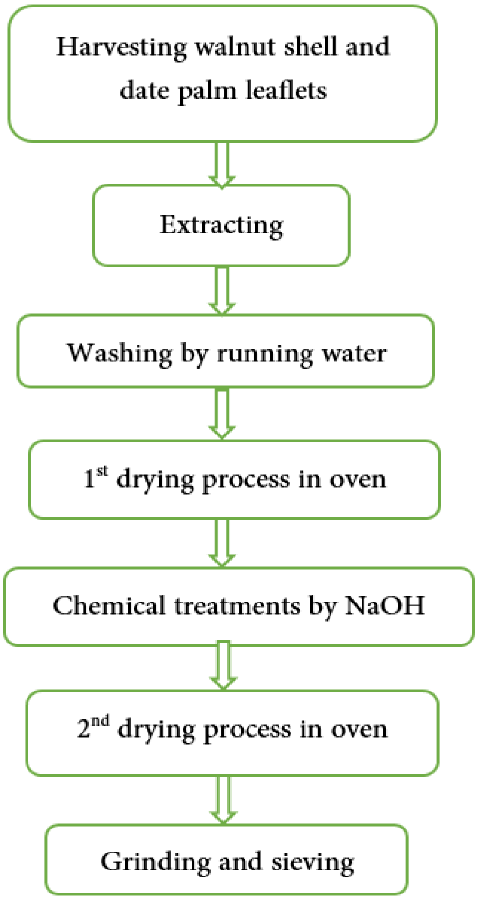


Fig.1. Bio fibers harvesting and treatments.

2.1.1. Bio fiber treatments

American walnut shells, and local date palm tree fibers were cut by scissors from fronds of date palm trees on Iraqi farms, as shown in Fig.2a, b respectively were collected and rinsed well with tap water to remove contaminates, sand, and dust from it, then dried in an oven at *70°C* for *2* hours and *45* minutes respectively. The basic ingredients of the walnut shell are hemicellulose, lignin, and cellulose [24], on the other hand, the chemical analysis of date palm fiber showed that contained *46%* cellulose, *20%* lignin, *20%* hemicellulose, and *5%* moisture [26].

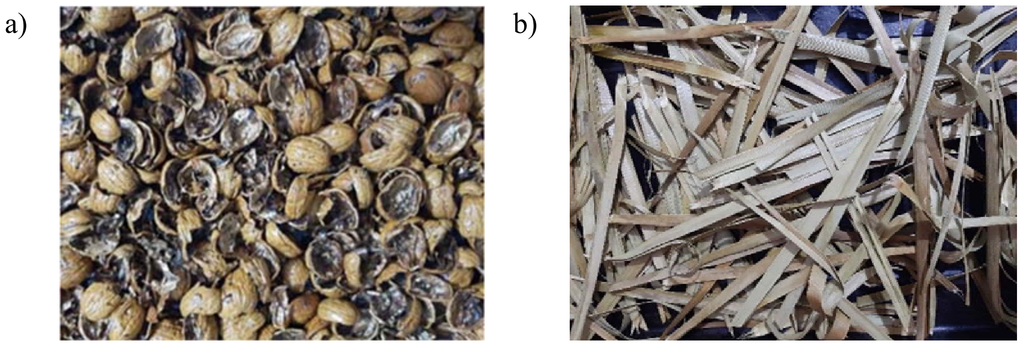


Fig.2. Bio fibers used in this study (a) walnut shell, (b) date palm fronds.

Chemical treatment was done for walnut shells and date palm fronds by immersing them in *2%* sodium hydroxide (NaOH) pellets provided by s d fine-chem limited (SDFCL), India; which dissolved in *98%* distilled water for *2* hours, the chemical treatment was done to increase the fiber strength and reduced the moisture of bio fibers below *2%*, as it removes the contaminants and achieves the molecular orientation stability [18]. Also depending on [15] and [24] that showed the treated biofiber (corn fiber) had a rougher and clearer surface comparable with the untreated one. Khakalo *et al.* [27] revealed that treated wood had tensile strength two times higher than the natural wood in the ambient temperature.

Then, the walnut shells and date palm fronds were taken out from the chemical solution and washed with stream tap water to remove excess sodium hydroxide from walnut shells and date palm fronds. Thereafter, another drying process was done in an oven at *50°C* for *1* hour and *30* minutes for the walnut shells and date palm fronds respectively. The grinding process is the last step of the walnut shells and date palm fronds preparation uses a blender to grind the biofibers into a small fiber, Fig.3 shows the biofibers after the grinding process, but not been sieved yet. Later the biofibers were sieved by a sieve to measure the fiber length, and it was found to be approximately *1 mm*.

a) b)

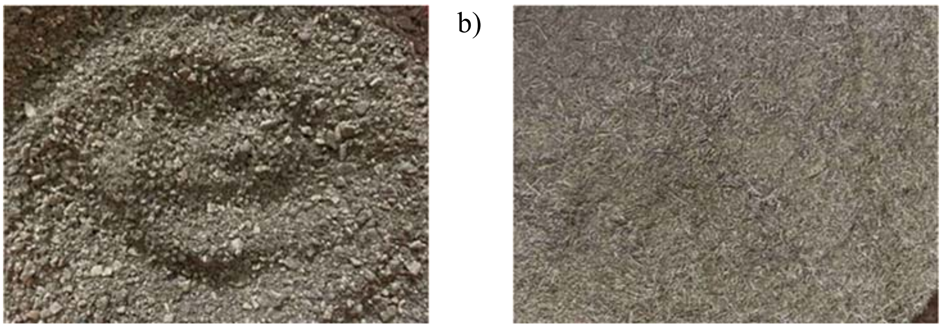


Fig.3. Bio fibers after grinding process (a) walnut shell fiber, (b) date palm fronds fiber.

2.1.2. Resin

The basic functions of resin are to transfer load between the fibers (reinforcement), act like glue to hold the fibers together, moreover to protect the fiber from mechanical and environmental damage, resin used in reinforced polymer composites are either thermosets or thermoplastics. Thermosets are used to fabricate most composite materials because they are cured by using catalysts, heat, pressure, or a combination of them, unlike thermoplastics which are converted from liquid to solid state by polymerization process.

Unsaturated polyester resin is a transparent thermoset used as a matrix in this study supplied by (TOPAZ-1110 TP general-purpose Resin-Saudi Arabia), which is a medium reactive resin based on phthalic Anhydride with excellent laminating properties with room temperature cure capability with gel time around (*15-20*) minutes, high strength and modulus, high water resistance, with catalyst as a hardener with weight percentage for unsaturated polyester to catalyst *100:2*. Table 1 summarized the details of each material used in this study.

Table 1. Materials used in this study.

material	type	supplied by
unsaturated polyester	liquid	TOPAZ-1110 TP general-purpose Resin-Saudi Arabia
catalysts	liquid	TOPAZ-1110 TP general-purpose Resin-Saudi Arabia
sodium hydroxide	pellets	s d fine-chem limited (SDFCL) India
walnut shell	particle	were obtained from a nearby local market (American type)
date palm fiber	particle	were collected from local date palm fronds on Iraqi farms.

2.2. Biocomposites fabrication

After the treatment of fibers, a glass rectangular mold with dimensions *420* (length) *mm* $\times$*297* (width) *mm*$\times$*5* (thickness) *mm* was prepared and covered the inner walls with nylon paper to prevent adhesion of the biocomposite with the walls after the biocomposite was hardened. Figure 4 shows the sequence of manufacturing and testing biocomposites.

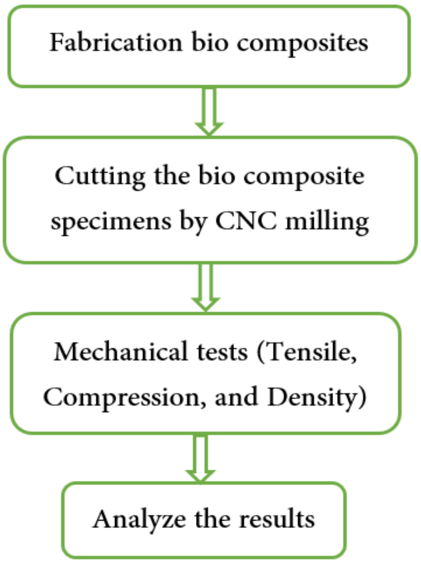


Fig.4. The sequence of fabrication and testing biocomposites.

Depending on the rule of the mixture to determine the weighted average of each composite's component which can define the properties of the manufactured composite materials and it is stated that the total mass of composite material is the sum of the mass of the reinforcement and matrix materials as cleared in Eq.(2.1) [28]

$$m_c = m_f + m_m \tag{2.1}$$

where m_c is the mass of the composite *g*, m_f is the mass of the reinforcement material *g*, and m_m is the mass of the matrix *g*.

Biocomposites with reinforcement contents of *30%* by weight were prepared in this study, the first type of biocomposite was walnut shell fiber reinforced polymer (WFRP) composite with *30* wt.*%* of walnut shell fiber as reinforcement, the second type of biocomposite was date palm fiber reinforcement polymer (DFRP) composite with *30* wt.*%* of date palm leaflet fiber, the last type was hybrid fiber reinforced polymer (HFRP) composite which made from *15* wt.*%* of walnut shell fiber, and *15* wt. *%* date palm leaflet fiber as reinforcement, for the three types of biocomposite the *70* wt.*%* an unsaturated polyester resin. The weight percentage of each bio composite sheet is summarized in Tab.2.

Table 2. Weight percentage of reinforcement of the biocomposites.

type of biocomposites		walnut shell fiber wt.%	date palm leaflet fiber wt.%
1	WFRP	*30*	-
2	DFRP	-	*30*
3	HFRP	*15*	*15*

The fabrication process of biocomposite was done by hand lay-up technique, the unsaturated polyester resin with a short cure time was mixed with a catalyst at a weight percentage of *100:2* and homogeneously mixed for a few minutes to ensure the distribution of the catalyst in all resin regions. Then the biofibers were added to the resin and continued mix all the ingredients, subsequently poured into the glass mold; using a vacuum bagging to get rid of the air bubbles that may occur while mixing the ingredients; and let the biocomposite materials solidified at room temperature at *23°C*, after *24* hours the biocomposites sheet were shrunk in the glass mold, this occurs due to the property of unsaturated polyester shrinkage cure around (*4-8%*) according to [28] the mold was opened and the biocomposite material sheets were taken out with a measured thickness of *5±0.2* as illustrated in Fig.5a.

2.3. Cutting process

The motivation to use a CNC milling machine for the biocomposite cutting process is to ensure a good surface finishing for mechanical test specimens rather than using traditional cutting methods. The cutting process was done on a 3-axis CNC milling machine with a spindle speed of *8000 rev/min* using a HSS end mill tool to perform the cutting processes of mechanical tests specimens according to ASTM standards; D3039 [29], D695 [30], and D792 [31], which was done without using coolant as shown in Fig.5. It was conducted CNC milling machine while cutting the specimen.

a) b)

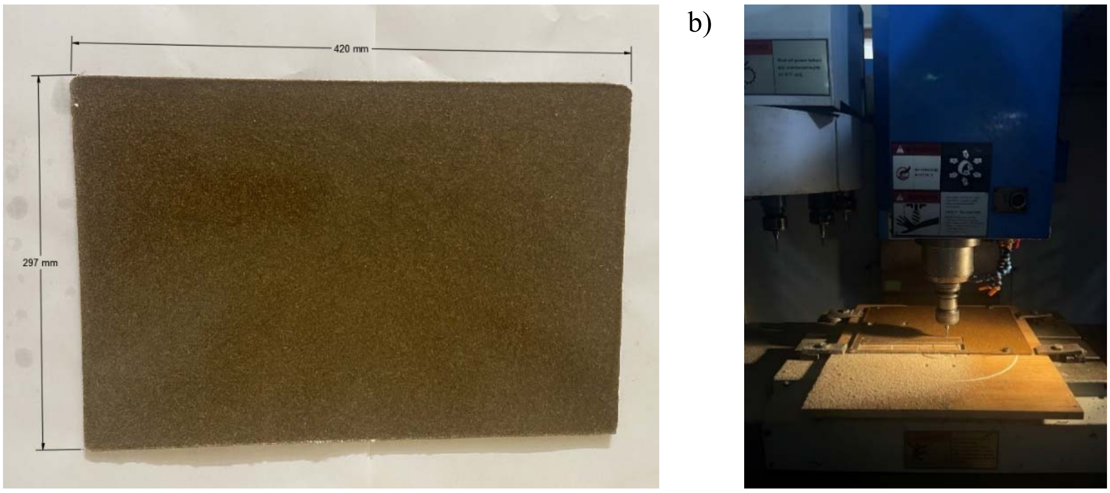


Fig.5. Experimental set up (a) manufactured biocomposite material (b) cutting process by CNC milling machine.

2.4. Characterizations

This paper investigated the mechanical properties of biocomposite types that were prepared followed by the cutting process mentioned in previous sections. All tests were conducted at an ambient temperature of approximately *22°C*, and humidity around *42%*.

2.4.1. Tensile test

The tensile test was used to indicate the material behavior during tension which involved subjecting a standard dimension's specimen to controlling load and measuring the applied load and the extended length of the sample through the distance between machine gauges. Many properties can be achieved through the tensile test such as young modulus, yield strength, ultimate strength, elongation, and elastic limit. To evaluate the tensile properties of the biocomposite; ASTM D3039 standard [29] was used in this paper.

Tensile tests are used for measuring the stress required to break composite material specimens and the extent to which the specimen elongates to that breaking point. In this study, the LARYEE tensile testing machine with a capacity of *100 kN* and strain rate of *2 mm/min* according to ASTM D3039 with sample dimensions *250 mm* (length)× *25* (width) *mm*× *5* (thickness) *mm*. This procedure was done for four specimens of each type of bio composite as shown in Fig.6, and then the average was taken.

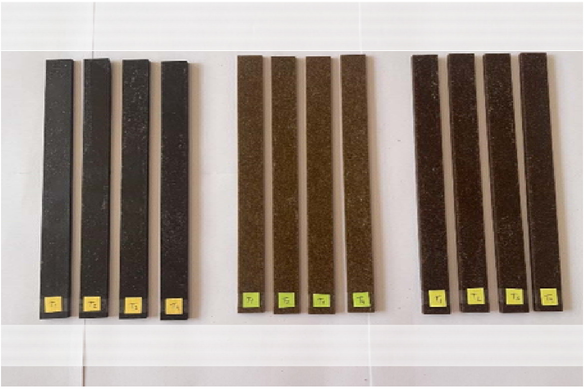
a)

b)


Fig.6. Tensile test (a) biocomposites tensile test specimens (b) tensile specimen during the test.

2.4.2. Compression test

Compression tests are used to determine specimen material's behavior under applied crushing loads, compressive strength quantifies the material's capacity to withstand size reduction when subjected to load without fracture, test specimen usually either cuboid or cylindrical geometry. A computer-controlled electronic universal testing (TIME GROUP INC., CHINA) with a capacity of *50 kN* was used for the compression test according to ASTM D695 [30] with specimen dimensions *10 mm* (length)× *5* (width) *mm*× *5* (thickness) *mm* at a cross-head speed of *1 mm/min*. Figure 7 shows the specimen during the compression test. Four replicates were tested for each type of biocomposite material.

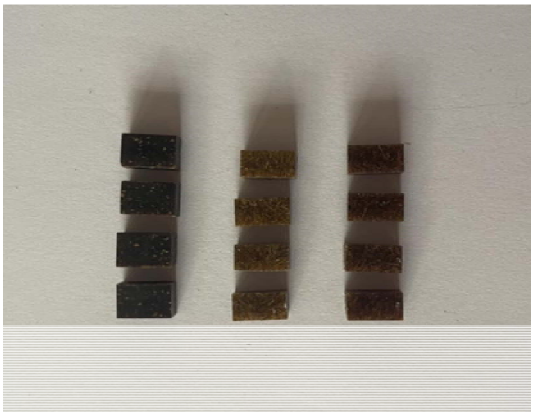
a)

b)


Fig. 7: Compression test (a) compression test specimens (b) compression specimen during the test.

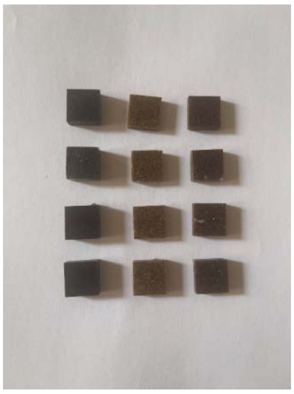
2.4.3. Density test

In order to find the density of the three biocomposite materials, this test was conducted at room temperature, the dimensions of density test samples were done according to ASTM D792 [31] of *10 mm* (length) × *10* (width) *mm* × *5* (thickness) *mm*. The test was conducted for four specimens for each type of biocomposite sheet then the average of the results was taken.

The density of biocomposite samples was determined by calculating their weight in the air, hanging in the distilled water as shown in Fig.8 and then calculating the density according to Eq.(2.2) for each type of biocomposite sheet.

$$\rho = \frac{Wa}{Wa + Ww + Wb}\rho_w \tag{2.2}$$

where ρ is the density of biocomposite g/cm^3, ρ_w is the density of distilled water $0.997045\ g/cm^3$. *Wa* is the weight of the biocomposite in the air *g*, *Ww* is the weight of the biocomposite hanging when immersed in distilled water *g* and *Wb* is the weight of the biocomposite when immersed in distilled water for 24 hours.

a) 

b) 

Fig.8. Density test (a) density test specimens, (b) specimen during hanging when immersed in distilled water.

3. Results and discussions

In this study, three types of biocomposites were investigated which are from walnut shell fiber and date palm fronds as reinforcement, and unsaturated polyester as matrix. The mechanical properties were carried out to find the tensile strength, compression strength, and density of the biocomposites. The DFRP composite showed the best tensile strength, on the other hand WFRP composite revealed the best compression strength and density as clear in Tab.3.

The tensile strength of the DFRP composite increased by 28.*6%* and *12.5%* comparable with WFRP and HFRP composites, and the compression strength of the WFRP composite increased by *21.5%* and *10.3%* comparable with DFRP and HFRP composites, the good bonding interface of walnut shell and date palm leaflets fibers with the matrix could explain why the mechanical property results were better than WFRP and HFRP composites. Additionally, the Density of the WFRP composite decreased by *18%* and *10.9%* compared with DFRP and HFRP composites respectively. The mechanical properties tests were done for four specimens each then the average of it was taken that cleared in Tab.3, the tensile and compression curves of the biocomposite materials illustrated in Fig.9 and Fig.10 respectively.

Table 3. Mechanical properties of biocomposites.

property	WFRP	DFRP	HFRP
tensile strength *MPa*	*35*	*45*	*40*
compression strength *MPa*	*103.67*	*85.33*	*94*
density g/cm^3	*4.60*	*5.61*	*5.16*

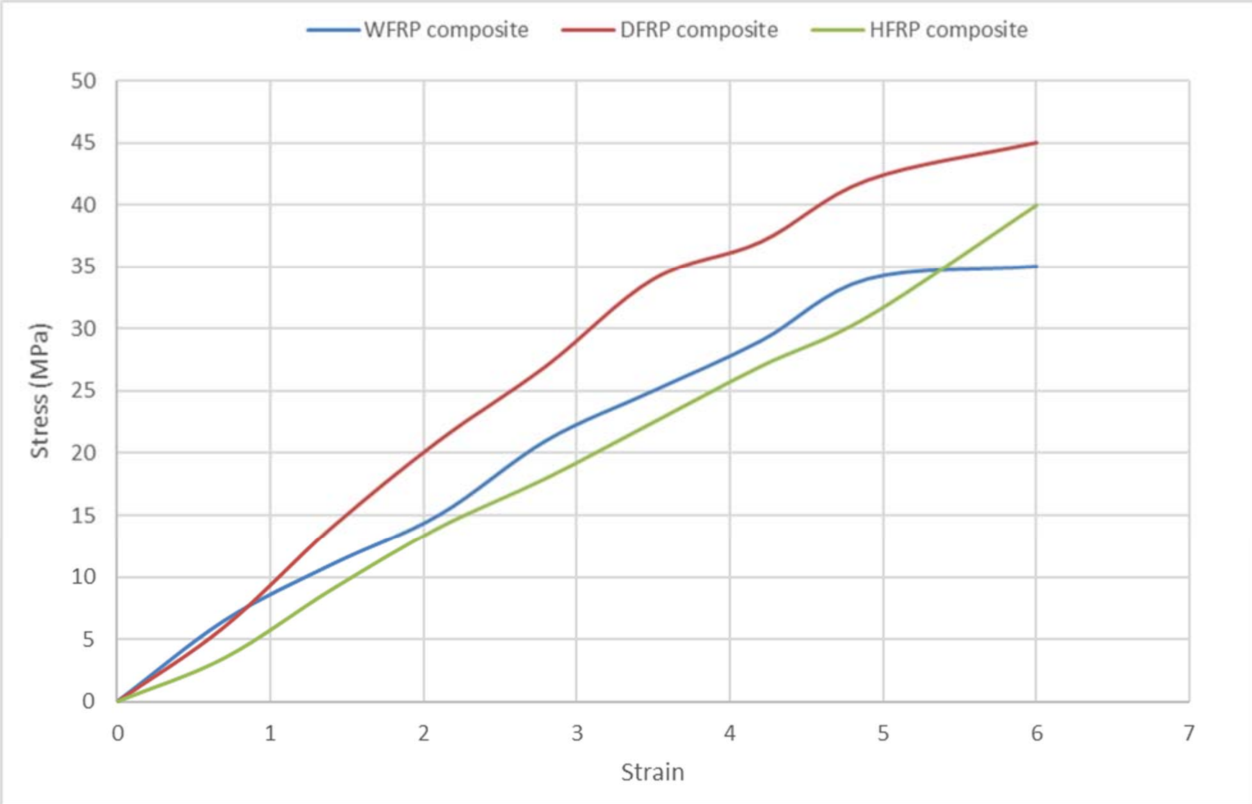


Fig.9. Biocomposites tensile test curves.

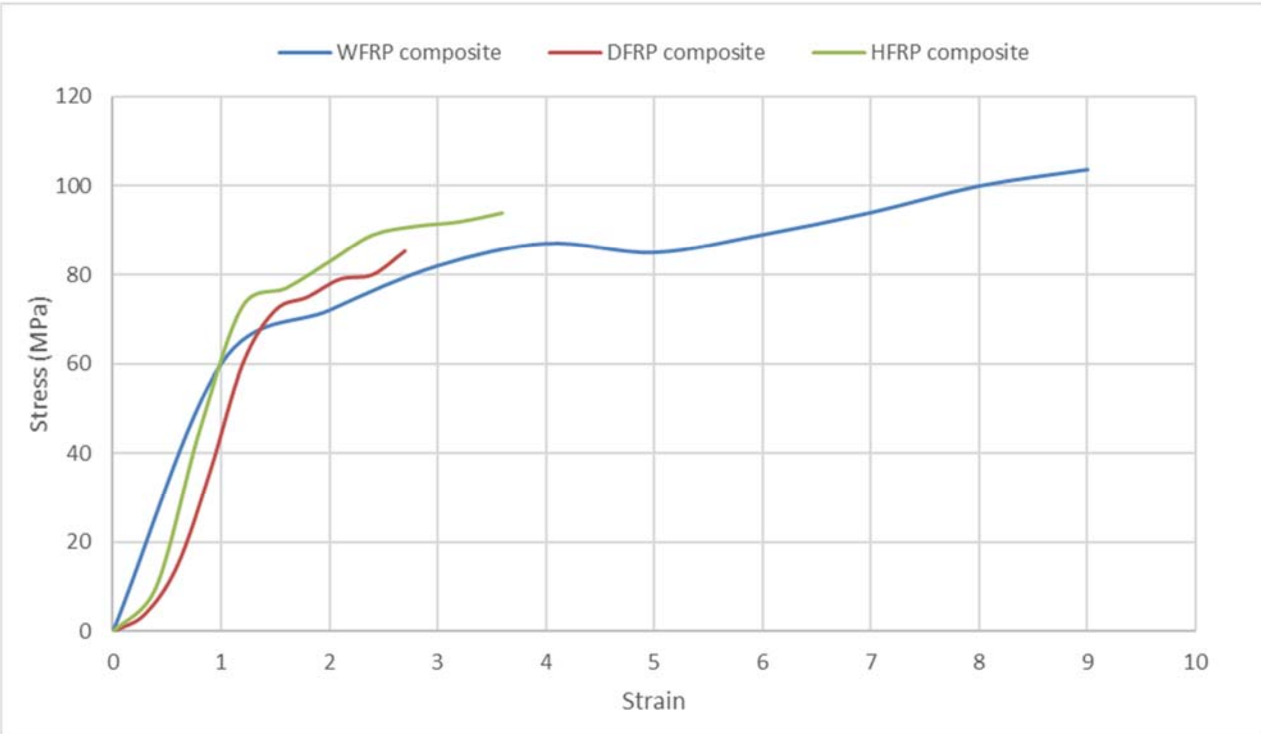


Fig.10. Biocomposites compression test curves.

4. Comparison with previous studies

Depending on previous studies [16], [18] and [24] used both heated and chemical treatments for biofibers to remove the moisture of biofibers and also improve the adhesion bond between fiber and matrix. The range of heat treatment was different in the previous studies due to the various types of bio fibers used, so in this research, many heat treatment experiments were done for walnut shell and date palm frond samples before fabricating the biocomposite, to find the proper heat treatment temperature for the nature of biofibers used in this paper.

Figure 11 shows the tensile strength values of biocomposite materials that used different types of biofibers as reinforcements compared with the DFRP composite used in this study, it was concluded that the current research has the maximum tensile strength of *45 MPa*.

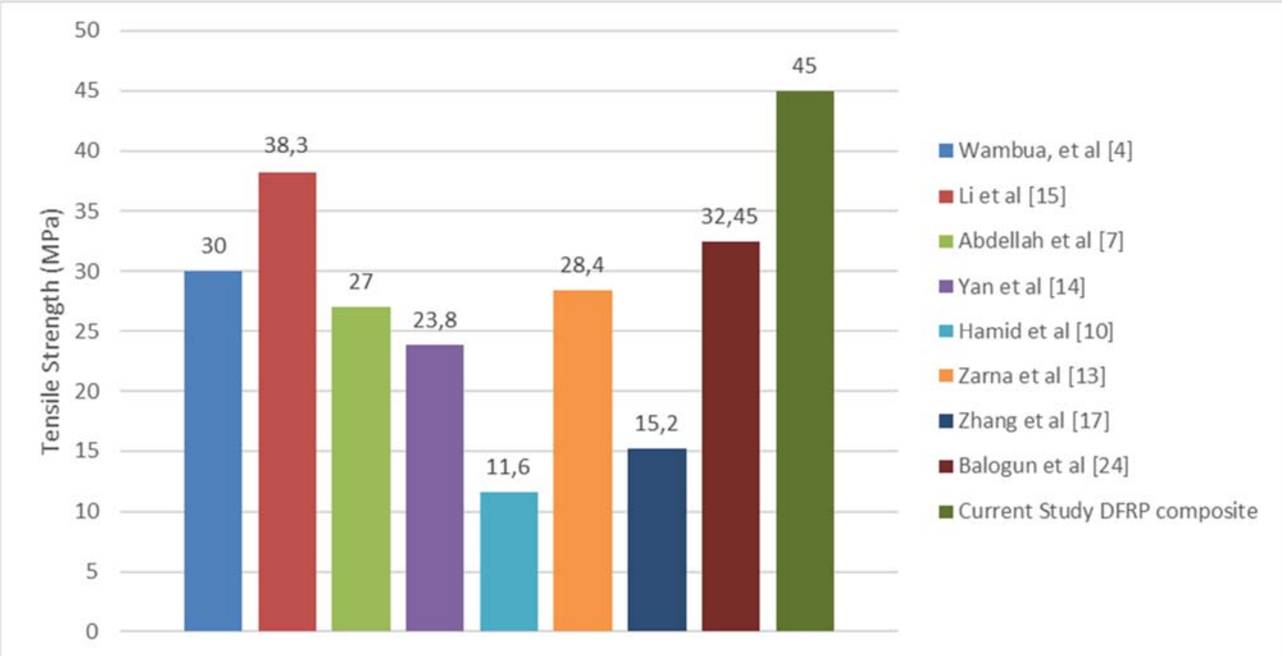


Fig.11. Comparison chart of DFRP composite tensile strength with previous studies.

5. Conclusions

In this paper, three types of biocomposites were conducted from walnut shell and date palm fronds reinforced unsaturated polyester composite. The excellent cost/performance ratio, as the low weight, environmentally friendly, biodegradable, and nonabrasive biofibers are suitable for various applications such as automotive, aerospace, transportation,

Furthermore, mechanical properties of biocomposite materials were found; tensile strength, compression strength, and density of biocomposites valued according to ASTM standards. Based on the results of this study, the following conclusions were investigated:

1. Chemical treatments by NaOH were done for walnut shells and date palm fronds to remove moisture to increase the strength of the fibers then dried in an oven for *1* hour and *30* minutes at *50°C* for the walnut shells and date palm fronds respectively.
2. The DFRP composite showed the best results of the ultimate tensile strength of *45 MPa* comparable with WFRP and HFRP composites.
3. The WFRP composite has a higher compression strength *103.67 MPa* and a lower weight of $4.60\ g/cm^3$ than DFRP and HFRP composites.

6. Recommendations for future works

1. Study the effect of the biocomposite materials in this paper on the end mill cutting tool wear at different cutting conditions.
2. Study the surface roughness of biocomposites after machining by different cutting conditions.

Declaration of competing interest

The authors declare no conflict of interest

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Nomenclature

Wa – the weight of the material in the air
Wb – the weight of the material when immersed in distilled water for 24 hours
Ww – the weight of the material hanging when immersed in distilled water
ρ – density of the material
ρ_w – the density of distilled water

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