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Utilization of Palm Seeds Nano powder Reinforced Polyester as a Green Composite

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ABSTRACT: Global warming and resource depletion are the main two issues that whole world try to reduce to keep our planet. So the production of synthetic fillers consumes energy, substances beside the pollution in air, soil and water. This work dealt with the preparation of green composite. The adding effect of palm seeds powder as natural additives on the mechanical properties of polyester resin were determined. Three different weight fractions of powder was added (3 %, 6 %, and 9 %). The composite were prepared using (Hand Lay-Up) approach. The utilized palm seed powder was pre-prepared using shear milling of well-dried palm seeds and then characterized using AFM analyzer. Granularity cumulating distribution report showed that the average diameter for 123 grains was 92.87 nm. 2D and 3D topography was also investigated. Tensile test, flexural test, impact test and hardness test were performed to determine the mechanical characteristics of composites. Obtained results revealed that the flexural modulus, impact strength and hardness properties enhanced with the increment of powder weight fraction incorporated in polyester matrix. While the values of tensile strength, tensile modulus, elongation percentage and flexural strength properties deteriorated with the increasing of weight fraction of powder.

KEYWORDS: Green composite, nanopowder, palm seeds, polymer composites, natural fillers.

INTRODUCTION

In the last decade, scientists and researchers paid rising attention towards environmental issues like global warming, climate change, greenhouses gases, water crisis and resource depletion. Also, due to the risk of hazardous substance on the human health, increasing interest for create more green materials. Green composites are one of these materials which filled with natural-organic fillers from biodegradable and renewable sources which can find versatile industrial applications [1-3]. Biomaterials are defined as materials natural or synthetic origin that are used as treatment, supplement, or replacing any part in human body lost its function as a healing, or to improve function through living tissue. In order to be considered a biomaterial it should be non-inflammable, non-toxic and not introduce allergenic symptoms in the body. Also it has to be biocompatible, sterilizable, bioactive, bio functional and bio inert. The common classes of materials used as biomedical materials are polymers, metals, ceramics, and composite materials that have been widely utilized in dentistry and medical applications [4].

The most important type of biocomposite material is biopolymer composite materials that have been widely used in medical disposable supply, prosthetic materials [5]. Due to the main characteristics of the biopolymer composite materials as compared with biometal or bioceramic composite materials are easy to manufacture to different design, easy to processing, low cost, and available with various mechanical and physical properties to be fit to the desired applications. The polymers which act as biomaterials are (poly methyl methacrylate, polyvinyl chloride and polyester) have many advantages such as easy to make complicated items, good physical and mechanical properties, also have some disadvantages like absorb water and proteins and difficult to sterilize [6]. The prosthetic socket is the interface between an amputee and his artificial limb. The ideal socket must be lightweight, easily worn and removed, biocompatible, resist impact and stress in all directions, relatively cheap and easily available, so the socket is typically made from polymer composite materials. Thermosets such as epoxy and polyester are cheap, offer high impact strength, rigidity, and modulus of elasticity, and thus are extensively favored in orthotic and prosthetic industries [7].

Numerous studies executed all over the world to enhance the low cost, eco-friendly prostheses products. Andrew Campbell *et al.* prepared plant-based composite materials as green composite for prosthetic limb sockets. They utilized polycarbonate-polyurethane copolymer resin reinforced with (10.0% by volume) plant fiber (bamboo, banana, corn, cotton, flax, ramie, Seacell and soya) instead of conventional synthetic fiber to enhance safety, performance and accessibility of product [8]. Rungsima Chollakup *et al.* reinforced Polyethylene green composites with cellulose fibers (coir and palm fibers). Surface modification (alkaline and bleaching treatment) and fiber content was investigated to show these effects on fiber-matrix interaction [9]. Also kenaf fiber can be incorporated into composites for high impact application instead of fiber-glass as one ply in prosthetic socket composite [10]. Other researchers used ramie biomaterial instead of fiberglass in polyester matrix. It can be considered as an alternative substance to substitute a prosthetic socket material due to its toughness (high strength /weight ratio), natural availability, comfortability, and acceptable appearance [11]. Cotton and nylon composite can be substitute by Bamboo fiber reinforced composite utilized in prosthetic and orthopedic due to their ductility and potential strength [12]. Others utilized rattan fiber to reinforced epoxy for lower limb prosthesis socket [13].

The previous findings of green biocomposite on prosthetic field could have potential to serve as a cost effective, applicable practically, and eco-friendly and it could be considered sustainable choice as green materials in both prosthetic and orthopedic applications. This work targets to enhance the polyester resin to have desirable properties for medical applications such as prosthetic socket. Also, due to rising concern towards global warming, climate change, resources depletion, pollution and energy and other environmental issues. Increasing interest was paid about green composite, which is filled with natural fillers. Polyester nanocomposites were prepared in current work for this reason, which is reinforced with natural nanopowder of palm seeds. Some mechanical properties were investigated including (tensile, flexural, impact and hardness) to recognize the enhancement extent.

MATERIALS AND METHODS

The utilized matrix is unsaturated polyester resins (UPR) is supported from Saudi Industrial Resins Limited (SIR) Company. This resin is in the form of transparent pink viscous liquid at room temperature and one of the types of thermosetting polymers. It is transformed from liquid to solid state by the adding liquid hardener, which is methyl ethyl keton peroxide (MEKP) by adding 2 g to 100 g of resin at room temperature. In order to increase the speed of solidification, cobalt naphthalite was added by 0.5 g per 100 g of resin as accelerator. This accelerator gives a violet color to polyester resins when absorb ultraviolet ray (UV) radiation from the atmosphere, which is a major factor in polyester solidification. The specification of used polyester is illustrated in the Table (1) as received from the providing company.

Table 1. Utilized unsaturated polyester specifications.

Density g/cm^3	Specific Heat J/ kg. k	Fracture Toughness $MPa.m^{1/2}$	Tensile Strength MPa	Percent Elongation (El%)	Modulus of Elasticity GPa.
1.2	710-920	0.6	41.4-89.7	< 2.6	2.06-4.41

The reinforcing filler was powder of date palm seeds. Palm tree are commonly found in Asia. Palm seed is a hard coated seed, which has weight (0.5 - 4 g) representing (6 to 20) % of the total weight of fruit. The date seeds are considered as a source of natural polyphenolic materials. These seeds can be incorporated in food products as natural antioxidants agents due to valuable containing polyphenolic fatty acids which react actively with free radicals. But higher amounts in human body can be harmful because are associated with carcinogenesis diseases and others [14]. Locally harvested date palm, were removed the fruit cover. The seeds were washed by potable water; air dried then put in oven at 90 C for 5 hours. Well dried seeds milled in shear miller for 3 hours. The palm seed and palm seed powder are shown in Figure 1.

Figure 1. Palm seeds before and after milling.

Pure polyester and polyester composite materials reinforced with 3 %, 6 % and 9 % Wt% of palm seeds were prepared in this work for prosthetic socket. Figure 2 shows the schematic diagram of technology path which had been followed in this work.

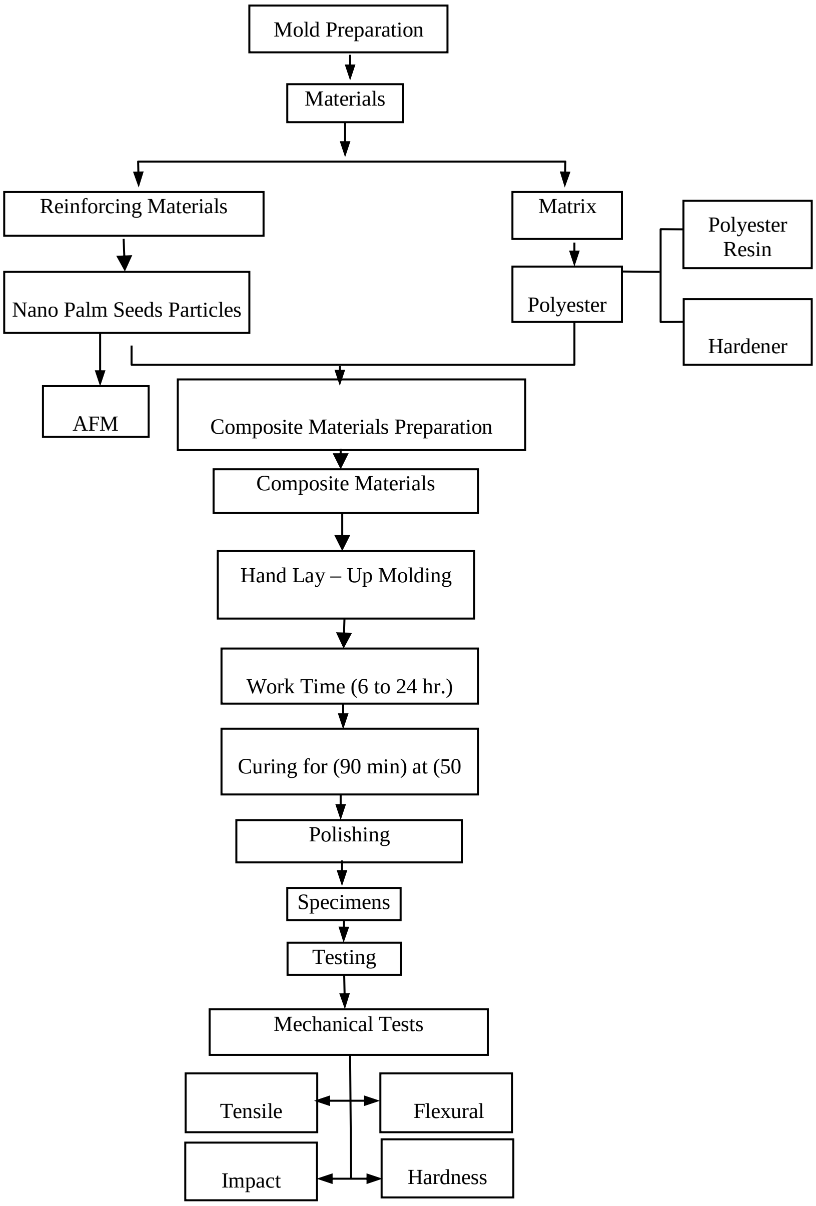

Figure 2. Schematic Diagram of Technology Path

Characterization and tests

AFM Analysis

The particle size and distribution of palm seeds powder were carried out using Atomic Force microscopy by Scanning Probe Microscopy (SPM). 2D and 3D topography were also investigated.

Tensile Test

Tensile test is performed according to (ASTM D638) utilizing tensile machine (Universal Testing Machine, Instron model) at a cross head speed of (5 mm/min) and (50 KN) as applied load until breakage of the sample occur. As recommended by (ADA Specification No.12, 1999), after complete finishing and polishing processes of test specimens, all samples must be stored in distilled water at temperature (37±1) °C for a period (48 hr). Each test was done in air at room temperature (23±2) °C. Average value for five specimens was taken as a final result [15].

Bending Test

The bending test is simple supports test method involves three-point bending test, which is loading pin is lowered from above at a constant rate. The specimen is placed on two supporting pins a set distance a part specimen, that fixture on a universal testing machine at across head peed (5 mm/min) and applied load (5 kN) which was gradually loaded until the breakage of specimen occur. This test was executed according to (ISO 178, 2003) at temperature (23±2) °C. Details of the test preparation, conditioning and load rate affect the test results. Averaged of five specimens was taken. The bending modulus can be calculated by the equation (1) [16, 17].

$$E_{bending} = \frac{F.\ L^3}{48\ I.\delta} \quad (1)$$

Where:

E_{bend}: bending modulus.

F: force applied on the specimen (N).

L: length of the specimen between (mm).

I: cross-section moment of inertia (mm^4).

δ: Deflection in the specimen due to applied loading (mm^2) [18].

The moment of inertia in the sample cross-section can be calculated by the equation (2):-

$$I = \frac{b\ .\ d^3}{12} \quad (2)$$

Where:

b: width of the specimen.

d: thickness of the specimen.

The flexural strength can be obtained by the equation (3):-

$$F.S = \frac{3\ F.\ L}{2\ b.\ d^2} \quad (3)$$

Impact Test

The impact test is performed to know the material behavior when exposes to shock forces that lead to deformation, initiate fracture and continue the fracture until the specimen is broken. Also to determine the absorb energy during a collision. Toughness, impact strength, fracture resistance, impact fracture resistance of the material can be determine by this energy. According to (ISO-180), the impact specimen is placed in the device with using Izod Impact approach by (XJU pendulum Izod/Charpy impact testing machine), then release the pendulum from a known height so that it collides with the specimen as sudden force and record the amount of energy that record to fracture of specimen [19]. Impact test specimens might be with or without notch. In this type of impact test, one end of specimen is fixed vertically as cantilevered beam; applied energy was (5.5J) and pendulum velocity (3.5 m/s). Average value for five samples was taken into account [20].

Hardness Test

Hardness is defining the surface resistance to scratching, cutting, wear, indentation and penetration from an applied force of sharp point and as indication of surface durability. The durometer is one of the several international standard methods that using for measuring the hardness of rubber, sponge, plastic, and other nonmetallic materials, there are several scales of durometer are used for materials with different properties. The specimen surface must be smooth and clear from any forge in matter in zone test. The hardness property is affected significantly by sample thickness, diameter and distance from the edge. This test is performed by use the hardness device type (shore-D) according to ASTM D2240 at applied load 50 N and pressing time 15sec to measure [21].

RESULTS AND DISCUSSION

The AFM analysis result is illustrated as granularity accumulation distribution, and surface topography (2D and 3D) which represented in Table 1, Fig. 3, Fig. 4 and Fig.5 respectively. The results showed that the average value of diameter was 92.87 nm. Also the well distributed granular size can be observed significantly between 82-145 nm. 2D and 3D topography revealed the well distrusted and non-agglomerated nanopowder.

Table 2. Granularity Cumulating Distribution, Avg. Dia. :92.87 nanometers

Dia. (nm)	Vo. l%	Cum. (%)	Dia. (nm)	Vol.%	Cum (%)	Dia. (nm)	Vol.%	Cum. (%)
80	17.89	17.89	100	9.76	72.36	120	4.07	97.56
85	21.14	39.02	105	8.94	81.30	130	0.81	98.37
90	13.82	52.85	110	4.88	86.18	140	0.81	99.19
95	9.76	62.60	115	7.32	93.50	145	0.81	100.00

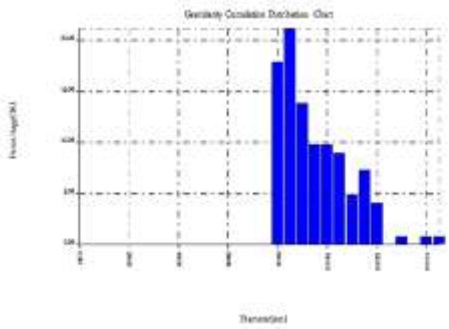

Figure 3. Granularity Cumulating Distribution Chart of palm seeds powder.

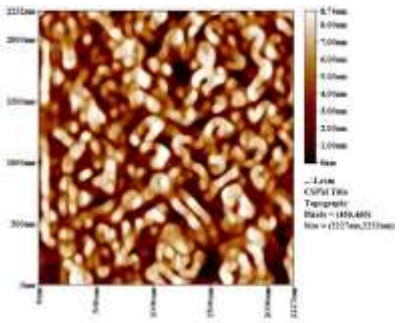

Figure 4. 2D surface topography by SPM.

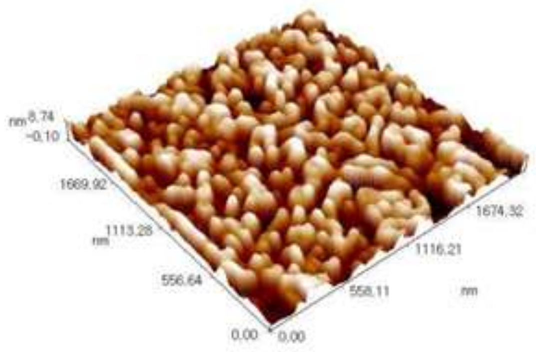

Figure 5. 3D surface topography by SPM

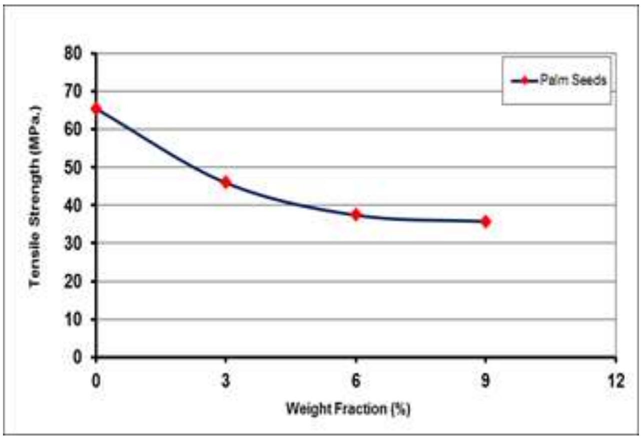

Figure 6. Tensile Strength of Polyester Composite vs. Palm Seeds Powder Wt. %

Table 3. Tensile strength of polyester reinforced with different Wt% of palm seeds powder

Specimen	Tensile Strength (MPa)
Polyester	65.5
Polyester + 3% Palm Seeds	46
Polyester + 6% Palm Seeds	37.5
Polyester + 9% Palm Seeds	35.8

Table 3 and Fig. 6 show the effect of adding different Wt. % of palm seeds powder on the tensile strength of polyester. From this figure can be notice that tensile strength decrease with the increasing of weight fractions of filler in polyester composite. This decreasing in the tensile strength of the polyester composite materials is due to the low tensile strength of palm seeds particles as compared to polyester resin and may be also due to the low the strengthening mechanism, low the bonding strength and created poor interface between these particles and polyester matrix. Table 4 and Fig. 7 revealed the modulus of elasticity of prepare composites at different Wt. % of filler. It can be observed significantly the decrementing of modulus values respect to the increasing the filler content. This result reflects the martial discontinuity when adding the filler that may due to the weak bonding between the filler and matrix which responsible of force transfer [22].

Table 4. Tensile modulus of polyester reinforced with different Wt% of palm seeds powder.

Specimen	Tensile Modulus (GPa)
Polyester	1.132
Polyester + 3% Palm Seeds	1.042
Polyester + 6% Palm Seeds	1.003
Polyester + 9% Palm Seeds	0.853

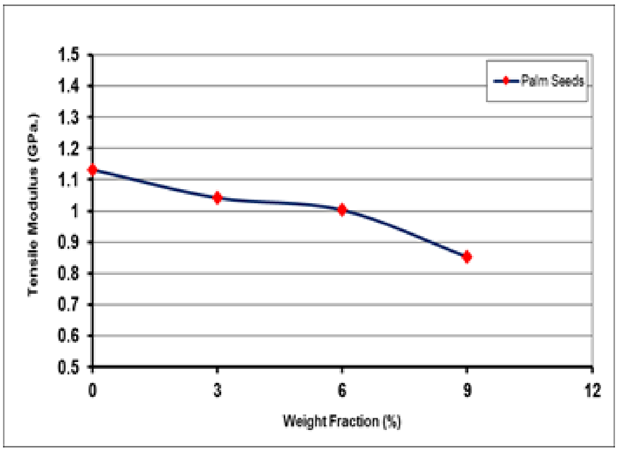

Figure 7. Tensile Modulus of Polyester Composite vs. Palm Seeds Powder Wt. %

Table 5 and Fig. 8 illustrate the elongation percentage of polyester composite against weight fraction of palm seeds powder. It can be observe that elongation percentage decreases with increasing the Wt. % of palm seeds powder in polyester composite. Based on previous tensile test values, the elongation was also estimated to be dimensioned because when adding more amount of powder, nanoparticles will number act as localized stress concentration regions because of bad bonding between the filler and matrix [23]. Table 6 and Fig. 9 show the relationship between the flexural modulus of polyester matrix composite versus palm seeds powder content. The flexural modulus values increase with increasing the weight fractions of palm seeds powder in polyester composite. The increasing in the flexural modulus of the polyester composite materials is due to nanopowder which imped the slippage of the polyester chains one of each other [24, 25]. Table 7 and Fig. 10 show the effect of adding of palm seeds powder with different Wt. % on flexural strength of polyester composites. It can be seen significantly from this figure the reduction in flexural strength values with increasing the weight fractions of powder. This is due to the interfaces bonding between the polyester matrix and powder [26].

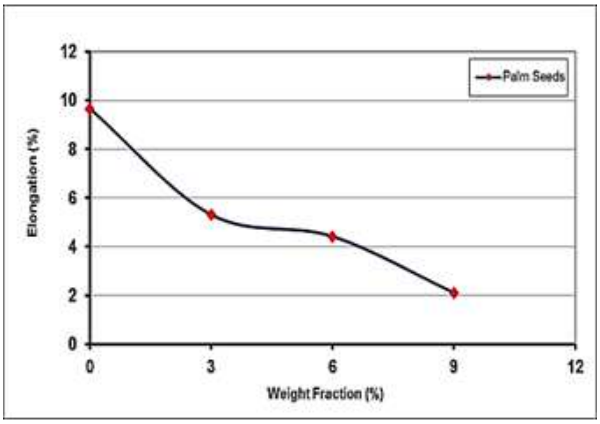

Figure 8. Elongation Percentage of Polyester Composite vs. Palm Seeds Powder Wt%

Table 5. Elongation percentage of polyester reinforced with different Wt. % of palm seeds

Specimen	Elongation Percentage (%)
Polyester	9.65
Polyester + 3% Palm Seeds	5.3
Polyester + 6% Palm Seeds	4.41
Polyester + 9% Palm Seeds	2.1

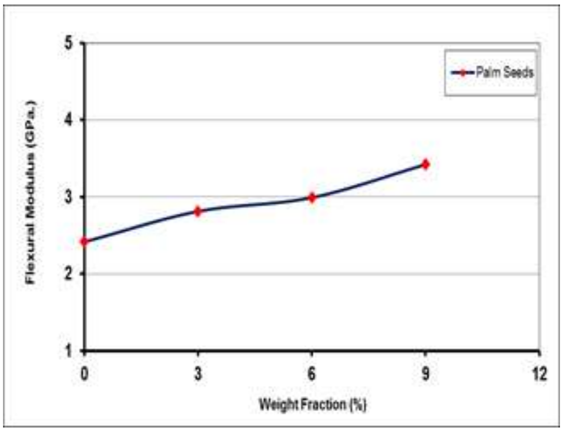

Figure 9. Flexural Modulus of Polyester Composite vs. Palm Seeds Powder Wt%

Table 6. Flexural modulus of polyester reinforced with different Wt. % of palm seeds

Specimen	Flexural Modulus (GPa)
Polyester	2.415
Polyester + 3% Palm Seeds	2.814
Polyester + 6% Palm Seeds	2.992
Polyester + 9% Palm Seeds	3.422

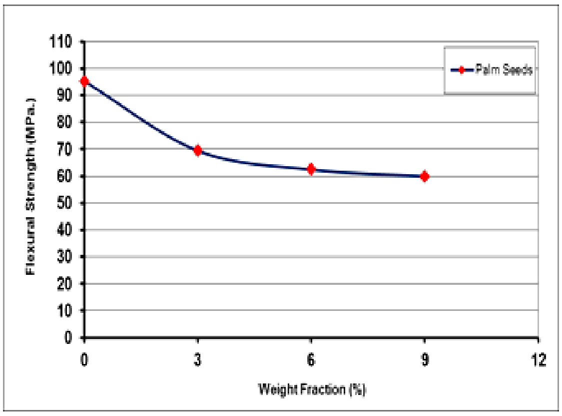

Figure 10. Flexural Strength of Polyester Composite vs. Palm Seeds Powder Wt. %

Table 7. Flexural Strength of polyester reinforced with different Wt. % of palm seeds

Specimen	Flexural Strength (MPa)
Polyester	95.24
Polyester + 3% Palm Seeds	69.5
Polyester + 6% Palm Seeds	62.5
Polyester + 9% Palm Seeds	60

Table 8 and Fig. 11 show the adding effect -palm seeds powder with different Wt. % on the impact strength of polyester composite. It can be observed clearly that the impact strength values incremented with the adding more Wt% of powder due to the distribution of impact load among particles and matrix to resist damage the as compared with polyester matrix [27-34]. Table 9 and Fig. 12 show the adding effect of palm seeds on the hardness (Shore-D) of polyester composite. hardness (Shore-D) values increase with increasing the Wt% of powder in polyester composite because of these particles make the surface harder by restricting the movement of the polyester chains towards the stress direction [35, 36].

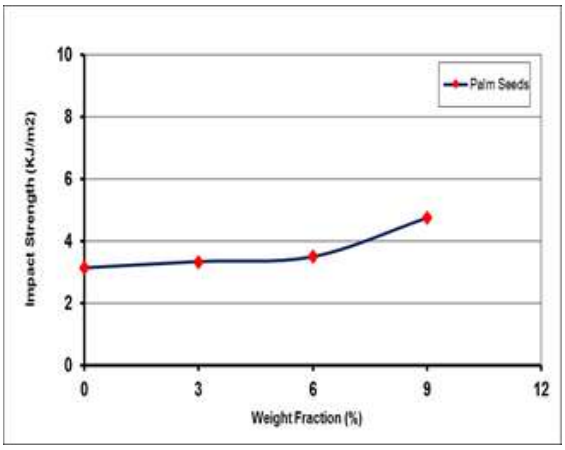

Figure 11. Impact Strength of Polyester Composite vs. Palm Seeds Powder Wt. %

Table 8. Impact strength of polyester reinforced with different Wt. % of palm seeds powder.

Specimen Names	Impact Strength (KJ/m^2)
Polyester	3.125
Polyester + 3% Palm Seeds	3.33
Polyester + 6% Palm Seeds	3.49
Polyester + 9% Palm Seeds	4.76

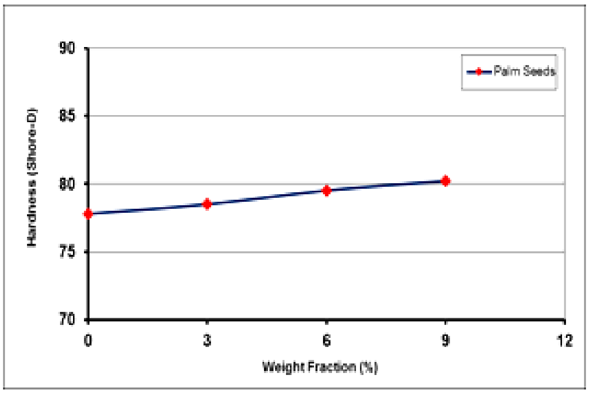

Figure 12. Hardness of Polyester Composite vs. Palm Seeds Powder Wt. %

Table 9. Hardness (Shore-D) Of Polyester Reinforced with Different Wt. % Of Palm Seeds Powder.

Specimen Names	Hardness (Shore-D)
Polyester	77.8
Polyester + 3% Palm Seeds	78.5
Polyester + 6% Palm Seeds	79.5
Polyester + 9% Palm Seeds	80.2

CONCLUSIONS

All findings below could lead the current work to produce eco-friend and low cost prosthetic socket made from natural nano powder based composites:

1- The tensile strength of polyester matrix composite prosthetic socket specimens decreased with adding more palm seeds powder. Thus, the tensile strength decreased from (65.5MPa) for polyester to (35.8 MPa) for (polyester /9Wt % palm seeds powder).

2- The tensile modulus of polyester matrix composite prosthetic socket specimens decreased with adding more palm seeds powder. Thus, the tensile modulus decreased from (1.132GPa) for polyester to (0. 853 GPa) for (polyester /9Wt % palm seeds powder).

3- The elongation percentage of polyester matrix composite prosthetic socket specimens decreased with adding more palm seeds powder. Thus, the elongation percentage decreased from (9.65 %) for polyester to (2.1) for (polyester /9Wt % palm seeds powder).

4- The flexural strength of polyester matrix composite prosthetic socket specimens decreased with adding more palm seeds powder. Therefore, the flexural strength deteriorated from (95.24 MPa) for polyester to (60 MPa) for (polyester /9Wt % palm seeds powder).

5- The flexural modulus of polyester matrix composite prosthetic socket specimens increased with adding more palm seeds powder. Thus, the flexural modulus increased from (2.415 GPa) for polyester to (3.422 GPa) for (polyester /9Wt % palm seeds powder).

6- The impact strength of polyester matrix composite prosthetic socket specimens increased with the increasing of the weight fraction of palm seeds particles. Thus, the impact strength increased from (3.125 KJ/m^2) for polyester to (4.76 KJ/m^2) for (polyester /9Wt % palm seeds powder).

7- The hardness of polyester matrix composite prosthetic socket specimens increased with adding more palm seeds powder. Thus, the hardness increased from (77.8) for polyester to (80.2) for (polyester /9Wt % palm seeds powder).

It is recommended to use chemical treatment of natural powder before to use and then characterize the functionality by FTIR, to increase bonding with many kinds of polymers. Also it highly recommended to use different parts of plants as eco-friendly filler for green composite not only for medical application but also for construction and building like 3D printing as well.

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