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Physico - Mechanical Properties of Dates Palm *(Phoenix dactylifera)* Pits Reinforced Unsaturated Polyester Composites

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Abstract: ***This paper investigates the potential of using waste from date fruits as filler for the production of thermosets composites. Unsaturated polyester composites reinforced with filler from date pits were prepared using hand lay up technique at filler contents 10, 20, 30, 40 & 50 %wt, the resulting composites were moulded in sheet and thereafter machined into sizes for analysis. Physico-mechanical test such as density, hardness, tensile and flexural tests were carried out on the composites. Impact properties were also determined. The composite gives maximum tensile strength at 10% filler ratio while the maximum tensile modulus was observed at 50wt% filler loading with minimum value at 20% filler content. Maximum values were observed for flexural modulus, flexural and impact strength at 10 & 20wt% filler content respectively, while the minimum value was observed at 50% filler content. The shore D hardness test gives the highest value at 50% filler loading. The density was equally observed to be highest at maximum filler ratio of 50. Result obtained from the mechanical tests shows a promising low cost composites material that can be used in non- structural applications.***

Keywords: Thermosets, Hardness, Flexural Strength, Tensile, Impact , Modulus.

1. Introduction

The quest for an economically feasible light weight composite that will compete favourably well with the conventional materials such as metals in terms of physico-mechanical properties for structural engineering and in building application led to the use of lingo-cellulosic materials as reinforcing fillers in the production of composites. The filling of polyesters with cellulosic fibres has been widely reported, polyester-jute, Polyester-sisal, polyester-coir, polyester-banana-cotton, polyester-straw, polyester-pineapple leaf, and polyester- cotton-kapok, have been widely reported [1]. Several other researches have been conducted in the utilisation of agro-waste filler in the production of ecofriendly composites.

Date Palm *(Phoenix dactylifera*) is a monocot plant in the Arecaceae family, cultivated in dry tropical regions worldwide for its edible sweet fruit. Its native range is difficult to ascertain, because it has been spread through cultivation for thousands of years, and no longer occurs in the wild, but it is generally considered to have originated in region around the Persian gulf-northern Africa, the Arabian peninsula, and northwest India. It is widely planted in hot and dry climate regions of Africa, the Middle East and Asia. Date palm fruit is an important food resource in these regions. Annual production of dates was about seven million tons in 2004. Besides food-grade date production, large amounts of dates end up as waste. The global waste palm date production is approximately two million tons per year [2].

Dates were important as a food (easily stored and transported), a source of building material the fronds provided fibre for thatching roofs and making baskets, trunks are used for construction. The date has been traditionally a staple food in Algeria, Morocco, Tunisia, Egypt, the Sudan, Arabia and Iran and in Nigeria.

Palm date fruits consist of three main parts: date flesh, date pit, and skin. That is, it is a drupe, an indehiscent fruit in which an outer fleshy part (exocarp, or skin; and mesocarp, or flesh) surrounds a shell (the pit, stone, or pyrene) of hardened endocarp. Date pit is mainly used as animal feed [3]. It contain a single seed (kernel) about 2–2.5cm long and 6–8mm thick. The kernel is a major by-product of the date palm-processing industry. They contained 7.1–10.3% moisture, 5.0–6.3% protein; 9.9–13.5% fat; 46–51% acid detergent fibre; 65–69% neutral detergent fibre; and 1.0–1.8% ash. Date pit is mainly used as animal feed [4].

Thus, the study intend to take the advantage of the unutilised pits that always ends up as waste as filler in the preparation composite.

Figure 1: Showing fruits and dried pits(seed) of date palm

Unsaturated polyesters are extremely versatile in properties and applications, fairly inexpensive, and have been a popular thermoset used as the polymer matrix in composites. They are widely produced industrially as they possess many advantages compared to other thermosetting resins which include room temperature cure capability, good mechanical properties and transparency. Unsaturated polyester (UP) resins are extensively used in composite industry because of their good mechanical properties, low cost and easy to use [5].

The objectives of this work is to prepare date pits filler reinforcement unsaturated polyester composites and thereafter characterise for them.

2. Materials and Methods

The date palm fruits were obtained from Gwagwalada market in Gwagwalada area council, F.C.T; Nigeria. The matrix material used in this study was based on a commercially available unsaturated polyester with density 1.03g/cm^3, and polyamine curing agent (hardener) that were procured from a local supplier in, Lagos State, Nigeria.

2.1 Filler Preparation

The date pits (DTP) were separated from their fruits manually, thereafter washed and cleaned to remove contaminants and they were dried and grounded with hammer mill to obtain filler powder. The fillers were made to pass through wire mesh screen to obtain about 150µm particle size, thereafter, oven dried for 4hrs at temperature of about 70^{o}C before use so as to reduce the moisture content. Samples were thereafter stored in a dessicator.

2.2 Preparation of Composites

Unsaturated polyester (UP) composites with varying degrees of filler loading were prepared (10, 20, 30, 40, & 50 wt %) each were made from fillers with the matrixes. Virgin resin without filler was equally prepared to serve as control. This was achieved by mixing the various ratios of the prepared fillers with the resin to form homogenous blends. The mixing was achieved via manual stirring method for 10minutes. 0.2% of the accelerator (cobalt naphthenate) was added to mixture and stirred for 3minutes before the final addition of the catalyst i.e methyl ethyl ketone peroxide in ratio 2% of the resin, the mixture was poured onto the cavity of glass mould previously covered with aluminium foil so as to serve as releasing agent. The mixture was allowed to cure at room temperature for 24hours before removal from the mould. The composites were kept for 14 days at room temperature for complete curing operation. The sheeted composites (140 x 70 × 4 mm) were thereafter machined into various dimensions according to ASTM standards for various polymer test.

COMPOUNDING OR MIXING
Mixing of matrix + Fillers
UP + PITS
↓
ADDITION OF CURING AGENT
↓
MOULDING
To obtain homogenous thickness of 4mm
↓
CURING
↓
CUTTING OF SAMPLE
↓
CHARACTERIZATION

2.4 Tensile Tests

The tensile test was carried out on a Universal testing machine (TIRA 2810) with maximum load of 10KN in accordance with ASTM D3039. A cross speed of 2mm/min was used. Five specimens for each composite were tested and statistical averages for each set of results were recorded.

2.5 Flexural tests

The tests were carried out on a universal testing machine (TIRA test 2810) with maximum load of 1KN in accordance with ASTM D790. A cross speed of 5mm/min was used. The specimens were position horizontally on the machine. Flexural strength (at break), flexural modulus were determined from the results.

Flexural Strength (F_s) = $\frac{3PL}{2bh^2}$

Flexural modulus (F_m) = $\frac{PL^3}{4bwh^3}$

Where P is the maximum load in Newton (N), L is the range (mm), while h is the specimen thickness (mm), b is the specimen width (mm) and w indicates the depth of the deflection (mm)

2.6 Impact strength test

The impact tests were performed according to ASTM D256 standard using impact testing machine (IMPat 15). The test method determined the charpy impact strength of the composites. An unnotched charpy impact type test in which the specimens were held as a cantilever beam (usually horizontal position) thereafter broken by a blow delivered at a fixed distance from the edge of the specimen.

Impact strength = (KJ/A) where, J=Energy absorbed (KJ) and A = Area of cross section of the specimen (m^2).

2.7 Hardness Test

The tests were performed in accordance to ASTM D2240. The indentations were carried out on three positions of the specimen surface and the average result for each samples were recorded.

2.8 Density of the pits and composites

This was carried out in accordance with ASTM D792. In this research, the weight of each specimen having been dried in an oven at 70°C to a constant weight was taken. Thereafter, the weights of the specimen when immersed in water were equally recorded. The density (ρ) is then determined from the relationship below.

$$\rho_c = \frac{W_c}{W_w} * \rho_w$$

Where
W_c = weight of composite in air
W_w = weight of composite when immersed in water
ρ_w = density of water at room temperature ($1000 kg/m^3$) or $1 g/cm^3$
ρ_c = density of the composites specimen.

2.9 Surface morphology of the composites

The images of the fractured surface of the composites were taken and analyzed. The SEM is a valuable tool in providing analyses such as the determination of failure mode, analysis of material defects or contaminants, and analysis of microstructures and material characterization.

3. Results and Discussion

Filler dispersion, filler content, degree of filler adhesion, degree of degradation of composites, interfacial relationship between filler and matrixes e.t.c are factors which determine the mechanical properties of polymer composites. The results of the tensile, modulus and flexural, modulus properties are as shown in figure 1 and 2 respectively, while figure 3, 4 and 5 depicts the impact strength, hardness and density of the composite respectively.

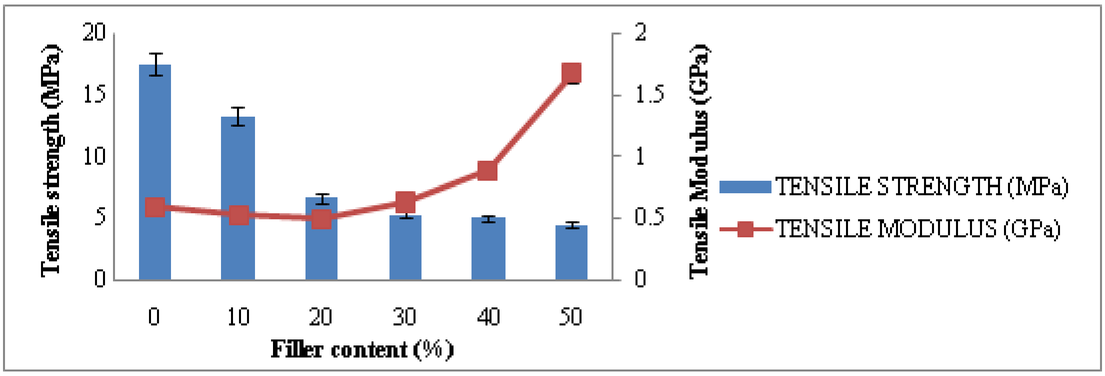

Figure 1: Effects of filler content on the tensile strength and modulus of date pits/ unsaturated polyester composites.

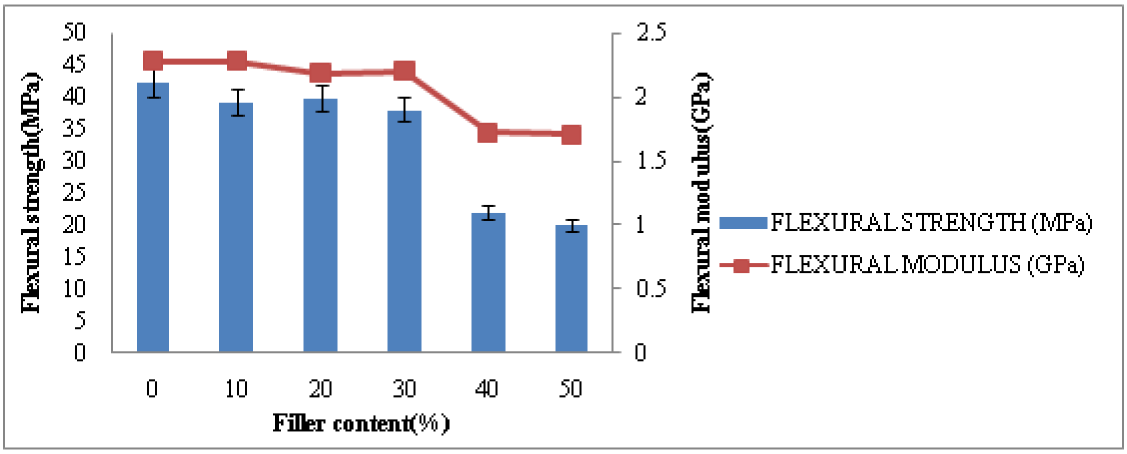

Figure 2: Effects of filler content on the flexural strength and modulus properties of date pits/unsaturated polyester composites.

3.1 Tensile properties

The effects of the filler content on the tensile strength and modulus of unsaturated polyester composites are as represented in Figure 1. From the results shown, an increase in the filler content by 10% produces a corresponding decrease of 24% in the tensile strength of the composite while modulus property increases as the filler ratio increases. Although, the modulus experience a slight reduction of about 11% and 17% as the filler content increases from 0 to 10 and from 10 to 20 respectively before the progressive increase in the tensile modulus as the filler content increase from 30 to 50 filler volume. The initial reduction might be attributed to inadequacy of the filler to saturate the matrix component thereby creating distance between the filler particles, hence, leading to poor load transfer to the bearing component or It may also be due to lack of proper dispersion of the filler in the matrix. Increases in tensile modulus as a result of the increase in the filler ratio impart stiffness to the composite. According to Adams, *et al.* (1969), the relative stiffness of a material is indicated by its modulus [6]. The incorporation of DTP has improved the stiffness of the matrix, since the tensile modulus of the composites increased as DTP filler loading was increased. The decreasing of tensile strength can be attributed to the physical properties of this filler and interaction of this filler with the matrix component [7]. The tensile strength and modulus of composite was maximum value at 10% and 50% with 13.34MPa and 1.683GPa respectively. In general, decrease in tensile strength with increase in filler loading was not surprising since other studies such as of Yang, *et al.*,(2006) and Raju, *et al.,* (2012) had also indicated that incorporation of ligno-cellulosic filler into matrix might not necessarily increase the tensile strength of the composites [8,9]. Similar works carried out by Imoisili, *et al.,*(2012) also showed a decrease in tensile strength as the filler ratio increases [10]. The relationship between the filler and unsaturated polyester resin shows that incorporation of the filler reduces the tensile strength of the resin to minimum of 4.5MPa.The later decrease in tensile strength and modulus at 50% filler weight is due to agglomerate formation at higher concentrations of the reinforcement of the composites [11]. This may also due to insufficient wetting of resin to the fillers for higher filler content. It was observed by many researchers that the modulus increases with an increase in filler loading [9]. This behaviour may possibly lead to the conclusion that tensile modulus perhaps depend on the on the filler content rather than the particle–matrix interface. Furthermore, increased modulus for higher filler content is due to the higher stiffness of the filling particles than the matrix material. The elastic properties of the composites decreases with the increased in modulus and thus, show that the stiffness of the composite increases by adding more filler. Stiffness is a good property generally when considering the application of the material. Owing to this, overall stiffness of the composite specimens increased and thus tensile modulus enhanced [12].

3.2 Flexural Properties

The flexural properties of composites depend critically on the microstructure of the composite and the interfacial bonding between the reinforcement and the matrix [13]. From Figure 2, it can be observed that the flexural strength of the neat resins was reduced from 42.31MPa to 40MPa on the addition of 20% filler showing a decline of 5%. Agro-particles mostly shows significant reduction in flexural strength of neat resin when used as filler in composite preparation and so they majorly served as bulking agent in composites. Thus, the slight reduction may due to the decreased interfacial adhesion/bonding between the particles and the matrix which does not facilitate load transfer. The decrease in flexural strength and modulus at 50% filler loading could be due agglomerate formation at higher concentrations of the filler in the composites which was also observed in the tensile behaviour [11]. Similar results have been reported by other researchers that the flexural strength decreased after 40 wt% filler loading [9]. The maximum flexural strength of 40MPa was observed at 20% filler content while the minimum value of 20.06MPa was observed at 50% filler content. The flexural modulus was at maximum at 10% filler content with value 2.286GPa.

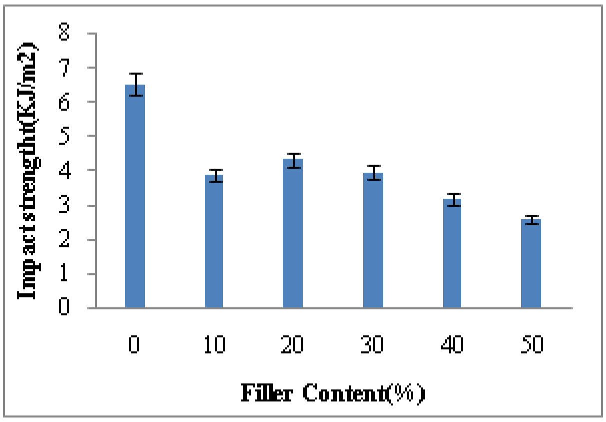

Figure 3: Effects of filler content on the impact strength of date pits/unsaturated polyester composites.

3.3 Impact Strength

The response to impact shock of the composites is as shown in Figure 3. From the results, the impact strength increases up to a maximum value of 4.328KJ m^{-2} at 20wt % and reduces as reinforcement increases which gives minimum value of 2.572KJ m^{-2}. The increase in impact strength of a composite is due to increase in elasticity of the composite thereby increasing the deformability of the matrix. The decrease in impact strength is due to the inability of the filler to block the crack propagation resulting in reduction of the impact strength [14].

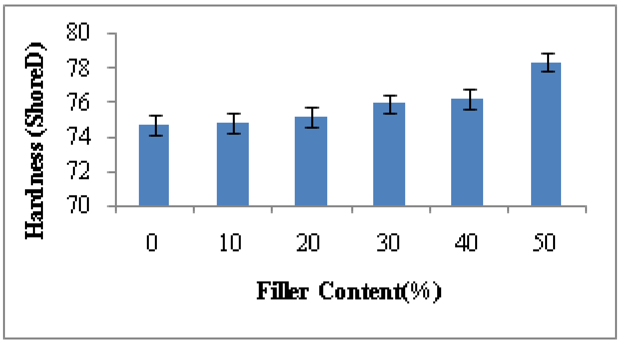

Figure 4: Effects of filler content on the hardness of date pits/unsaturated polyester composites

3.4 Hardness

Hardness implies a resistance to indentation, permanent or plastic deformation of material. In a composite material, filler weight fraction significantly affects the hardness of the composite material. The effects filler loading on the hardness of the composites are presented in Figure 4. It was found from that hardness of neat resin was 74.7. The values from the result show that an increase in filler increases the hardness. That is, hardness increases with high wt% of date pits particulate in the composite. Hardness test is a simple one and gives good information on the microstructure relationships of polymer composites [15].

The date pits/ unsaturated polyester composite increases up to maximum of 78.4 at 50% filler content. This might be attributed to the nature of the filler since the mechanical properties of composite depends on nature and type of the filler. Naturally, date pit is a hard stony material that can be termed as composite containing cellulose as reinforcement in a lignin matrix. From the results, it can be deduced that hardness is influenced by the filler ratio, and also more importantly the types of filler i.e the nature of the filler [16].

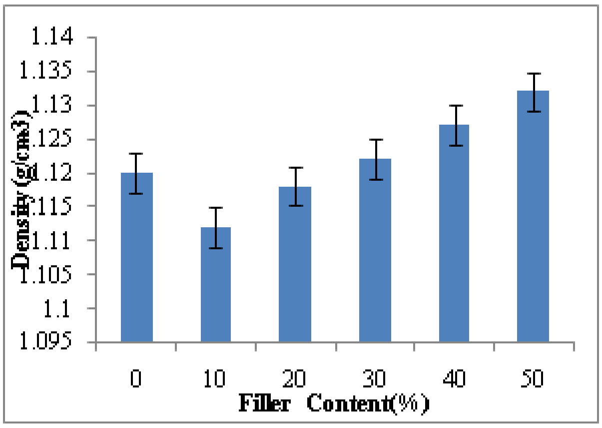

Figure 5: Effects of filler content on the density of date pits/unsaturated polyester composites.

3.5 Density

The density of date pits reinforced composite with different composition is as shown in Figure 5. It can be seen that the density at 50% filler weight content is 1.132 g/cm^3 while that of the minimum is 1.112 g/cm^3 at 10% filler weight content. The result reveals that the density of the composites increases as the filler ratio increases, this is because the density of the unsaturated polyester resin used is less dense compare to the date pits particulate. In the present investigation density of date pits is 1.14g/cm^3 while that of unsaturated polyester is 1.120g/cm^3. Hence, it can be concluded that proper combination of the both fillers in composite making may have a varieties of industrial application where weight and strength would be the critical parameter in the design.

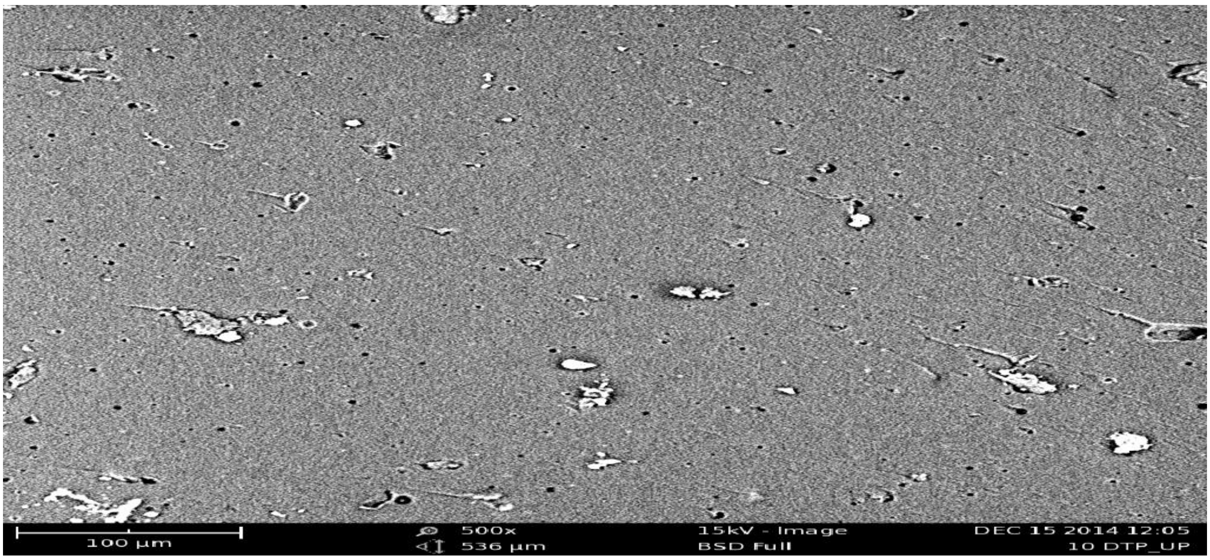

Figure 6: SEM micrograph of 10wt% date pit loading at 500X

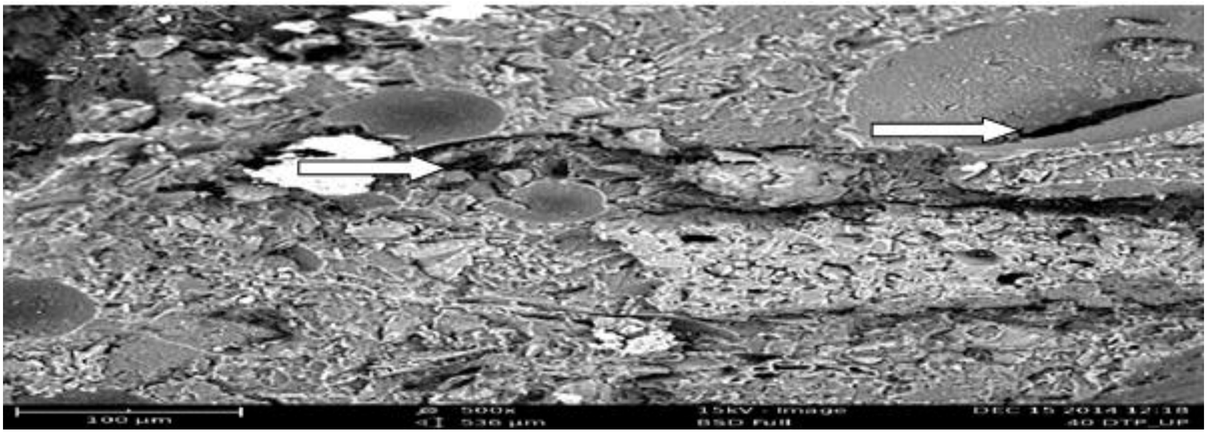

Figure 7: SEM micrograph of 40wt% date pit loading at 500X

3.6 Morphology

Studies of the fracture surfaces of the composites were carried out and the results are as shown in Figure 6 and 7 above. The scanning was done on the impact fracture surface of the composite. Figure 6 indicates the state of dispersion of 10wt% filler at 500X while Figure 7 shows the morphology of 40wt% date pits filler in the resin. Thus, the micrographs reveal the state of dispersion of date pits filler (DTP) into the resin matrix which plays vital role on the mechanical properties of the composite. It can be seen from Figure 6 that the filler dispersed uniformly in the matrix and a good interfacial bonding exits between the filler and the resin. However, several pores or voids were observed in the micrographs that might have occurred as a result of the technique used in the fabrication, that will ultimately affects the mechanical property adversely. Also, the presence of voids in a composite can open doors for crack propagation that will reduce the functional ability of the composite. On the other hand, Figure 7 shows the interaction of 40wt% filler with the resin, and from the results, positionof crack propagation can be observed as depicted with the arrow which might be due to non- uniform dispersion of the filler in the resin. It might also be due to the formation of agglomerate at higher concentration of the filler which has been observed in tensile strength and modulus at 40% filler weight [11]. It may also due to insufficient wetting of resin to the fillers for higher filler content. This occurrence will permit the crack to broadcast at quicker rate (less adhesion), which affects the mechanical strength of the material.

4. Conclusion

This study reveals that it is possible to produce composite panels using date pits with unsaturated polyester resin. Properties such as modulus, hardness increased as filler loading increases while properties such as tensile strength and flexural strength decreased as the filler loading decreases. The presumption of this investigation was that the material composite to be produced might be used in automobile construction if promising result were attained. However, the composite might still be useful in automobile industry for vehicle interior constructions such as dash board. The optimum ratio of the fillers that the resin can accommodate is 50wt% showing that the filler will serve as good bulking agent in composites production and beyond this value the resin will not be able to accommodate the filler.

The composites material produced may also be useful for several application both for indoor and outdoor usage such as ceiling construction, table top, particle board, wall tiles e.t.c. Nigeria is one of the largest consumer of dates fruit in the world, the use of wastes such as the pits from date fruits for producing useful components would be very attractive for the economy.

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