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STUDY THE EFFECT OF THE PARTICLE SIZE ON MECHANICAL PROPERTIES OF PARTICULATE NATURAL COMPOSITE MATERIALS

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Abstract: *This research studied the mechanical properties of particulates composite materials made from natural fillers (eggshell and sawdust) reinforced epoxy resin. The composite specimens were prepared by hand lay-up technique with one volume fraction Vf, and different particle size of fillers.*

Modulus of elasticity, tensile strength, percentage of elongation and hardness were measured for each type of composite. The tensile tests results had shown an incremental increase with size of particles decreasing also hardness results had shown an incremental increase with smaller particle size.

This work institutes a good substitution for synthetics fillers avoiding their highly cost and their toxicity, at the same time reducing of the environmental pollution..

Keywords: natural fillers, particles size, mechanical properties, particulate composite materials.

1. INTRODUCTION

In previous years there is a necessity of materials with unconventional properties that satisfy an ever more difficult market show the way to the utilization of composite materials. For instance, composites use at automotive, aeronautic and civil construction. Composite materials are multiphase materials that prove advanced properties than their individual phases, providing a synergistic effect. [1]

From meaning, composites are materials be made of at least two chemically separate elements on a macro level with a different boundary separating them and having a good performance which is noticeably dissimilar from those of individual elements. The major phase of composite material is usually poor in stiffness, weak, more ductile and having a continuous character is called matrix. The matrix forms the large fraction in composite. The minor phase is a discontinuous form which is fixed in the matrix. The dispersed phase is normally stiffer and stronger than the matrix phase therefore is called reinforcement. It employs to reinforce the matrix and improves the mechanical performance of the matrix. [2]

Polymers which used as matrix materials consist of two main classes such as thermoplastics and thermosets. Thermoplastics (polypropylene, nylons, acrylics etc.) by effect of heat they can be repeatedly softened and reformed. Conversely, thermosets (phenolics, epoxies, unsaturated polyester etc.), are materials that experience a curing process through their production, behind which they are rigid and cannot be reformed. [1]

In practice, for uses most polymers in engineering applications require greatly filled with protective particles. These particles provide to improve several important properties as well as density, strength

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modulus, thermal expansion, and thermal conductivity. For many years, material scientists create many studies covered composite systems, unexpectedly slight essential thoughtful present about the effects of the size, shape, distribution, surface chemical nature, and concentration of the filler particles on the mechanical and rheological performance of composite and polymers through the curing process. [3] Inorganic materials frequently need chemical modifications to enhance the interface between filler and polymer. [4] The chemical modification acts as a connection between the inorganic filler and the organic polymer matrix. The connection must hold or link to the filler and in order must strongly attach with the polymer. [5]

Inorganic mineral fillers similar to calcium carbonate (CC), silica, and talc have achieved importance because inexpensive fillers for thermoplastics. [6] A late present engineering research recognized that the natural filler reinforced polymer composites, for the reason that natural fibers or "bio-fibers" involve several compensation over conventional polymer fillers for instance glass fiber, these comprise low-priced, little energy use, non-harsh nature, security in usage, light weight, potentially higher volume fraction, high specific properties, adding up to the importance of environmental factors,. [7]

La Mantia[8] classify these materials, which employ normal resources, like green composites. A green composites seeking-out for materials coming from renewable resources will be favorite as opposite to the exhaustible fossil products. 9]

More studies had studied the effect of type and particle size of particles on physical and mechanical properties of composite materials.

Ahmed.R, 2004 [10] made an investigation into the properties for unsaturated polyester resin reinforced with rice husk. Results showed that composite materials of rice husk gain better mechanical properties compared with composite prepared from unsaturated polyester resin without filler.
M. Mohammed , A. Hadi ,2012,[11]calculated the ultimate tensile strength, modulus of elasticity, elongation at break, hardness, resilience and specific gravity for natural composite materials and study the effect of egg shells powder on these mechanical and physical properties of natural rubber(NR).

The results of testing showed that the hardness and elastic modulus, specific weight improved with increasing of the powder. While it was observed that the tensile strength, elongation and resilience reduced with rising of powder ratio.

S.B. Hassan, V.S. Aigbodion, 2013[12], fabricated particulate composites materials with a total of 2–12 wt.% eggshell particles (ES) additions. And the effects of (ES) on the microstructures and properties of Al–Cu–Mg/ES particulate composites were examined. The physical and mechanical properties measured integrated: density, tensile strength, impact energy and hardness. The results discovered that the tensile strength and hardness were increased for uncarbonized ES more than for carbonized ES while the impact energy was decrease by 23.5% at 12 wt.% uncarbonized ES and 24.67% at 12 wt.% carbonized ES particles, respectively

K. A. Iyer, J. M. Torkelson,2014,[13] prepared green composite materials from egg shell and used complex grinding–sieving and/or chemical modification to assist in dispersing ES in polymers such as polypropylene (PP). The high-quality dispersion of filler guide to a principal raise in Young's modulus (87% enhance relative to orderly PP for 40 wt% ES) and a fine enhance in hardness; results display drop in strength of yielding, elongation, and impact strength.

Ch. Charoenwong, S.Pisuchpen[14] , in that research, used four levels of rubber wood sawdust particle with sizes (<10, 10-20, 20-35 and > 35 meshes) with polymeric materials and prepared a medium density particleboards from. The modulus of rupture, modulus of elasticity, thickness swelling and water absorption were examined. It was established that the particles sizes really affected in the mechanical properties of boards but the resistance of water of boards was unfavorably affected by the particle sizes. It recommended that the finer the particles sizes, the weaker and the higher water resistance.

2. THEORETICAL PART

2.1. PROPERTIES OF USED MATERIALS

2.1.1. MATRIX MATERIAL (EPOXY):

One of the thermosetting polymers is epoxy resin that, prior to curing, has one or more active epoxide or oxirane groups at the end(s) of the molecule and a few repeated units in the middle of the molecule [15].

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Comparison with further materials, epoxy has many exclusive chemical and physical properties. Epoxy resins be able to formed to have outstanding chemical resistance, excellent adhesion, high thermal and electrical resistance, low shrinkage, and good mechanical properties, for example high strength and toughness. These good properties effect in epoxy resins include wide applications in industry, packaging, aerospace, construction, etc. They have found significant applications as bonding and adhesives, protective coatings, electrical laminates, apparel finishes, fiber-reinforced plastics, flooring and paving, and composite pipes .mechanical properties of epoxy were showed in table (1) [16].

Table (1): Mechanical properties of epoxy

Properties	
Glass transition temperature (Tg)	120 - 130 °C
Tensile strength	85 N/mm^2
Tensile Modulus	10,500 N/mm^2
Elongation at break	0.8%
Flexural strength	112 N/mm^2
Flexural Modulus	10,000 N/mm^2
Compressive Strength	190 N/mm^2
Coefficient of linear thermal expansion	34 *10-6 K-1
Water absorption - 24 hours at 23°C	5-10 mg (0.06-0.068%) ISO 62 (1980)

2.1.2. REINFORCING MATERIALS:

A : EGGSHELL FILLER

One of the bad environmental troubles, which have been scheduled universally, is chicken eggshell (ES) is an aviculture byproduct, particularly in those countries where the egg production is fine developed. About 150,000 tons of this material is disposed in landfills only in the United State .Eggshell has about 95% calcium carbonate in the form of calcite and 5% organic materials such as type X collagen, sulfated polysaccharides, and other proteins[table 2] [17,18]. Though there have been some efforts to employ eggshell components for unusual submissions [19–20].

Along with further properties, that egg shell has light weight contrast to mineral calcium carbonate (values of density got by ASTM 679 are 0.4236 g/cm3 for eggshell put side by side with 0.4670 g/cm3 of commercial calcium carbonate or 0.4581 g/cm3 of talk). These characteristics succeed eggshell as an excellent applicant for bulk quantity, low-cost and low load-bearing composite applications, such as the automotive industry, trucks, homes, offices, and factories. The chemical composition of eggshell investigated by using X-ray Fluorescence Spectrometer was listed at table (2) [21]

B : SAW DUST FILLER

The waste sawdust is a significant source of raw material. FAOSTAT (Food and Agriculture Organization of the United Nations) wrote a report that explains that the quantity of diverse types of wood cut by sawmills is about 125.36 million m3only in Europe in the year 2010 [22]. The sawdust losses resulting from sawing processes are between 5-11% of the total log volume. At a least loss value of 5% results in a volume of 6.27 million m3 sawdust. As result sawdust is a significant renewable raw material and be able to utilize in modern functions additionally than heating.

Lignocelluloses fibers have a set of advantages as contrast with conventional glass fibers utilized to

Table (2): Chemical composition of the eggshell

Chemical Composition	Concentration (%)
Al2O3	0.001
SiO2	0.001
S	0.001
Cl	0.009
CaO	99.83
Cr2O3	0.003
MnO	0.001
CuO	0.001
LOI	0.153

strengthen composite materials. Their ecological character, biodegradability, low-priced non-abrasive nature, safe treatment, use with various possibilities as fillers, processing with low power consumption, vital properties, low density and a large number of types of fiber are very important factors for their receiving in markets where a large amount of materials is required such as automotive industry.

On the other hand certain disadvantages, such as the tendency of agglomeration throughout manufacture, low resistance to moisture and quality changes due to the seasons of growth, reduce the potential for these fibers [23, 24]. The chemical composition of saw dust was listed at table (3) [25]

Table (3): Chemical composition of the wood powder

Chemical Composition	Concentration (%)
Cellulose	47%
Lignin	21%
Hemi- Cellulose and other compounds	30%
Extractives	2%
Ash	0.4%

3. EXPERIMENTAL PROCEDURE

3.1. MATERIALS

The essential materials used in the preparation of samples in this research consisting of epoxy resin , with a density of (1.2 gm / cm3) made in Jordan in the shape of transparent viscous liquid at room temperature which is a thermally hardened polymers (Thermosets). The powder was used were egg shell (30, 50 and 100µm), and wood powder (sawdust) (30, 50 and 100µm) as shown in figure (1).

3.2. PREPARATION OF NATURAL MATERIALS

A: EGGSHELL POWDER

The eggshell used in this research was brown eggshell that they were collected and washed and sun

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dried to remove the membranes, crushed, grinded by milling machine and then the set of sieves employed to obtain a powder of egg shells in the variously size.

Figure (1): The eggshell and sawdust powder before and after milling

B: WOOD POWDER (SAWDUST)

Wood powder is a consequence of cutting, grinding, drilling of wood with a saw or other tool; Subsequently, sawdust was screened to three levels by sieving with screens having 30, 50, and 100 μm openings and oven-dried to less than 5% moisture content.The powder that using in this work was shown in figure (1).

3.3. PREPARATION OF SAMPLES

Moulds required for obtaining the samples were prepared from glass with dimensions of (200×250×5) mm. The internal face of the mould was covered with a thin layer of nylon (thermal paper) made from polyvinyl alcohol (PVA) so as to avoid sticking between cast material and the mould as shown in figure (2).

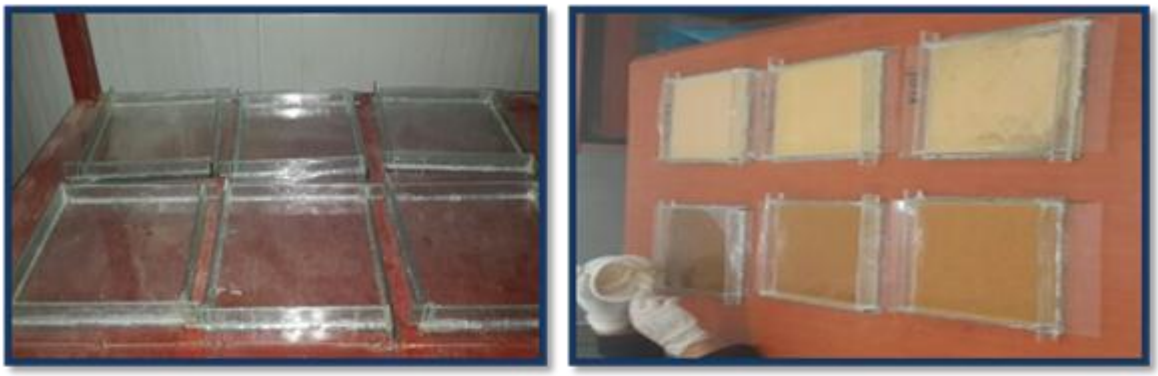

Figure (2): The shape of the prepared mould.

The technique applied in the preparation of the specimens, in this study is the (Hand lay-up Molding) by addition the epoxy with the hardener and mixing it continuously and slowly by using a glass rod to keep away from forming the bubbles.

And adding the powder intermittently into the mixture and moving it for a period of (10-15) minutes to achieve homogeneity. It is very essential that the mixture must have a good viscosity for the purpose of

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protecting the particles from precipitation which may result in the heterogeneity of the mixture that leads to the agglomeration after hardening.

For the purpose of completing the process of hardening, finally is leaving the sample in the mould for a period of (24) hour at room temperature. Samples are then extracted from the mould and then heat treated in an oven at (60°Ċ) for a period of (60) minutes. This process is very important for the purpose of obtaining the best cross linking between polymeric chains, and to remove the stresses generated from the preparation process and complete the full hardening of the samples.[26]

3.4. EQUIPMENT

The equipment used in this study included:
Ball milling machine, sieves, measuring cylinder of 200 ml capacity, furnace, a digital weighing balance, molding (casting) boxes, universal tensile testing machine, and a digital hardness instrument.

MECHANICAL TEST

TENSILE TEST

Tensile test is widely used to provide the designer with information about material strength and maximum elongation and others. The stress used in such curve is a longitudinal stress in test specimen and expressed as:

$$\sigma = P/A \quad \text{.............................. (1)}$$

Where:-

σ: Longitudinal stress for specimen (MPa).

P: Applied load (N).

A: Original cross sectional area before testing (m^2).

The strain which is used in such stress-strain curve is a linear strain and can be expressed as:

$$\varepsilon = \frac{\Delta L}{L_\circ} \quad \text{.................. (2)}$$

where:-

ε : Strain.

$$\Delta L = L - L_\circ$$

L : The final length (m).

$L_\circ$: The original length (m).

From the tensile curve, the following can be calculated:

1. Tensile strength (MPa).

2. Modulus of elasticity (MPa).

3. Elongation percentage at break (%).

This test is performed according to (ASTM D638M- 87b) at room temperature [27]. The used tensile machine is (INSTRON 1195, made in England). Figure (3) shows a standard specimen for tensile test.

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Figure (3): Tensile specimens and test instrument

HARDNESS TEST (SHORE D)

This test is performed by using hardness (Shore D) and according to (ASTM D-2240) standard at room temperature [28]. Samples have been cut into a disk with diameter (40 mm) and (5 mm) as thickness. Figure (4) shows hardness device and sample used in this research. For each specimen five hardness measurements were taken and the average hardness is calculated.

Figure (4): Hardness specimens and test instrument

4. RESULTS AND DISCUSSION

4.1. TENSILE TEST

Tensile test was executed on the specimen prepared from Epoxy before and after adding the particulate fillers of the two types. Curves of tensile test are illustrated in Figures (5) to (9).

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Figures (5) and (6) illustrate that the addition of (Eggshell +Sawdust) powder in (6 %) volume fraction respectively. where at the same fillers volume fraction leads to increased the tensile strength with decreasing particles size. Such behavior is due to the nature of bonding force between the matrix and fillers particles which is strong bonding in case of small particle size that does not allow forming internal defects (cracks) in quick manner and in turn the composite material will have high tensile strength.[29]

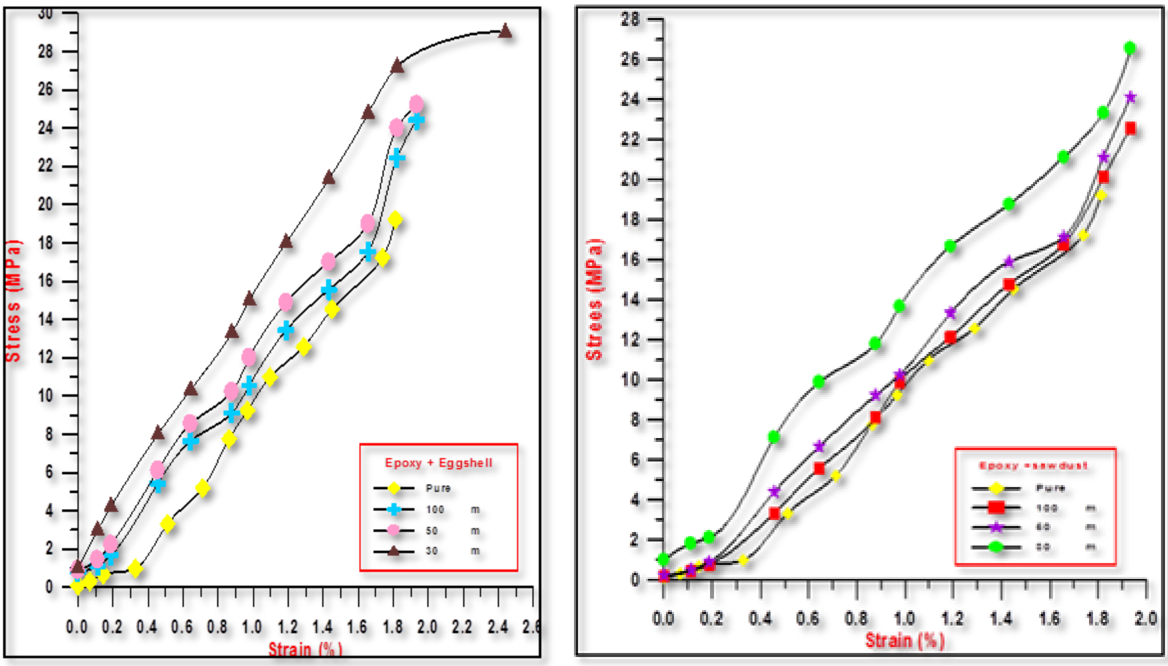

Figure (5,6): Stress- strain curve of epoxy resin reinforced with eggshell and sawdust fillers at volume fraction of (6 %) and particles size (30-100) µm.

The relationship between the modulus of elasticity and the particles size of reinforcement fillers is shown in Figure (7). From this figure, it be able to be seen that small particles of reinforcing fillers improve the modulus of elasticity more than large particles, this is due to the strengthening mechanism as follows firstly: large particles strengthening for reinforcing fillers tend to restrain the movement of the matrix phase in the area of each particles, whereas the matrix transfers some of the applied load to the particles and bear fraction of it, and secondly: the small fillers hinder or impede slipping of matrix chains and require high stress to bow them in narrow space among particles compared with large particles of fillers and the matrix bears the major portion of the applied load [29].

Both egg shell, and saw dust powder enhanced the tensile modulus of epoxy. However, egg shell powder was superior to saw dust powder in increasing the modulus of elasticity of epoxy. Where, it can be noted the modulus of elasticity for eggshell fillers composite is higher than that for the sawdust fillers composites, where the maximum values are (1.48 and 1.37 GPa) at (25 µm) for eggshell and sawdust fillers respectively.[21]

Figure (8) shows the relationship between the tensile strength and the size of the filler particles .The figure illustrates that, the tensile strength increases with decreasing particles size, where the smallest particle size has a noticeable effect on the tensile strength more than the large particles due to the same reason mentioned before.

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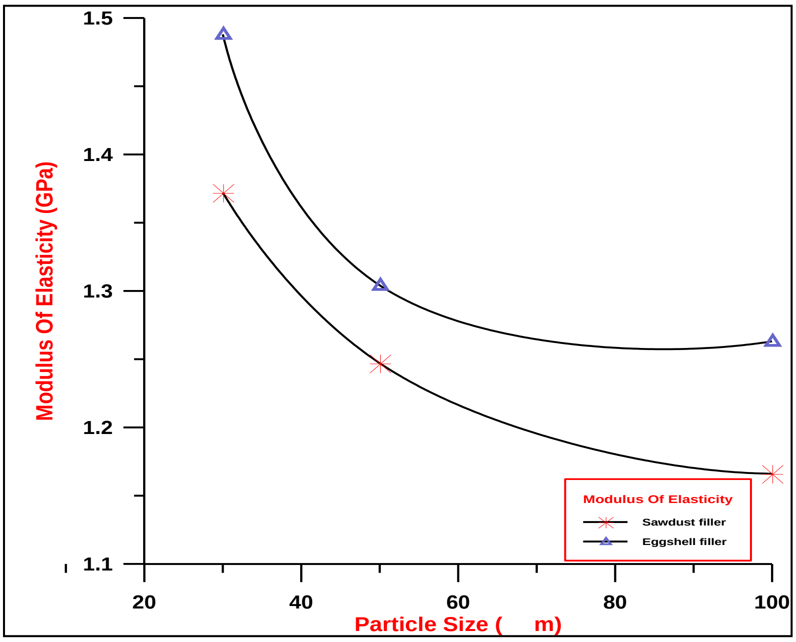

Figure (7): The relationship between the modulus of elasticity and particle size for epoxy resin filled by eggshell and sawdust fillers.

And it can be seen clearly that the strengthening by egg shell powder is more active than by sawdust, so that the tensile strength at (30 µm) particles size reaches (29 and 26.5 MPa), while (24.2 and 22.5 MPa) are at (100 µm) for eggshell and sawdust fillers composite respectively

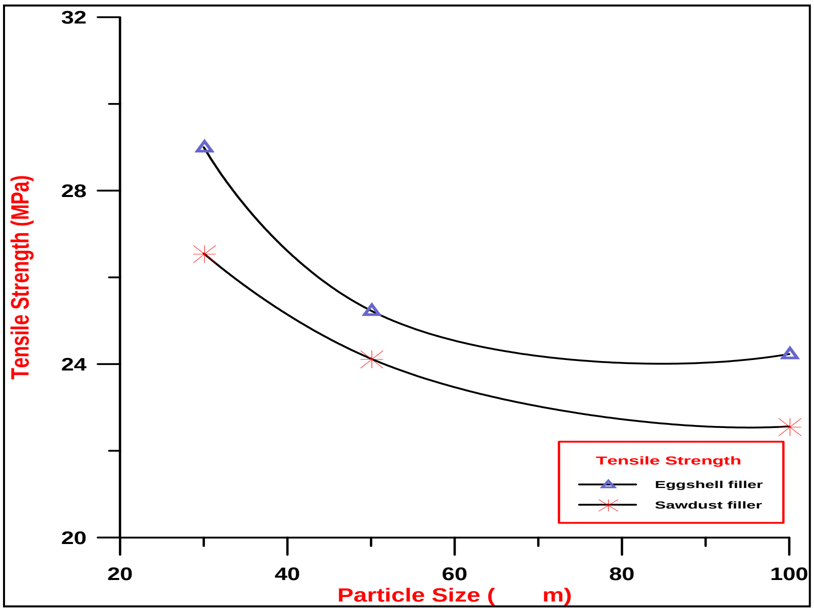

Figure (8): The relationship between the tensile strength and particle size for epoxy resin filled by eggshell and sawdust fillers.

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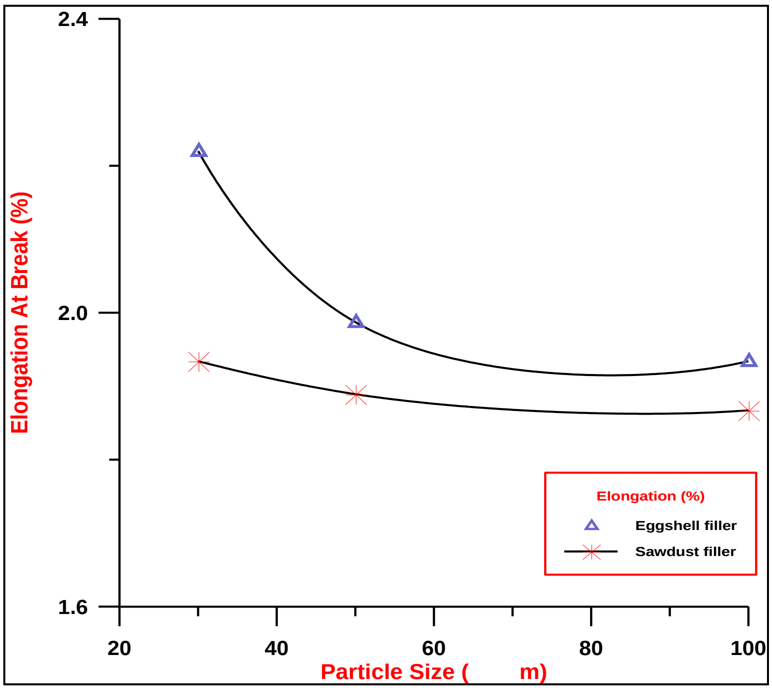

Figure (9): The relationship between the elongation percentage and particle size for epoxy resin filled by eggshell and sawdust fillers.

Figure (9) shows the relationship between the elongation percentage calculated at break point and the size of the filler particles of prepared composite materials. It could be observed from this figure that the finer particle size increases the elongation percentage at break more than larger particles size of two types of reinforcing fillers; this is due to the nature of bonding force between the matrix and fillers particles.

Also, the composite material filled with eggshell filler particles has higher elongation percentage than the other which was filled with sawdust filler particles.

4.2. HARDNESS SHORE (D)

Hardness test type shore (D) has been carried out on epoxy before and after powder fillers were added and the average of five readings in each case was taken to obtain higher accuracy results. Table (4) shows the hardness values shore (D) for the prepared specimens (pure epoxy, epoxy +6% natural powder) composites.

Figure (10) shows the effect of filler type and particle sizes on the hardness of filled epoxy. The hardness of unfilled epoxy is 61 (Shore D). The figure shows that the hardness of all filled epoxy at a given filler particle size increased more than pure epoxy. , this may be due to the fact that the hardness is generally considered to be a property of the surface therefore this behavior of hardness is expected. The addition of the filler leads to an increase in the elasticity and the matrix surface resistance to the indentation [30], thus specimen (epoxy +6% powder filler) have higher hardness than specimen with pure epoxy. The explanation of such behavior agrees with the results of modulus of elasticity because hardness gives indication to modulus of elasticity for polymer under simple strain condition.

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Table (4): Hardness of the prepared composites specimens

Specimens	Hardness Shore(D)
Pure Epoxy	61
Epoxy +Egg shell (30µm)	86.4
Epoxy +Egg shell (50µm)	82
Epoxy +Egg shell (100µm)	78.4
Epoxy +Sawdust (30µm)	79
Epoxy +Sawdust (50µm)	77.2
Epoxy +Sawdust (100µm)	75.4

Such increases in composites hardness with increase in filler content have been reported by Chakraborty et al [31], and Igwe and Njoku[32]. Generally, the hardness of all the composites decreased with increase in the filler particle size at any type of filler had used. At any given filler particle size considered, the order in the enhancement of hardness of epoxy is egg shell > sawdust powder.

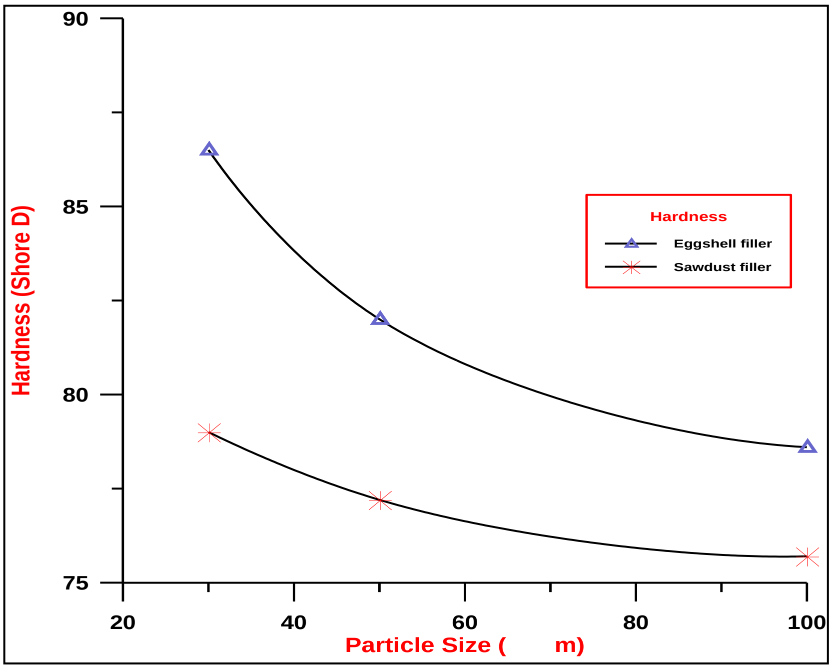

Figure (10): The relationship between the hardness and particle size for epoxy resin filled by eggshell and sawdust fillers.

5. CONCLCLUSIONS

1. The addition of the reinforcing fillers (eggshell + sawdust) to the epoxy polymer leads to develop the mechanical properties.
2. Ultimate tensile strength, modulus of elasticity and elongation percentages at break for two types of fillers increase with the addition of the reinforcing fillers. The small fillers improve the tensile properties

of pure epoxy more than large fillers. Where it reach the maximum value at (epoxy +6% eggshell in 30 μm particle size)

3. Result shows that the best hardness value for prepared composite material of (epoxy +6% eggshell in 30 μm particle size) was (86.4 shore D) also it mean that the hardness of the prepared composite material increases with the decreasing in the size of particle of two fillers.
4. From result can be noted that the best properties value for epoxy composite materials are improved by using eggshell particle when compared with sawdust particles uses.
5. The use of egg shell, and sawdust powder as fillers for the plastic industry will not only provide a renewable source of filler for the plastic industry but also generate a non – food source of economic development for the famers in the rural areas.

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