



## Preparation of Polyester/ Micro Eggshell Fillers Composite as Natural Surface Coating

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### Abstract

To fabricate an inexpensive surface coating with excellent mechanical properties with good water resistance and thermal diffusion, white eggshell fibers with particle size (~1micrometer) has been added by different weight percentages (1,2,3,4,5,6,7 and 8 %) to Unsaturated Polyester.

The weight ratio (4%) of eggshell powder is a good ratio to be added to polyester to improve its mechanical properties, such as hardness, impact strength, and wear resistance. The hardness was improved by (3.75%); impact strength has the same value as polyester, flexural strength by (8.43%) and high improvement in wear resistance (74.4%).

, as well as to get further improvements in mechanical properties of polyester, the eggshell powder was added with (5%), where the hardness was improved by (3.63%), impact strength improved by (~23%) and the flexural strength by (23.23%)but the wear resistance by (2.15%) only .

The resistance to thermal of polyester was improved to (9.95%) and (13.77%) by adding the eggshell powder with ratios (of 4%) and (5%), respectively.

(Polyester/ 4% eggshells). And (Polyester/ 5% eggshells) composites have a higher resistance to water diffusion after being immersed in water between (1-4 weeks).

The electron microscope image was used to observe the degree of homogeneity in polyester composite microstructure, which improved the results mechanical and thermal results.

**Keywords:** natural fibers, polyester, eggshell, mechanical properties, thermal conductivity and water diffusion.

## 1. Introduction

Chicken eggshell (E.S) is a biomaterial consisting of calcium carbonate with a weight fraction (of ~95%) and other materials like ( $\text{SiO}_2$ ,  $\text{AL}_2\text{O}_3$ , P, S, CL) with a weight ratio (~5%) [1, 2].

Eggshell has many properties, such as non-toxicity, lightweight, costless, and high strength. Calcium carbonate in eggshells has a low density compared to the mineral calcium carbonate, which makes eggshells of great use in many applications, including the use of eggshells by many researchers [3, 4].

Chicken eggshell fibers have been used with different percentages and particle sizes to reinforce polypropylene (PP), where the Young's modulus increased because of the improvement of the bonding between eggshell powder and polypropylene matrix [4].

The eggshell has been used with weight ratios (10, 20, and 30 %) as fillers in poly lactic acid (PLA), and they have found that the ratio (30% eggshell) was the best to improve the (PLA) strength [5].

The epoxy flexural and tensile moduli are improved using the eggshell by the two weight ratios of 10 and 20 % [6].

In this research, the eggshell waste powder was employed in a certain weight ratio to prepare a surface coating with high mechanical properties (hardness, impact strength, flexural strength, and wear resistance) and high resistance to both heat and water diffusion.

## 2. Experimental work

### 2.1. Materials

#### 2.1.1. Matrix

The thermoset unsaturated polyester resin with trade name (siropol-8341) made by Saudi industrial resins limited was used as a matrix 2% cobalt was added to polyester as an accelerator. The resin converts from viscous liquid to solid by adding methyl ethyl ketone peroxide (MEKP) with a concentration (of 2.5% Wt.); the gel time was (15 min) at a temperature (of 25° C).

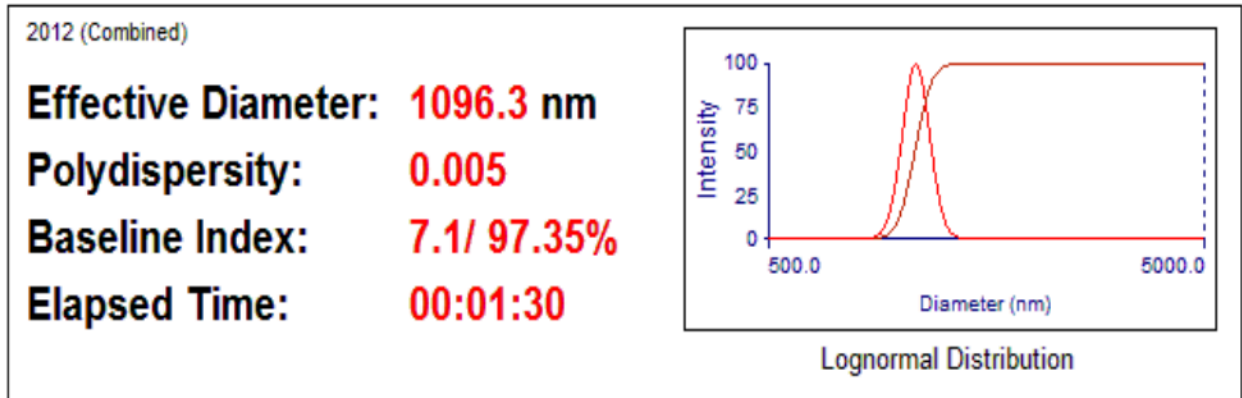
#### 2.1.2. Filler

Waste white eggshell (E.S) powder was used as a source of bio-caco<sub>3</sub> source.

The eggshell was cleaned with water and dried by the sun first, then using the oven for four hours at a temperature of 60° C. Dried eggshells were crushed and ground for one hour using a special milling device (NQM-0.4 ball mill) to obtain a fine particle size (~1micro meter).



Figure 1. a) crushed eggshell b) milling device c) ground eggshell



**Figure 2.** Volume size distribution of eggshell powder.

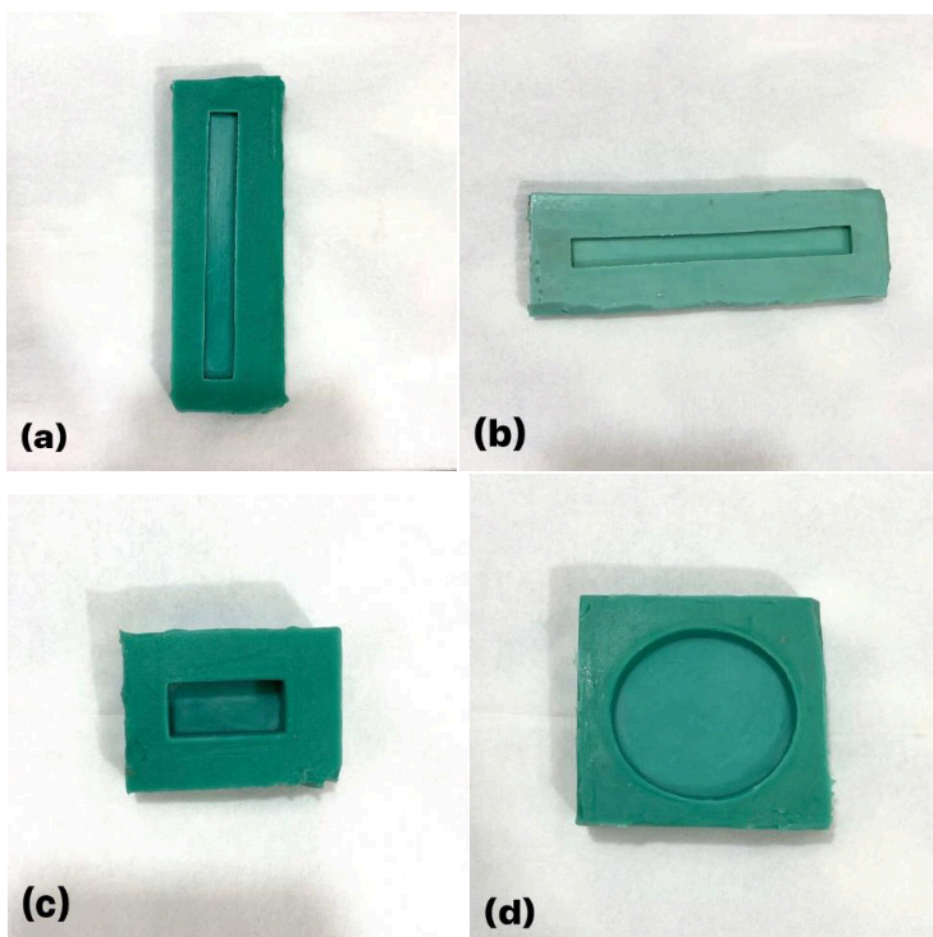
## 2.2. Method

The unsaturated polyester resin was mixed with a catalyst (MEKP), without any filler to prepare neat polyester, and with eggshell powder to prepare the composites (polyester/eggshell). As shown in figure (3), the mixtures were poured into silicone molds to easy composites sheets removal. Vaseline was used to rubber the caps in molds.

Different weight percentages of eggshell powder (1, 2, 3, 4, 5, 6, 7, and 8 %) were used to obtain the best percentage of polyester reinforcement when subjected to various tests.

The (PE/E.S) mixtures were allowed to cure for (3 hours) at room temperature. To prepare the gel coat, the best weight ratio of the eggshell powder was chosen to mix with the polyester after conducting mechanical and thermal tests on it.





**Figure 3.** Molds of silicon of the tests a) Impact strength b) flexural strength c) wear resistance d)thermal conductivity

### 3. Tests and Measurements

#### 3.1. Mechanical test

##### 3.1.1. Hardness

Shore (D) hardness test was performed on the composites using the device (TH2IO, Italy). Four measurements of hardness have been taken at different positions on composite surfaces to get the average hardness value.

##### 3.1.2. Impact Test

Impact test was carried out on polyester/ micro eggshell fillers (PE/ ME.SF) using a Charpy impact instrument. This test was performed according to the international system (ISO-179). The samples were cast with dimensions (100×10×8 mm).

##### 3.1.3. Flexural Test

To determine the flexural strength of (polyester/ eggshell powder) composite, with dimensions (100×10×4 mm), three-point bending was used according to (ASTM-D790).

##### 3.1.4. Wear Test

The wear test was performed on the composite specimens using the (pin-on-disk) by applying (500 gm) as a load on the sample. The distance from the center of the sample to the center of the metal disk was ( $r=500$  cm).

The sample under test rotates around the disk center by ( $n=375$  cycle/min) turns for five minutes.

To determine the wear loss of specimen, Equation (1) was used:

$$\text{Wear loss} = \frac{w_0 - w}{2\pi nrt} \dots\dots\dots (1) [7].$$

Where  
 $w_0$ : the original sample weight  
 $w$ : the final sample weight

**3.2. Physical Properties**

**3.2.1. Density Measurement**

Density be measured by dividing the sample mass by volume, the density of eggshell powder is pressing using piston to make a circular shape.

**3.2.2. Thermal Conductivity**

The thermal conductivity of (polyester/eggshell powder) composites was determined using Lee’s disk device.

**3.2.3. Water Diffusion**

The samples of (5×5×5 mm<sup>2</sup>) were cut from (PE/ ME.SF) composites. These samples were weighed before and after being immersed in water for different times (1, 2, 3, 4, 5, and 6 weeks).

The ASTM D570 was used to determine the water uptake using equation (1).

$$\text{Water diffusion} = (W_{\text{after}} - W_{\text{before}}) / W_{\text{before}} \dots\dots\dots(1) [8].$$

**3.3. Morphology**

The microstructure of (the PE/ ME.SF) composite was checked by electronic photography using the electronic scanning instrument (SEM) (Tescan) manufactured in British.

**4. Results and Discussions**

**4.1. Hardness**

The resistance of the material to indentation is hardness. Figure (4) shows hardness values increased from (79.27) for neat polyester to (~85) for polyester by adding eggshell at a weight ratio of (3%). This means the improvement in the hardness of polyester by (~ 7.22 %), by increasing the weight ratios of addition to (4%) and (5%), the improvement in hardness of epoxy decrease to (3.76) and (3.6), respectively.

The hardness maintenance is approximately the same value by increasing the weight ratios of adding eggshells, where the hardness values for all ratios are higher than its value for pure polyester, where the hardness values ranged between (82-85), except for polyester with (8%) of eggshell, where the value of hardness reaches to (87.42). This may be due to the agglomeration of eggshell particles. These results are in agreement with the research [1].

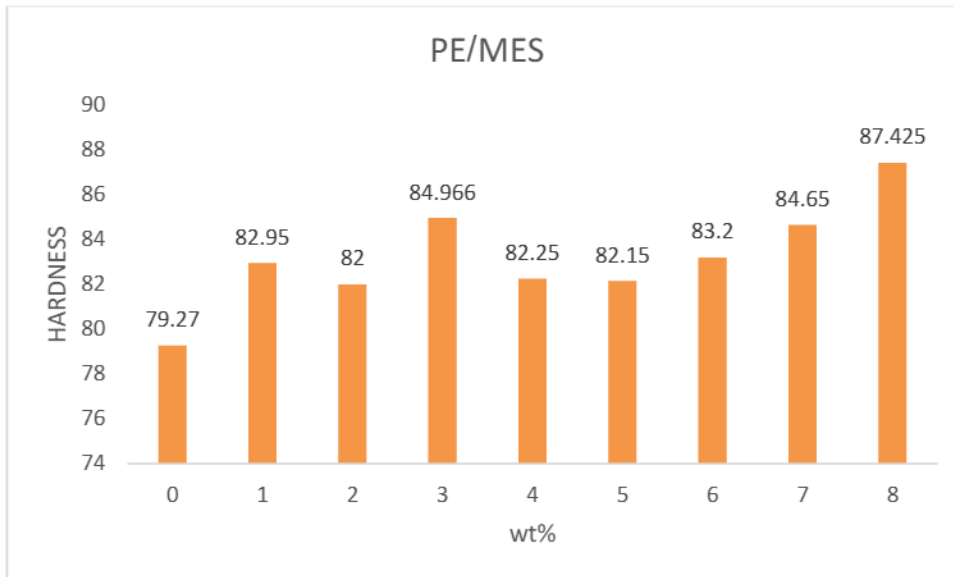


Figure 4. Hardness values of polyester / micro eggshell composites

#### 4.2. Impact Test

Impact strength can be defined as the ability of a material to absorb energy before breaking or falling [9].

Figure (5) shows the increase in the value of the impact strength from (8.59 KJ/m<sup>2</sup>) for neat polyester to a higher value (10.57 KJ/m<sup>2</sup>) for polyester reinforced with eggshell powder added by weight (5 %). Then it decreased to (9.15 KJ/m<sup>2</sup>) by adding (6%E.SF) to polyester. According to the researchers, this rise and then decline in impact strength is similar to the behavior of polyester composites [10].

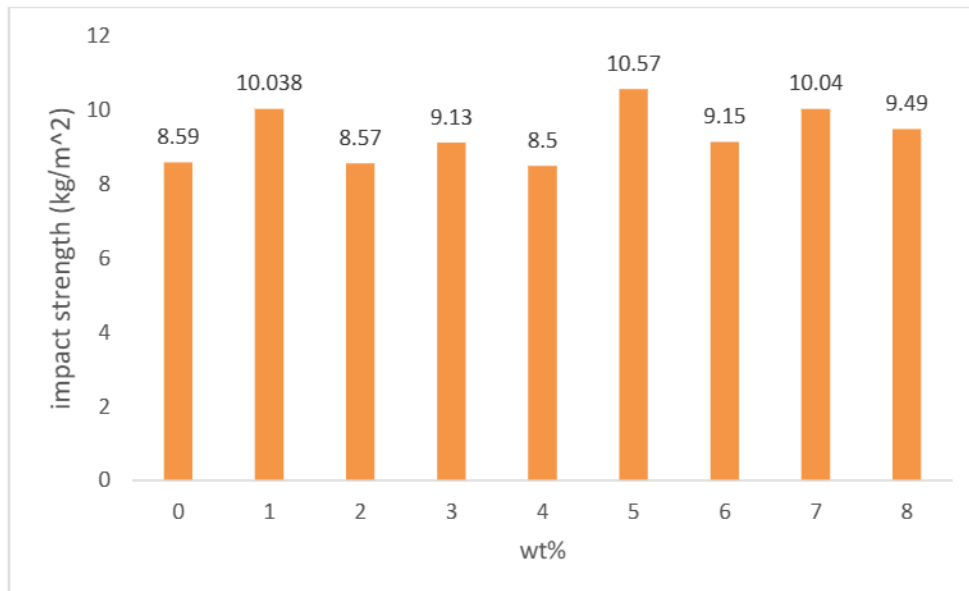


Figure 5. impact strength values of polyester / micro eggshell composites

#### 4.3. Flexural Strength

Flexural strength can be defined as the ability of a material to bend when applying external stress [9]. Figure (6) shows the increase in the value of the flexural strength from (55.48 KJ/m<sup>2</sup>) for neat polyester to a higher value (68.37 KJ/m<sup>2</sup>) for polyester reinforced with eggshell powder added by weight (5 %), then decreased to (37.06 KJ/m<sup>2</sup>) with adding (7 % E.SF) to polyester. The flexural

strength of polyester was improved by (8.43%) and (23.23%) by adding (4 %) and (5 %) of micro-sized eggshell particles, respectively.

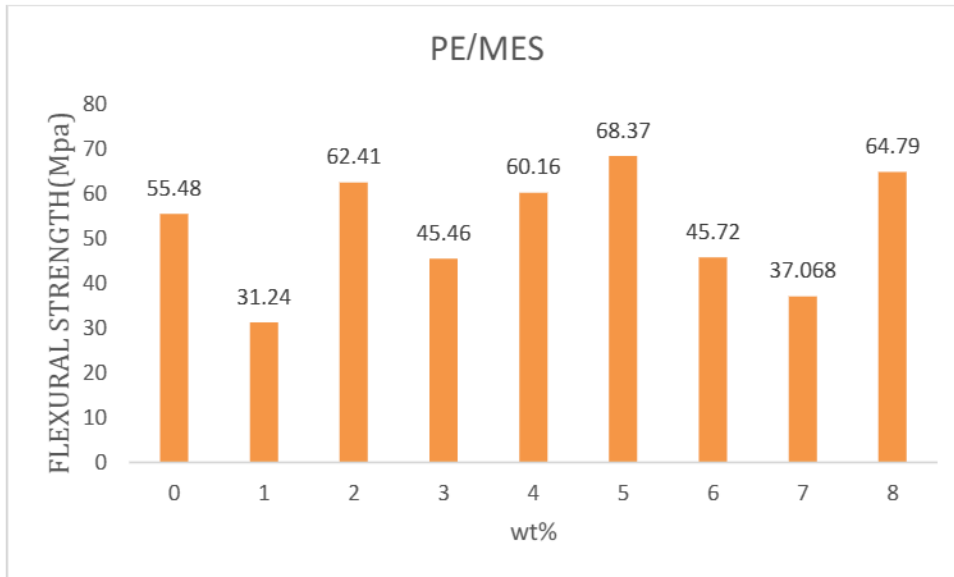


Figure 6. flexural strength values of polyester / micro eggshell composites

#### 4.4. Wear Test

The wear loss as a function of the weight percentage of micro eggshell fibers with particle size (~1micron) in polyester is shown in Figure (7), the value of wear loss for neat polyester is ( $3.72 \times 10^{-7}$  gm) which is decreased four times by adding (4 %) of the eggshell powder and then it goes back to the amount ( $3.64 \times 10^{-7}$  gm) by increasing the percentage of adding the powder to (5%).

This means that adding (4%) and (5%) of eggshell powder improves the wear resistance of polyester by (74.4%) and (2.15%), respectively. These results agreed with [11-12].

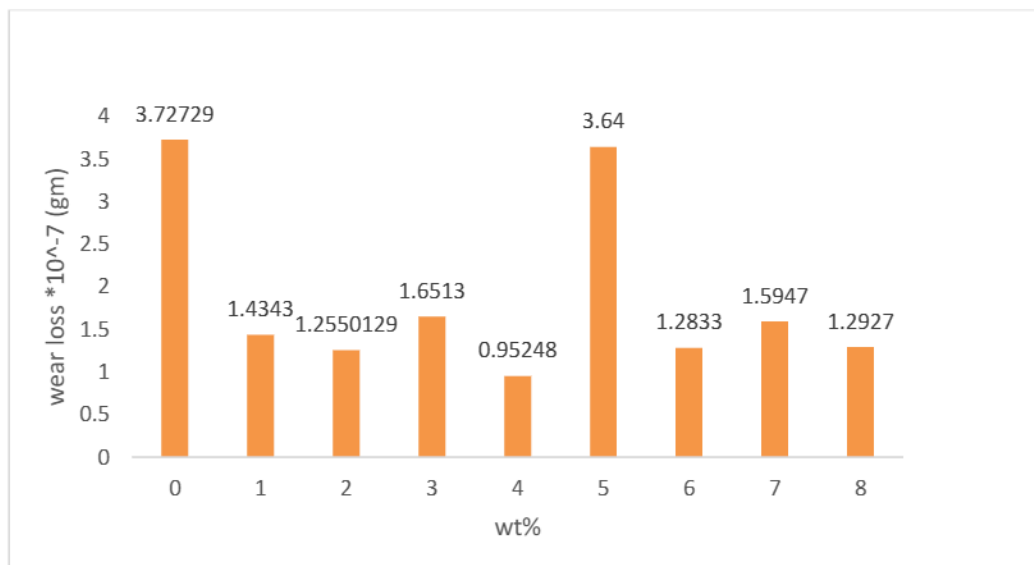


Figure 7. wear loss values of polyester / micro eggshell composites

**3.2. Physical Properties**

**3.2.1. Density Measurement**

The density ( $\rho$ ) of polyester with micro eggshells particles and different weight percentages are calculated by rule of matrix [13].

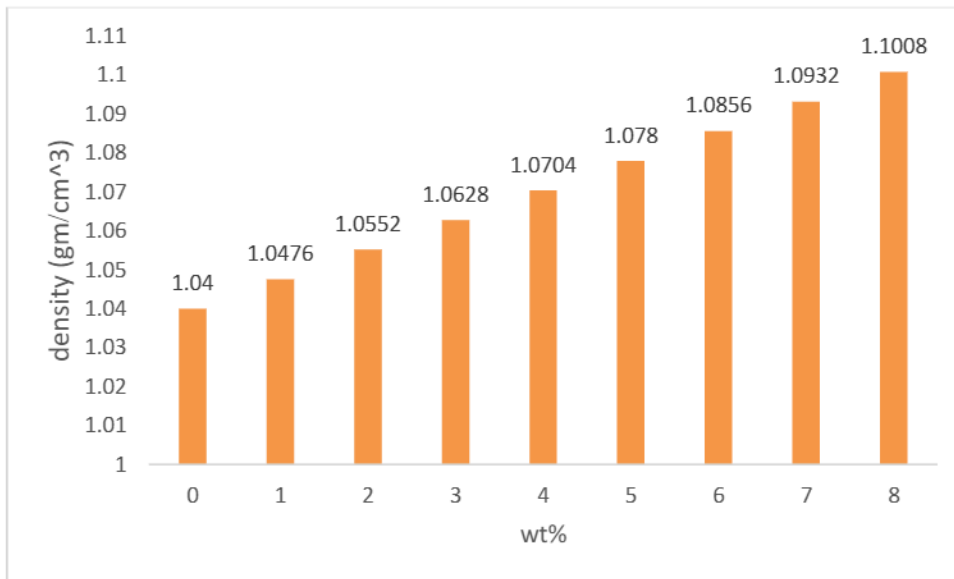
$$\rho = \rho_m w_m + \rho_r w_r \dots \dots \dots (3)$$

Where

$\rho_m$  And  $\rho_r$  : are the density of matrix and filler, respectively.

$w_m$  And  $w_r$ : are the weight fractions of matrix and filler, respectively.

**Figure (8)** shows that density of neat polyester increased from (1.04 gm / cm<sup>3</sup>) to a higher value (1.1gm/cm<sup>3</sup>) with the addition of the eggshell fibers to epoxy at a weight ratio of 8 %, these results agreed with the results (Sunny et al.)([13]).



**Figure 8.**densities values of polyester / micro eggshell composites

**3.2.2. Thermal Conductivity**

**Figure (9)** shows that the thermal conductivity value of polyester (0.4727 watt/m. °C) gradually decreases to (0.425 watt/ m. °C) and (0.407 watt/ m. °C) with the increased weight ratios of adding the eggshell powder to (4%) and (5%) respectively. The thermal conductivity increases to its highest level (0.4819 watt/ m. °C) with the addition (of 6%) of eggshell powder. This means that the polyester with small percentages of addition, like (4%) and (5%), have higher resistances against the



thermal transition. This is because of the excellent dispersion of eggshell particles in the polyester matrix [3].

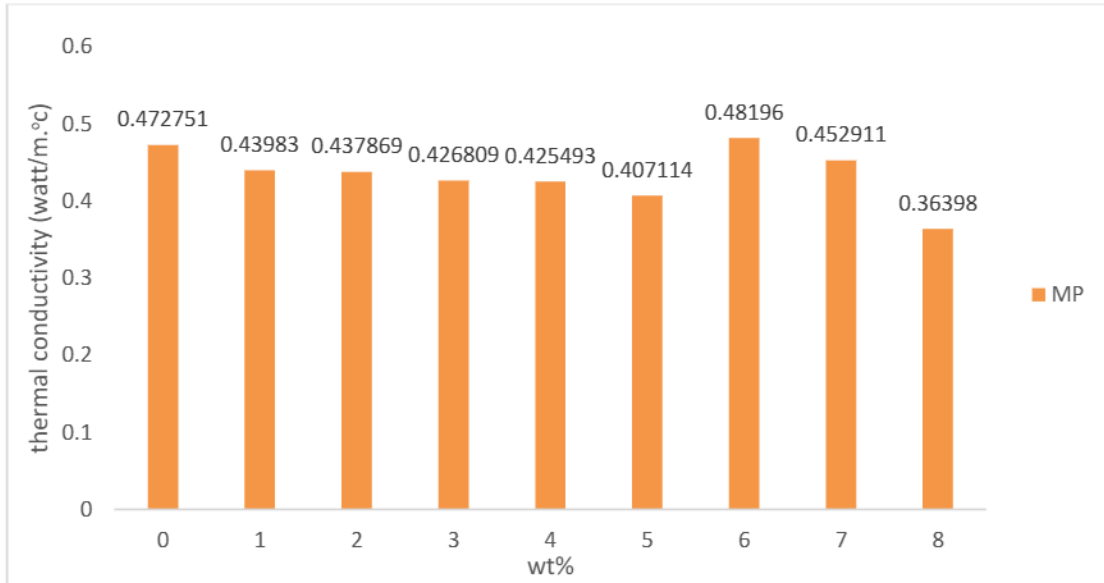


Figure 9. Thermal conductivity of polyester with different percentages of eggshell fibers

### 3.2.3. Water Diffusion

Figures (10-13) show the increase in water diffusion of (polyester/eggshell fiber) composites with an increase in the weight ratios of eggshell fibers. This is because of the osmotic process, where the water molecules are interred to the small voids and cavities of samples.

Figure (10) shows that the water diffusion of polyester with eggshells with ratios (of 4%) and (5%) (for one-week immersion in tap water), both composites have a higher resistance to water diffusion than the other addition ratios. Figures (11and 12) show the improvement in polyester resistance to water diffusion by adding eggshell by weight ratio (5%) with an increase in immersion time to ( 2 and 3) weeks, with an increase in the immersion time to (4 weeks) the eggshell ratio (4%) will improve the resistance of polyester against the water diffusion. This is because the capillary process occurs due to the diffusion of water molecules into the interface between the pumpkin fibers and epoxy matrix. These results agreed with ( Md .Milon et al.) [14].

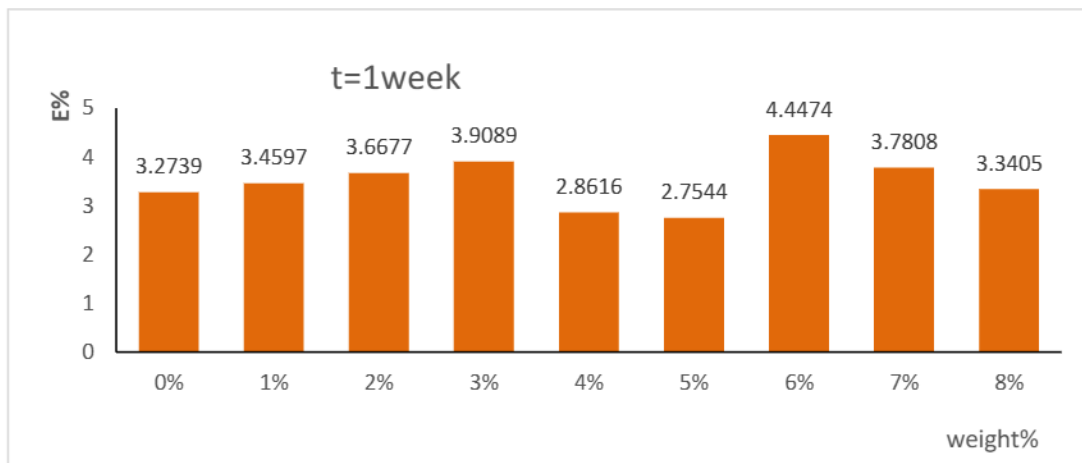


Figure 10. water diffusion of polyester with different percentages of eggshell fibers at ( 1 week)

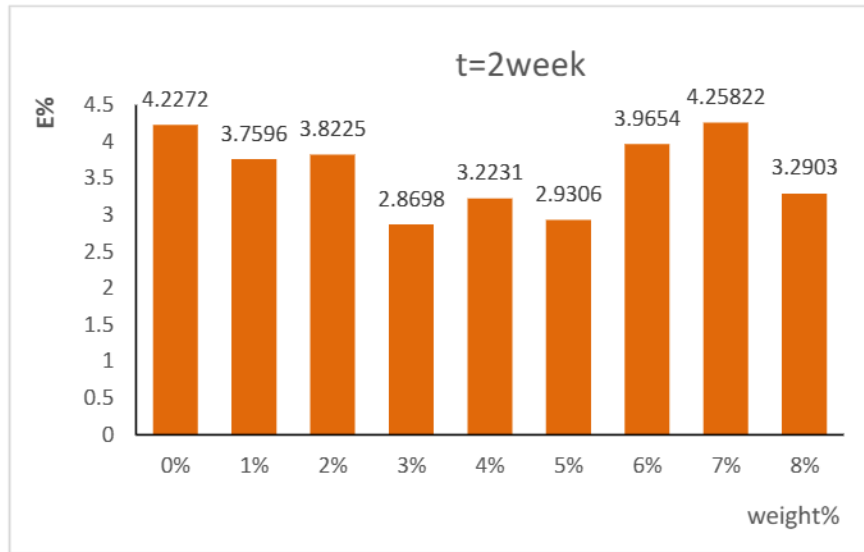


Figure 11. Water diffusion of polyester with different percentages of eggshell fibers at ( 2 week)

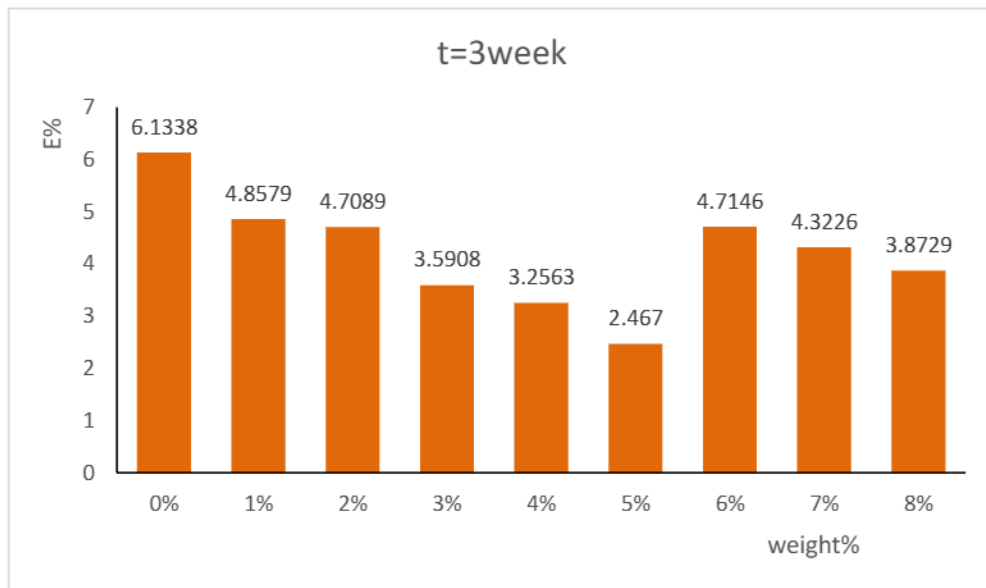
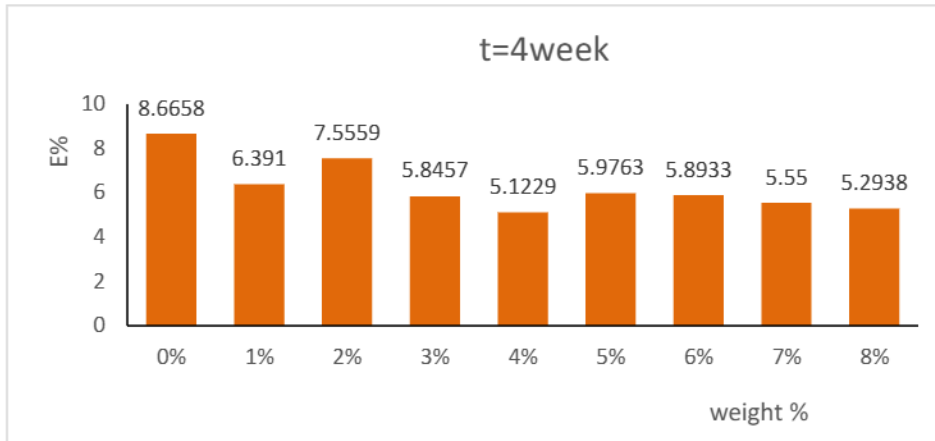


Figure 12. water diffusion of polyester with different percentages of eggshell fibers at ( 3 week)

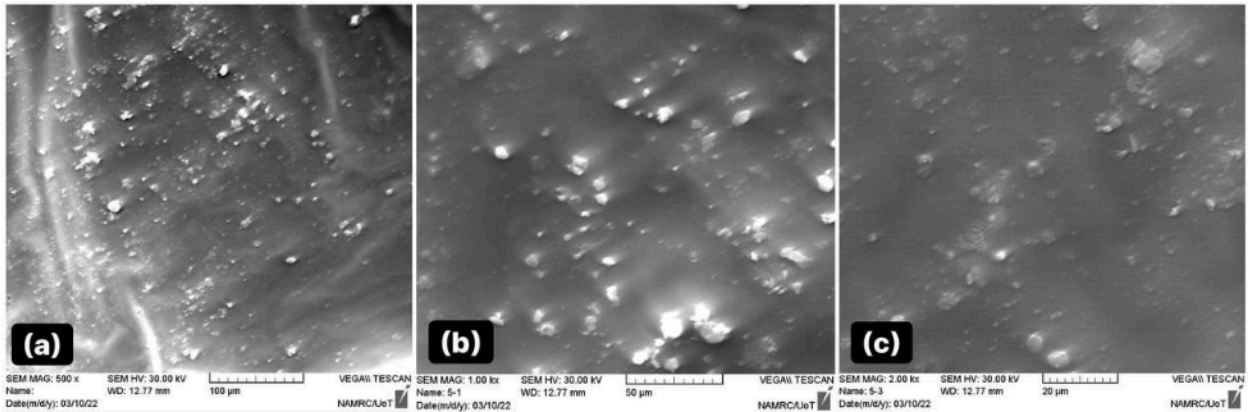


**Figure 13.** water diffusion of polyester with different percentages of eggshell fibers at ( 4 week)

#### 4. Scanning Electron Microscopy

There is a strong correlation between the exact composition of the polyester composite and its mechanical and thermal properties.

**Figure (14)** shows the polyester when adding eggshell powder by (1%) with the forces of magnification (500×, 1000×, and 2000×). The photographs indicate the homogeneous distribution of the powder within the material at this percentage of addition, which strengthens the polyester and improves its properties as a whole.



**Figure 9.** SEM images of polyester with (5%wt. ) eggshells with magnification (a)500 ×(b)1000 × (c)2000×

#### Conclusions

- 1- The density of polyester increased with the weight ratios of eggshell fibers.
- 2- Thermal insulation and impact strength of (polyester/5% E.S) was improved by a ratio (13.77%) and (~23%) compared with (polyester/4% E.S). Therefore, the (polyester/5% E.S ) composite was used to work in conditions that required thermal insulation while maintaining high impact strength, like painting the interior doors of homes.
- 3- for (the polyester/4 % E.S) composite, the resistance to wear and water diffusion (four weeks) was improved by a ratio (of 73.8%) and (14.29%) respectively, compared

with (polyester /5% E.S). Therefore, the (polyester/4 % E.S) composite was used as a kitchen floor coating.

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