# Preparation and Characterization of Eggshell Powder (ESP) and Study its Effect on Unsaturated Polyester Composites Material

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***Microcomposites of unsaturated polyester (UP) with different loading of eggshell powder of grain size (36μ) ranging from 0 to 25wt% were prepared by hand – layup method. The mechanical properties of the composites such as: tensile strength, young's modulus and impact strength were studied as a function of filler loading. All of these mechanical properties values were decreased with increasing the filler concentration. To improve adhesion between the filler and matrix some coupling agents have been used like: NaOH, silane and stearic acid. The results showed that tensile strength and young modulus for stearic acid treated filler increase with increasing of filler loading followed by a decrease beyond 15wt%. Silane treated filler showed similar results o fimpact strength compared to untreated system. Impact strength of all coupling agent systems were increased with increasing the filler concentration, while decrease for untreated system. Finally, stearic acid treated filler have higher values of impact strength as compared to the other composites.***

***Keywords***: Composite materials, Polyester, Eggshell powder, Mechanical properties

## 1. Introduction

An important attribute of polymers is the ability to modify their inherent physical properties by the addition of fillers while retaining their characteristic processing ease. Polymers can be colored, made stronger, stiffer, electronically conductive, magnetically permeable, flame retardant, harder, and more wear resistant by the incorporation of various additives. Most of these modifications are made by the addition of inorganic fillers to the polymer. These fillers, present in varying degrees, also affect the basic mechanical properties of the polymer. In many cases, the changes in the mechanical properties of the filled polymer can be predicted from basic principles. In other cases, the property changes must be experimentally measured, because there is not much sufficient knowledge about the polymer–filler interactions to calculate the effect of filler concentration on property changes [1]. Reinforcement depends on the transmission of mechanical energy from the polymer to the filler through the interface [2]. An increase in the strength of the bond between polymer and filler leads to an increase in the reinforcing of the polymer [3]. The introduction of filler into a resin creates an interface between two very dissimilar substances [4]. Water may accumulate at the interface, resulting in debonding of the filler thereby reducing the reinforcing action of the filler. Under a tensile force, the filler will "pull out rather than accept the stress [5]. Coupling agents contain chemical functional groups that can react with a given surface. Attachments to the surface can therefore made through covalent bonds. In addition, coupling agents contain at least one other functional group, which could react with the resin. The coupling agent may therefore acts as a bridge to bond a surface to a resin with a chain of primary bonds. This could be expected to lead to the strongest interfacial bond [6]. Unsaturated polyester resins are widely used commercial thermosetting resins which are used neat, with fillers and reinforced using fibers. Apart from the addition of a separate phase in the polymer matrix, the unsaturated resins have been modified by the use of various reactive diluents, reactants and by tailoring the polymer backbone to suit requirements of various applications [7]. Eggshell is a biomaterial containing 95% by weight of calcium carbonate in the form of calcite and 5% by weight of organic materials, such as ($Al_2O_3$, $SiO_2$, S, Cl, $Cr_2O_3$, MnO). The generalized eggshell structure, which varies widely among specie, is a protein matrix lined with mineral crystals, usually of a calcium compound such as calcium carbonate [8].

Water absorption and mechanical properties of high density polyethelene/eggshell composite were analyzed by Abdullah et al. [9]. Hassan S.B. investigated the development of polyester/eggshell particulate composites [10]. Siti studied LDPE-Isiphthalic acid modified eggshell powder composites [11]. Mechanism of interaction of eggshell microparticles with epoxy resin were studied by Genzhong et al. [12]. Properties of eggshell powder filled low density polyethylene/acrylonitrile b0utadiene styrene composites were analyzed by Mohammed [13]. Sivarao and Vijayaram studied the determination of tensile, flexural properties and microstructure

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characterization of calcium carbonate filler reinforced polypropylene matrix composites [14]. Evaluation of mechanical properties of polymide eggshell powder composite materials studied by Challa et al. [15]. Investigation on the mechanical properties of eggshell powder reinforced polymeric composites were analyzed by Asha and Chandra [16].

The main goal of the current research is to study the effect of chemical treatment of the eggshell powder on the mechanical properties of the unsaturated polyester composites for different volume fraction.

## 2. Experimental Work

The unsaturated polyester (UP) resin was used, the curing of (UP) was done at room temperature (25C°) by the incorporation of 2 volume percent methyl ethyl ketone peroxide (MEKP). A 1% (volume percent) cobalt naphenate was added as a catalyst to accelerate the hardening process (molecular formula $C_{22}H_{14}O_4Co$, density: 0.921g/ml, melting point: 140°C, boiling point >150°C). The eggshell powder was obtained from a local market.

The collected eggshells were washed thoroughly, initially in tap water and later in distilled water. The adhering membrane was separated manually and shells were dried in a furnace at 80°C for two hours. Later the shells were crushed using a domestic mixer. A sieve of 63µm was used to obtain the required average grain size. The powder was then analyzed using X-ray fluorescence spectrometer and the results are shown in table 1.The eggshell lpowder (ESP) was treated with three different solutions of 7% NaOH, 1% silane and stearic acid. The molecular formula of silane and stearic acid are $SiH_4$ and $C_{18}H_{36}O_2$, respectively.

**Table (1) Inorganic compounds in eggshell powder analyzed by X-ray fluorescence spectrometer**

| Elements | Concentration% |
|---|---|
| $Al_2O_3$ | 0.001 |
| $SiO_2$ | 0.002 |
| S | 0.001 |
| Cl | 0.008 |
| CaO | 99.82 |
| $Cr_2O_3$ | 0.0030.001 |
| MnO | 0.001 |
| CuO | 0.001 |
| LOI | 0.163 |

Note: LOI- loss of Ignition

Hardener and catalyst were added to unsaturated polyester for getting the sample as fast as possible. Contents were mixed very well to avoid bubbles; finally these contents were reinforced with eggshell powder with different weight fractions (5, 10, 15, 20 and 25%). The composites were cured at room temperature (25°C) until they were dried.

The tensile strength was carried out at room temperature by INSTRON tester (1195) testing machine with gage length of 32mm, applied load of 50kg with speed of 12.5mm/min. Measurements dimensions of tensile bar were: length 32 mm; width 7.8 mm and thickness 3.2 mm according to ASTM-D638.

The charpy impact test on unnotched specimens was determined using 5 J pendulum impact testing machine. The measurement dimensions of impact specimen were 55x10 mm according to ISO-179.

## 3. Results and Discussion

Tensile strength results for all compositions of UP/ESP composites were reported in Fig. (1). It can be seen that the tensile strength decreases with increasing filler loading for untreated, NaOH and silane treated ESP composites. The results could be described on the ability to receive a stretching force. The insertion of filler between molecular chains of polymer is effected to intermolecular force and loosely chain entanglement. These results are similar to report by Dangtungee [17]. As can be seen from Fig. (1), silane treated ESP showed similar results as compared to untreated system. Surface treatment is only one of the remedies for poor mechanical performance. It can therefore be concluded that the inclusion of silane coupling agent does not offer any additional advantage for ESP reinforced composites. Silane did, however, improve the tensile strength of ESP reinforced composites at lower filler loadings [18].

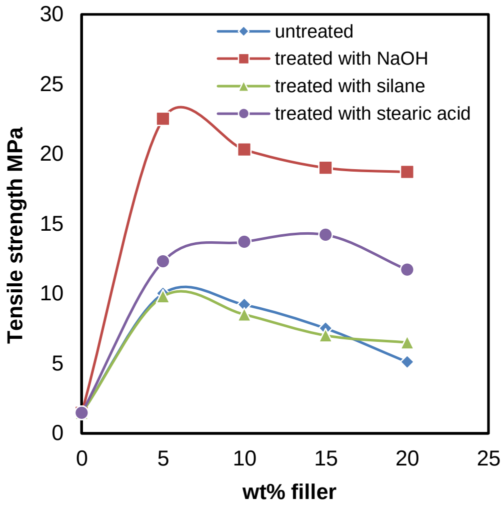


**Fig. (1) The tensile strength for treated and untreated eggshell powder filled UP composites with different filler loading**

The tensile strength for NaOH treated ESP composites is higher than for untreated system. This is probably because of a better interfacial adhesion between filler and matrix after chemical modification. Strong adhesion between filler and matrix interface can cause better stress transfer from the matrix to the filler leads to a higher tensile strength [19]. Also, from the same figure, it is observed that an increasing the filler loading of stearic acid treated ESP the tensile strength increases up to a certain value because the filler has reinforcing ability. The treatment improved substantially the extent of reinforcement. After attaining the maximum value (corresponding to 15wt %) the decline started. This decline is because of de-wetting effect, which has resulted from inadequate matrix material to hold filler particle [20].

Figure (2) shows the effect of filler loading on the young modulus of all composites. Young Modulus for the NaOH treated filler increases with the increasing filler loading. This is because at high filler loading, the composites will be able to withstand greater loads. This is behavior is similar to a result reported by Shuhadah et al [11].

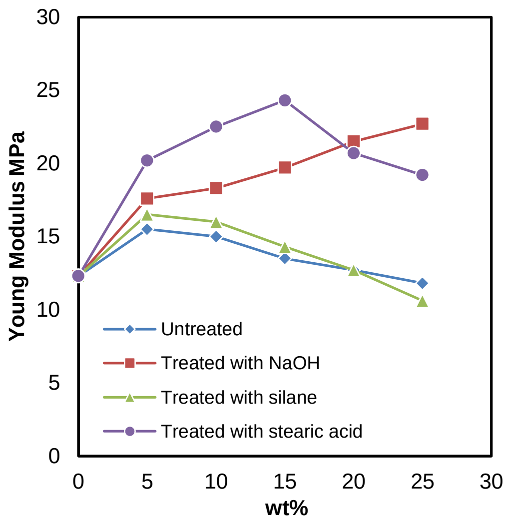


**Fig. (2) The Young modulus for treated and untreated eggshell powder filled UP composites with different filler loading**

In the stearic acid coupled system the modulus values showed an increasing trend up to 15wt% filler content followed by a decrease beyond 15wt%. For all the composites, the modulus values are higher than those containing no coupling agent. The stearic acid binds the inorganic material and the polymer matrix so that the reinforcement is enhanced. The increase in modulus is attributed to the various parameters of the filler added to the polymer. They include the aspect ratio of the filler, the orientation of the fillers, interfacial interaction and the nature of the failure. In these composites, there exists a matrix filler interaction, which enables the young modulus increased. The Young modulus values show an optimum value at 15wt% of the filler; this may be due to the saturation of the reinforcing action of the filler. After that, the coupling agent is not effective [1]. In the case of untreated and silane treated filler, the young modulus decreases with increasing filler content. According to Abdullah et al. [9], this decrement in young modulus can be referred to increase the resistance of material to deformation. The increased modulus corresponds to more filler where its intrinsic properties as a request agent exhibit high stiffness (modulus) compared to polymeric material. This is because at high filler loading, the composite will be able to withstand greater loads.

Figure (3) shows the impact strength as a function of filler content. It can be seen that the impact strength shows a decrease with increase of filler loading for untreated composites. Impact strength depends on the brittleness of the polymer matrix. The reason for the decrease in the property can be attributed to the poor adhesion or bonding at the interface between the matrix and the untreated filler [21]. Again by incorporating the coupling agents better filler/ matrix adhesion is achieved and the impact strength increased for all treated compositions. This agrees with conclusion of earlier work Asha and Chandra [16].

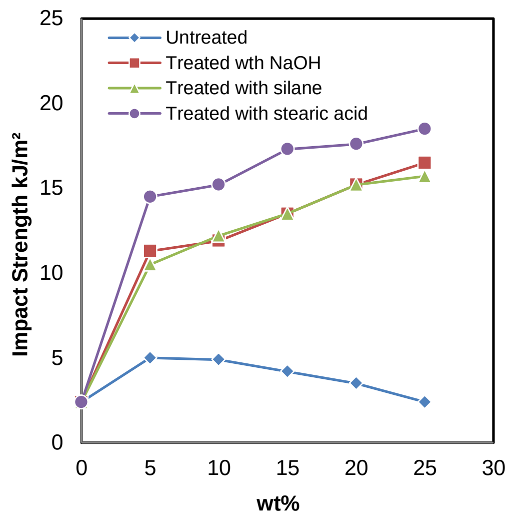


**Fig. (3) The impact strength for treated and untreated eggshell powder filled UP composites with different filler loading**

In the presence of coupling agents, the interfacial bonding between the matrix and the filler increase and thus facilitates better transfer of stress [11]. According to the same Figure we can see that stearic acid treated filler composites exhibited greater impact strength than other composites. Due to treating with stearic acid on the surface of the filler, tend to better dispersion and distribution of filler, the absorption of shock load or impact force could be greater [17].

## 4. Conclusion

Tensile strength of all composites except stearic acid treated ESP was reduced with increase in filler loading. Tensile strength and Young modulus for stearic acid treated ESP increase with increasing in filler loading followed by a decrease beyond 15wt%. Silane treated ESP showed similar results of tensile strength as compared to the non- surface treated system. Young modulus of untreated and silane treated ESP composites were reduced with the incorporation of filler, while increased for NaOH treated ESP composites. Impact strength of all coupling agent systems were increased with increasing filler content, and decreased for untreated system. Stearic acid treated ESP composites have higher values of impact strength as compared to other composites examined in this study. The tensile strength, young modulus and impact strength of untreated and treated composites are more as compared with pure unsaturated polyester.

## References


[1] Thomas, D., Kuruvilla, J. and Sabu, T, Mater. Lett., 58 (2004) 281-289.

[2] Lutz, J.T., **"Thermoplastic Polymer Additive"**, Marcel Dekker (NY) (1989).

[3] Ukanszyk, B.P., Composite, 1 (2004) 255-262.

[4] Plueddemann, E.P., **"Silane Coupling Agent"**, Plenum Press (NY) (1982).

[5] Kricheldorf, H.R., Nuyken, O. and Swift, G., **"Handbook of Polymer Synthesis"**, 2nd ed., Marcel Dekker (2005).

[6] Osman, M.A., Atallah and Suter, U.W., Polymer, 45 (2004) 1177-1183.

[7] Shenoy, M.A. and D'Melo, D.J., Express Polymer Lett., 1(9) (2007) 622-688.

[8] Shuhadah, S., Supri, M. and Kamarddin, H., Proc. MUCET, UniMAP, Kangar; Perlis; 15-16 (2008).

[9] A. Hussien et al. J. Basrah Res. (Sciences), 37(3A) (2011) 36-42.

[10] Hassan S.B. et al., Tribol. in Industry, 34(4) (2012) 217-225.

[11] Shuhadah, S. and Supri, A.G., J. Phys. Sci., 20(1) (2009) 87-98.

[12] Zhu, G. et al., J. Polymer Eng. and Sci., 49(7) (2009) 1383-1388.

[13] M.J. Al-Jumaily., “Properties of eggshell powder filled low density polyethelene/acrylonitrile butadiene styrene composites”, Thesis, Materials Engineering University, Perlis, Malaysia (2009).

[14] Sivarao, V.T.R., Trans. Indian Inst. of Metals, 26(3) (2009) 245-248.

[15] Challa, R., Vamsi, M. and Sreenivasulu, The Int. J. Sci. Technol., 2(11) (2014) 90-95.

[16] Asha A. and Chandra V., Int. J. Eng. Res. Technol. (IJERT), 3(12) (2014) 288-291.

[17] Dangtungee, R. and Shawaphun, S., J. KMUTNB, 18(3) (2008) 9-15.

[18] Nabil, A.N., Hundiwale, D.G. and Kapadi, U.R., J. Sci. Indust. Res., 36 (2004) 603-609.

[19] Kwan, S. et al., Polymer, 43 (2002) 6901-6909.

[20] Arencon, D. and Ignacio, J., J. Mater., 2 (2009) 2046-2049.

[21] Liang, J.Z., J. Thermoplast. Composite Mater., 19 (2006) 703-713.


 