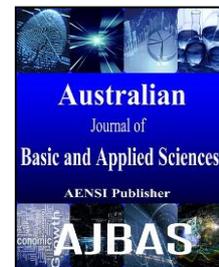




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### The Effect of Filler loading on the Structure and Properties of Polyvinylchloride/Chicken Eggshell powder (PVC/ESP) Composites

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#### ABSTRACT

This paper investigated the effect of filler loading on the structure and properties of polyvinylchloride/chicken eggshell powder composite (PVC/ESP). The compounding of composites were done using Rheomix mixture internal mixer at 180°C and rotor speed of 30 rpm for 10 minutes to allow the mixing torque to reach a steady state. The mechanical and morphological properties of PVC/ESP composites under different filler loading have been characterized by a light weight tensile tester and scanning electron microscopy (SEM). As the filler loading increases, the tensile strength and elongation at break decreases where as the tensile modulus increases. The tensile modulus for PVC/ESP composites prepared with 10% filler loading exhibit lower stiffness compare to PVC/ESP composites prepared with 40% filler loading. The addition of ESP filler has increases the temperature at which the decomposition of the composite starts under thermogravimetric analysis (TGA). In addition, higher filler loading has much higher thermal stability compare to lower filler loading.

#### INTRODUCTION

An assortment of polymer types which are used broadly in today's industry are plastics, elastomers or rubbers, fibers, coatings, adhesives, foams and films. A specific polymer can be used for more than one application type depending on its properties. PVC (poly (vinyl chloride)) is one of the polymer types which can be use in multiple application due to its nature properties. The major characteristics of PVC are that it can be obtained at low cost, versatile, high stiffness, flame retardancy and chemical resistance (Tongamp *et al.*, 2008). Processing PVC in the raw form using heat and pressure; result in severe degradation of the polymer. In addition, PVC has low impact strength and low heat resistance due to its rigid characteristic. This has limited PVC application in industry. The flexibility, ductility and toughness of polymers may be improved with the aid of additives. There are several additives that can be used such as fillers, plasticizers and stabilizers. Plasticizers are generally liquids having low vapour pressures and low molecular weights. Plasticizers are commonly used in polymers that are brittle at room temperature such as PVC. Effect of plasticizer on the mechanical properties of the polymer is governed by the action of the plasticizer itself on the molecules of the polymers, which is believed to act as spacer between molecules of the polymers by forming links with polymer molecules. The formed links weaken the bond forces of the polymer atoms and thus addition of the plasticizer to the polymer increases the free volume which leads to lowering the hardness, modulus of elasticity and tensile strength

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(Saunders, 1973). In addition, high level of plasticization results in the plasticizer leaching can be a serious health risk (Gibbons and Kusy, 1996). To overcome the plasticizer problem, this research will be focusing in the usage of filler additives.

Filler additives are most often added to polymers to improve tensile and compressive strengths, abrasion resistance, toughness, dimensional stability, thermal stabilities and other properties. Polymers that contain fillers may also be classified as composite materials. It requires high loading amount to achieve a significant improved performance. To overcome the drawback, a new class of reinforced thermoplastics has been extensively investigated using fillers in nanometer size range preferably less than 100nm (Chrissafis *et al.*, 2011). Properties of thermoplastics which have shown substantial improvements due to the incorporation of nanocomposites include mechanical properties, thermal stability and heat distortion temperature, flame retardancy, chemical resistance, surface appearance, electrical and thermal conductivity and etc.

Previous study shows that by varying the filler content in compounding it affects the mechanical properties, morphology structure and thermal degradation of the compounded material. Ofem and Umar (2012) investigated the effect of filler content on the mechanical properties of periwinkle shell reinforced CNSL resin composites. Compression molding technique was used to develop the composite resin with varying filler content (10% to 40%). They reported that as the filler contents increases the mechanical properties such as tensile strength, flexural strength, percentage of elongation at break, tensile modulus and impact strength shows slight increment. The optimum properties were observed at 30% filler loading. On the other hand, Sutapun *et al.* (2013) investigated the application of calcined eggshell powder as functional filler for high density polyethylene (HDPE). The compounding of HDPE/calcined eggshell powder composite was prepared via 10-40% filler content. The result shows that Young's modulus and ultimate stress of the filled HDPE increased with increasing filler content while thermal decomposition temperature and yield strength of the filled HDPE were not significantly influenced by the addition of the filler. Farahana *et al.* (2015) studied the impact of ESP filler loading on the tensile and water absorption properties of recycled high density polyethylene. The ESP filler loading was varied from 0 to 25 phr. The results show that as the ESP filler loading increases, the tensile modulus and water absorption increases but decreases the tensile strength and the elongation at break. Hassan and Aigbodion (2015) investigated the effects of eggshell on the microstructures and properties of Al-Cu-Mg/eggshell particulate composites. A total of 2 – 12 wt% ESP were added. Through addition of ESP into the compound, it shows a tremendous improvement in the strength and hardness properties but decreases the density and impact properties. Chai Ai Bao *et al.* (2016) examined the effect of oil palm fiber/eggshell powder loading on the mechanical properties of natural rubber composites. They fabricated natural rubber composite with 5 different loading. The result reflects that as the ESP filler loading increases it also increases the tensile strength and elongation at break but decreases the hardness and hysteresis loss properties.

In this paper, the effects of filler loading in compounding of PVC/ESP composite were studied. To evaluate the effect of filler loading, mechanical properties, thermal decomposition and morphology of PVC/ESP composites prepared through different filler loading were compared.

### **Experimental Methods:**

#### **Materials:**

PVC resin (the K value is in the range of 60-67) and dioctylphthalate (DOP) with >99% purity was purchased from Chem Soln C6388. Stearic acid, AR grade, were obtained from Syntrec Enterprise. Dibutyltin maleate (act as stabilizer during compounding) was obtained from Medigne Sdn Bhd. The chicken eggshell is collected from Universiti Tunku Abdul Rahman (UTAR) cafeteria.

#### **Preparation of chicken eggshell powder:**

The collected chicken eggshell was washed several times under the tap water. During the washing process the chicken eggshell membrane is removed. Then the washed chicken eggshell without the membrane was blended using Waring laboratory blender HGB 2WTS3 to smaller pieces. The blended chicken eggshell was then dried in hot air oven at 80°C for 24 hours. The dried chicken eggshell was grounded to powder form using the Retsch grinder ZM200 at 6000rpm speed. The chicken eggshell powder which passed 45 mesh sieve using sieve vibrating machine RX-29-10-220V was used in the study. The chicken eggshell powder was dried under a vacuum oven at 80°C for 24 hours to eliminate moisture before compounding process in internal mixer.

#### **PVC/ESP composite compounding:**

The PVC/ESP composites were prepared using Rheomix mixture internal mixer model Brabender 815652. The compounding of PVC/ESP was carried by missing the entire ingredient in Table 1 at 180°C and rotor speed of 30 rpm for 10 minutes to allow the mixing torque to reach a steady state. The blending was carried out by mixing PVC, stearic acid and dibutyltin maleate first in a beaker for 10 minutes. Then this mixture was discharge into the mixing chamber of the internal mixer. In another beaker a mixture of ESP and plasticizer was

prepared and after 4 minutes this mixture was added into the internal mixer chamber spoon by spoon. The mixing continued for another 6 minutes until a steady state of mixing torque is obtained.

**Table 1:** Ingredient of PVC/ESP compounding

Ingredients	Formulation (phr)
PVC	100
DOP	30
Stearic Acid	3.0
Dibutyltin Maleate	0.3
ESP	0, 10, 20, 30 and 40

The sample of the compounded composite is then molded using electrically heated hydraulic hot and cold press GT-7014-A30C. Hot press procedure involved 3 stages; preheating, compress and cooling. Preheating is done at 180°C for 7 minutes, followed by compress for 2 minutes under the same temperature subsequent cooling under pressure for 2 minutes.

#### **Tensile test:**

The tensile properties were determined using the Light weight tensile tester H10KS according to the ASTM D638 standard. The specimen used for testing is cut into dumbbell shape. A crosshead speed of 50mm/min was used. Average of 5 samples was used during the test to minimize the error. Tensile tests were conducted at ambient temperature 20°C to 25°C. Tensile strength, elongation at break and modulus results had been recorded from the test.

#### **Morphology study:**

The morphology of the tensile fracture surface of PVC/ESP composites was studied using field emission scanning electron microscopy (FESEM) at a magnification of 500x. for scanning electron interpretation. The samples of the tensile fracture surface were coated with a thin layer of platinum before proceeding to the SEM to enhance the conductivity.

#### **Thermogravimetric Analysis (TGA):**

The thermal decomposition characteristics of the PVC/ESP composites were studied using thermogravimetric analyzer. The weight loss against temperature was measured at a heating rate of 10°C/min in dynamic nitrogen atmosphere with the flow rate of 20ml/min from 25 to 900°C. The sample mass used should be in between 6 to 10mg.

#### **Results:**

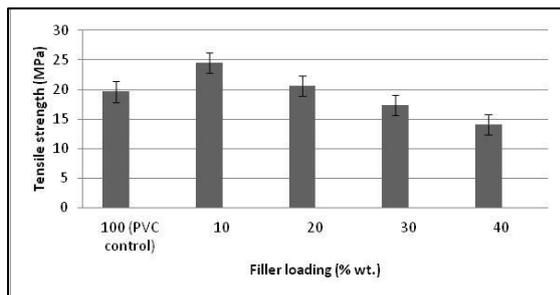
##### **Mechanical Properties:**

Fig. 1 illustrates the effect of filler loading on the tensile strength of PVC/ESP composites. As shown in the diagram, as the filler loading increases the tensile strength decreases. Similar results were reported by Deshmukh *et al.* (2010), Abdullah *et al.* (2011) and Rohani *et al.* (2014). Deshmukh *et al.* (2010) studies the effect of particle size and concentration on mechanical and electrical properties of the mica filled PVC. They reported that as the mica loading increases, stiffness and Young's modulus of the PVC mica composites increases while the elongation at break and tensile strength decreases. On the other hand, Abdullah *et al.* (2011) uses high-density polyethylene as their matrix resin and eggshell as their filler to study the effect of water absorption and mechanical properties on high-density polyethylene/ eggshell (HDPE/ESP) composite. They use 5 different filler loading to compound HDPE/ESP composite. The result shows that as the filler loading increases, it decreases the tensile strength, modulus of elasticity, shore-D hardness whereas increases the elongation at break and impact strength. The amount of water absorbed increases as the filler loading increases under constant exposure time. Rohani *et al.* (2014) studies the effect of polyethylene grafted maleic anhydride on the mechanical, morphological and swelling properties of poly (vinyl chloride)/epoxidized natural rubber/kenaf core powder composites. The research was carried out under four different filler loading. The results shows that as the filler loading increases the tensile strength and elongation at break decreases whereas Young's modulus increases. This is due to the fact that as the filler loading increases, the filler tend to agglomerate which increases the resistance of dispersion. Therefore at higher filler loading the filler does not dispersed well in the matrix resin and indirectly decreases the interfacial adhesion between the filler and matrix resin. As the adhesion bond between the filler and matrix is weak, the bond will be easier to break when load is applied on it. Weak adhesion bonding is caused by the agglomeration of filler particles. Therefore at higher filler loading, the tensile strength decreases. The optimum filler loading to obtain the best tensile strength is at 10% filler loading of PVC/ESP composite.

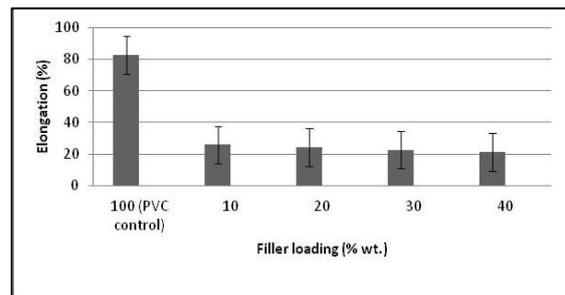
Fig. 2 illustrates the elongation at break of PVC/ESP composites under different filler loading. From the diagram, it shows that as the filler loading increases the elongation at break decreases for PVC/ESP composites.

In addition the elongation at break for pure PVC matrix resin is higher compare to PVC/ESP composites. PVC/ESP composite exhibit better dispersion of filler in matrix resin which improve the adhesion bonding between the filler and matrix resin. In addition it enhances the stress transfer from the matrix to the filler phase. But at higher filler loading, the elongation at break decreases due to the fact that stress transmission from the matrix to the filler was disturbed because of poor dispersion of filler and also due to filler irregular structure.

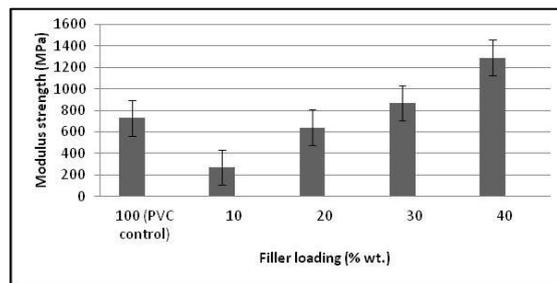
Fig. 3 represents the tensile modulus of PVC/ESP composites under different filler loading. The diagram illustrates an increasing trend of tensile modulus of PVC/ESP composites as the filler loading increases. The tensile modulus for pure PVC matrix resin exhibit higher value when compare it with 10% and 20% filler loading. The optimum result of tensile modulus is achieved at 30% and 40% filler loading. As the filler loading increases, the stiffness of the PVC/ESP composites increases hence it exhibit higher tensile modulus at higher filler loading. More ESP particle are dispersed in PVC matrix resin at higher filler loading compare to lower filler loading even though with the presence of agglomeration of ESP filler. Hence, dispersion of ESP rigid filler particle into the matrix resin has enhanced the stiffness of the PVC/ESP composites. At higher filler loading, PVC/ESP composites can withstand higher load.



**Fig. 1:** Tensile strength of PVC/ESP composites



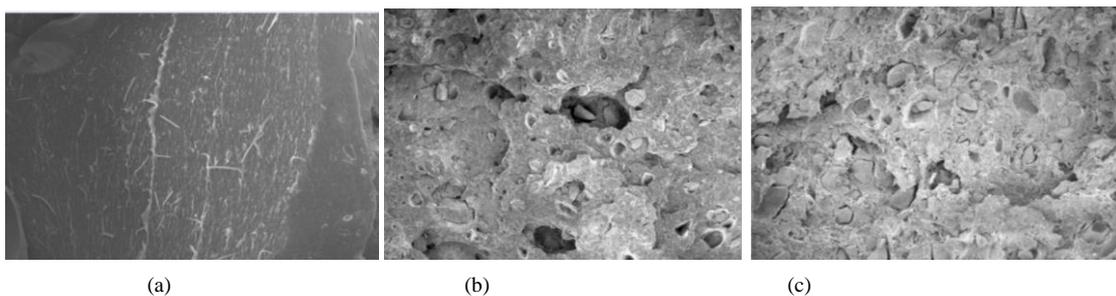
**Fig. 2:** Elongation at break of PVC/ESP composites



**Fig. 3:** Tensile modulus of PVC/ESP composites.

#### Morphology Studies:

Fig. 4 illustrates the tensile fracture micrograph of the PVC/ESP composites. Fig. 4(a) represents the tensile fracture micrograph of the PVC pristine. It is observed that the fracture surface morphology for PVC pristine is smooth. Fig. 4(b) illustrates the tensile fracture micrograph of the PVC/ESP with 10% filler loading. As can be seen from Fig. 4(b) many holes are present on the surface fracture of PVC/ESP composites. This is due to the detachment of filler from the matrix surface and perhaps voids have been introduced during processing. Fig. 4(c) illustrates the tensile fracture micrograph of the PVC/ESP composite with 40% filler loading. It is observed from Fig. 4(c), the filler are well distributed and not many holes are present on the surface of PVC/ESP composite. This supported the higher tensile modulus results for composite with 40% filler loading than 10% filler loading.



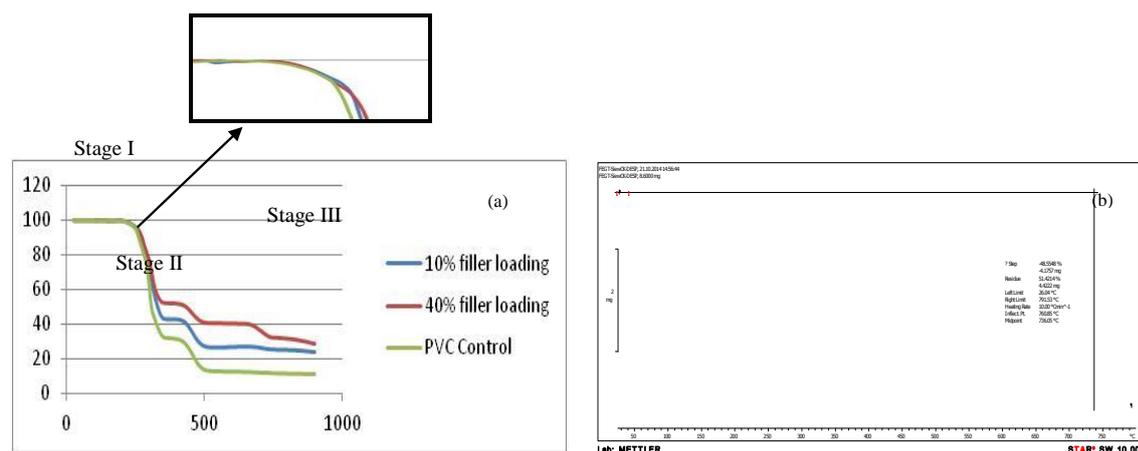
**Fig. 4:** SEM micrographs (500x magnification) tensile fracture surfaces of PVC/ESP composites.

(a) PVC pristine; (b) PVC/ESP with 10% filler loading; and (c) PVC/ESP with 40% filler loading.

### Thermogravimetric Analysis:

Fig. 5(a) shows the result of TGA for PVC pristine and PVC/ESP composites with different filler loading while Fig. 5(b) shows the result of TGA for 100% ESP. As shown in Fig. 5(a), the PVC pristine and PVC/ESP composites with 10% filler loading experience two weight loss stages (I and II) where as PVC/ESP composites with 40% filler loading experience three weight loss stages (I, II and III). The first stage (Stage I) weight decomposition starts at about 195°C and ends around 330°C. The weight loss of PVC pristine and PVC/ESP composites is about an average of 65% during the first decomposition. As the temperature increased, it causes the polymer chain of PVC to be weak and loosed. Therefore, it leads to break off of C-Cl bond from H-Cl covalent bond (Freire and Holanda, 2006). The sample is said to be thermally stable where it experiences no weight lost at the temperature between 350°C and 425°C. From Fig. 5(a), it can be seen that PVC/ESP polymer composites are more heat stable compare to pure pristine PVC. The second stage (Stage II) weight decomposition starts at about 425°C and ends around 500°C. Due to the increase of heat, the covalent bond of the new polymer formed starts to break. Above 520°C, a stable residue is formed that corresponds to carbon black for pure PVC and some PVC/ESP composite. At higher filler loading it is observed that, the polymer composite will undergo third stage decomposition which takes place between 670°C and 750°C. Above 750°C, a stable residue is formed that corresponds to carbon black for higher filler loading. On the other hand, in Fig. 5 (b) the ESP sample was thermally stable until 545°C. The first decomposition stage at (~65°C) is attributed to the removal of physically adsorbed water on the particles of the waste powder. The second decomposition stage (~544.73°C) is related to decomposition of organic matter. The third decomposition stage (~699.14°C) was caused by decomposition of calcium.

Also from the graph, it can be summarized that the thermal stability of PVC/ESP composites is much stable comparable pure PVC. The addition of ESP filler has increases the temperature at which the decomposition of the composite starts. In addition, higher filler loading has much higher thermal stability compare to lower filler loading.



**Fig. 5:** TGA of (a) pure PVC and PVC/ESP composite; and (b) 100% ESP.

### Conclusion:

In this paper, the effect of filler loading on the structure and properties of PVC/ESP composites were investigated using light weight tensile tester, SEM and TGA. The results obtain from light weight tensile tester shows that as the filler loading of the PVC/ESP composite increases, the tensile strength and elongation at break decreases whereas the tensile modulus increases. The PVC/ESP composites prepared with 10% filler loading exhibits better strength and elongation at break compared to PVC/ESP composites prepared with 40% filler loading. The tensile modulus for PVC/ESP composites prepared with 10% filler loading exhibit lower stiffness compare to PVC/ESP composites prepared with 40% filler loading. These results are further supported by FESEM images which shows better dispersion of filler in composites prepared with 40% filler loading compare to composite prepared with 10% filler loading. The addition of ESP filler has increases the temperature at which the decomposition of the composite starts. In addition, higher filler loading has much higher thermal stability compare to lower filler loading.

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