Characterization of Vinyl Ester Composites Filled with Carbonized *Jatropha* seed shell: effect of accelerated weathering

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**Abstract.** The effect of accelerated weathering test of carbonized *jatropha* seed shell filled vinyl ester biocomposites was investigated. In this study, four loading of carbonized *jatropha* seed shell and one without loading of vinyl ester biocomposites were used. The samples exposure at several circles time in QUV chamber. The durability of vinyl ester biocomposites filled carbonized *jatropha* seed shell changes in mechanical properties and weight loss during exposure in UV and condensation. The tensile test and flexural indicated decrease with increasing of carbonized *jatropha* seed shell loading. The SEM fracture surface of biocomposites looks rough and some carbonized out of the matrix.

1. **Introduction**

Although inorganic fillers currently dominated the polymer industry, biomass filler have become more accepted in recent years and have made significant in roads into specialty market. This material applications continues to increase as in the construction, transportation, industrial and consumer applications. In other words, this material can be improved strength and resistance to environmental [1]. Loading of biomass in material composites will effect on the strengthening and flexibility of the materials [2]. Durability of environment is an important aspect of overall performance of material biocomposites used in both interior and exterior application where sunlight and condensation could have detrimental effects. The fact that interaction between biomass filler and matrix is not only important for the the properties of the composites but also for conserving the integrity of the biocomposites by maximizing filler-matrix adhesion [3]. Accelerated weathering for composites are the way of exposure testing that attempt to rapidly occur the changes material composites on outdoor and indoor exposure equipment [4]. Accelerated weathering for biocomposites are necessary to ensure the materials and products meet their expected functionality and lifetime. The strength and flexibility of the composite materials useful for applications outdoors. Because of they will be influence on the weather and the material composites to be easily damaged [5]. The effect of lighting from an artificial light source and this may be attended by water spraying and circular experimental conditions, because of changes in temperature, relative humidity, and irradiation energy. The degradation processes of materials composites by exposing to artificially intensive weathering condition, allowing a fast
evaluation of the service durability of material composites in a rationally short period of time. In this study, accelerated weathering for carbonized *jatropha* seed shell filled vinyl ester composites has been investigated durability of biocomposites with the effect of UV exposure and condensation. Accelerated weathering for carbonized *jatropha* seed shell filled vinyl ester has been studied for carbonized particle loading. The analysis for this study includes change in physical and mechanical properties and morphology of vinyl ester composites.

2. Experimental

Biomass filler was prepared from *jatropha* seed shell obtained from Aceh Province, Indonesia. The size of biomass filler after carbonization is > 300 mesh. Vinyl ester resin with 42% styrene monomer content, methyl ethyl ketone peroxide (MEKP), and cobalt napthenate were purchased from Zarm Scientific & Supplies Sdn. Bhd. Malaysia.

2.1. Accelerated weathering processes

The weathering accelerated chamber model MEGASOL was used in this study (James H. Heal & Co. Ltd, Halifax England) with burner life 1500 hours, megasol light & weathering fastness tester. Vinyl ester composites were prepared following the previous method [2]. The composites were in size 160 x 20 mm samples for flexural test and 120 x 15 mm for tensile test were exposed in UV cabinet for 96, 240, 360 and 720 h using a cycle of 2-h UV exposure followed by 30 minutes condensation at 29°C and humidity 32% RH, which is cycle recommended in ASTM D 4329-1999. After exposure, the panels were allowed to equilibrate back to ambient condition and reweighed. Analysis for flexural, tensile and morphology follow the mechanical and morphology analysis for vinyl ester composites below.

2.2. Mechanical Properties

Mechanical properties include tensile strength and flexural properties of biocomposites were performed using the INSTRON 5582 Universal Testing Machine (USA). The size of biocomposites sample by dimensions 120 mm x 20 mm x 7 mm (ASTM D638). Tensile properties including tensile strength, tensile modulus were acquired from the recorded data. Five specimens were tested and the average value was tabulated. ASTM D790 standard method were followed for flexural properties including flexural strength and tensile modulus and crosshead speed was 2 mm/min [1].

2.3. Scanning electron microscopy

Scanning electron microscopy (SEM) (model EVO MA10, Carl-ZEISS SMT, Germany) was used for tensile fracture surface morphology of biocomposites. The SEM accelerating voltage is 20 KV. To avoid electrostatic charge, the specimens of fractured surface were mounted on aluminum stubs and sputter coated with thin layer of palladium and gold.

3. Results and discussion

3.1 Weight change before and after weathering

Figure 1 shows the weight changes of carbonized *jatropha* seed shell before and after accelerated weathering. All of the composites would have small changes in their weight after exposure for 96 hours. The higher weight loss after exposure for 360 hours in weathering chamber were composites with carbonized particle loading 40%, the change is about 0.25%. After 360 hours exposure to UV and condensation condition, all carbonized vinyl ester composites had lost weight below 1%. The temperature of the surface of the specimens exposed, increased step by step from normal levels to be used when performing the test temperature by increasing weathering (room temperature), this cause weight loss from the composites, although UV exposure followed condensation for the composite [6].
3.2 Tensile properties for weathering test

The change of tensile strength for carbonized *jatropha* seed shell filled in vinyl ester biocomposites compared to neat vinyl ester were illustrated in Figure 2a. Neat vinyl ester increase on tensile strength after weathering for 96 hours but slightly change when exposed for 240 hours and 360 hours. But, for carbonized *jatropha* seed shell filled vinyl ester biocomposites, the tensile strength slightly decreases after weathering is accelerated.

![Figure 1. Weight change of carbonized *jatropha* seed shell filled vinyl ester composite](image1)

![Figure 2. Change of tensile strength (a) and Tensile modulus (b) before and after accelerated weathering](image2)

The change of tensile strength of the carbonized *jatropha* seed shell filled vinyl ester composites are about 0.01 to 0.15% at carbonized particle loading from 0 to 40% respectively to vinyl ester matrix. The higher carbonized particle loading, the larger the losses in tensile strength during exposure to weathering. With increasing weathering time, the tensile strength of carbonized *jatropha* seed shell filled vinyl ester composites continuously lost their strength. It is decreases because of embrittlement of the vinyl ester matrix and could be because of the degradation of biofiller-matrix interfacial bonds [7]. Figure 2b show the tensile modulus of neat vinyl ester composite and carbonized *jatropha* seed shell filled vinyl ester composites. Tensile modulus also decreases after UV and condentation condition for all vinyl ester composites. Losses in tensile modulus 0.06% for neat vinyl ester and 0.12 to 0.22% for carbonized particle loading in the vinyl ester matrix composites with increasing weathering time. This suggests that the decrease in molecular weight vinyl ester matrix-chain reaction is reduced due to the scission induced by photo-oxidation from UV radiation [8].

Karbhari et al. (2003) [9] implied that degradation on the surface components polymeric which have been shown to affect mechanical properties are disproportionate; as deficiency resulting from ultraviolet radiation can be compounded by the action of temperature, freeze-thaw, wind-borne abrasives, moisture, and other environmental components. However, with UV exposure, carbonized *jatropha* seed shell filled in vinyl ester matrix can damage, e.g. tensile and flexural strength. According to Maxwell et al. (2005) [10], UV radiation is known to degrade the polymer materials,
although only the outer layer is likely to be affected. Signs of photo-degradation include embrittlement (cracking of the surface), changes in color, and the loss of transparency. These factors caused the decrease in mechanical properties of materials.

### 3.3 Flexural properties for weathering test

Figure 3 show the change of flexural properties of carbonized *jatropha* seed shell filled vinyl ester composites. Flexural strength (Figure 3a) of vinyl ester composites, decrease after exposure to UV radiation and condensation with water spray with addition of weathering time. The losses of flexural strength is 0.02% for neat vinyl ester and 0.07 to 0.27% for carbonized particle loading in vinyl ester composites. The loss in strength was likely due to moisture penetration into the matrix, which degraded the filler–polymer interface. This decreases the stress transfer efficiency from matrix [11]. At high temperature, the rate of moisture absorption and accelerated degradation properties of materials were increase [10] and [4].

![Figure 3](image)

**Figure 3.** Change of flexural strength (a) and flexural modulus (b) before and after weathering accelerated

After exposure to UV radiation for 2h followed by condensation for 30 minute, the flexural modulus (Figure 3b) of vinyl ester composite will decrease. The degradation was most hard for composites exposed to condensation and cyclic UV radiation. After 360 hours of cyclic exposure the transverse flexural modulus was decreased by 0.09% for neat vinyl ester composite, 0.12 to 0.29% for carbonized particle loading in vinyl ester composites. The influence of temperature on the radiation aging effect of materials investigated for several polymer materials for technical applications and for being used as reference material in the accelerated weathering tests. The higher carbonized particle loading after exposure to weathering test makes composites ductility more than small carbonized particle loading. The mechanical properties decrease is due to the embrittlement of the polymeric matrix and to degradation of fiber-matrix interfacial bonds [7].

### 3.4. Scanning electron microscopy

Figure 4 and 5 show the SEM image of neat vinyl ester and carbonized *jatropha* seed shell filled vinyl ester composites before and after exposure to accelerated weathering chamber. From the Figure 4(a) and 4(b), the SEM image of vinyl ester composite exhibits a regular and smooth fracture surface with no significant defects. After exposure for 360 hours, the surface looks rough and smooth less (Figure 4b and Figure 5b). Degradation of composite fracture surface was less distinguishable after UV radiation and condensation to 360 hours and the carbonized filler already defects on vinyl ester matrix (Figure 5b). This could mean that it is still in the early stages of degradation by photo-oxidation of the accelerated. Accelerated weathering causes the formation of surface defects on matrix of vinyl ester, and some white particles can be observed due to the formation of carbonized particle out of the matrix [12], which allows further degradation (including biodegradation) in the environment [7]. According to Al-Shabanat (2011) [13], it is obvious that there are cracks and fissures and micro can be seen on the surface of a composite of degraded greatly increased after exposure and some of the particles can be observed which might be caused by a large number of fragments of the fracture surface [14]. This entire surface damage from UV exposure during spontaneous as a result the surface layer of composites, which leads to shrinkage [5]. Although cracks on surface are clear in all composites after
being accelerated weathered for 360 hours, the severity of cracks varies from one another where the
damage was done by weathering on matrix vinyl ester contained carbonized particle filler of *jatropha*
seed shell.

![Figure 4](image1.png)

**Figure 4.** SEM image of neat vinyl ester composite: (a) before and (b) after exposure 360 hours

![Figure 5](image2.png)

**Figure 5.** SEM image carbonized *jatropha* seed shell filled vinyl ester composite: (a) before and (b) after exposure 360 hours

4. Conclusions
The weight change of carbonized *jatropha* seed shell occurred after exposure in weathering chamber.
Losing weight is happening this because surface temperature specimens affected increases step by
step than the normal rate used when doing test weathering by increasing temperature. The mechanical
properties of carbonized *jatropha* seed shell filled vinyl ester biocomposites decrease when subjected
in UV light and condensation by exposure time. When ultraviolet radiation, the biocomposites can
compound by the action of temperature, moisture, wind-borne abrasives, freeze-thaw, and other
environmental components. The higher carbonized particles loading after exposure in weathering test
make composites ductility. The mechanical properties decrease is due to embrittlement of the
polymeric matrix and to degradation of fiber-matrix interfacial bonds. The degradation of the
composite fracture surface was less distinguishable after UV radiation and condensation. Accelerated
weathering causes the formation of surface defects on matrix of vinyl ester, some white particles being
observed due to formation of carbonized particle out of the matrix, which allows further degradation
(including biodegradation) in the environment.

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