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Surface Treatment of Fly Ash for Improving the Tensile Strength of Fly Ash/Unsaturated Polyester Composites

Sugiman Sugiman
Department of Mechanical Engineering, Faculty of Engineering, University of Mataram, Mataram 83125, Indonesia, s.sugiman@tm.ftunram.ac.id

Paryanto Dwi Setyawan
Department of Mechanical Engineering, Faculty of Engineering, University of Mataram, Mataram 83125, Indonesia

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Surface Treatment of Fly Ash for Improving the Tensile Strength of Fly Ash/Unsaturated Polyester Composites

Sugiman* and Paryanto Dwi Setyawan

Department of Mechanical Engineering, Faculty of Engineering, University of Mataram, Mataram 83125, Indonesia

**e-mail: s.sugiman@tm.ftunram.ac.id*

Abstract

The paper presents the surface treatment of fly ashes using acid and alkali solution on the tensile strength of the fly ash/unsaturated polyester (UP) composites. Sulfuric acid (H_2SO_4), hydrofluoric acid (HF) and sodium hydroxide (NaOH) solution with concentration of 0%, 5%, 10%, 15%, and 20% (by weight) were used for surface treatment of fly ashes. Generally, the tensile strength of the treated fly-ash/UP composites shows a significant increase compared to the untreated fly ash. Of all surface treatments studied, the optimum tensile strength was obtained at a concentration of 10%. At this concentration, at a fly ash content of 30% (weight), the surface treatment using NaOH gave the highest tensile strength, approximately 18.69 MPa, or increased about 91% compared to the untreated fly ash. The morphology of fracture surfaces were evaluated using a scanning electron microscope (SEM). The fracture surfaces of the treated fly ash composite were rougher than that of the untreated fly ash indicating the improvement of the fly ashes-UP interaction.

Abstrak

Perlakuan Permukaan *Fly Ash* untuk Meningkatkan Kekuatan Tarik Komposit *Fly Ash/*Poliester Tak Jenuh. Paper ini menyajikan perlakuan permukaan *fly ash* (abu terbang) menggunakan larutan asam dan alkali pada kekuatan tarik komposit abu terbang/poliester tak jenuh. Larutan asam sulfat (H_2SO_4), asam fluorida (HF) dan natrium hidroksida (NaOH) dengan konsentrasi 0%, 5%, 10%, 15%, dan 20% (berat) digunakan untuk perlakuan permukaan abu terbang. Secara umum, kekuatan tarik komposit abu terbang/poliester tak jenuh dengan perlakuan permukaan menunjukkan peningkatan yang signifikan dibanding abu terbang tanpa perlakuan. Dari semua perlakuan permukaan yang dipelajari, kekuatan tarik optimum diperoleh pada konsentrasi larutan 10%. Pada konsentrasi tersebut, pada kandungan abu terbang 30% berat, perlakuan permukaan dengan larutan NaOH memberikan kekuatan yang paling tinggi sekitar 18,69 MPa atau meningkat 91% dibanding *fly ash* tanpa perlakuan. Berdasar pada morfologi patahan sampel yang dievaluasi dengan *scanning electron microscope* (SEM), permukaan patah komposit abu terbang dengan perlakuan lebih kasar dibanding tanpa perlakuan yang menunjukkan perbaikan interaksi antara abu terbang dan resin poliester tak jenuh.

Keywords: composites, fly ash, surface treatment, tensile strength, unsaturated polyester

1. Introduction

Filler has been widely used in the plastic industry to reduce the production cost and to increase certain mechanical properties of plastic products. Fillers are cheap and hugely available in many different types, forms and sizes [1-2]. Filler can be originated from organic and inorganic substances. It can be in form of spherical, cubic, amorf, block, flake and fiber with an aspect ratio from 1 (spherical) to 1600 (fibre). Some examples of filler are talc, flour, silica, $CaCO_3$, quartz, mica, carbon black, fly ash and clays (bentonite and monmorilonite) [2].

Fly ashes are waste products of coal combustion in electric power plants and are categorized as hazardous materials; therefore, they become a problem if they are directly dumped to the environment, as landfill materials. Nowadays, fly ashes are used as partial replacement of cement to manufacture high strength concrete [3]. Fly ashes are also used as raw materials for geopolymer, and refractories [4]. Fly ashes contain some metal elements, ceramic fiber, alumina and carbon [5-6], therefore they can be further treated to obtain those materials. The applications of fly ashes as filler for thermoplastic have been reported in polypropylene [7], in blend of polypropylene-polymethylmetacrylate (PMMA) [8], and in polycarbonate as flame retardant [9].

It has been widely known that the bonding between filler and polymeric matrix plays an important role on the strength of its composites. Therefore, the aim of the study is to investigate the tensile strength of the chemically treated fly ash/unsaturated polyester composites and their fracture morphology.

2. Experiment

Firstly, the study determined the effect of the fly ash content on the tensile strength of the untreated fly ash/UP composites. Secondly, the fly ashes were treated using chemical solution (NaOH, H_2SO_4 and HF), prior to be mixed with the UP and then their tensile strength were investigated. Further, the surfaces of the broken specimens were examined using a SEM.

Specimen preparation. Fly ashes were supplied by the Paiton Electric Power Plants (East Java, Indonesia). The diameter of fly ashes is less than 50 μm. The composition of the same fly ash (from Paiton) was obtained from literature [10] as shown in Table 1. Before fly ashes were used to reinforce the commercial UP, they were dried in the oven at 115 ^oC for 5 hours to ensure that moisture has been desorbed from the fly ashes. Further, the fly ashes were immersed and stirred in the acetone to remove oil, and other contaminants (rinsing) from the surface of fly ashes. This was followed by drying them in the oven at a temperature of 70 ^oC for an hour. To study the effect of the fly ash content on the tensile strength of the fly ash/UP composites, five variations of the untreated fly ash content (10, 20, 30, 40, and 50% (by weight)) were prepared. Fly ashes were mixed with the UP and stirred well before adding the hardener (methyl ethyl ketone peroxide). The ratio of UP to hardener was 100 : 1 (by volume). The mixture then was poured to the pre-shape mold according to ASTM D638.

Prior to surface treatment, the fly ashes were treated following the above procedures (drying and rinsing). Surface treatment of fly ash was performed using NaOH, H_2SO_4 and HF within the range of concentration from 5% to 20% (by weight) with the increment of 5%. Then, they were immersed in those solutions at a room temperature, and stirred for approximately 30 minutes. Drying of the treated fly ashes was carried out in the oven at a temperature of 115 ^oC for 5 hours. The dry fly ashes then were mixed with UP and hardener, and stirred well before pouring into the mold. In this case, the fly ashes content was kept at 30% (by weight) for each type of treated fly ash.

Testing. The testing included the tensile tests and the fracture surfaces examination using a SEM. The tensile tests were carried out using a Hung Ta universal testing machine (Taiwan) with a load capacity of 10 tones. The displacement rate was 2 mm/min. Morphology of the fracture specimen was determined using a SEM, JEOL JSM-35C, at operating voltage of 10-20 kV. Prior to scanning, the specimens were coated with a gold (Au)-paladium (Pd) (Au/Pd = 4) using Ion Sputter JFC–1100 machine at a voltage of 1.2 kV, electric current of 6-7.5 mA, and a vacuum pressure of 0.2 torr for 4 minutes.

3. Results and Discussion

This section presents the results of the tensile strength of the untreated fly ash/UP with the fly ash content and then the effect of surface treatment.

Effect of fly ash content. Figure 1a shows the average tensile strength of untreated fly ash/UP with the fly ash content. The vertical bars at each data point indicate the variation of the data. It can be seen that the tensile strength of fly ash/UP decrease with the increase of the fly ash content. The decrease occurs steeply until the content of 30% then it tends to be flat. At the content of 30% the decrease of tensile strength was approximately 46%. This is possibly due to the weak interfacial bonding between the fly ash particles and UP. As seen in Figure 2, the morphology of fracture surfaces of the fly ashes-filled UP (Figure 2b and c) seems rougher than that of the neat UP (see Figure 2a), which indicates more complex failure mechanism of the filled UP. The weak interfacial bonding between fly ash and UP (see Figure 2d) is a site of high local stress concentration as debonding occurs easily. This further induces the micro crack initiation to occur. With increasing the fly ash content, the number of this site increases and causes more micro cracks formed which finally lead to early failure (lower the tensile strength of the fly ash/UP composites). These results are also consistent with the observed trends in the polyurea filled with fly ash [11]. The increasing volume fraction of fly ash up to 20% in the polyurea decreased both the tensile stress at break and the elastic modulus due to debonding of fly ash with the polyurea matrix.

Table 1. Composition of Fly Ash Obtained from Paiton's Power Plant [10]

SiO_2	Al_2O_3	TiO_2	Fe_2O_3	CaO	Na_2O	K_2O	MgO	SO_3	LOI
38.10	21.22	1.64	13.65	12.3	2.68	0.76	4.36	3.17	1.06

LOI = Loss of ignition

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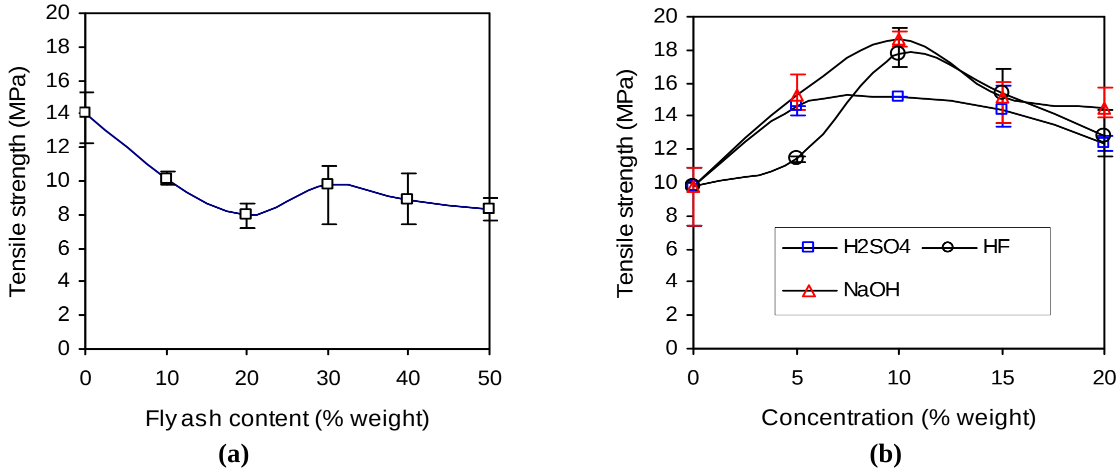

Figure 1. Showing the Tensile Strength of (a) Untreated Fly Ash/UP with the Fly Ash Content, (b) Treated Fly Ash/UP with the Concentration of the Modifier

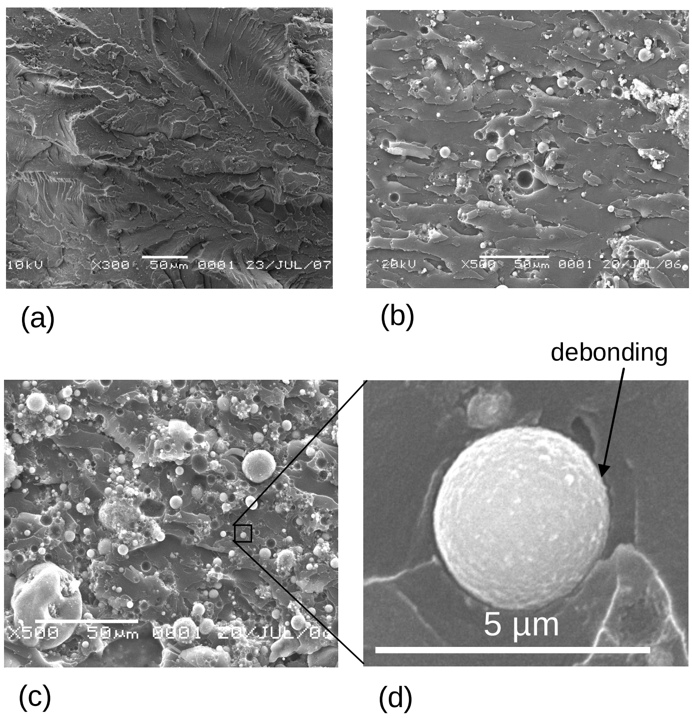

Figure 2. SEM Micrographs of Fly Ash/UP at the Fly Ash Content of (a) 0% (the Neat UP), (b) 20%, (c) and (d) 40%

Effect of surface treatment. The interfacial bonding between fly ash and UP can be improved using a proper surface treatment. As seen in Figure 1b, at the fly ash content of 30%, when the concentrations of NaOH, HF and H_2SO_4 are up to 10%, the tensile strength of fly ash/UP increases, and when the concentrations are between 10% and 20%, it tends to decrease. However, within this concentration range (0-20%), the tensile strength of the fly ash/UP is still higher than that of the untreated fly ash. The strong acid and alkali solution are able to leach the alumina and silica in the fly ash and possible to degrade the fly ash structure. The best result of each surface treatment methods was obtained at a concentration of 10%. At this concentration, the tensile strength is even better than the neat UP. It marginally increases approximately 29%, 26%, and 9% for NaOH, HF and H_2SO_4 respectively, compared to the neat UP or about 91%, 81%, and 55% respectively compared to the untreated fly ash at the same content (30%). This indicates that of the chemical solution used in this study, NaOH solution gives the best improvement to the tensile strength followed by HF and H_2SO_4. Chemical surface treatment (acid and alkali solution) increases the surface area and produces micro pores in the particles, which can promote an intimate contact with the matrix [12]. In addition, the chemical activation of fly ashes also induce functional groups such as –OH, which also become sites for the chemical bonding with the functional chain structure of the polymer matrix [7]. Hence, the chemical treatment is able to increase the surface energy of fly ash, surface area and micro pores and then improve the interaction with the UP matrix yielding to high interfacial bonding. Guhanathan and Devi [13] investigated the silane-treated fly ash on the mechanical properties of fly ash/UP and showed the increase of tensile strength about 75% compared to the untreated fly ash. This increase is lower than that of reported here (i.e. using NaOH and HF at a concentration of 10%). The use of silane coupling agent is to introduce an adhesion promoter that incorporates the fly ash particles and polyester resin by covalent bonding. Thus, the interaction mechanism may differ with the use of acid and alkali solution in altering the surface chemistry of the fly ash, and so with the improvement of the interfacial bonding. This will be of interest for the future investigation.

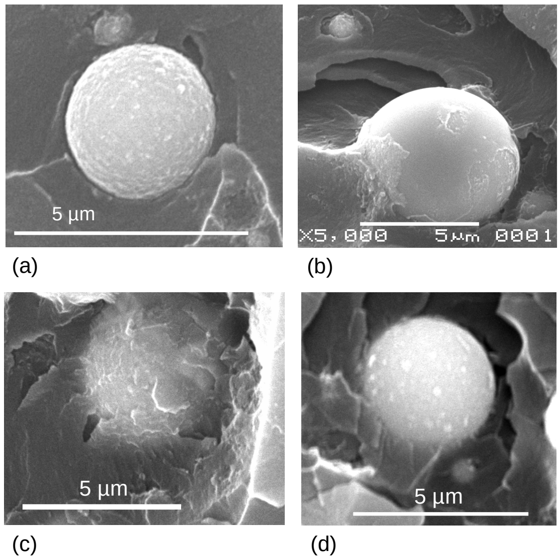

Figure 3. SEM Micrographs of the Fracture Surfaces of Fly Ash/UP with Fly Ash Treated Using (a) Untreated, (b) 10% of HF, (c) 10% of NaOH, (d) 10% of H_2SO_4

Figure 3 shows the SEM micrograph of the fracture surfaces of the fly ash/UP at the various surface treatments. It can be seen that debonding occurs at the interface between the fly ash particle and the UP matrix for untreated fly ash (Figure 3a), which indicates the weak interfacial bonding. Therefore, it only needs low external forces to break the specimen. Further, it can be seen in Figure 3b-d, the surface treatment of fly ash improves the interfacial bonding, as the debonding at the interface region of the treated fly ash particle/UP was not observed. The smooth surface of fly ash treated using HF (Figure 3b) is possible due to the excessive etching of SiO_2 with HF because HF is very reactive with the silica. Again, in Figure 3c, the fly ash particles were treated using NaOH at a concentration of 10% and the morphology shows that they are well bonded with the UP matrix as most of failure occurs at the matrix region. This further clarifies why surface treatment using NaOH 10% gives the highest tensile strength among the others. The morphology between NaOH and H_2SO_4-treated fly ash is similar; however, why NaOH treatment gives the better results requires further investigation.

4. Conclusions

Addition of the untreated fly ashes to the unsaturated polyester decreases the tensile strength of its composites. However, the decrease can be relieved by surface treatment of the fly ashes. Chemical solutions (NaOH, HF and H_2SO_4) improve the surface condition of fly ashes, which lead to increasing the interfacial composites. This is shown by the increase of tensile bonding with the unsaturated polyester matrix yielding to increasing the tensile strength of the fly ash/UP strength approximately 91%, 82%, and 55% for NaOH, HF, and H_2SO_4 respectively at a fly ash content of 30% (weight). The surface treatment using NaOH solution at a concentration of 10% (by weight) gives the better interfacial bonding between fly ash particles and UP matrix, clarified using a scanning electron microscope.

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