

Study the Effect of the Untreated and Treated Fly Ash on the Mechanical Properties of the Polymer Composites Based on a Mixture of Bisphenol a and Bisphenol F Epoxy Resin Cured by Kingcure K11 Hardener

Bach Trong Phuc, Pham Duc Trinh, Nguyen Thanh Liem

Centre for Polymer, Composite and Paper Technology/School of Chemical Engineering/
Hanoi University of Science and Technology, Hanoi, Vietnam

ABSTRACT

Ordinary fly ash type C is modified the surface by the agents KOH 3M, Silane Silquest in acidic condition pH = 4 and Stearic acid 2%, respectively. Mixture of bisphenol A epoxy resin (GELR 128 - resin A) and Bisphenol F epoxy resin (EPOTEC 170LV - F resin) is prepared by agitating well in a glass with a agitating speed of 200 rpm at a temperature of 50°C and a time of 30 minutes. Modified fly ash was dispersed into mixture of epoxy resin and then solidified with Kingcure K11 hardener. The results showed that the mechanical properties of the polymer composites based on mixture of bisphenol A and bisphenol F with treated fly ash (10%, 20%, 30% by weight) cured by Kingcure K11 hardener were higher than that of untreated fly ash composites.

KEYWORDS: mixture, bisphenol A epoxy resin, bisphenol F epoxy resin, fly ash, treated, polymer composite

How to cite this paper: Bach Trong Phuc | Pham Duc Trinh | Nguyen Thanh Liem "Study the Effect of the Untreated and Treated Fly Ash on the Mechanical Properties of the Polymer Composites Based on a Mixture of Bisphenol a and Bisphenol F Epoxy Resin Cured by Kingcure K11 Hardener" Published in International Journal of Trend in Scientific Research and Development (ijtsrd), ISSN: 2456-6470, Volume-5 | Issue-3, April 2021, pp.759-762, URL: www.ijtsrd.com/papers/ijtsrd39950.pdf



IJTSRD39950

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I. INTRODUCTION

Fly ash is a type of dust from burning coal by energy production industries emitted to the environment. It is recovered in the exhaust fume department of thermal power plants [1,2]. The main chemical composition of fly ash is a mixture of inorganic oxides such as SiO₂, Al₂O₃, Fe₂O₃, TiO₂, MgO, CaO, K₂O [3]. Carbon content in fly ash is less than 4%. There are 2 types of fly ash: type C (Ca and Mg content is high up to 20%) and fly ash type F is much smaller than fly ash type C) [4]

Diglycidyl ether of bisphenol A – DGEBA, based on epichlorohydrin, is the most widely epoxy resin, whose viscosity depends on the number of basic links n and the molecular mass. The bisphenol F epoxy resin (Diglycidyl ether of bisphenol F - DGEBF) has a molecular structure similar to DGEBA but with the difference that there are 2 methyl groups attached to the carbon atom located between the alternative benzene groups for the hydrogen atom. The bisphenol F epoxy resin has a lower molecular weight and viscosity than the bisphenol A epoxy resin. Because of its high viscosity, the bisphenol A epoxy resin is difficult to mix

with the filling powder or difficult to penetrate the fiber reinforcing substance in composite materials. The bisphenol F epoxy resin has lower viscosity and higher activity than the bisphenol A epoxy resin, so it is easily mixed with filling substances, easily formed and solidified at room temperature. Thus, the mixing of these two epoxy resins will create low viscosity mixtures and also contribute to anti-crystallization of DGEBA, giving composites better properties, overcoming the drawbacks of individual type of plastic [5].

This study presents the effect of the untreated and treated fly ash on the mechanical properties of the polymer composites based on a mixture of bisphenol A and bisphenol F epoxy resin cured by Kingcure K11 hardener at 25° C

II. Experiment

2.1. Raw materials and chemicals

- The diglycidyl ether epoxy resin of bisphenol A has the trade name of GELR 128, originating from China. GELR is a transparent liquid without impurities, viscosity 11000 - 15000 mPa.s at 25° C, epoxy equivalent mass 184-190

g/eq, color (Gardner) 1,0 max., hydrolyzed chlorine content (ppm) 300-1000. Diglycidyl epoxy resin ether of bisphenol F has trade name of EPOTEC YDF 170LV, originating from Thailand. EPOTEC YDF 170LV is a transparent liquid with 100% non-volatile substance content, viscosity 2000 - 3000 mPa.s at 25° C, epoxy equivalent mass 160-170 g/eq, color (Gardner) 1,0 max, hydrolyzed chlorine content 0.1% max.

- Fly ash was supplied from Song Da-12 Cao Cuong Joint stock company.
- KOH 3M (from China).
- SilaneSilquest A-186: Momentive Brand (Germany). (β - (3, 4- Epoxycyclohexyl) etyl trimetoxysilane) Formula: $C_6H_9(O)-CH_2-CH_2-Si-(O-CH_3)_3$ MW: 246.1, d: 1.065g/cm³; Fire point: 113°C, Boiling point 310°C.
- Stearic acid $CH_3-(CH_2)_{16}-COOH$ (China) d: 0,8390 g/cm³, M = 284,48; Melting point: 69,6°C, Boiling point 361,1°C, easy to be fired.
- Kingcure K11 (KK1) is an accelerated aliphatic amine curing agent in an amber-colored liquid form was purchased from Sanho Chemical Co., LTD (Taiwan) (density 1.04 g/cm³ at 250C, viscosity at 250C: 1000-2000 mPa.s, amine value: 430±20 mgKOH/g and Active Hydrogen Equivalent Weight (AHEW): 93).

2.2. Fly ash surface treatment process

2.2.1. Fly ash surface treatment by KOH 3M solution

The fly ash particles were mixed with KOH 3M solution at the ratio of 1 (g) FA: 15 ml of KOH solution in a 3-neck flask. Then, the mixture was heated and agitated continuously for 6 hours at 90°C. The treated solution was cooled to room temperature and filtered, washed several times with distilled water until pH = 7. Fly ash treated with KOH solution was dried in an oven at 100°C for 12 hours and was denoted as FAN [6].

2.2.2. Fly ash surface treatment by silane compound

The silane compounds are hydrolyzed in 100 ml ethanol (with acetic acid supplementation to create a condition of pH = 4) at 50°C for 30 minutes, then add 100 grams of fly ash to the mixture and continue agitating well for 4 hours at 50°C. Silane content is taken according to the amount of fly ash to be modified. The reaction mixture is dried naturally, then continue to dry at 60°C, then filter, wash to remove excess silane. The fly ash product after modification is denoted as FAS [6].

2.2.3. Fly ash surface treatment by stearic acid

Stearic acid with a volume of 2 grams is mixed a mixture of acetone and toluene with a volume ratio of 3:1 to make a solution 2% (wt) stearic acid. The whole mixture was agitated well for 30 minutes. Then add 100 grams of fly ash to the mixture and continue for 30 minutes. The obtained material block is stabilized at room temperature for 24 hours and then filtered, washed and dried at 100 ° C. The modified fly ash product is denoted as FASA [7].

2.3. Prepare the epoxy resin mixture

To prepare the mixture, weigh each epoxy resin, epoxy bisphenol A (GELR 128), denoted resin A and epoxy resin bisphenol F (EPOTEC YDF 170LV) at a weight ratio of 20:80, gradually pour into a glass placed in a water-proof autoclave heated at 50°C and mechanically agitated. Keep the mixture at 50°C and agitating at speed of 200 rpm for 30 minutes. At the end of the process, pour the mixture into the containing bottle, store for at least 24 hours before utilizing. Then

weight the weight of fly ash and epoxy resin according to the calculated ratio before then stirr 200 rpm in 30 minutes at a temperature of 50°C, vacuum the mixture for 5 minutes. At the end of the process, pour the mixture into the containing bottle, store for at least 48 hours before utilizing.

2.4. Preparation of polymer composite (PC) samples from epoxy resin solidified by Kingcure at 25°C

First clean the molds by wiping the surface with acetone and then using WAX 8 anti-stick agent to prevent sticking to the mold. Weigh the calculated Kingcure, mix well in a beaker, agitate well and pour mixture slowly into the molds. Combining both vacuum and vibration in the solidification process of epoxy resin with calculated time. At the end of the process, take the sample from the mold, let it stabilize for 7 days, then take the Polycomposit samples to determine the properties of the material.

2.5. Methods and devices for analysis of contact angles, surface images and mechanical strength.

Contact angle is determined according to ASTM C813-90 standard on DROP SHAPE ANALYZER device (Germany) – DSA100 s (Kruss) resolution 0.01 mN/m.

The morphologies at the fracture surfaces of the epoxy samples were evaluated from Scanning Electron Microscopy (SEM) on Hitachi (Japan) S4800 at the main laboratory, Institute of Materials Science – Vietnam Academy of Science and Technology.

Tensile stress is determined according to ISO 527-2012 standard on INSTRON 5582-100kN device (USA) with a tensile speed of 2mm/min at Polymer-Composit and Paper Technology Center, Hanoi University of Technology.

Flexural strength is determined according to ISO 178-2012 standard on INSTRON 5582-100kN device (USA) with a flexural speed of 2mm/min at Polymer-Composit and Paper Technology Center, Hanoi University of Science and Technology.

Notched and non-notched impact resistance was determined according to ISO 180-2012 standard on Tinius Olsen Model 92T device (USA) at Polymer-Composit and Paper Technology Center, Hanoi University of Science and Technology.

III. Results

3.1. The modification results of fly ash samples compared with ordinary ash

3.1.1. Results of measuring the contact angle of fly original ash sample and modified fly ash samples

Proceed modification of ordinary ash with various modifying agents and use a drop of distilled water to measure contact angles with the fly ash original and modified fly ash then compare. We get the corresponding results as the image below, which are the contact angle measurement values of the samples.

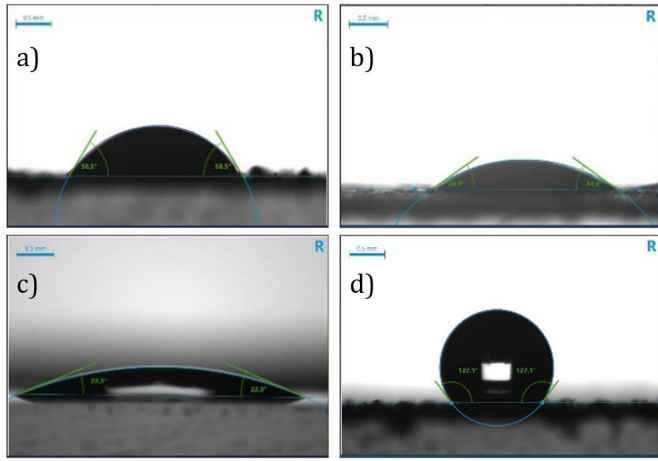


Fig.1 The contact angles of fly ash samples

- A. Non-modified ordinary fly ash (Contact angle = 58,5°)
- B. Modified ordinary fly ash by KOH 3M solution (Contact angle = 34,9°)
- C. Modified ordinary fly ash by Silane (Contact angle = 22,3°)
- D. Modified ordinary fly ash by Stearic acid 2% (Contact angle = 127,1°)

From **Fig.1**, it was found that when using water as a dripping medium to measure the contact angle, compared with ordinary fly ash, the modified fly ash has a considerable change in the contact angle. Fly ash samples modified with KOH 3M and Silane become more hydrophilic so the contact angle is smaller, and samples modified with stearic acid increase the contact angle. The agents have chemical effects on the surface of fly ash leading to the appearance of hydrophilic or hydrophobic tips on the fly ash surface, leading to significant changes in surface structure and surface composition of fly ash particles.

3.1.2. SEM analysis images of original fly sample and modified fly ash samples

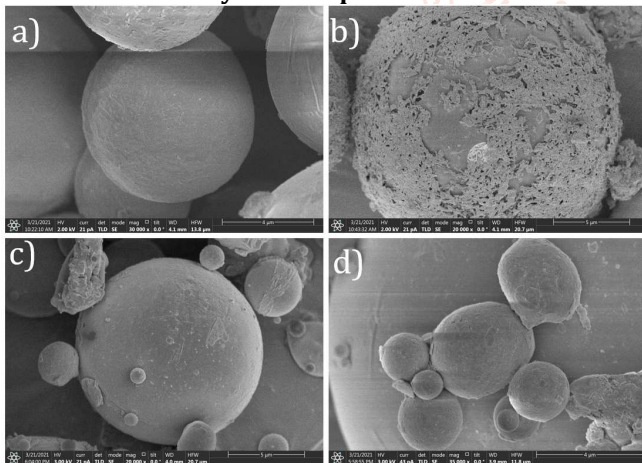


Fig.2 SEM images of fly ash samples

- A. Non-modified ordinary fly ash
- B. Modified ordinary fly ash by KOH 3M solution
- C. Modified ordinary fly ash by Silane
- D. Modified ordinary fly ash by Stearic acid 2%

From **Fig.2**, it is realized that compared to the non-modified ordinary fly ash, the fly ash particles, after surface modification, show a considerable change in appearance through the SEM electron microscope. Ordinary fly ash sample surface is smooth. With the fly ash sample modified by KOH 3M, under chemical erosion the unstable oxides participating in the reaction leave undulating positions on

the surface to increase significantly the surface area of this sample. Samples modified with Silane and stearic acid show signs of forming additional roughness on the surface of fly ash particles in Fig.2 c), d). This is one of the factors involved in the process of bonding with epoxy in the solidifying process.

3.2. Research the effect on mechanical strength of composite based on substrate resin mixed with modified fly ash compared with surface non-modified fly ash

The mixture of epoxy resin bisphenol A (GELR 128), denoted resin A and epoxy resin bisphenol F (EPOTEC YDF 170LV) was mixed with some types of fly ash 10%, 20%, 30% by weight, respectively, solidify by the modified Amine Kingcure K11 at a temperature of 25°C.

The order of the samples is numbered and named as follows:

1. Bisphenol A mix with Bis Phenol F 2:8 in volume
2. Mixture A / F 2: 8 + 10% modified fly ash by KOH 3M
3. Mixture of A / F 2: 8 + 10% of non-modified ordinary fly ash
4. Mixture of A / F 2: 8 + 10% fly ash modified by Silane
5. Mixture A / F 2: 8 + 10% fly ash modified by Stearic Acid 2%
6. Mixture A / F 2: 8 + 20% non-modified ordinary fly ash
7. Mixture A / F 2: 8 + 20% modified fly ash by KOH 3M
8. Mixture of A / F 2: 8 + 20% fly ash modified by Silane
9. Mixed A / F 2: 8 + 20% fly ash modified by Stearic Acid 2%
10. Mixture of A / F 2: 8 + 30% of non-modified ordinary fly ash
11. Mixed A / F 2: 8 + 30% fly ash modified by KOH 3M
12. Mixed A / F 2: 8 + 30% fly ash modified by Silane
13. Mixed A / F 2: 8 + 30% fly ash modified by Stearic Acid 2%

3.2.1. Measurement results of flexural strength

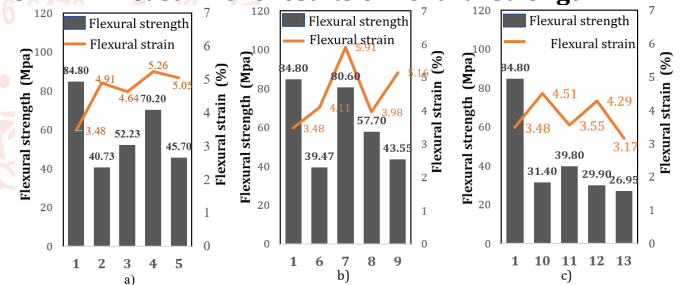


Fig 3 Flexural strength and module of the composite is compounded by epoxy resin and solidified by Kingcure K11 when adding original fly sample and modified fly ash samples

Through the results found in **Fig.3**, 3a, 3b the flexural strength of the composite samples when mixing fly ash compared with the flexural strength of the sample without mixing fly ash has decreased, but comparing the flexural strength of epoxy sample – non-modified fly ash and epoxy samples combined with modified fly ash, epoxy samples and modified fly ash have much higher flexural strength at the percentage of 10% and 20% in weight, especially epoxy blended 10% of silane modified fly ash has a flexural strength of 70.2 Mpa, the epoxy sample mixed with 20% of the fly ash modified by KOH 3M has a flexural strength of 80.6 Mpa, while the epoxy sample without mixing fly ash after solidifying has a strength of 84.8 Mpa. The highest bending modulus at sample 7 (Mixture A/F 2: 8 + 20% fly ash modified by KOH 3M)

3.2.2. Measurement results of tensile stress

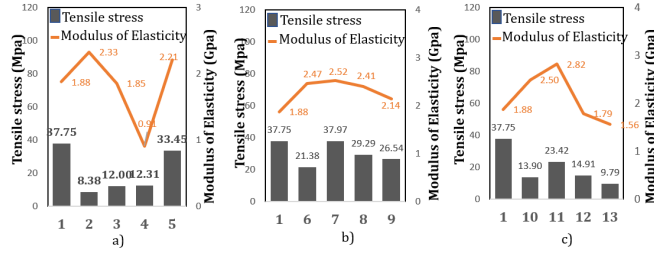


Fig.4 Tensile stress and module elasticity of the composite is compounded by epoxy resin and solidified by Kingcure K11 when adding fly ash original fly sample and modified fly ash samples

Through the results obtained in Fig.4, it was found that the tensile stress of the composite sample blended with fly ash was lower than that of the sample without mixing fly ash, but compared with ordinary fly ash samples, the modified fly ash samples had tensile stress and tensile modulus are better at a blend ratio of 20% by weight, especially a composite sample with 10% mass Stearic acid modified fly ash reached 33.45 Mpa. And in these tensile stress of composites that have 20% modified fly ash almost higher than tensile stress of composite contents 20% weight original fly ash. Tensile stress of composite contented 20% (wt) fly ash modified by KOH 3M solution reached 37.97Mpa and at this sample the module elasticity also high (2.47Gpa).

3.2.3. Measurement results of notched and non-notched impact resistance

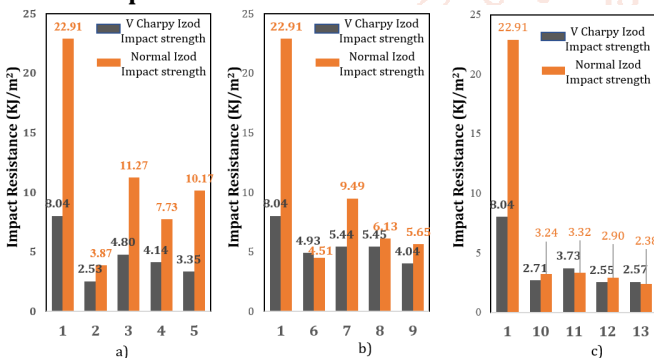


Fig.5. Notched and non-notched impact resistance of the composite is compounded by epoxy resin and solidified by Kingcure K11 when adding original fly sample and modified fly ash samples

From the results of the impact resistance measurement of composite samples mixed with denatured fly ash at about 10-20% by weight, the impact resistance is higher than that of composite samples mixed with non-modified ordinary fly ash. In composite samples with 10% and 20% content of the composite sample containing fly ash modified by KOH 3M solution are higher than others, notched and non-notched

impact resistance is 11,27 KJ/m², and 9,49 KJ/m², respectively.

Conclusion

At 25°C, when blending with a mixture of epoxy resin bisphenol A (GELR 128) and epoxy resin bisphenol F (EPOTEC (YDF 170LV) with fly ash samples and solidified at 25°C, the results showed that the composite composed of the mixture of resin and surface modified fly ash have higher mechanical strength than that of composite samples made up of a mixture of resin and non-modified fly ash. The highest flexural strength is in the composite sample when mixing fly ash 20% of weight modified by KOH 3M with the value of 80.6 Mpa, the Flexural strain value is 5.91 %. The highest tensile stress in a composite sample when mixing 20% weight fly ash modified by KOH 3M is stood at 37.97Mpa and the elasticity module is achieved 2.52 Gpa. The highest notched impact resistance in a composite sample composed of epoxy resin and 20% weight of fly ash modified by silane is 5,45 KJ/m². The highest non-notched impact resistance in the mixture of resin with 10% fly ash modified by KOH 3M reaches 11,27 KJ/m².

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