

FIRE RESISTANCE OF FLY ASH - POLYESTER GEOPOLYMER COMPOSITE

Kuncoro Diharjo¹, Jamasri², Feris Firdaus³

¹Lecturer in Mechanical Engineering Department, Engineering Faculty, Sebelas Maret University, Email: kuncorodiharjo@uns.ac.id

²Profesor in Mechanical Engineering Department, Engineering Faculty, Gadjah Mada University

³Researcher in Research Directorate of Indonesia Islamic University

ABSTRACT

The development of PLTU using coal in Indonesia results the increasing of fly ash waste. The waste should be used carefully to eliminate environment pollution. The objective of this research is to investigate the fire resistance of the fly ash – polyester geopolymer composite. The fly ash-polyester composite was made by using a press mold method for 0, 30, 40, 50, 60, and 70 % of fly ash content (w/w). The fire resistance test was prepared by according to JIS K-6911 standard for 2 minutes of burning test time. The analysis in this research covers fire resistance, heat release, composite mass burned and smoke test. The fire resistance increases with the increasing of fly ash content. The longest burning time of the composite (42,92 second) occurs on the composite containing the highest fly ash content (70% of fly ash content). The heat release decreases with the increasing of fly ash content. The highest heat release (88,67%) occurs on the composite without fly ash. The least mass of the composite burned (12,34%) occurs on the composite containing 30% of fly ash content. The mouses smoked by smoke resulted of burning composite for 2 minutes indicate no difference behavior.

Keywords: geopolymer composite - fly ash - fire resistance - heat release - composite mass burned - smoke test

INTRODUCTION

Background

In engineering, composite materials have been applied to substitute some metal components of automotive car body panels. Industries are very interest to apply the materials because of their advantages, like light, high strength and low cost. Beside of high strength, the composite panel should also have high fire resistance to eliminate fire of transportation

accidents. For example, when the Garuda Indonesia Aircraft had an accident caused by hard landing on International Adisucipto Air Port Yogyakarta in 2007, the fire burned all interior car body composite panels in 5 minutes. Whereas, the panels had been be made from the glass fiber as reinforcement and resin containing high fire resistance material as bonding material (matrix). Other accident of train also shows that the fire, caused by short

circuit of electrical system, burned the interior car body panels in few minutes. According to the accident cases, it is very important to develop the composite panel, which has high fire resistance.

Disposal of this fly ash waste, like in PLTU Payton East Java and PLTU Suralaya West Java, causes air and water pollution. The fly ash is categorized as danger waste by government. In other side, the development of PLTU in Indonesia increases the productivity of fly ash. In 2000, the productivity of fly ash was 1.66 million tons and increase in 2006 resulting 2 million ton of fly ash. In PLTU Suralaya, the mean productivity of fly ash is around 219,000 ton/year since 2000 to 2006 (Ardha ; <http://www.tekmira.esdm.go.id>). When dry season, the fly ash can be carried by the wind, and when rainy season, it can be carried by water causing soil pollution. As the fly ash is produced in high capacity, the negative effect caused by the fly ash is difficult to be protected. It can also cause the decreasing of some ecosystem. A lot of the fly ash has been used in other industry, like cement industry. The fly ash is classified into small size powder, containing more than 80% of silicate and alumina oxide. According to the problem, engineers and scientists should have responsible to research the useful of the fly ash for making other engineering product.

In India, more than 100 million tons of fly ash produced, use of fly ash for the preparation of polypyrrole-fly ash composites will in no way help in its bulk utilization. Murugendrappa et. al (2005) also have made an effort towards the better utility of fly ash by synthesizing polypyrrole-fly ash composites. The results show that the properties of the composites prepared by fly ash are

significantly influenced by the weight percentage of fly ash in polypyrrole. The higher fly ash content in the polymer, the higher extent of moisture ingression (Kishore, 2005). Whereas strength in dry condition increases with initial addition of ash particles, those for the medium ingressed show a decrement. Interface debonding occurring around ash particles has been traced to be the cause for such changes in response in the wet samples vis-a-vis the dry ones. The impact strength increases linearly with increasing the fly ash content up to 50 wt% and then decreases for further increment (Ramakrishna, 2006). This decrease in impact strength when the fly ash content increases from 50 to 60 wt% can be attributed to the immobilization of the macromolecular chains by the fillers, which limit their ability to adopt to the deformations and hence makes the material more brittle and each particle or aggregate of particles is the site of stress concentration and can act as a micro crack initiator (Maiti and Sharma,1992; Svehlova and Poloucek,1987).

According to the problems, fly ash produced in high number should be used to produce a new innovation product, such as geopolymer composite panel for car body automotive. This study aims to investigate the effect of fly ash content to fire resistance of fly ash – polyester geopolymer composite.

Fly Ash and Geopolymer Composite

According to the chemistry, fly ash is an inorganic oxide material containing active oxide of silica and alumina. The oxide can easily reactive with others for making composite material to result high temperature resistance of new material. The SiO₂ (silica), Al₂O₃ (alumina) and

CaO (coarse) are the main components of fly ash material, which have high temperature resistance. The highest and the lowest melting point of fly ash are around 14,000 – 15,000 °C and 11,700 – 12,650 °C, respectively. The biological test of fly ash resulted by PLTU-Ombilin and PLTU Bukit Asam, to the fish and mouse shows that the fly ash is no dangerous to them. Federal Aviation Administration has improved the geopolymer composite prepared by fly ash for designing high temperature resistance material. The composite panel has been used for interior panel of aircraft and structure component of Formula-1 grand prix car (Kasim, 2001; Khaerunisa, <http://www.tekmira.esdm.go.id>; Lyon et. al, 1997).

The research resulted by Cornell University/ National Institute of Standards and Technology (NIST) shows that the geopolymer composite, prepared by 90% clay and 10% plastic in weight, can protected 60-80% on fire failure. In other side, the gropolymer composite prepared by using clay has higher dynamical-mechanical properties compared to that prepared without clay. The other inorganic materials, such as boron and zirconium oxides, composing with nylon, polyethylene, and polypropylene, also result similar characteristic.

The fly ash has a good potential as reinforcement particulate material for designing geopolymer composite. Smaller grain size of fly ash, higher strength and strain of fly ash – polyester geopolymer composite. For 40% of fly ash (w/w), the composite reinforced by 0.1 mm of fly ash grain size has 127.24 MPa in flexural strength and 4.11 GPa in flexural modulus. The flexural strength and modulus of the geopolymer composite increase with the increasing of fly ash content. The

composite has highest flexural strength for 40% of fly ash content (Diharjo et. al, 2007a).

Until 60% of fly ash (w/w), the tensile strength of fly ash – polyester composite increases with the increasing of fly ash content. The strength increases significantly up to 50% of fly ash content. The tensile strength of composite reinforced by 20, 30, 40, 50 and 60% of fly ash content are 21.79, 22.29, 26.54, 27.73 and 28.43 MPa, respectively. The result is very suitable with Rule of Mixture (ROM) theory (Sanadi et. al., 1985; Diharjo et. al., 2007b).

Fire Resistance

The advantage of composite reinforced by fly ash is low smoke density and the disadvantage results poisonous gas. The NF F16-101, applied for train in France, gives evaluation for protecting the composite material to fire resistance properties with combining reaction of fire and poisonous gas. Now, the fire safety has been required by public. As consequence, there are some requirement of fire retardant resin and filler system (QinetiQ, 2004). Last time, British Standard B 6851 has introduced an R- index concept, where the qualification of poisonous gas risk is correlated to the composite material used for interior panel of train car body. The R-index is classified as:

- a. Category 1a : train using corridor, $R < 1.0$
- b. Category 1b : train using tunnel, $R < 1.6$
- c. Category 2 : train using underground railway, $R < 3.6$

R-index is descendant of 8 gas types analysis, where the critical concentration have been decided by National Institute for Occupational Safety and Health

Administration (OSHA) and reported to the Immediately Dangerous Life and Health (IDLH). The values have been evaluated and researched continuously for reducing the emission gas size. For example, the oxide nitrogen made in 1997 is classified as poison gas. Whereas, the use of the nitrogen mixture has been applied for all train types in United Kingdom (Reinforced Plastics Magazine, 2002).

Heat Release

Heat release is defined as capability of a material to release the heat after burning. The percentage of heat release can be measured by using the equation (1).

Heat release (HR) =

$$\left(1 - \frac{T_1}{T_0}\right) \times 100\% \dots\dots\dots(1)$$

Where : HR = Heat Release
 T₁ = temperature on the material after the fire is out
 T₀ = burning temperature

Toxicity

Toxicity of EC_{50%} is a method used to know what a material contains poison. It is taken by burning the material to observe the effect of smoke resulted to animal. The object of toxicity test is usually tested to the white mouse, daphnia and fish. There are four grades of toxicity, i.e. toxic, toxic

relative, no toxic relative and no toxic. According to the Indonesia Government Regulation No. 85 - 1999, the fly ash resulted by PLTU is categorized as dangerous waste because it contains weight metal oxide causing environment pollution. Therefore, it is needed to know what the material contains poison.

METHOD

Materials used in this research were fly ash (FA) waste, unsaturated polyester resin (UPRs) and Methyl Ethyl Keton Peroxide (MEKPO) hardener. The FA was sieved by using a sieving machine for 500 µm of mesh size. The grain size resulted was equal or less than 500 µm. Furthermore, the FA was heated at 100 °C for 30 minutes to evaporate the water content.

The FA – UPRs composite was produced by using a press mold method for 0, 30, 40, 50, 60 and 70% of fly ash weight content (w/w). The MEKPO hardener added in UPRs was 1% (v/v). The mixing of FA-UPRs-MEKPO was poured in a mold, made from glasses. The composite size resulted by the process is 100 mm in length, 20 mm in width and 3 mm in thickness. The burning specimen has the same size with the composite size.



Figure 1. Burning test specimen and burning tester set.

The burning test specimen was tested by using a bunsen burning tester whose 6.5-11.5 mm in diameter. The fuel used to burn the composite was methanol. The bunsen was put at a holder and it's angle made 30° to vertical line. The fire has blue color and 25 mm in high. The burning test was tested in a room to avoid wind. According to the JIS K-6911 standard, the composite specimen was put on a holder with horizontal position (JIS, 1998). The data observed in the burning test is time needed to burn the specimen.

The heat release was tested by burning the specimen for 120 second, and then the fire was removed. Fire, which had burned the specimen for 120 second, was also extinguished. The temperatures observed are the temperature when the specimen was being burned and the temperature when the fire of the specimen was out. The heat release can be calculated by using equation (1). When the heat release test was going on, mass of the specimen was also weighted to investigate the mass reduction caused by the burning test. When the length of composite panel burned is equal or less then 25 mm, the sample can be categorized as a material whose high fire resistance.

Smoking on two white mice for 2 minutes was tested to investigate the

toxicity test. The smoke used is a result of fire burning the composite panel. Here, the difference of mouse behavior was observed in this research.

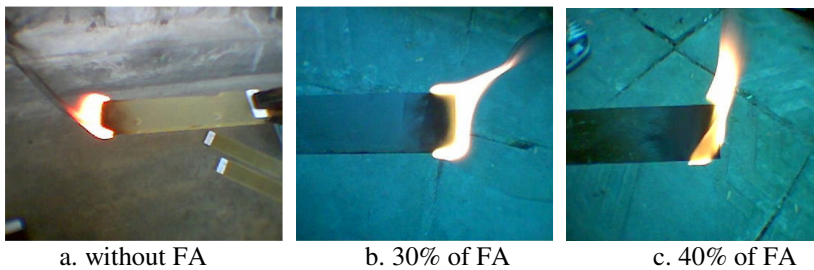
RESULT AND DISCUSION

Fire Resistance

The increasing of fly ash content increases the time to burn the composite, as shown in figure 3 and table 1. The longest time (42.92 second) for burning the composite occurs on the composite containing 70% of fly ash, and the least time (12.50 second) for burning the composite occurs on the composite without fly ash. The polyester, used in this research, has low temperature resistance (160 °C) and high flammable property (Surdia and Saito, 2000). This result shows that addition of fly ash on the polyester increases fire resistance property of the fly ash – polyester composite.

Table1. Time needed to burn the composite

Fly ash content (w/w), %	Time to burn the composite (second)
0	12,50
30	19,33
40	27,60
50	31,04
60	39,47
70	42,92



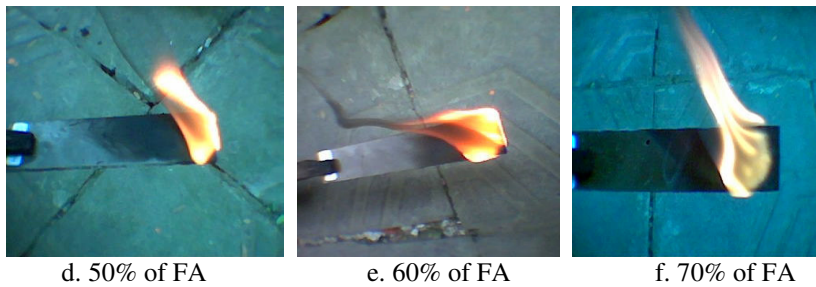


Figure 2. Burning test of composite.

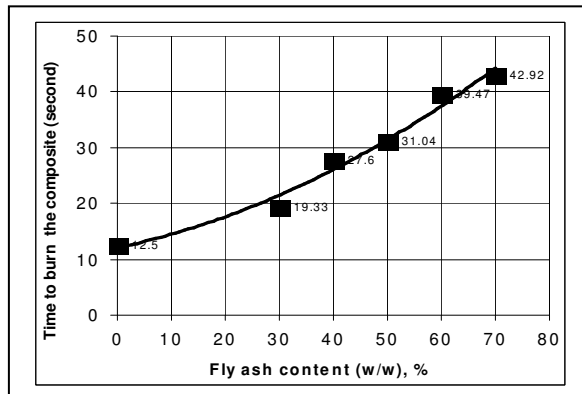


Figure 3. Curve of time to burn the composite.

Heat Release

Tabel 2. Heat release of the composite.

Fly Ash Content (w/w), %	Heat Release (%)
0	88,67
30	87,13
40	86,73
50	86,20
60	85,80
70	85,47

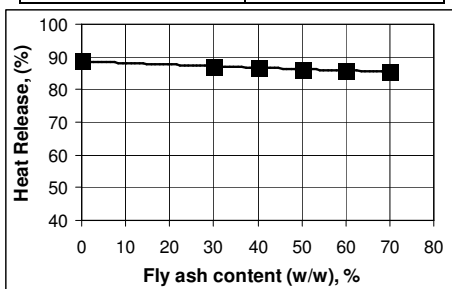


Figure 4. Heat release curve.

After the composite panel was burned for 120 second and the fire was out for 10 second, the temperature of the composite decreases. The temperature of the composite becomes the same with room temperature when the fire, burning the composite, was out for 2 minutes. According to the figure 4, heat release decreases with the increasing of fly ash content and the highest heat release occurs on the composite panel without fly ash. It shows that the increasing of fly ash content reduces the cooling rate of composite panel. It is reasonable because the decreasing of heat release is caused by silicate (Si) and Lumina (Al), which absorb heat on the burning process of the composite.

Composite Mass Burned

Table 3. Composite mass burned.

FA Content (w/w), %	Initial Mass gram	Final Mass gram	Mass Panel Burned gram	Percentage of composite mass burned, %
0	7,24	5,77	1,47	20,3
30	9,07	7,95	1,12	12,34
40	9,65	8,43	1,22	12,64
50	10,40	8,65	1,75	16,83
60	10,63	8,71	1,92	18,06
70	12,86	10,67	2,19	17,02

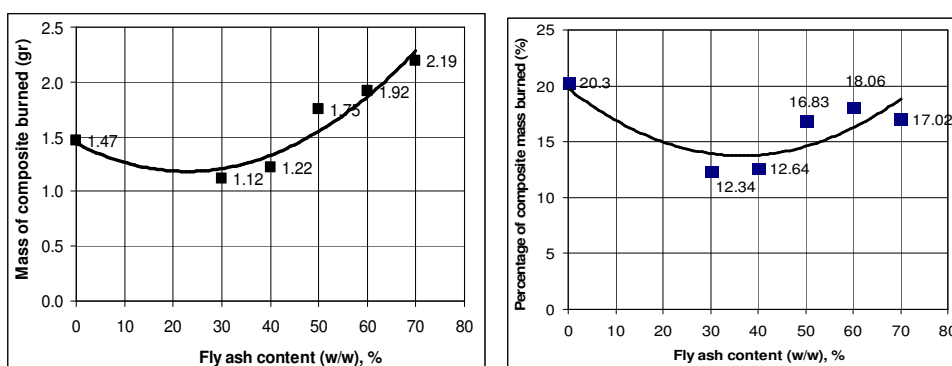


Figure 5. The curve of composite panel mass burned.

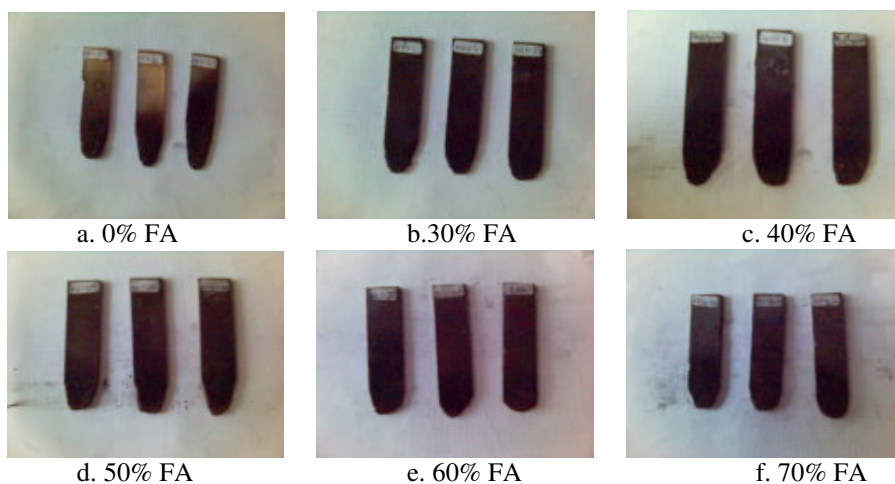


Figure 6. Burned composite specimens.

The composite containing 30% of fly ash has the least mass burned (12.34%). In contrast, the composite without fly ash has higher number of mass loss (20.3%). The

smoke analysis shows that the burning of composite resulting highest smoke density with darkest color occurs on the composite containing highest fly ash content (70% of

fly ash). The darker smoke indicates that the material is more difficult to be burned.

This result has a good correlation with that resulted by fire resistance testing.

Smoke Test

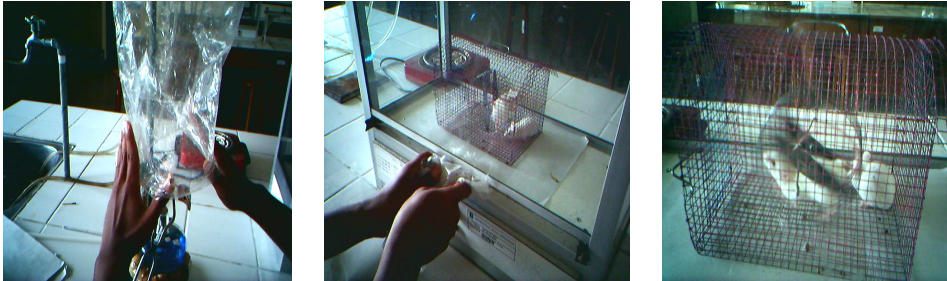


Figure 7. Smoking test on white mice.

When two mice were smoked using smoke resulted by burning of the composite panel in glasses room for 120 second, they had no difference behaviour. And then, they also were in health condition. It can be concluded that the fly ash-UPRs composite is not dangerous to the mice for 120 second of smoking test.

The composite containing 30% of fly ash has the least mass burned (12.34%). The increasing of fly ash content increases the number of burned composite mass. The burning of composite resulting highest smoke density with darkest color occurs on the composite containing highest fly ash content (70% of fly ash). The fly ash-UPRs composite is not dangerous to the mice for 120 second of smoking test.

CONCLUSION

The increasing of fly ash content increases time to burn the fly ash-polyester geopolymer composite. The longest time (42.92 second) and the least time (12.50 second) for burning the composite occurs on the composite containing 70% of fly ash and without fly ash, respectively. Heat release decreases with the increasing of fly ash content and the highest heat release occurs on the composite without fly ash.

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REFERENCES

- Ardha I.G.N., "The use of Fly Ash Produced PLTU Suralaya For Castable Refractory", Center Research and Development of Coal and Mineral Technology, Bandung, Indonesia, Web Master; <http://www.tekmira.esdm.go.id>.
- Diharjo K., Jamasri, dan Firdaus F., 2007a. "Effect of Particulate Size of Fly Ash and Fly Ash Weight Fraction To Flexural Properties of Fly Ash – Polyester Composite", Proceeding. International Conference on Chemical Science", Gadjah Mada University, Indonesia, May 24-26, 2007.

- Diharjo K., Jamasri, dan Firdaus F., 2007b. "Tensile Properties of Fly Ash – Polyester Geopolymer Composite", Proceeding of Conference, The 6th National Seminar Design and Application of Technology 2007, Widya Mandala Catholic University Surabaya, July 19, 2007.
- JIS, 1998, Japan International Standard, JIS K-6911.
- Kasim A.A., 2001, "The melting Point Study of Coal Ash For Three Oxide Component", da", JBPTITBPP, Bandung, Indonesia.
- Khaerunisa H., "Toxicity of Fly Ash and Bottom Ash Waste Resulted by Coal-PLTU using in Sumatra and Kalimantan using Biological analysis", Center Research and Development of Coal and Mineral Technology, Bandung, Indonesia, Web Master; <http://www.tekmira.esdm.go.id>.
- Kishore, Barpanda P., and Kulkarni S. M. (2005). Compression Strength of Saline Water-exposed Epoxy System Containing Fly Ash Particles, *Journal of Reinforced Plastics and Composites*; 24; 1567
- Lyon, R. E., Balaguru, P. N., Foden, A., Sorathia, U., Davidovits, J. & Davidovics, M., 1997, "Fire Resistant Alumino-Silicate Composites", Center for Advanced Infrastructure and Transportation (CAIT), Civil & Environmental Engineering, Rutgers, The State University, Piscataway, NJ.
- Maiti, S. N. and Sharma, K. K. (1992). Studies on Polypropylene Composites Filled with Talc Particles, *J. Mater. Sci.*, 27: 4605.
- Murugendrappa M.V., Khasim S., and Prasad M.V.N.A. (2005). Synthesis, characterization and conductivity studies of polypyrrole–fly ash composites, *Bull. Mater. Sci.*, Vol. 28, No. 6pp. 565–569, Indian Academy of Sciences.
- QinetiQ, 2004, "Macro-Scale Multi Component Material in Fire", Warrington Fire Research/QinetiQ Proprietary, British.
- Ramakrishna H.V., Priya S.P., and Rai S.K., (2006). Effect of Fly Ash Content on Impact, Compression, and Water Absorption Properties of Epoxy Toughened with Epoxy Phenol Cashew Nut Shell Liquid–Fly Ash Composites, *Journal of Reinforced Plastics and Composites*, Vol. 25, No. 5, pp. 455 – 462. www.sagepub.com
- Reinforced Plastics Magazine*, 2002, "Fire-Safe composite for mass transit vehicles", *Reinforced Plastics Magazine*, Vol 46.
- Sanadi A.r., Prasad S.V. dan Rohatgi P.K., 1986. "Sunhemp Fibre-Reinforced Polyester", *Journal of Materials Science* 21, pp. 4299-4304, UK.
- Surdia T. and Saito Y., 2000. "Intoduction to Rngineering Materials", Pradnya Paramita, Jakarta, Indonesia.
- Svehlova, V. and Poloucek, E. (1987). *Angew. Makromol. Chem.*, 153(2505): 197.