

# On The Physico-Mechanics, Thermal and Microstructure Properties of Hybrid Composite Epoxy-Geopolymer for Geothermal Pipe Application

Ira Firawati<sup>1,\*</sup>, Jasruddin<sup>1</sup>, and Subaer<sup>1</sup>

<sup>1</sup>Laboratorium Fisika Material, FMIPA, Universitas Negeri Makassar Jalan Daeng Tata Raya, Makassar 90223, Indonesia

**Abstract.** The objective of this study is to determine the effect of epoxy resin on the physico-mechanics, thermal and microstructure properties of geopolymers hybrid composites for geothermal pipe application. Hybrid composite epoxy-geopolymers pipes were produced through alkali activation method of class-C fly ash and epoxy resin. The mass of epoxy-resin was varied relative to the mass of fly ash namely 0% (SPG01), 5% (SPG02), 10% (SPG03), 15% (SPG04), and 20% (SPG05). The resulting materials were stored in open air for 28 days before conducting any measurements. The densities of the product composites were measured before and after the samples immersed in boiling water for 3 hours. The mechanical strength of the resulting geothermal pipes was measured by using splitting tensile measurement. The thermal properties of the pipes were measured by means of thermal conductivity measurement, differential scanning calorimetry (DSC) and fire resistance measurements. The chemical resistance was measured by immersing the samples into 1M H<sub>2</sub>SO<sub>4</sub> solution for 4 days. The microstructure properties of the resulting materials were examined by using x-ray diffraction (XRD) and Scanning Electron Microscopy-Energy Dispersive Spectroscopy (SEM-EDS). The results of this study showed that hybrid composite epoxy-geopolymers SPG02 and SPG03 are suitable to be applied as geothermal pipes.

## 1 Introduction

The needs for energy consumption is increasing rapidly every year and forcing new efforts to search for new and renewable energy resources [1]. One of the energy resources which readily available in many countries, including Indonesia is geothermal. Kamojang in West Java Province is one of the geothermal sites in Indonesia, which releases hydrothermal fluid with a temperature between 180 °C to 220 °C [2]. The hydrothermal fluid moves upward and reaches the surface of the earth after passing the drill of impermeable stones [3].

---

\* Corresponding author: ira.firawati28@gmail.com

The hydrothermal fluid requires heat and chemical corrosion resistant pipes known as geothermal pipes [4]. Geopolymer is inorganic polymer which has good mechanical properties, excellent fire and heat resistant and able to stand on strong chemical attacks [5-8]. Geopolymers can be synthesized from pure aluminosilicate minerals such as kaolinite and clays or industrial waste such as fly ash, furnace slag and red mud [9,10].

Fly ash is an inorganic material comprising of active  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  species as a result of coal burning in power plants and has been used as geopolymers raw material for many years [10, 11, 12]. Fly ash is abundantly available and categorized as dangerous waste and hence requires careful and thorough handling. Data from the Ministry of Environment shows that in 2006, Indonesia produced 52.2 tons fly ash per day and bottom ash as high as 5.8 tons per day. This figure is increasing in recent years [13, 14].

Geopolymers can be fabricated as a hybrid composite through the addition of certain organic material into the geopolymers paste such as commercial epoxy-resin [15, 16]. The used of phenolic resin and Mekpo hardener for example was found to improve the heat resistance of producing geopolymers. Beside that epoxy-resin has been used in space craft industries to develop fire resistance exterior composites [16].

## **2 Experimental**

Geopolymers were produced from class C-fly ash activated with alkali solution and mixed manually until the mixture was homogenous. Epoxy-resin (Union®) was added to the geopolymer paste by varying its mass relative to the mass of fly ash. The samples destination were SPG01 (0% epoxy-resin), SPG02 (5% epoxy-resin), SPG03 (10% epoxy resin), SPG04 (15% epoxy-resin), and SPG05 (20% epoxy resin). The mixture was molded to produce pipe shaped hybrid composite samples having an outer diameter of 4.3 cm and inner diameter of 3.3 cm. The samples were cured at 70°C for 2 hours before remolded. The produce composites were then stored in open air for 28 days before any measurements were performed [17]. The splitting tensile measurements were conducted by using Tokyo Testing Machine (TTM) with a maximum load of 1000 kN. The chemical resistance of the sample was measured by immersing the sample into 1M  $\text{H}_2\text{SO}_4$  solution for 4 days, the length of the time was chosen to be enough for measurement. Thermal properties of the samples were examined through thermal conductivity measurement, differential scanning calorimetry (DSC) (Perkin Elmer DSC 4000 series), and fire resistance by using torcher flame 1500°C for 30 minutes. The microstructures of the raw and produced materials were examined by using Mini-Flex II x-ray diffraction (XRD) and Tescan SB3 Scanning Electron Microscopy-Energy Dispersive Spectroscopy (SEM-EDS).

## **3 Results and discussion**

Figure 1 shows examples of hybrid composites, epoxy-geopolymer produced in this research with a length of 9.0 cm and prepared for various measurements. It can be seen that the surface of all samples is smooth, shining and free from apparent pores.



**Fig. 1.** Samples of hybrid composites, epoxy-geopolymer pipes

Table 1 shows the density of the samples before and after immersing them into boiling water for 3 hours. It can be seen that the density of hybrid composite epoxy resin-geopolymer is lower than pure geopolymers (SPG01). It was also observed that there were no physical damage or cracks occurred on all samples after 3 hours in boiling water although their densities were slightly increased except for sample SPG05.

**Table 1.** The density, splitting tensile strength and thermal conductivity of hybrid composites, epoxy-geopolymer samples

Sample	Density (g/cm <sup>3</sup> )		Splitting Tensile Strength (MPa)	Thermal Conductivity (W/m)
	Before soaked in boiling water	After soaked in boiling water		
SPG01	2.13	2.24	2.56	0.34
SPG02	1.96	2.02	3.33	0.32
SPG03	1.96	1.95	2.22	0.31
SPG04	1.88	1.92	2.22	0.31
SPG05	1.91	1.87	1.91	0.33

The splitting tensile strength of the samples was measured horizontally by using the following equation [18].

$$P = \frac{2F}{d \times L} \tag{1}$$

Where, F is applied force (N), d is the thickness of the pipe (mm), and L is the length of the pipe (mm).

The measuring results showed that best splitting tensile strength of the samples is 3.33 MPa for sample SPG02 with the addition of 5% epoxy resin relative the mass of fly ash. The splitting tensile strength of the rest of the samples decreases as the mass of epoxy resin increase. These results were expected since epoxy resin is an organic material with low mechanical strength. Similar measurement has been conducted by [19] and achieved the splitting tensile strength for their samples 1.10 MPa.

The thermal conductivity (k) measurements were performed by using a steady state method with the following equation [8],

$$k = \frac{Q \times l}{A \times \Delta T} \quad (2)$$

Where, Q is heat energy (W), l is length (mm), A is cross section (mm<sup>2</sup>), and ΔT is temperature difference.

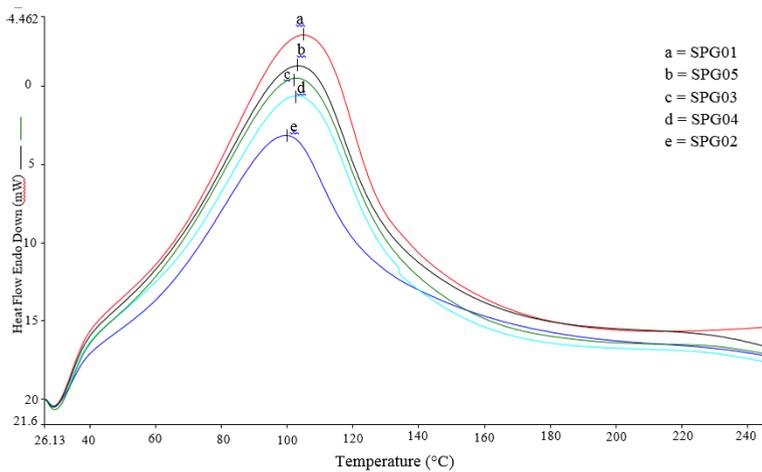
Table 1 shows that the thermal conductivity of all samples ranges from 0.31 to 0.33 W/mK which means that the ability of the material to conduct heat is low. The thermal conductivity of geopolymer can be tailored depending on raw material, chemical composition of alkali, type and size of aggregate (minerals) and the volume of pores [10].

The acid resistance was examined by soaking the sample into 1 M H<sub>2</sub>SO<sub>4</sub>. Figure 2 shows the picture of the sample after 4 days. Thin layer of epoxy resin appeared to peel out from SPG04 and SPG05 while other samples remain physically unchanged. These results indicate that the maximum addition of epoxy-resin into the network of geopolymer in order to stand the acid attack was 15%.



**Fig. 2.** The condition of hybrid composites, epoxy-geopolymer after soaking in 1 M H<sub>2</sub>SO<sub>4</sub> solution for 4 days

Differential Scanning Calorimetry (DSC) measurements were performed to study the nature and the ability of the composites to absorb or release heat from its network. The measurements were conducted at a temperature range between 30 to 400°C with a heating rate 20°C/minute. Figure 3 is the results of DSC measurements for all samples showing a single broad exothermic curve. The peaks of exothermic curve and the enthalpy (ΔH) decrease as the mass of epoxy resin increase. This indicates that the presence of epoxy resin reduces the ability of the composites to inhibit heat and fire.

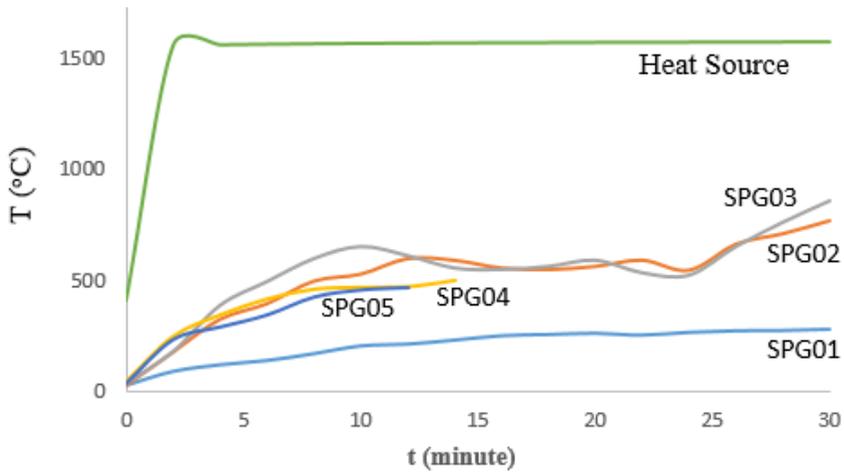


**Fig. 3.** DSC results of as-prepared hybrid composite epoxy-geopolymers

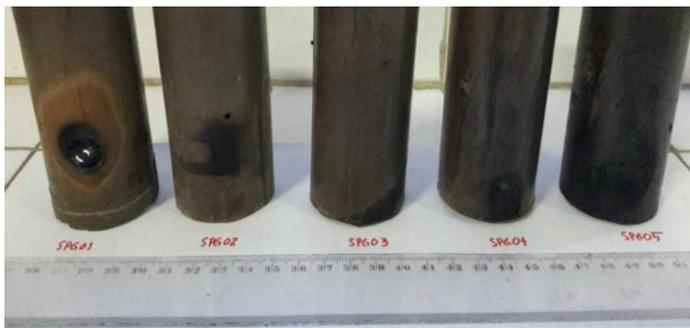
**Table 2.** The result of DSC of as-prepared hybrid composite epoxy- geopolymers

Sample	Peak ( $^{\circ}\text{C}$ )	Peak Height (mW)	$\Delta H$ (J/g)
SPG01 (a)	108.80	-18.5412	-157.089
SPG05 (b)	103.43	-16.3115	-133.647
SPG03 (c)	102.75	-15.6946	-123.202
SPG04 (d)	102.72	-15.2520	-121.717
SPG02 (e)	99.67	-15.2252	-108.911

Figure 4 shows the results of fire resistant measurement of the composite. The measurements were performed by using a torcher flame reaching  $1500^{\circ}\text{C}$  in a few minutes. The surface of the samples were exposed to fire for 30 minutes and a thermocouple was inserted into the inner diameter of the sample to register the temperature inside the pipe. The results showed that sample SPG01, SPG02, and SPG03 were able to resist the temperature between  $200 - 800^{\circ}\text{C}$  without burning or significant physical damage. Sample SPG04 and SPG05 which contain 15% and 20% of epoxy-resin were caught on fire and released the smoke at temperature around  $400^{\circ}\text{C}$ . Figure 5 shows the photograph of the samples after fire resistance measurements. It can be seen that sample SPG01 which did not contain epoxy resin formed a new phase on its surface after 30 minutes exposed to fire. Sample SPG03 which contain 10% epoxy-resin was found to be the best composite produced for high temperature application. This sample showed to be excellent as fire and chemical resistance.

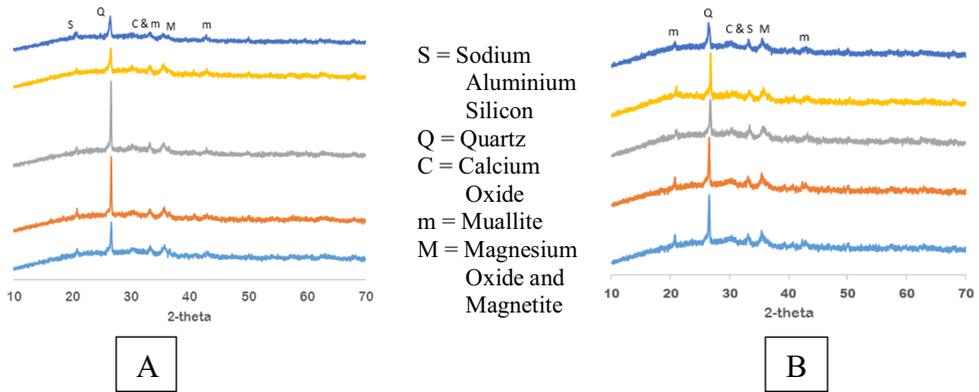


**Fig. 4.** Temperature versus time during fire resistance measurement of hybrid composites, epoxy-geopolymer



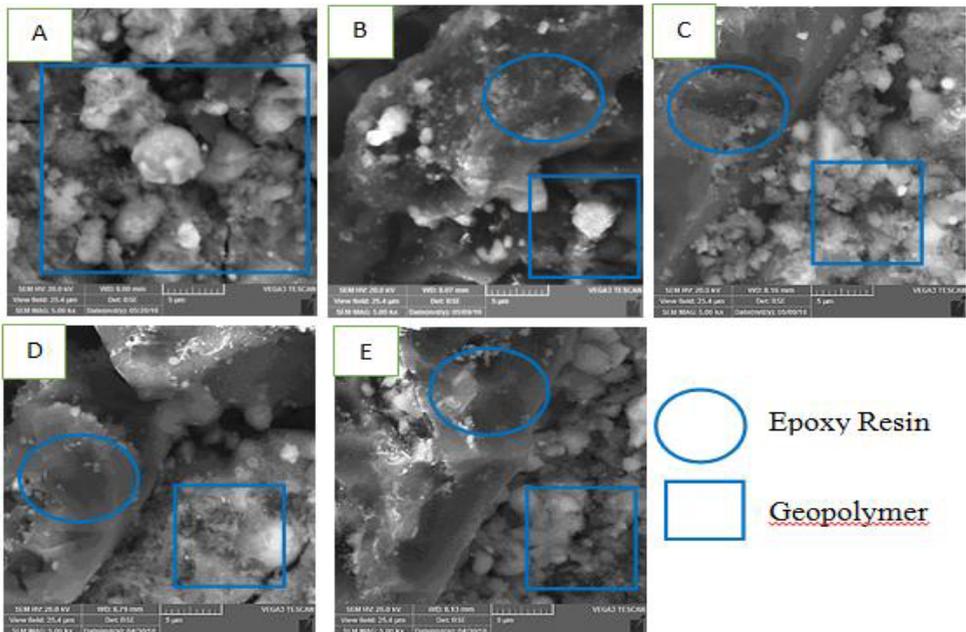
**Fig. 5.** Photograph of samples surface after fire testing

XRD examinations were performed for as prepared samples and after the samples were soaked in 1M  $H_2SO_4$  solution for 4 days. This was intended to examine the formation of any new compound (phase) due to strong acid attack on the surface of the samples. Diffractogram of the samples before and after soaking in the acid solution is shown in figure 6 (A) and (B), respectively. It can be seen clearly that XRD results did not show any discernible differences between the samples before and after acid attack. This result indicates although few layers of epoxy peeled out from the surface of the sample, the structure of polymeric network of hybrid composite epoxy-geopolymer did not change due to the intrusion of  $H_2SO_4$  solution, at least during the time scale of measurement.

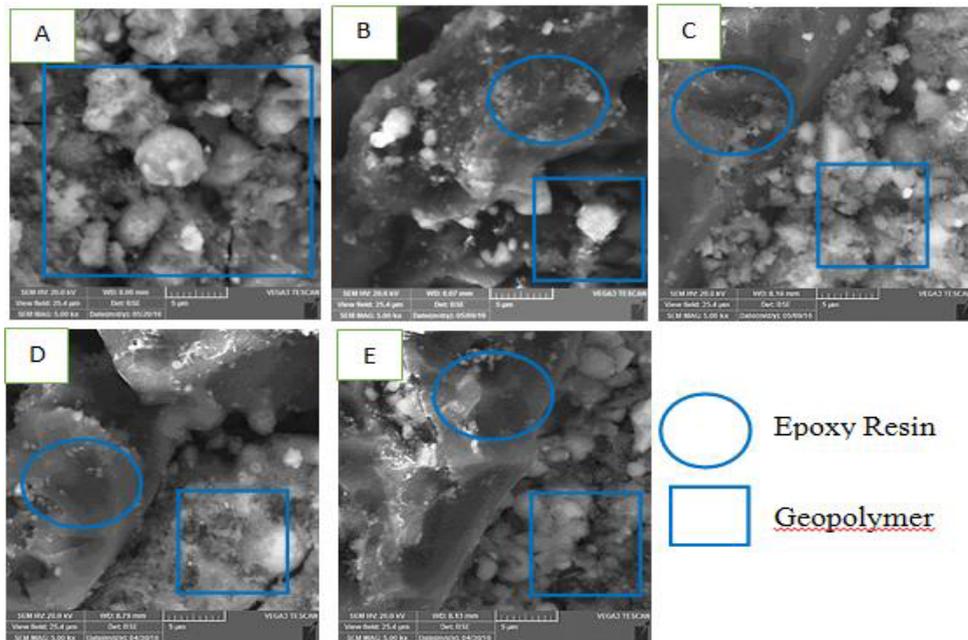


**Fig. 6.** The diffractogram of hybrid composite epoxy-geopolymer; (A) before, (B) after soaking in 1 M H<sub>2</sub>SO<sub>4</sub> solution for 4 days

Microstructure examination of the samples before and after acid attack was performed by using SEM-EDS. Figure 7 shows SEM micrographs for as-prepared samples. The micrographs shows that epoxy resin create a strong mechanical bond with the geopolymer matrix. The morphology of the composites also shows the presence of substantial unreacted fly ash particles which is normally observed on geopolymers made from fly ash. Figure 8 shows the surface morphology of the sample after immersing in 1M H<sub>2</sub>SO<sub>4</sub> solution for 4 days. It can be seen that the attack of strong acid did not form any new phase on the surface of the sample. The surface morphology of all samples remains similar to those of as-prepared sample. This indicates that at the time scale of the measurement, H<sub>2</sub>SO<sub>4</sub> did not affect the integrity of geopolymer structure. Further study needs to be conducted by immersing the samples into a strong acid solution for much longer time.



**Fig. 7.** SEM image of hybrid composite epoxy-geopolymer before soaking in 1 M H<sub>2</sub>SO<sub>4</sub> solution for 4 days (A)SPG01 (B)SPG02 (C)SPG03 (D)SPG04 (E)SPG05



**Fig. 8.** SEM image of hybrid composite epoxy-geopolymer after soaking in 1 M H<sub>2</sub>SO<sub>4</sub> solution for 4 days (A)SPG01 (B) SPG02 (C)SPG03 (D)SPG04 (E)SPG05

## 4 Conclusion

Hybrid composite epoxy resin-geopolymers have been successfully produced and characterized. The splitting tensile strength of the composite reached 3.33 MPa for the sample contain 5% epoxy resin. The fire resistance, thermal conductivity and DSC measurements showed that (figure 3) the thermal properties of the composites were suitable to be applied as geothermal pipe. The microstructure examinations for as-prepared samples and after immersing the samples into 1M H<sub>2</sub>SO<sub>4</sub> solutions for 4 days showed that the produced composites remain stable and did not show the formation of a new phase or physical damage. The results of this study indicate that hybrid composite epoxy resin-geopolymer offer a high potential to be applied as geothermal pipe.

## References

1. ESDMMAG. *Eksplorasi Panas Bumi; Potensi Energi Baru Terbaharukan; Upaya Mengentaskan Kemiskinan Energi*. Media Komunikasi Kementerian Energi dan Sumber Daya Mineral (2012)
2. D. Yudiantoro, S. Yuwono, I. Takashima, D. Ishiyama, Y. Kamah, J. Hutabarat, *Indonesian Journal on Geoscience*, **8** (2013)
3. S. Suparno, Universitas Indonesia, Jakarta (2009)
4. Bastarina, Annisa Destavia, *Energi Panas Bumi A Present from the Heart of The Earth*. Universitas Indonesia. **1** (2008)
5. A. Haris, A. Irhamsyah, J.J. Ekaputri, *Mater. Sci. Forum.*, **841**, 170 (2016)
6. V. Sreevidya, R. Anuradha, D. Dinakar, R. Venkatasubramani, *Int. J. Eng. Sci. Res. Technol.*, **4** (2012)

7. Luhar, Salmabanu, Urvashi Khandelwal, Salmabanu Luhar, *Int. J. Eng. Res. Ind. Appl.*, **8**, 17 (2015)
8. A. Fernandez Jimenez, A. Palomo, *Cem. Concr. Res.*, **35**, 1984 (2005)
9. I. Nikolic, D. Durovic, R. Zejak, L. Karanovic, M. Tadic, D. Blecic, V.R. Radmilovic, *J. Serb. Chem. Soc.*, **78**, 851 (2013)
10. Subaer, *Pengantar Fisika Geopolimer*. Makasar: Badan Penerbit UNM. (2014)
11. Y. Yusuf, Z. Zuki, G. Refnita, *Pengaruh Penambahan Abu Terbang (Fly Ash) Terhadap Kuat Tekan Mortar Semen Tipe PCC Serta Analisis Air Laut yang Digunakan untuk Perendaman*, Prosiding SEMIRATA. **1** (2013)
12. K. Diharjo, B. Kusharjanta, R.A.P. Tarigan, A.R. Andhika, *Pengaruh Kandungan dan Ukuran Serbuk Genteng Sokka terhadap Ketahanan Bakar Komposit Geopolimer*. *Rekayasa Mesin*, **4**, 27-34 (2013)
13. J. Setiaka, I. Ulfan, N. Widiastuti, *Adsorpsi Ion Logam Cu (II) dalam Larutan pada Abu Dasar Batubara Menggunakan Metode Kolom*. Prosiding Tugas Akhir. (2010)
14. A. Lasryza, D. Sawitri, *Pemanfaatan Fly Ash Batubara Sebagai Adsorben Emisi Gas CO Pada Kendaraan Bermotor*. *Jurnal Teknik Pomits*. **1** (2012)
15. G. Roviello, L. Ricciotti, C. Ferone, F. Colangelo, R. Cioffi, O. Tarallo, *Materials*, **6**, 3943 (2013)
16. C.M. Thomson, Jr. Smith, J.W. Connell, P.W. Hergenrother, *Flame Retardant Epoxy Resin*. NASA Research Centre. (2007)
17. B.S. Nasional, *Tata Cara Perhitungan Struktur Beton untuk Bangunan Gedung*. **SNI 03-2847-2002** (2002)
18. SNI (Standar Nasional Indonesia). *Metode Kuat Tarik Belah Beton*. **SNI 03-2492-2002** (2001)
19. A. Elkadi, J. Van Mier, *Int. J. Fract.*, **1**, 55 (2004)