IOP Conference Series:
Materials Science and
Engineering

Bonding and Phases Analysis of Geopolymer Materials

To cite this article: Nor Shida Dalila Mohd Azhar *et al* 2020 *IOP Conf. Ser.: Mater. Sci. Eng.* **957** 012052

Bonding and Phases Analysis of Geopolymer Materials

Nor Shida Dalila Mohd Azhar[1,*], *Farah Farhana* Zainal[1], *Mohd Mustafa Al Bakri* Abdullah[1]

[1]Center of Excellence Geopolymer and Green Technology (CEGeoGTech), School of Materials Engineering, Universiti Malaysia Perlis (UniMAP), P.O. Box 77, D/A Pejabat Pos Besar, 01000 Kangar, Perlis, Malaysia

Abstract. This paper presents the bondings and mineral phases exist in geopolymer material consist of raw fly ash, raw metakaolin and also fly ash and metakaolin blend geopolymer. Fly ash used is a waste materials from Lumut power plant by combusting coal while metakaolin was obtained by calcining the kaolin at 750 ºC for 4 hours. Alkaline activators that act as binder were sodium silicate (Na_2SiO_3) solution and sodium hydroxide (NaOH) solution with the ratio of Na_2SiO_3/NaOH is 2.5 to 1. The samples undergo Functional Group Identification (FTIR) and Phase Analysis (XRD). FTIR results show that after geopolymerization process, the band shifted to lower wavenumber (776 cm^{-1}) due to interaction of Al atoms in the silicate geopolymer network because of higher cross-linking. XRD results show mineral phases of quartz and mullite in geopolymer after geopolymerization process and it can be said that not all mineral phases from raw material participating in formation of geopolymer matrix.

1 Introduction

The term geopolymer refers to inorganic aluminosilicate polymers. These inorganic polymers are generally obtained by pozzolanic reaction between aluminosilicate and basic alkaline solution [1]. Pozzolan materials that commonly used for the geopolymer synthesis are fly ash, blast furnace slag or metakaolin [2]. The chemical composition of fly ash and metakaolin are varies usually leads to different physical and mechanical properties from one aluminosilicate material to another [3].

Fly ash is the main product of coal combustion which was vastly used in many industries especially in construction area of interest. This is due to their good pozzolanic and cementitious properties [4,5]. Fly ash is a by-product of coal combustion that contains mainly quartz, hematite, mullite, and amorphous, that have high levels of silicate and aluminate.

On the other hand, metakaolin also have good pozzolanic properties where the calcination condition of kaolin affect the characteristics of resulting inorganic aluminosilicate polymers [6]. Based on previous studies state that geopolymers with optimal characteristics are obtained and found at temperature range 700 °C to 800 °C [1, 5]. Castelein et al investigated

---

[*] Corresponding author: norshidadalila@gmail.com

the effect of kaolin to reveal its influence on the formation of mullite also neighboring kaolinite [1, 5]. Metakaolin is prepared by dehydroxylation process of kaolin to remove the chemically bonded hydroxyl ions [6-8].

Because of SiO_2 and Al_2O_3 are the main chemical constituents of fly ash and metakaolin, they are considered as important raw materials for geopolymer [9, 10]. This paper will discussed the bonding and mineral phases between the raw material and the fly ash-metakaolin blend geopolymer by using FTIR and XRD analysis.

2 Methodology

2.1 Materials Selection and Samples Preparation

The materials that used to prepare the geopolymer paste was made from the mixture of fly ash and metakaolin. Fly ash raw used in this study was obtained from Lumut Power Plant, Perak, Malaysia while metakaolins used in this study were obtained from kaolin provided by the from Associated Kaolin Industries Sdn. Bhd. Malaysia. Before using the kaolin, this material was enriched with kaolinite and calcinated at 750 °C in electric muffle furnace [11]. Calcination process lasted 4 hours at the rates of calcination 10 °C/min.

The alkaline solution was prepared by mixing sodium silicate and sodium hydroxide solution (12 M). The sodium hydroxide solution was obtained by dissolving sodium hydroxide pellets with a purity of 99% in distilled water and kept it at room temperature for 24 hours [12].

2.2 Functional Group Identification (FTIR)

FTIR spectroscopy is an analysis method which searches for the major reaction zones of Si-O and Al-O geopolymers. Functional groups shifting of the materials were identified using spectrometer. The specimens was powdered samples scanned from 4000-600 cm^{-1} .

2.3 Phase Analysis (XRD)

The phase analysis was conducted by using Shimadzu X-ray diffractometer. Specimen for analysis also in powder form. X-ray diffraction are very useful to detect structural changes of the material. Characteristics peaks in the XRD pattern of geopolymer materials commonly show quartz, mullite, hematite, magnetite also silliminite. The XRD pattern was analyzed using HighScore Plus software.

3 Results and Discussion

3.1 FTIR Analysis

Fig.1 and Table 1 represent the FTIR spectra of raw fly ash and metakaolin also fly ash and metakaolin blend geopolymers. From Fig.1, the infrared spectrum of fly ash shows the main absorption band occurred at 959-1020 cm^{-1}.These spectrum band are corresponding to symmetric stretching vibration of Si-O-Si and Si-O-Al bonds of SiO_4 and AlO_4 tetrahedrons of the geopolymer network [12]. However, different to fly ash, the FTIR spectra of the metakaolin exhibited absorption bands at 3624 cm^{-1} which are the result of the network of kaolinite.

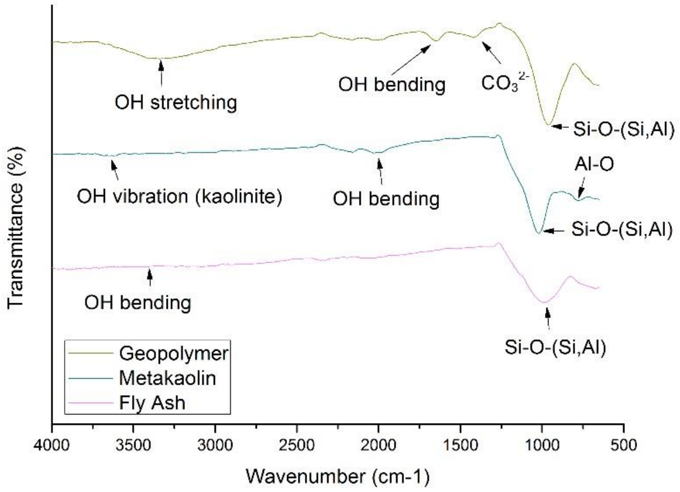

Fig. 1. FTIR spectra of fly ash, metakaolin and Geopolymer.

Table 1. Assignment of main FTIR bands.

Band	Assignment
3624 cm^{-1}	Vibration of OH^- groups of kaolinite
3340-3480 cm^{-1}	OH^- stretching vibration
1648,2020 cm^{-1}	OH^- bending vibration of H-O-H
1390-1420 cm^{-1}	Stretching vibration of CO_3^{2-}
1020 cm^{-1}	Stretching vibration of Si-O-Si and Si-O-Al
959 cm^{-1}	Stretching vibration of Si-O-Si and Si-O-Al
776 cm^{-1}	4-coordinated Al-O bond

In FTIR spectra of geopolymers (Fig. 1) the absorption bands at 3340–3480 ,2020 and 1648 cm^{-1} corresponded to the stretching and bending vibrations of O-H and H-O-H bonds of water molecules [13]. The absorption band at 1390 –1420 cm^{-1} also refer to stretching vibrations of C-O bond of CO_3^{2-} ion which results from the reaction of atmospheric CO_2 with residual sodium content [13]. After geopolymerization process, the band shifted to lower wavenumber (776 cm^{-1}) due to the silicate geopolymer network of Al atoms and it is also indicated formation of higher cross-linking and larger molecular [12].

3.2 XRD Analysis

Fig.2 presents the XRD diffractograms of fly ash, metakaolin and geopolymer. Fly ash and metakaolin was used as primary binder materials. Fly ash shows a range at 2θ between 15°

to 36° indicates the presence of amorphous phase [14]. According to the obtained patterns, the raw fly ash mainly contain of quartz, mullite and hematite. Hematite is the mineral forms of iron oxides, while quartz and mullite indicate the presence of silica and alumina.

Metakaolin showed that the material is rich in kaolinite, quartz, muscovite and hematite peaks. At 2θ,the pattern shows a broad area located in the range 12° to 45°. After the activation process, the crystalline phases were dissolved in the alkaline solution and the aluminosilicate phase was formed in the surface of blend fly ash and metakaolin paste by geopolymerization reaction [15].

However, the differences between raw materials and geopolymer sample show certain changes with the peak representing the amorphous phase. Mineral phases of quartz, mullite were still present in geopolymer but with reduced in intensity and also there were sillimanite phases presence. These results reflected not all mineral phases are reacting in the geopolymerization process.

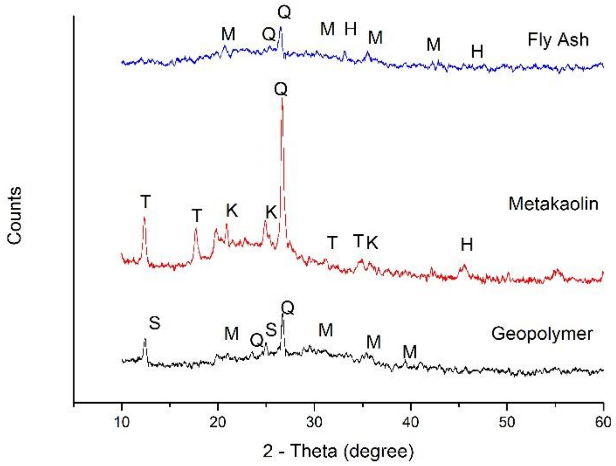

Fig. 2. XRD diffractograms of fly ash, metakaolin and geopolymer (Q – Quartz (PDF no. 01-083-2468), M – Mullite (PDF no. 01-074-2419), S – Sillimanite (PDF no. 01-084-0983), H – Hematite (PDF no. 01-086-0550), T – Muscovite (PDF no. 00-046-1311), K – Kaolinite (PDF no. 00-029-1311)).

4 Conclusions

Thus, as for XRD and FTIR analysis showed that after geopolymerization process, there will be slightly changes in structural phases and the functional group.

The authors would like to thank the staffs of Center of Excellence Geopolymer & Green Technology (CEGeoGTech), School of Materials Engineering, Universiti Malaysia Perlis (UniMAP) for their involvement in the research. This work was supported and funded by the Fundamental Research Grant Scheme (FRGS) under a grant number of **FRGS/1/2018/TK06/UNIMAP/02/2** from the Ministry of Education Malaysia.

References

1. B. K. Diffo, A. Elimbi, M. Cyr, J. D. Manga, and H. T. Kouamo, J Asian Ceram. Soc. **3(1)**, 130–138 (2015)
2. Ding, Y.-C., Chang, W.-H., Lee, W.-H., Wang, S., & Cheng, T.-W. Struct Cooncrere, **19(5),** 1521–1528 (2018)
3. S. N. Fifinatasha *et al.*, Adv. Environ. Biol, **7**(12) 28–29 (2013)
4. D. M. J. Sumajouw, D. Hardjito, S. E. Wallah, and B. V. Rangan, J. Mater. Sci*,* **42**(9), 3124–3130 (2011)
5. Luna-Galiano, Y., Cornejo, A., Leiva, C., Vilches, L. F., & Fernández-Pereira, C. Mater Construcc**, *65*(319)** (2015)
6. Boonanunwong, P., Keawpapasson, P., Tippayasam, C., Thavorniti, P., Chindaprasirt, P., & Chaysuwan, D. Key. Eng. Mater. **690,** 179–186 (2016)
7. S.E. Wallah and B.V Rangan, 220-225 (2013)
8. J.M. Khatib, O. Baalbaki, A.A Elkordi, 493–511 (2018)
9. Ma, C.-K., Awang, A. Z., & Omar, W. Constr Build Mater **186,** 90–102 (2018)
10. Zainal, F. F., Hussin, K., Rahmat, A., Abdullah, M. M. A. B., & Sandu, A. V. Appl. Mech. Mater. **754-755**, 892–896 (2015)
11. Diffo, B. K., Elimbi, A., Cyr, M., Manga, J. D., & Kouamo, H. T. J. Asian Ceram. Soc. **3(1),** 130–138 (2015)
12. Cheng-Yong, H., Yun-Ming, L., Abdullah, M. M. A. B., & Hussin, K. Sci. Rep. **7(1)** (2017)
13. N.A. Jaya, L. Y. Ming, H. C. Yong, M. M. A Bakri, " (2018)
14. Kljajević, L. M., Nenadović, S. S., Nenadović, M. T., Bundaleski, N. K., Todorović, B. Ž., Pavlović, V. B., & Rakočević, Z. L. Ceram Int. **43(9)**, 6700–6708 (2017)
15. M. E. Alouani, S. Alehyen, M. E. Achouri, and M. H. Taibi, J. Chem, 1–14, (2019)