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PREPARATION AND CHARACTERIZATION OF CARBON NANOFIBERS / METAKAOLIN GEOPOLYMER BASED NANOCOMPOSITE

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Abstract: Geopolymer can be considered as a novel material and used as alternative to Ordinary Portland Cement (OPC) due to its environment friendly properties. In this study metakaolin-geopolymer reinforced with carbon nanofibers (CNFs) were prepared and characterized by using X-Ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscope (SEM). Metakaolin-geopolymer based nanocomposites were produced by mixing the alkaline activator contains of 10 M of sodium hydroxide (NaOH) and 8M of sodium silicate (Na_2SiO_3) and subsequently reinforced with 1.0 % of CNFs. The CNFs / Metakaolin Geopolymer based nanocomposites later on sintered at temperature of 700 ^{0}C, 800 ^{0}C, 900 ^{0}C and 1000 ^{0}C. Results from XRD showed that nepheline phases are formed at temperature 900 ^{0}C and 1000 ^{0}C. It was also observed that the crystallinity of geopolymer has been improved. FTIR analysis has exhibited strong bands of IR in range of 725 cm^{-1} to 1006 cm^{-1} which is ascribed to symmetric stretching of Si-O-Si and Si-O-Al. Based on SEM observation, the carbon nanofibers were not well dispersed within the geopolymer and this is probably due to the agglomeration of CNFs itself.
Keywords: metakaolin, geopolymer, carbon nanofibers.

Introduction

Geopolymers are amorphous to semi crystalline three-dimensional aluminosilicate polymers and this term was initially invented by Davidovits in the late 1970s (Davidovits, 1991). Geopolymer scan be synthesized by process called geopolymerisation. This process occurs in highly alkaline solution with aluminosilicate oxides and silicates (either solid or liquid) as the reactants. Geopolymers also can be formed by calcination of clay or coal fly ash with alkaline solution. At calcination temperature ranged between 600-800 ^{o}C, the kaolin which one of geopolymer material can be transformed into metakaol in (Sun et *al.,* 2014) as shown below:

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$$\underset{\text{kaolin}}{Al_2Si_2O_5(OH_4)} \xrightarrow{600\ \text{C}-800\ C} \underset{\text{metakaolin}}{Al_2O_3.2SiO_2} + 2H_2O$$

Geopolymers have received tremendous attention because it can be used as alternative to Ordinary Portland Cement (OPC), as it is environmental friendly with no emission of CO2 (Jumrat et. al., 2011)Other applications of metakaolinare as fire resistans materials, coatings and hydroceramics (Duxson et *al*., 2007).

Numerous studies have been carried out on the alkaline activation of metakaolin to produce geopolymer cement (Zhang et. *al.*, 2010; Zuhua et. *al.,* 2009; Rovnanik, 2010). The properties of geopolymers based nanocomposites containing different fibers such as basalt fiber (Uddin and Shaik, 2013) and carbon nanotubes (Saafi et. al, 2013) also had investigated. The properties of geopolymer-based nanocomposite (Uddin and Shaik, 2013) is improved by adding the mentioned fiber to its. However there are not many study related to geopolymer reinforced with carbon nanofibers (CNFs) reported. Carbon nanofibers belong to graphene family and grown by direct-current plasma-enhanced chemical vapor deposition (DC-PECVD). It often shows stacked cone structure and also known as herringbone or bamboo structure (Hafner et.*al.,* 2010) Promising superior characteristic is expected should CNFs is used as nanofiller in preparing the geopolymerbased nanocomposite due to their sought-after mechanical, thermal and electrical properties. In addition, CNFs is a cheaper than carbon nanotubes (CNTs), thus geopolymer based nanocomposites blended with CNFs could be produced at lower cost.

Therefore, this paper presents the preparation and characterization properties of CNFs / Metakaolin Geopolymer based nanocomposites at different sintering temperatures by using X-Ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscope (SEM).

Method

a) Sample Preparation

Metakaolin was produced by calcination of commercial kaolin at 800°C for 200 minutes. Metakaolin-based geopolymer was prepared by manually mixing the alkaline activator whichcontains of 10 M of sodium hydroxide (NaOH) and 8M of sodium silicate (Na_2SiO_3). This mixture was often used as alkaline solution (Lylord and Rangan, 2010).In this research, the ratio of NaOH and Na_2SiO_3 to form alkali activator was 1:1. The alkaline activators were prepared 24 hours prior to be mixed with metakaolin. The ratio of metakaolin and alkaline activator was 1:2. Carbon nanofibers (CNFs) that used in this study have electrical

conductivity more than 100 S/cm, with density was about 2.1 g/cm^3, outside diameter from 200 to 600 nm and length from 5 to 50 μmCNFs with 1.0% wt. were added into geopolymer slurry, stirred and poured into a mould after being fully mixed. After that, both mixtures were shakedto eliminate air bubbles before the curing process was done at 60^{0}C for 24 hours, which is the optimum temperature according to Housi et.*al* (2013). After curing, the CNFs/Metakaolin geopolymer based nanocomposites were pelleted and subsequently sintered at temperature of 700 ^{0}C, 800 ^{0}C, 900 ^{0}C and 1000 ^{0}C for 1 hour. Then the nanocomposites were left to cool down to room temperature.

b) Samples Characterization

X-ray diffraction (XRD) Philip PW 3040/60 was used to investigate the crystallinity structure and mineralogy of CNFs/Metakaolin Geoplymer based nanocomposite. The Spectroscopic analysis was performed by Fourier Transform Infra-Red Spectroscopy (FTIR) Model Perkin Elmer Spectrum 100. The band spectral was recorded in the range of 4000-650 cm^{-1}.The Scanning Electron Microscope Model Carl Zeiss MA10 was employed to examine the dispersion of carbon nanofiber within metakaolin geopolymer. Before scanning, samples were coated with gold by Sputter Auto Fine Coater.

Results and Discussion

a) X-Ray Diffraction

X-ray diffraction patterns of geopolymer-based nanocomposite at temperature of 700 ^{0}C, 800 ^{0}C, 900 ^{0}C and 1000 ^{0}Care presented in Figure 1. At 700 ^{0}C and 800 ^{0}C, the mostintense peak detected at 2Θ of 26.65^{o}and this is representing a quartz mineral. It can be seen that when geopolymer heated at 900 ^{0}C and 1000^{0}C and the intensity of quartz have decreased with temperature. This is in a good agreement with study conducted by A. El-Maghraby *et*. al, (2013) on metakaolin-based geopolymer where the intensity of quartz also found to be decreased when heated at 900^{0}C. Nepheline ($NaAlSiO_4$) was formed once subjected to 900 ^{0}C, whileat1000 ^{0}C a large number of nepheline peaks appeared as well. In contrast with samples sintered at lower temperature (700 ^{0}C and 800^{0}C), the nephelinephases have not be identified. Based on research studied by Rahier et *al.,* (2007), they found that geopolymer was completely transformed into nephelineafterheatingto1000 ^{0}C. The XRD data for minerals in nanocompositeis tabulated in Table 1.From the XRD patterns, it also can be observed that the crystallinity of geopolymerswas improved when sintering temperature increased. According to Liewet. *al*., (2012) the crystalline phases were beneficial to the mechanical properties of geopolymer.

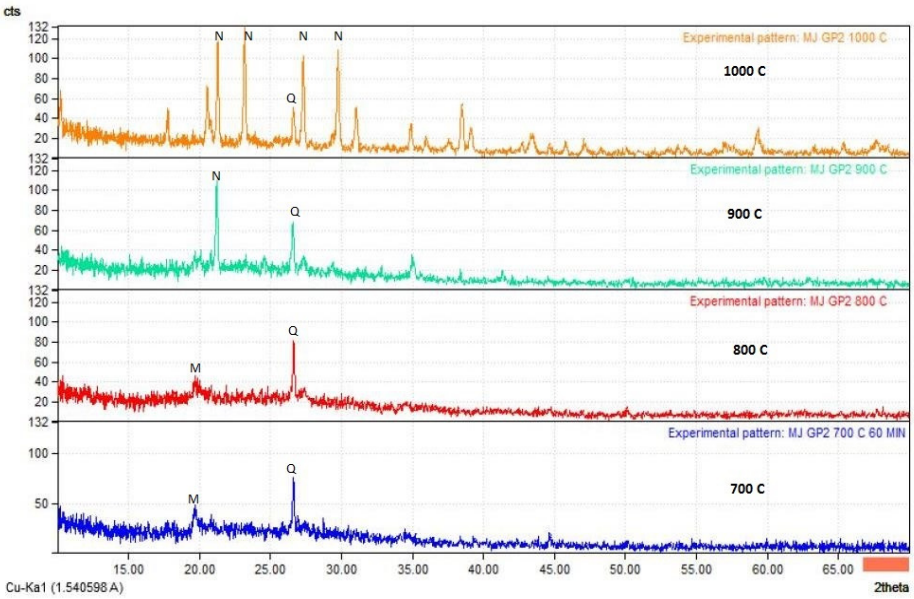

FIGURE 1. X-ray diffraction patterns of metakaolin geopolymer with 1.0-wt% of CNFs at various sintering temperatures.

TABLE 1. XRD data of minerals

Temperature (^{o}C)	**2-theta (2θ)**	***d*-spacing (Å)**	**Mineral**
700	19.61 26.59	4.52 3.35	Muscovite Quartz
800	19.83 26.59	4.47 3.34	Muscovite Quartz
900	21.21 26.57	4.18 3.35	Nepheline Quartz
1000	21.21 23.19 27.30 29.77 26.57	4.18 3.83 3.26 3.00 3.35	Nepheline Nepheline Nepheline Nepheline Quartz

b) FTIR Analysis

Series of the IR spectras of CNFs/Metakaolin geopolymer based nanocomposites after sintered at various temperatures are illustrated in Figure 2. The absorption bands of each nanocomposite are summarized in Table 2. In IR spectrum, all samples exhibit strong bands of symmetric stretching of Si-O-Si which recorded at 985 cm^{-1}(700^{o}C), 1010 cm^{-1}(800^{o}C), 992 cm^{-1}(900^{o}C) and 1006 cm^{-1} (1000^{o}C). Asymmetric stretching of Si-O-Alwas observed at 725cm^{-1}, 766cm^{-1} and 751cm^{-1}. According to Xu and Van Deventer (2000),

geopolymerisation involves a chemical reaction between various alumino-silicate oxides with silicates under highly alkaline conditions, yieldingpolymeric Si–O–Al–O bonds. However, Si-O-Alband was not detected in sample heated at 900°C. The broad bands IR spectra existed at 3166 cm^{-1} and 3577 cm^{-1}. These are ascribed to the OH- stretching and 3742 cm^{-1} which caused by O-H-O bending.

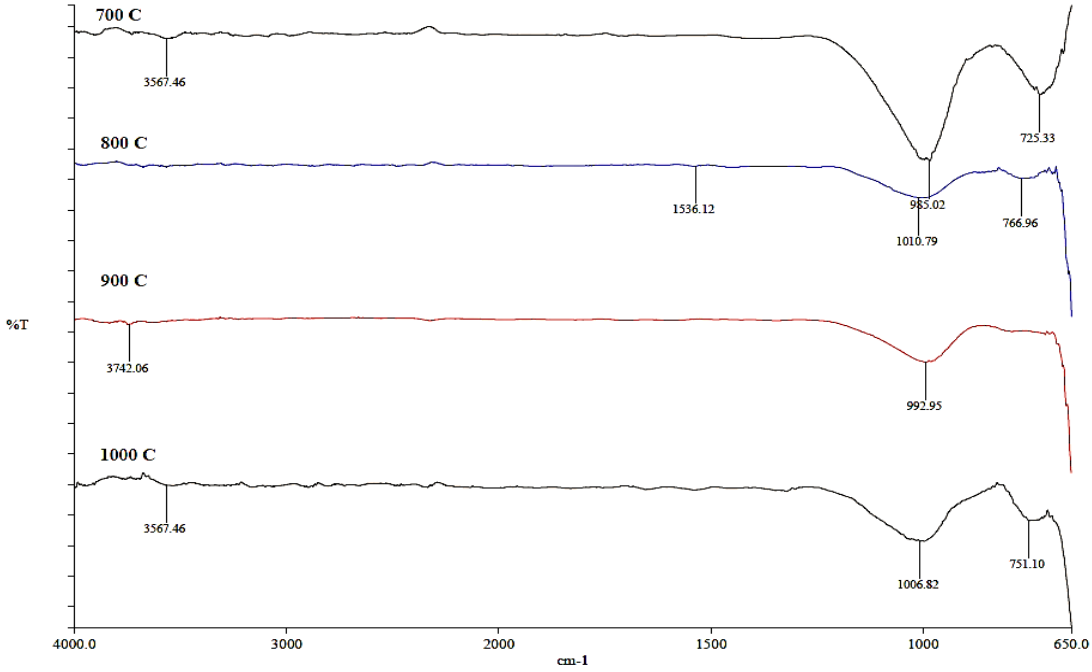

FIGURE 2. FTIR overlapping spectra of CNFs/Metakaolin geopolymer at various sintering temperature

TABLE 2. Bands of FTIR Spectra at different sintering temperatures

Temperature (°C)	Bands	Wavenumber (cm^{-1})
700	OH^- Si-O-Si Si-O-Al	3567 985 725
800	C-C Si-O-Si Si-O-Al	1536 1010 766
900	O-H-O Si-O-Si	3742 992
1000	OH^- Si-O-Si Si-O-Al	3567 1006 751

c) Microstructure Analysis

Figure 3(a)-(d) show SEM micrographs at 3000x magnification of geopolymer nanocomposites which sintered at 700 ^{0}C, 800 ^{0}C, 900 ^{0}C and 1000 ^{0}C, respectively. An image of pure carbon nanofibers observed through SEM is shown in Figure 4. Based on the SEM observation, the carbon nanofibers were not well dispersed within the geopolymer matrix probably due to the agglomeration of CNFs.

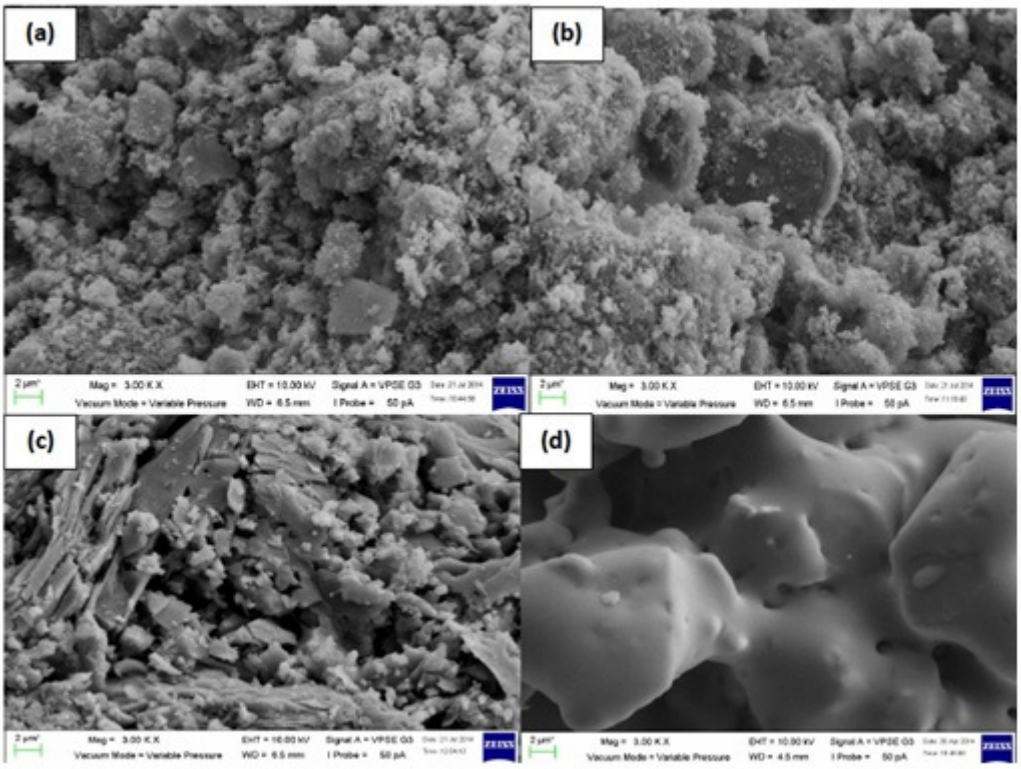

FIGURE 3. SEM micrographs of geopolymernanocomposites with 1.0% CNFs sintered at (a) 700°C (b) 800°C (c) 900°C and (d) 1000°C.

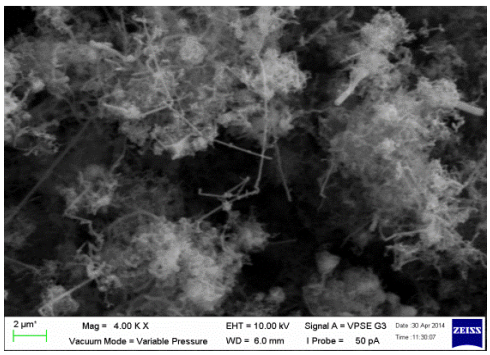

FIGURE 4. SEM observation shows the agglomeration of carbon nanofibers (CNFs)

Conclusion

Geopolymer based nanocomposite has successfully prepared and characterized. Based on the results, the crystallinity has been improved with temperature which showing that the best sintering temperature to produce geopolymer based nanocomposite with addition of 1.0% CNFs is 1000^{o}C. The geopolymerisation process has occurred as the bands of Si-O-Si and Si-O-Al have detected from FTIR analysis.

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References

[1] A. El-Maghraby, K.F. Khaled and Khaled M. Elsabawy. 2013. Formation of leucite crystals from metakaolin-based geopolymer using kaolin and bentonite. International Journal Chemical Science 11 **(2)**: 740-750

[2] Davidovits, J. 1991. Geopolymers: Inorganic polymeric new materials. *Journal of Thermal Analysis and Calorimetry,* **37**, 1633–1656.

[3] Duxson, P., Ferna´ndez-Jime´nez, A., Provis, J.L., Lukey, G.C., Palomo, A., Van Deventer, J.S.J., 2007c. Geopolymer technology: the current state of the art. *Journal of Materials Science.***42**: 2917–2933.

[4] Hafner, J., Chen, E., Lal, R., and Jin, S., 2010. Springer Handbook of Nanotechnology: *General and Special Probes in Scanning Microscopies*. Springer, London: 624-626

[5] Housi, A.D. Lecomte-Nana, G.L, Djeteli, G, Blanchart, P. 2013. Kaolin-based geopolymers: Effect on mechanical activation and curing process. *Construction Building Material*, **42**:105-113.

[6] Xu, H and van Deventer, J.S.J, 2000. The Geopolymerisation of Alumino-Silicate Minerals. *International Journal of Mineral Processing*, **59(3):**247-266.

[7] Jumrat, S., Chatveera, B. and Rattanadecho, P. 2011. Dielectric properties and temperature profile of fly ash-based geopolymer mortar. *International Communications in Heat and Mass Transfer*, **38**: 242-248

[8] Lloyd, N.A. and B.V. Rangan. 2010. Geopolymer Concrete with Fly Ash. in *Second International Conference on Sustainable Construction Materials and Technologies* Universita Politecnicadelle Marche, Ancona, Italy.

[9] Liew, Y.M., Kamarudin, H., Mustafa Al Bakri, A.M., Luqman, M., KhairulNizar, I., C.M. Ruzaidi, C.M., Heah, C.Y, 2012. Processing and Characterization of calcined kaolin cement powder. *Construction Building Materials*, **30**: 794-802

[10] Rahier, H., Wastiels, J., Biesemans, M., Willem, R., van Assche, G., van Mele, B. 2007. Reaction, mechanism, kinetics and high temperature transformations of geopolymers. *Journal of Material Science*, **42** :2982-2996

[11] Rovnanik, P., 2010. Effect of Curing Temperature on the Development of Hard Structure of Metakaolin-Based Geopolymer. *Construction and Building Materials*, **24**: 1176-1183.

[12] Saafi, M., Andrew, K., Tang, P.K., McGhon, D., Taylor, S., Rahman, M., Yang, S and X. Zhou, X., 2013. Multifunctional properties of carbon nanotube/fly ash geopolymernanocomposites. *Construction and Building Materials*, **49**:46-55.

[13] Sun, T., Chen, J., Lei, X., Zhou, C., 2014. Detoxification and Immobilization of Chromite Ore Processing Residue with Metakaolin-Based Geopolymer. *Journal of Environmental Chemical Engineering*, **2:** 304-309.

[14] Uddin F, Shaik. A. 2013. Review of the mechanical properties of short fibers reinforced geopolymer composites. *Construction and Building Materials*, **43**:37-49.

[15] Zhang, Y.S., W. Sun and L. Zongjin, 2010. Composition Design and Microstructural Characterization of Calcined Kaolin-Based Geopolymer Cement. Applied Clay Science, **47**: 271-275.

[16] Zuhua, Z, Xiaoa, Y, Huajuna, H, Yue C. 2009. Role of Water in the Synthesis of Calcined Kaolin-based Geopolymer. *Applied Clay Science*, **43 (2)**: 218-223.