

Characteristics of Novel Geopolymer Composites Synthesized from Red Mud and Diatomaceous Earth in Autoclave Conditions without Using Alkaline Activators

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Abstract

The geopolymer-based materials have been studied and developed since the last decades of the 20th century and applied in many fields of life. The production of geopolymer also has many different levels of technology from low to high technology which depends on quality and the applied fields of products. The geopolymer composites are also considered as environmentally friendly products related to low energy consumption production technology; reuse industrial wastes as raw materials; low or even zero CO₂ emission; many better properties compared to the same traditional materials, and others. This investigation would like to introduce a novel type of geopolymer-based materials synthesized from red mud and diatomaceous earth in autoclave conditions without using alkaline activators. The geopolymer composites were fabricated in three various temperatures of 120, 150, and 200°C and evaluated characteristics via different analytical methods. The method of X-ray diffraction (XRD) was used to evaluate phase transitions among raw materials (RM and DE) and the geopolymer composite materials. Characteristics of bondings in the materials were detected by using Fourier transform infrared spectroscopy (FTIR). The differential thermal analysis-thermogravimetric analysis (DTA-TG) is method used to look for the physico-chemical changes of the geopolymer composite materials exposed at high temperature. In addition, the geopolymer based materials were observed for morphologies and microstructures in its solid phase lattice matrix using scanning electron microscope (SEM).

Keywords: Alkaline activator, autoclave, diatomaceous earth, geopolymer-based materials, red mud

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INTRODUCTION

The term of "geopolymer" is used to describe a family of alkaline aluminosilicate bonds formed from reaction processes of the alkaline activators and aluminosilicate minerals [1]. The formation of geopolymer is the result of a heterogeneous complex chemical reaction among the solid Si-Al phases and a strong alkaline silicate solution [1–3]. The geopolymer reaction or geopolymerization is an exothermic reaction and it occurs at several special conditions of pressure and temperature. Although the geopolymer reactions of various aluminosilicate materials have been studied a lot in recent years, the exact mechanism in

geopolymerization is not fully understood [4]. The most proposed geopolymer synthesis mechanism consists of four stages, which are parallel and therefore indistinguishable and known as [5, 6]:

- Dissolve silica and alumina from solid aluminosilicate raw materials in a highly alkaline solution;
- Formation of the base chains or oligomers (Si-Si) or (Si-Al) in the liquid phase;
- The polymeric condensation of the oligomers to form 3-dimensional aluminosilicate lattices;
- Bonding among solid particles into the geopolymer frames and hardening

throughout the system to form solid inorganic polymer structural networks [1, 7–9].

The concept of alkaline activator is understood as the dissolution of aluminosilicate raw materials in a strongly alkaline environment which is prepared from a solution of sodium hydroxide or potassium hydroxide with water. Geopolymer belongs to groups of inorganic polymers which are bound into large molecules by covalent bonds and have the main circuit of -Si-O-M-O-. In which, M is understood mainly as cations of aluminium (Al) and secondly iron (Fe) [5, 8, 10–13]. The difference among geopolymer and other inorganic polymers is related to the forms of silicon and aluminium precursors used to

synthesize them. The conventional inorganic polymers are synthesized by the sol-gel method using silicon and aluminium oxides in alcohol and water solutions as precursors. Geopolymer is synthesized by the active ingredients of solid aluminosilicate raw material in high alkaline solution of sodium hydroxide, potassium hydroxide, sodium silicate, and potassium silicate [5, 14–17]. Silica precursors are sodium silicate and potassium silicate dissolved with the aluminosilicate raw material. Meanwhile, the alumina precursor is only from the dissolved aluminosilicate material, although in some cases the active solution also contains impurity of Al cation [1, 5, 18]. Applied fields of geopolymer-based materials are shown in Figure 1.

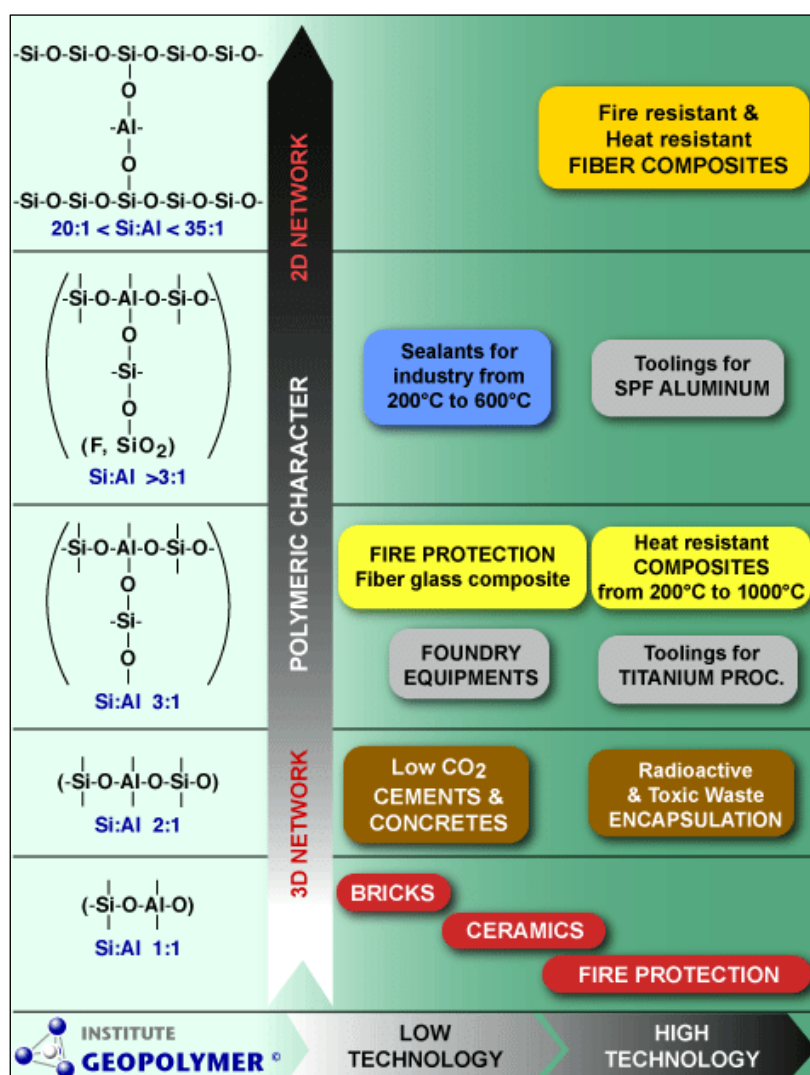


Fig. 1: Applied Fields of Geopolymer-Based Materials Proposed by Geopolymer Institute, France [1].

The geopolymer materials with 3-dimensional structure are able to replace ordinary materials based on ceramic, cement and concrete. The mechanical properties are very positive which are mainly related to the compressive strength. In other cases, the tensile strength is 2 to 3 times higher than the corresponding Portland cement [1, 5, 17–20]. In addition, the geopolymer composites have low apparent porosity and nanoscale pores resulting in low water permeability of about 9–12 cm/s. Moreover, the geopolymer-based materials are also applied as refractory, heat resistant, heat absorbent materials that can be used to prevent fire for buildings made of reinforced concrete such as building tunnels. Recently, the geopolymer materials are also used to store radioactive and toxic substances [1, 16–18, 21–23].

The geopolymer composite materials with 2-dimensional cross-linked structure have better heat and fire resistance properties than the geopolymer materials with 3-dimensional structures. In the structures, the 2-D geopolymer contains the physical and chemical bonds of water so that water molecules can move to the surface and evaporate without harming the material frame. The 2-dimensional longitudinally linked geopolymer with fiber reinforcement (carbon, glass, steel) has more complete mechanical properties. The geopolymer is used to manufacture special fire protection materials and applied in industrial fields such as transportation (aviation, ships, cars, trains, and others), nuclear, pharmaceutical chemistry etc. The 2-D geopolymer-based materials are also used to manufacture molds, lightweight materials, and auto parts [1, 3, 6, 24–26].

The geopolymer composite materials have attracted many scientists to participate in research during the past decades. There are many different types of aluminosilicate resources that can be used to synthesize geopolymers including solid rich-aluminosilicate raw materials of industrial minerals and wastes such as kaolin, ash, bentonite, peclite, fly ash, red mud, metallurgical slag, ores etc. mined from bentonite, peclite and others [1, 3, 7, 8, 12, 15, 27–35]. Currently, the groups of raw materials

related to wastes are considered as a potential, mainly to produce the geopolymer-based materials which are considered as positive solutions for environmental problems.

Indeed, the European Union has identified the harmful effects that are caused by industrial waste and requires the members to establish a legal framework to protect human health and the environment [25]. The European Union also promotes the recovery and reuse of industrial waste to conserve natural resources. The geopolymer synthesis technique uses solid industrial wastes containing aluminosilicate as raw materials for the manufacture of environment friendly products that can replace traditional building materials with compatible mechanical and thermal properties.

This study used red mud and diatomaceous earth or diatomite as raw materials for the geopolymerization processes. Red sludge or red mud is the name of an industrial waste of Bayer technology, a method mainly used in the refining process of bauxite to produce aluminium. It contains a mixture of solid and metal impurities and red mud is one of the most harmful waste problems in the aluminium smelting industry. The red color of sludge is due to the presence of the oxidized iron which can account for up to 60% mass of the sludge. Red mud is a rather problematic pollution problem because it occupies the land and cannot be built or grown anything on it even when the mud is dried. With very limited usability (brick and concrete production), red mud is an almost useless waste. Although red mud has many ingredients such as iron, titanium and aluminium (leftover), humans have not found an economically efficient way to utilize these substances yet. However, there are many advantageous specifications of red mud which are suitable for the geopolymerization technology. Because the red mud contains residual of NaOH (3–8%), Al₂O₃ (18–22%), SiO₂ (8–10%) and fine particle size (less than 40 μm) [11, 13, 14, 16, 21, 24, 27, 28]. However, when red mud is used as raw material for the geopolymerization reaction, it is necessary to add an additional amount of active SiO₂ in order to improve the reaction efficiency. On the other hand, diatomite contains high silica (over 70% SiO₂) [11, 13, 28–31] which can meet the

requirements of geopolymeric reaction to form aluminosilicate networks with red mud. These are key reasons that this study would like to report an investigation on utilization of red mud and diatomaceous earth as raw materials for production of the geopolymer composite materials. More specially, the study did not use any alkaline activators for geopolymer reaction because red mud already contains high sodium hydroxide in its compositions.

MATERIALS AND METHODOLOGY

Red mud (RM) was used in this study come from Tan Rai Bauxite Mining Plant, Lam

Dong province, Vietnam with the specifications as reported in the previous investigations [11, 13, 14, 16, 21, 24, 27, 28, 32–34]. Notably, Tan Rai red mud contains about 2.5% Na₂O, 20.8% Al₂O₃, 10.4% SiO₂, 42.3% Fe₂O₃ and others with the mineral compositions of quartz (SiO₂), hematite (FeOOH), gibbsite (Al(OH)₃). Both analyses of chemical and mineral compositions were carried out using methods of X-ray fluorescence (XRF) and X-ray diffraction, respectively. Tan Rai red mud had value of surface area at 6853.0 cm²/cm³, mean particle size at 8.2 μm with the smallest and largest particle size of 0.3 and 67.5 μm.

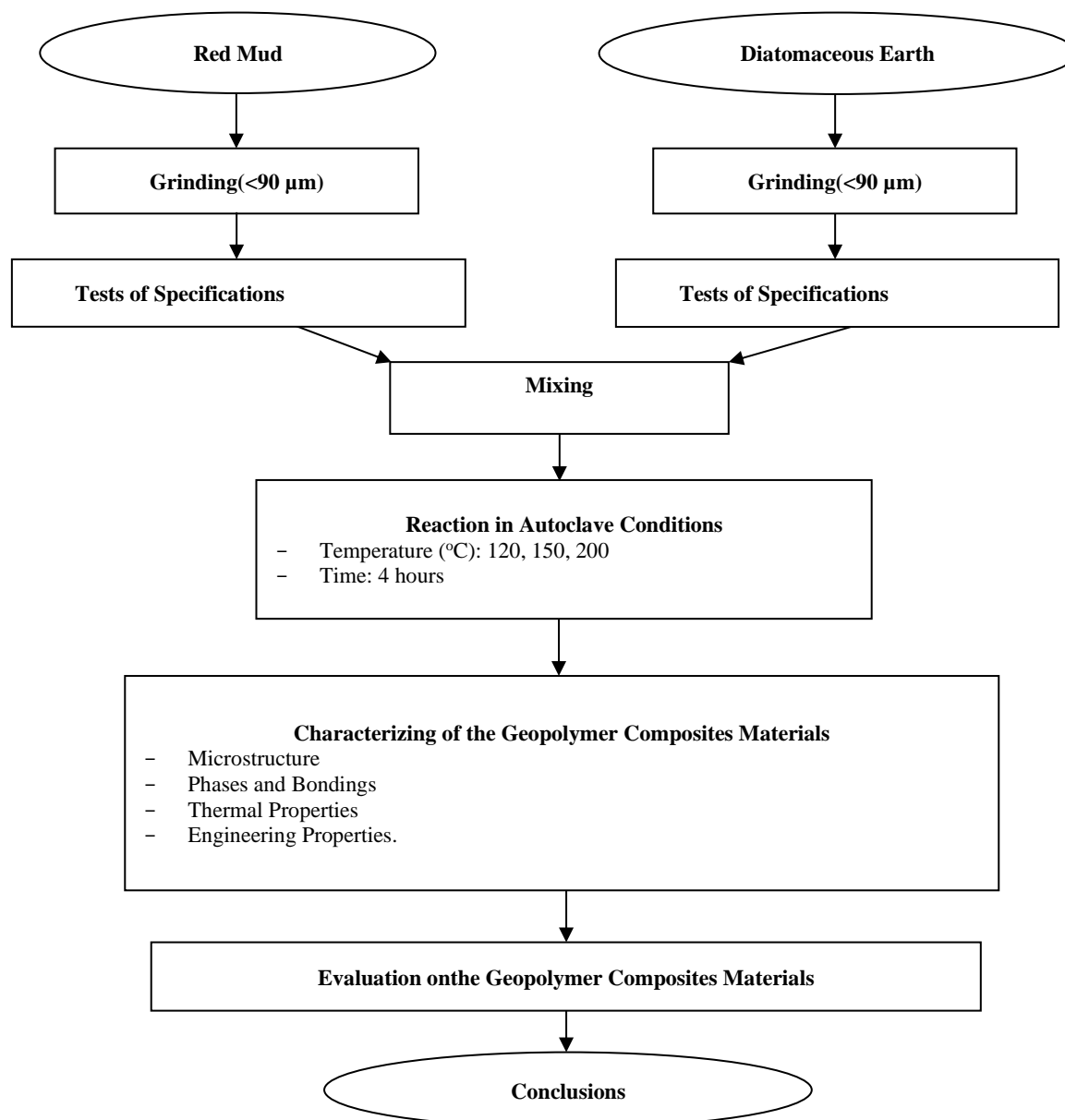


Fig. 2: Experimental Processes for Fabrication of the Geopolymer Composite Materials.

Diatomaceous earth (or diatomite, DE) was from Tam Bo mineral mining company, Lam Dong province, Vietnam with the specifications as reported in the previous investigations [11, 13, 28–31]. In which, DE contains 61.5% SiO₂, 15.8% Al₂O₃, 5.7% Fe₂O₃ and others with the minerals known as quartz (SiO₂), kaolinite (Al₂Si₂O₅(OH)₄), halloysite (Al₂Si₂O₅(OH)₄.2H₂O), and nontronite (Na_{0.3}Fe₂Si₄O₁₀(OH)₂.4H₂O). It is noted that DE has high porosity over 85% with the pore sizes less than 1 μm, surface area at 16698.0 cm²/cm³, mean particle size at 8.2 μm with the smallest and largest particle size of 1.7 and 174.6 μm.

The geopolymer composite materials had mixture design with the fixed ratio of red mud and DE at 1:1. The molar composition was converted from weight of raw materials as follows:

$$\frac{SiO_2}{Al_2O_3} = \frac{\frac{\%SiO_2_{RM}}{M_{SiO_2}} + \frac{\%SiO_2_{DE}}{M_{SiO_2}}}{\frac{\%Al_2O_3_{RM}}{M_{Al_2O_3}} + \frac{\%Al_2O_3_{DE}}{M_{Al_2O_3}}} = \frac{\frac{10,41}{60,08} + \frac{61,47}{60,08}}{\frac{20,85}{101,96} + \frac{15,85}{101,96}} = 3,32$$

$$\frac{Si}{Al} = \frac{1}{2} \frac{\frac{\%SiO_2_{RM}}{M_{SiO_2}} + \frac{\%SiO_2_{DE}}{M_{SiO_2}}}{\frac{\%Al_2O_3_{RM}}{M_{Al_2O_3}} + \frac{\%Al_2O_3_{DE}}{M_{Al_2O_3}}} = \frac{1}{2} \times \frac{\frac{10,41}{60,08} + \frac{61,47}{60,08}}{\frac{20,85}{101,96} + \frac{15,85}{101,96}} = 1,66$$

Raw materials (RM and DE) were ground and passed through the sieve of 90 μm. The samples of DE and RM were tested for specifications of water content, particle size distribution, chemical composition, mineral composition, pH value and heating microscopy before mixing into the fresh geopolymer mixtures. The fresh geopolymer mixtures were put in autoclave with adjustments of temperatures at 120, 150 and 200°C and the reaction time of 4 h. After the reaction process in autoclave, the geopolymer mixtures were divided into two sample groups. The first samples were characterized for microstructure, phases, bondings and thermal properties using the methods of scanning electron microscope

(SEM), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), differential thermal analysis-thermogravimetric analysis (DTA-TG) and heating microscope. The group of second samples was immediately formed in the standard molds of 40×40×160 mm for testing of mechanical properties known as flexural strength, compressive strength and others. The experimental processes are shown in Figure 2.

RESULTS AND DISCUSSION

The Phase Transitions among Raw Materials (RM and DE) and the Geopolymer Composite Materials via Reactions in the Different Autoclave Conditions Using X-Ray Diffraction (XRD)

There were changes significantly about phase compositions among raw materials (RM, DE) and the geopolymer-based materials as shown in Figure 3. The XRD pattern of RM detected crystal structures of gibbsite (Al(OH)₃), hematite (FeOOH), quartz (SiO₂) and amorphous phase with with background noise and expansion. DE has clay minerals such as kaolinite (Al₂Si₂O₅(OH)₄), halloysite (Al₂Si₂O₅(OH)₄.2H₂O), nontronite (Na_{0.3}Fe₂Si₄O₁₀(OH)₂.4H₂O) and others in its crystal compositions. It is noted that DE contains high silica (SiO₂) with amorphous structure of silica gel [1–4]. However, the crystal phases in raw materials of RM and DE were dissolved and reacted in autoclave condition to form the aluminosilicate structures of geopolymeric matrix. In autoclave condition with temperature at 120°C, the products of geopolymer composite material still have crystal phase of halloysite (Al₂Si₂O₅(OH)₄.2H₂O). There was formation of new crystal of analcine (NaAlSi₂O₆.H₂O) related to the reaction among alkaline in RM and aluminosilicate resources in RM and DE as shown in the following reactions:

$$2SiO_2 + [AlO_2]^- + [Na]^+ + H_2O \rightarrow NaAlSi_2O_6.H_2O$$

In autoclave condition with temperature at 150°C, the geopolymer still had crystals of halloysite and analcine but the their diffraction intensities were decreased significantly. These crystal phases were completely dissolved and reacted to form the geopolymer structures when

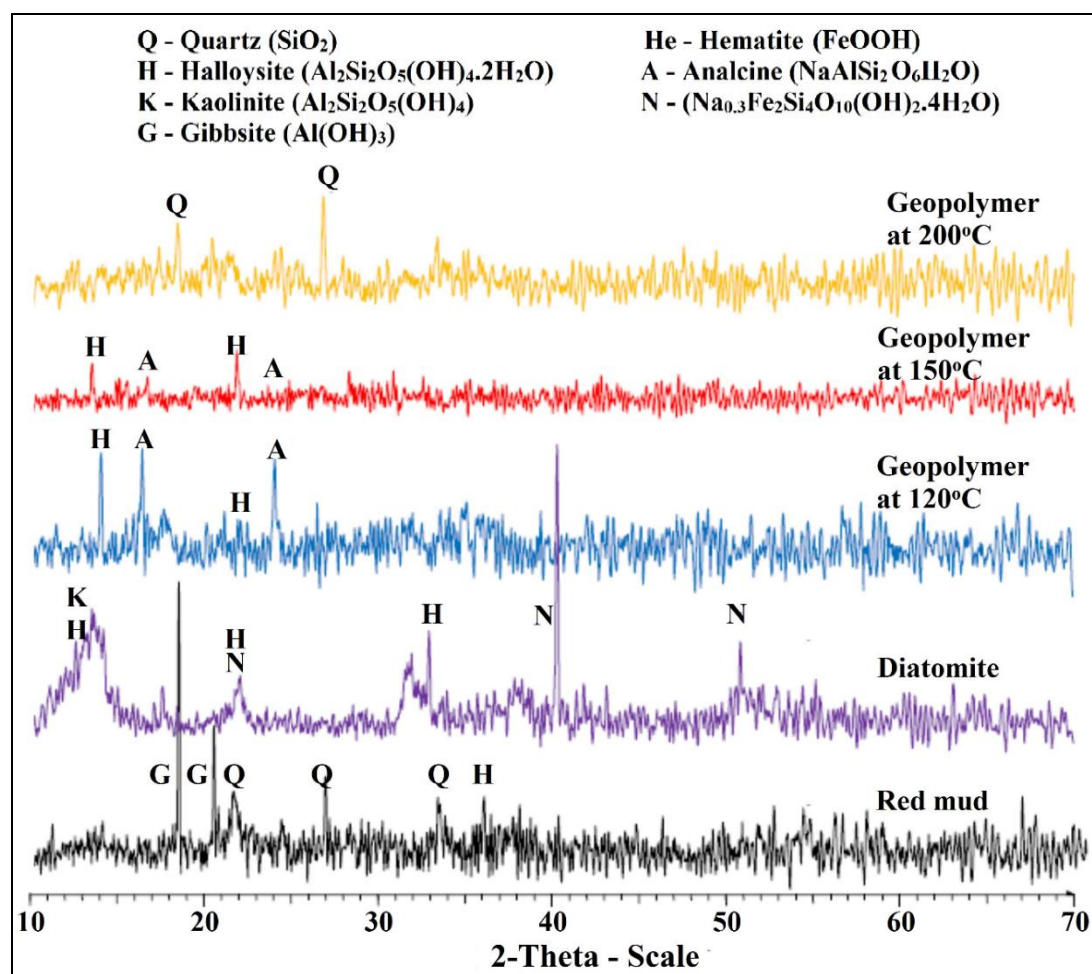


Fig. 3: The XRD Patterns of Raw Materials (RM and DE) and the Geopolymer Composite Materials with the Phase Transitions among Structures.

it was put in autoclave condition at 200°C. In general, the geopolymer composite materials contain mainly structures of amorphous phase and several crystals of quartz (SiO₂), analcine (NaAlSi₂O₆·H₂O), and clay minerals.

Characteristics of Bondings in the Geopolymeric Matrix of Aluminosilicate Structural Networks using Fourier Transform Infrared Spectroscopy (FTIR)

Figure 4 is the FTIR spectrum of RM, DE, and geopolymer-based materials reacted at 120, 150 and 200°C in autoclave condition. RM has the vibrations of bondings H-O-H at wavenumbers of 1639 cm⁻¹, 3434–3620 cm⁻¹; Si-O, Si-O-Si at wavenumbers of 1002 and 455 cm⁻¹; Si-O-Fe at wavenumbers of 455 cm⁻¹, and Al-O-H at wavenumbers of 802 cm⁻¹. DE contains the bondings of H-O-H, -OH, Si-O, Al-O, Si-O-Al, Si-O-Fe at wavenumbers of 466, 535, 696, 755, 797, 913, 1037, 1641, 3441, 3622, and

3698 cm⁻¹. There were displacements and changes of the wavenumbers of the bonding oscillations in the geopolymer composite materials. The wavenumber is from 400 to 1400 cm⁻¹ due to symmetric stretching, the geopolymerization process changed this region with vibrations of the double 4-membered ring and Si-O-Si, O-Si-O in silicate structure at 535, 913, 1032 cm⁻¹; the Al-O stretching at 799 and 914 cm⁻¹ wavenumber. The broadness in the wavenumber range of 950–1200 cm⁻¹ band reflected the wide distribution of Si and Al tetrahedral in the membered ring or chain structural units of geopolymers. The vibrations of Si-O-Fe bondings were detected as wavenumbers of 466 and 468 cm⁻¹. The remaining wavenumbers at 1635 and 3525, 3621, and 3697 cm⁻¹ are vibrations of the bondings H-O-H and -OH related to water content and OH-bonds in structures of geopolymer-based materials. In general, all

geopolymer samples in the various autoclave conditions have the same FTIR spectrum with the bonding vibrations as reported. This is scientific evidence that shows stability in the topology of the geopolymer composite materials despite experiencing different reaction temperature conditions at 120, 150, and 200°C in autoclave condition.

Physico-chemical Reactions of the Geopolymer Composite Materials Exposed at High Temperature Using Differential Thermal Analysis-Thermogravimetric Analysis (DTA-TG)

In this study, the geopolymer sample reacted at 150°C was analyzed for its thermal properties using method of differential thermal analysis i.e., thermogravimetric analysis (DTA-TG). The DTA-TG results are shown in Figure 5 with two curves of differential thermal analysis and thermogravimetric analysis from room

temperature (25°C) to 1000°C. The TG curve shows that the geopolymer had total mass loss of 12.89% including water content and loss on ignition (L.O.I). The value of total mass loss is considered to be suitable the used raw materials of RM and DE which have the L.O.I values at 11.19 and 7.60%, respectively. In this geopolymer sample, there is over 11% of total mass loss recorded at temperature less than 500°C. This is related to moisture evaporation, water loss in micrometer hollow structures and clay minerals, decomposition of the -OH structural group into H₂O and burning of organic impurities. The decomposition of the -OH structural group into H₂O was recorded with the endothermic effect at temperature of 292.9°C as shown in the DTA curve. The reaction occurred according to the following transformation process:

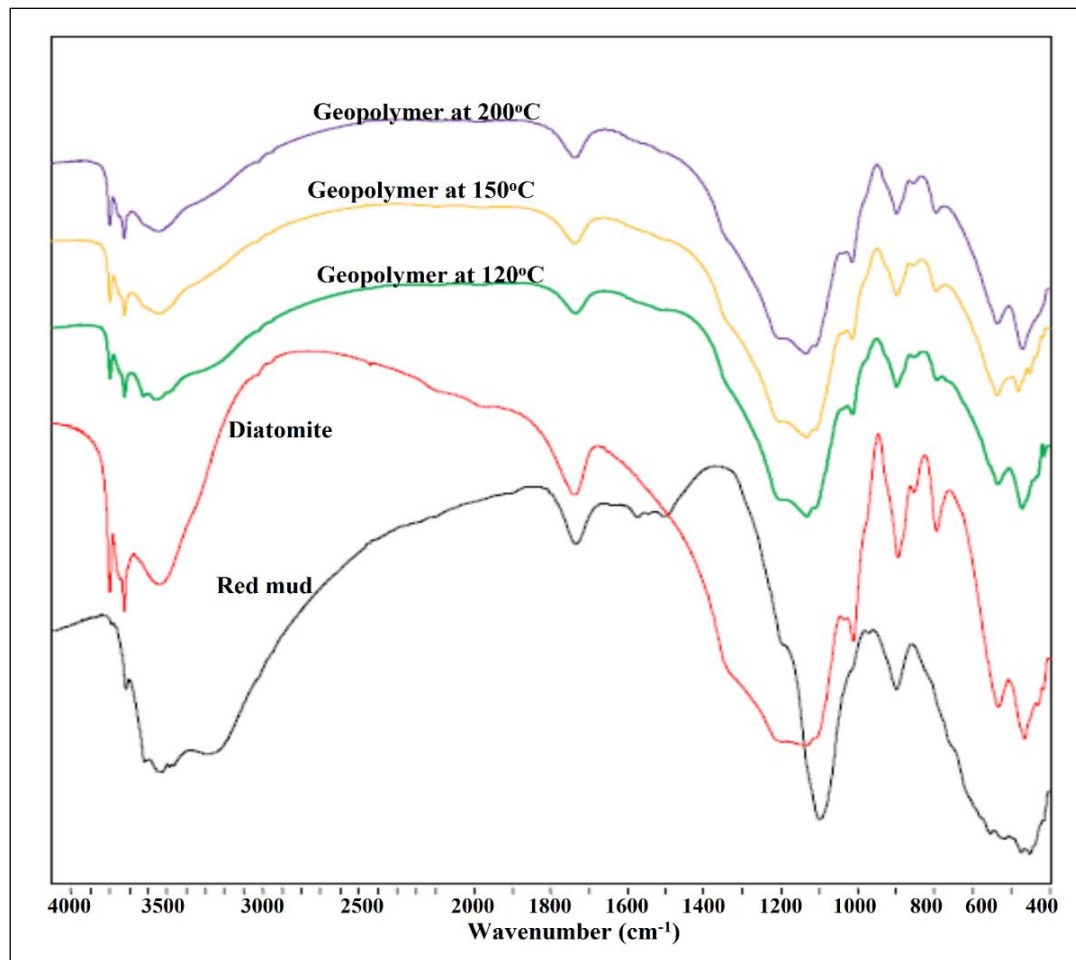
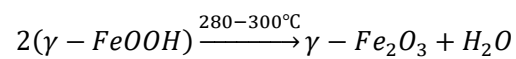


Fig. 4: Characteristics of Bondings in the Geopolymeric Matrix of Aluminosilicate Structural Networks.

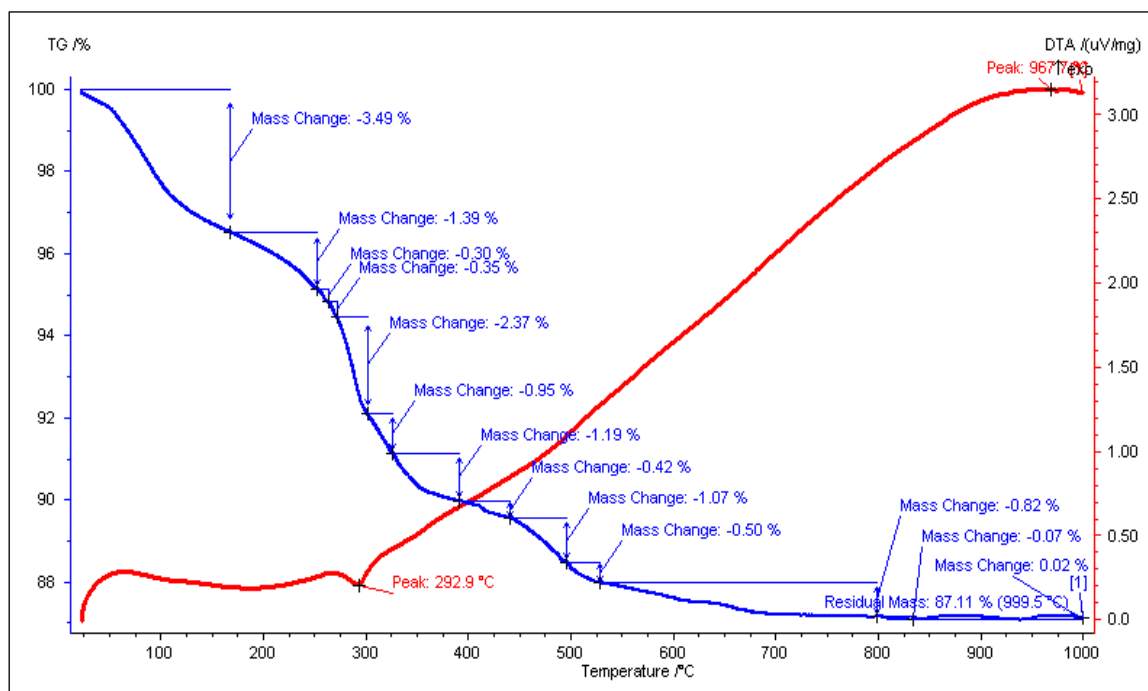


Fig. 5: The Physico-Chemical Reactions of the Geopolymer Composite Materials Exposed at High Temperature.

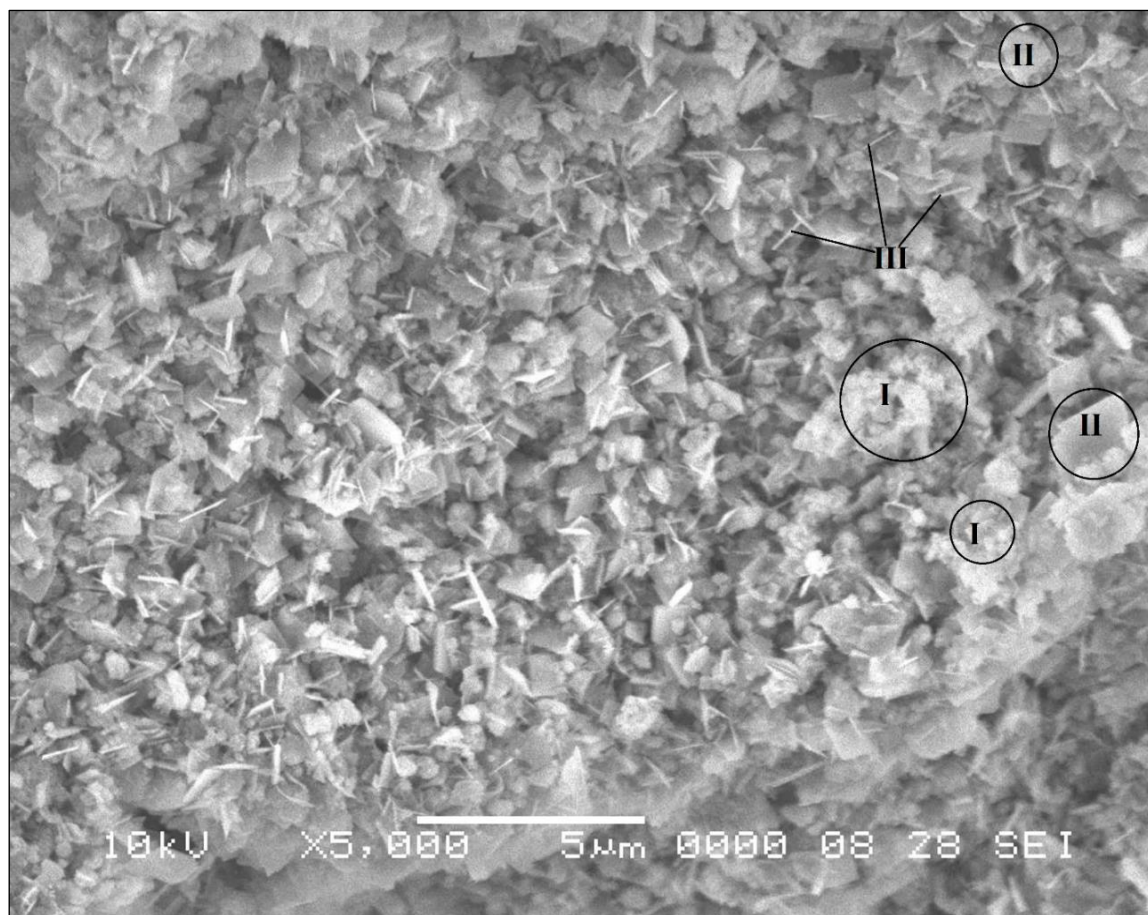


Fig. 6: Morphologies and Microstructures in the Solid Phase Lattice Matrix of Geopolymer Based Materials.

From 500 to 1000°C, the mass change is very low value, around 1%, and there is no any physico-chemical reaction recorded via the DTA curve. Thus, the thermal analysis results showed that the geopolymer-based materials have high structural stability without any physico-chemical reactions even at high temperature.

Morphologies and Microstructures in the Solid Phase Lattice Matrix of Geopolymer based Materials Using Scanning Electron Microscope (SEM)

The solid phase lattice matrix of geopolymer based materials were observed by method of image using SEM with magnification at 5000 time as shown in Figure 6; in which, the geopolymer sample was reacted in autoclave condition at 150°C. The SEM image shows the geopolymer based material contents and some typical morphologies in its microstructure. The fine powders are denoted (I) bonded with thin plate structures which are marked (II) as shown in Figure 6. The sizes of fine powder are less than 1 µm and the thin plate structures are around 2 µm with thickness less than 0.1 µm. In addition, there are many small rod structures with length of 2 µm and diameter around 0.1 µm. The rods were developed and distributed relatively evenly among the structural background of the fine powders and the thin plates. Finally, the geopolymer-based materials also have the pores distributed among spaces of the rods, the fine powders, and the thin plates.

CONCLUSION

Red mud is a kind of hazardous industrial waste which is a big problem of bauxite mining plants all over the world. This study succeeded in coming up with a useful solution for using RM as a raw material to produce the geopolymer-based materials. RM mixed with DE (contains high silica) without using alkaline activators is a novel method which has conducted no studies in the world yet. The geopolymer-based materials have their own characteristics in phase compositions, structural bondings, morphologies and microstructures as well as thermal properties at high temperatures. The geopolymer composites contain high amorphous phase and several crystal structures of quartz, analcine, clay minerals and others.

The FTIR spectrum recorded many vibrations or oscillations of bondings related to the double 4-membered rings, chains of aluminosilicate structural units in the geopolymeric matrix networks. The thermal analysis results showed that the geopolymer-based materials have high structural stability without any physico-chemical reactions even at high temperature. The geopolymer composite materials also contain specific morphologies and microstructures of the small rods, the fine powders, the thin plates and the pores. Further research should be carried out to test for engineering properties such as compressive strength, flexural strength, water absorption, volumetric weight, and others.

NOMENCLATURE

2-D	: 2-Dimension
3-D	: 3-Dimension
DE	: Diatomaceous Earth
DTA	: Differential Thermal Analysis
FTIR	: Fourier Transform InfraRed Spectroscopy
L.O.I	: Loss On Ignition
RM	: Red Mud
SEM	: Scanning Electron Microscopy
TG	: ThermoGravimetric
XRD	: X-Ray Diffraction
XRF	: X-Ray Fluorescence

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Cite this Article

Do Quang Minh, Nguyen Hoc Thang. Characteristics of Novel Geopolymer Composites Synthesized from Red Mud and Diatomaceous Earth in Autoclave Conditions without Using Alkaline Activators. *Journal of Polymer & Composites.* 2020; 8(3): 81–91p.