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Characterization of MgO and Al_2O_3 based Refractory waste as partial replacement for Fly ash based Geopolymer

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Green process is a manufacturing technologies method without harming the environmental, one of the green process is reclaiming and reuse the manufacturing waste into applicable products. Geopolymer is one of the examples of green process products. Geopolymer are a material from synthesized aluminosilicate and alkali silicate that formed polymeric SiO_4 and AlO_4 structure. Geopolymer can be applicated for building or refractory material. Aluminosilicate material such as fly ash is used for Geopolymer precursor. In this study, fly ash as precursor is substituted by magnesium oxide (MgO) and aluminum oxide ($Al2O_3$)-based refractory waste. It was then activated by activator consist of sodium hydroxide (NaOH) and Na_2SiO_3 (water glass). Results shown that the compressive strength of Al_2O_3 refractory waste based geopolymer are generally higher than MgO refractory waste based geopolymer. The result showed an addition of Al_2O_3 refractory waste improved the compressive strength of geopolymer specimen. XRD and FTIR characterization conducted to analyse the morphological compound and bonding of resulting geopolymer. The substance that contained in MgO and Al_2O_3 refractory based geopolymer are Quartz, Periclase, Corundum and Albite. FTIR results shows the siloxo and sialate bond as proof of geopolymerization has successfully occured.

Keywords: geopolymer; fly ash; refractory waste; compressive strength; FTIR.

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Introduction

The development of industrial technology today is very rapid. Sometimes with the progress of development, humans often forget the state of the environment. Technology Waste generated from industry can actually be reused. Processing waste into a new product has advantages in terms of being economical and environmentally friendly. Industry should be able to process and make a product with a process that is environmentally friendly and does not produce waste, this process is called a green process [1]. One of the green process products is geopolymer as a substitute for refractory materials and cement concrete.

In general, concrete is a material with the main composition of portland cement, aggregate, and water. The use of concrete is very wide and is used in general. Materials for making concrete are relatively easy to find and the manufacture of concrete is relatively simple. The continuous production of concrete can damage the environment because to produce 1 ton of cement, approximately 1 ton of greenhouse gases are produced. Emissions from the manufacture of concrete can damage the environment, therefore we need alternative materials that can replace concrete but do not damage the environment. A good example as a concrete substitute is Geopolymer.

Geopolymer is an environmentally friendly material that is commonly used as a substitute for cement-concrete. Geopolymers can be defined as materials resulting from the geosynthesis of aluminosilicates and alkali-silicates resulting in tetrahedrally bonded SiO_4 and AlO_4 polymer frameworks [2]. One of the main precursor materials of geopolymer is fly ash. Fly ash is a residual waste from burning and combustion residue of fine-sized coal [3]. In Indonesia, Fly ash production always increases every year, it is known that fly ash from coal combustion in 2006 was 1,700,000 tons and in 2009 it reached 2,780,000 tons. The price of fly ash is relatively less expensive than Ordinary Portland Cement (OPC) [4]. Fly ash-based Geopolymer is a solution to waste utilization and make materials at a more economical price. Geopolymer can be used as a substitute for refractory materials because it has high mechanical properties and durability [5]. Geopolymers can be applied as heat resistant coatings, radioactive waste containers, acoustic absorbents and as building materials [6, 7, 8, 9]. In this study, authors study the geopolymers with fly ash precursor partially substituted by MgO and Al_2O_3-based refractory waste from Iron and Steel Industries.

I. Methodology

Materials

The class-F fly ash with density of 2.5 g/cm^3 and passed from the sieve no. 325 was obtained from Suralaya Power Plant Banten, Indonesia. The oxides composition of these materials is shown in Table 1 by using X-Ray Fluorescence (XRF) method. MgO and Al_2O_3 Refractory waste was obtained from a private steelmaking industry in Cilegon, Banten, Indonesia. Sodium hydroxide (NaOH) and water glass (Na_2SiO_3) were purchased from Brataco Chemical, Bandung. Fly ash as precursor has some irregular particles. This may affect the setting time even though the lime content is less than 10% [10, 11]

Table 1.

Chemical composition of raw material

Oxide (%)	Class-F fly ash	Al_2O_3 refractories	MgO refractories
SiO_2	52.30	5.25	1.74
Al_2O_3	26.57	87.10	0.44
CaO	6.00	5.09	0.87
Fe_2O_3	7.28	0.65	0.07
MgO	2.13	0.73	85.00
K_2O	0.76	0.10	0.83

Methods

Since both of MgO and Al_2O_3 refractory waste are obtained as block (Figure 1a), it was grinded using Ball Mill until passed from the sieve #325 (Figure 1b) to enable its utilization as fly ash replacement.

Mix design of this study is listed in Table 2. Precursor consists grinded MgO and Al_2O_3 Refractory waste to replace the fly ash's weight from 0-75%. Alkali activator solution was prepared by mixing 12M sodium hydroxide (NaOH) solution with sodium silicate (Na_2SiO_3). The mass ratio of Na_2SiO_3 to NaOH was kept constant at 2.0.

Fig. 1. MgO and Al_2O_3 Refractory waste in (a) block form (b) ball mill grinded form.

Table 2.

Mix design of Geopolymer

No	Code	Precursor			NaOH: Na_2SiO_3 ratio	Curing Method
		Fly Ash (%)	MgO (%)	Al_2O_3 (%)		
1	MgO-0-A	100	0	0	2.0	Ambient
2	MgO-25-A	75	25	0		Ambient
3	MgO-50-A	50	50	0		Ambient
4	MgO-75-A	25	75	0		Ambient
5	MgO-0-D	100	0	0		Dry
6	MgO-25-D	75	25	0		Dry
7	MgO-50-D	50	50	0		Dry
8	MgO-75-D	25	75	0		Dry
9	Al_2O_3-0-A	100	0	0		Ambient
10	Al_2O_3-25-A	75	0	25		Ambient
11	Al_2O_3-50-A	50	0	50		Ambient
12	Al_2O_3-75-A	25	0	75		Ambient
13	Al_2O_3-0-D	100	0	0		Dry
14	Al_2O_3-25-D	75	0	25		Dry
15	Al_2O_3-50-D	50	0	50		Dry
16	Al_2O_3-75-D	25	0	75		Dry

Specimens are made according to the mix design by mixing all the ingredients into a basin and stirring homogenously and poured into 5x5x5 cm^3 mold. Each mix design can produce 3 cubes (Figure 2).

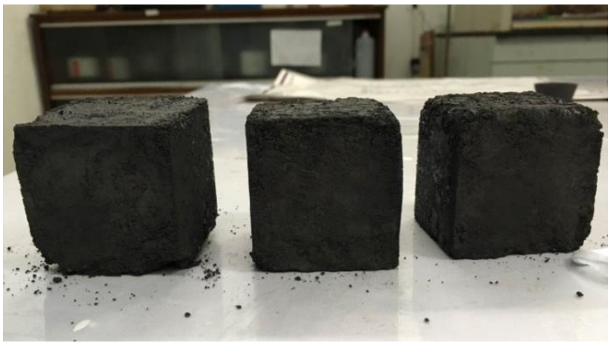

Fig. 2. Geopolymer removed from mold after 24 hours.

Curing method used are ambient and dry curing. For ambient curing, the geopolymer was kept in cling wrap at room temperature, then after 24 hours the Geopolymers were removed from the mold. For dry curing, the geopolymer with the mold sealed in a cling wrap and transferred into 80°C oven for 24 hours then kept at room temperature [12]. After 28 days maturity, the Geopolymer undergo a compressive test to measure its compressive strength at Materials Engineering Laboratory, Institut Teknologi Bandung using Tarnogrocki Universal Testing Machine (UTM) according to ASTM C109-02 [13].

Fraction from compressive strength was collected for X-Ray Diffraction (XRD) and Fourier Transform Infrared (FTIR) spectroscopy characterization. XRD was performed on Philips Diffractometer PW1710 using Cu anode at the Center of Advanced Sciences (CAS) Institut Teknologi Bandung, Indonesia. Resulting diffraction pattern was compared to the Joint Committee on Powder Diffraction Standards (JCPDS). FTIR was conducted to determine the chemical bonds of resulting Geopolymers using the Prestige 21 Shimadzu was carried out at the Analytical Chemistry Laboratory, Chemistry Department, Institut Teknologi Bandung, Indonesia.

II. Result and discussions

Compressive test result

Compressive test is an indicator which expresses the effect of refractory waste replacement towards Fly Ash based Geopolymers. The test was conducted at 28-days. Compressive strength properties of specimens are displayed in Figure 3 and 4.

Figures 3 and 4 shows that the strength of geopolymers using the dry curing method tends to be stronger than those using the ambient method. The dry curing method produces a higher compressive strength value because the temperature at the time of curing affects the geopolymerization reaction [14, 15]. Heat from outside causes the alkaline to be more reactive and the geopolymer formation reaction can occur completely. The curing temperature used during the experiment was 80°C, by heating during curing it caused the water in the geopolymer to evaporate so that when a compressive test was carried out the strength of the geopolymer was higher.

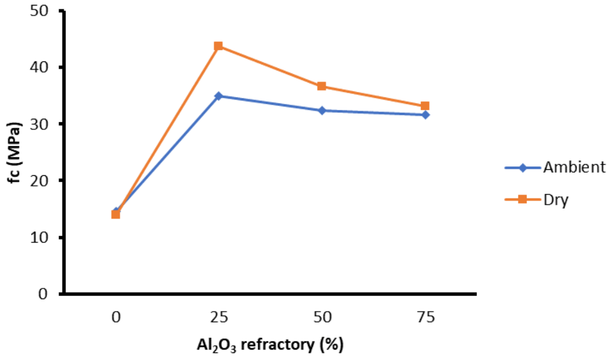

Fig. 3. Compressive Strength result of MgO refractory waste partial replacement Geopolymer at 28 days.

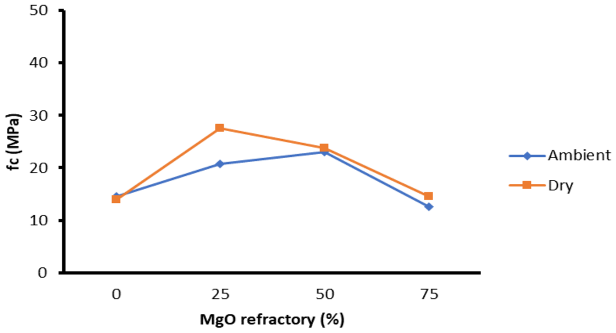

Fig. 4. Compressive Strength result of MgO refractory waste partial replacement Geopolymer at 28 days.

The best compressive strength for MgO based refractory waste as partial replacement for fly ash in Geopolymer was 27.6 MPa achieved by MgO-25-D. This happens because the addition of MgO reduces the shrinkage of the geopolymer volume so that when curing there are no cracks due to shrinkage [16]. MgO can reduce the shrinkage rate because MgO reacts with H_2O to form ($Mg(OH)_2$). The presence of cracks can cause stress concentrations when tested in compression [17]. Since the shrinkage rate is reduced, the strength of the MgO-25-D is higher than without MgO refractory waste partial replacement addition.

The best compressive strength for Al_2O_3 based refractory waste as partial replacement for fly ash in Geopolymer was 43.8 MPa achieved by Al_2O_3-25-D. The compressive strength of geopolymer with Al_2O_3 refractory waste tends to be higher than that of MgO refractory waste geopolymer. The strength of the MgO-based geopolymer when added with metakaolin increased the compressive strength value not as significantly as the Al_2O_3-based geopolymer. The addition of Al_2O_3 can increase the strength of the geopolymer by reacting actively with the alkaline solution to convert sodium hydroxide into geopolymer products. Al_2O_3 also functions to increase the Si:Al ratio. MgO-based geopolymer is not as strong as Al_2O_3-based geopolymer because MgO reacts with Si-O and Al-O to form magnesium-silicate-hydrate thereby reducing the reactivity of AlO^{4-} and SiO^{4-} which function as monomers in the geopolymer. Hence, not all Si and Al are formed into geopolymers. The addition of MgO can reduce the aluminosilicate content of the geopolymer [18]. Al_2O_3 reacts actively with alkaline solutions so that during the geopolymerization process, it produces reactive bonds in the monomer chain and intermolecular bonds increase and the compressive strength of the geopolymer increases. The addition of Al_2O_3 accelerates the synthesis of sodium hydroxide to become sodium-aluminum-silica-hydroxide [19]. Which explains the compressive strength of geopolymers containing Al_2O_3 refractory waste is higher than geopolymers with MgO refractory waste.

The geopolymer strength reached its maximum value at 25% partial replacement of fly ash, both for MgO and Al_2O_3 refractory waste. Addition of more than 25% partial replacement caused the compressive strength of the geopolymer to decrease. This shows that the most optimum addition of partial replacement is 25%. The compressive strength value decreased after the partial replacement exceeded 25% due to the clinker dilution effect phenomenon. This dilution effect causes some parts of the geopolymer to be replaced by partial replacement material, which cannot fully react in the geopolymer so that there is still unreacted MgO or Al_2O_3 left. As explained in the paragraph above, the effect of adding Al_2O_3 is to convert sodium hydroxide to sodium-aluminum-silicate-hydroxide, but if the addition of Al_2O_3 exceeds the optimum composition, the remaining Al_2O_3 will decrease the strength of the geopolymer [20].

XRD analysis

X-Ray Diffraction (XRD) analysis was performed to identify the phases and compounds contained in the research specimens to determine whether a geopolymerization reaction occurred. XRD testing was carried out on 8 types of specimens that had been pressure tested. The specimens tested for XRD were MgO and Al_2O_3 refractory waste based geopolymers with a composition of 25% and 75%, at ambiend and dry curing. XRD results for Al_2O_3 refractory waste based as fly ash partial replacement in Geopolymer is presented in Figure 5.

Figure 5 shows the diffractogram of Al_2O_3 refractory waste partial replacement for fly ash Geopolymer. Resulting XRD peaks showed the presence of Quartz, Albite, Corundum, and Kyanite compounds. The composition of Si and Al plays an important role in the formation of geopolymers and the compressive strength of geopolymers [21]. Quartz compounds are derived from fly ash which is used as a geopolymer precursor because the main composition of fly ash is SiO_2.

Corundum are derived both from fly ash precursor and Al_2O_3 based refractory waste. Presence of Corundum is also important in geopolymerization. Quartz functions as a monomer forming and silicate polymerization, while corundum functions as a link between silicate monomers which then forms a three-dimensional geopolymer structure.

Albite shows that the geopolymerization reaction has occurred. It has a compound structure as mark of geopolymerization mechanism [22]. Na element comes from alkaline activator solution reacts with SiO_2 and Al2O_3 from precursors. Presence of Albite in Al_2O_3 refractory waste based geopolymer shows that the geopolymer has been formed so that when compared to the compressive strength of the MgO-based geopolymer, the compressive strength is higher. More albite shows that the more geopolymerization occurred, as this affects the compressive strength of the geopolymer.

Figure 6 show the presence of periclase and quartz compounds. Periclase (MgO) comes from the refractories. Periclase compounds in MgO refractory waste based geopolymers are very dominant because the electronegativity of Mg is greater than Na^+ ($Mg^{2+} = 1.2$, $Na^+ = 0.9$) so that Na^+ ions are not visible. Na^+ plays a role in dissolving Si^{4+} and Al^{3+} ions during the gepolymerization process to form geopolymer compounds. When MgO is dominant in geopolymers, it acts as a filler that has higher composition than fly ash. Quartz plays an important role in the geopolymerization process. It is required for monomer formation and silicate polymerization. Quartz in the geopolymer is obtained from fly ash precursor. The geopolymerization reaction cannot be seen from the XRD analysis because the MgO compound is too dominant. Hence, the Geopolymer formation needs to be characterized by FTIR to check its bonding formation.

FTIR analysis

Fourier Transform Infrared (FTIR) spectroscopy analysis was performed to identify the resulting chemical bonding from the geopolymerization process. Identification of geopolymerization can be done by

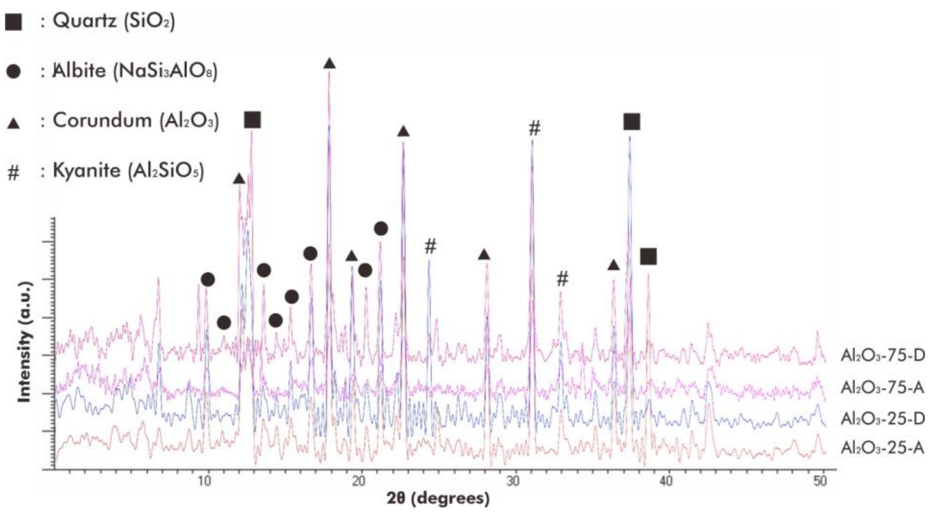

Fig. 5. X-Ray Diffractogram of Al_2O_3 refractory waste partial replacement for fly ash Geopolymer.

looking at the region of the wave length and matching it with common compound bonds in geopolymers such as Si-O-Si (siloxo) and Si-O-Al (sialate) [23]. Specimens that have been tested for compression are subjected to FTIR testing to determine the groups and bonds formed. The FTIR data obtained are presented in Figure 7 and 8.

Figure 7 shows the results of FTIR geopolymer based on Al_2O_3 refractory waste with a composition of 25% and 75% using dry and ambient curing methods. Resume of resulting bonds are covered in Table 3.

Based on Figure 7 and Table 3, it is known that the Al_2O_3-25-A shows that there is stretching –OH and bending of H-O-H in the geopolymer. As explained similarly above, stretching –OH occured at wavelength number 3700 – 2300 cm^{-1} and bending H-O-H is at wavelength number 1650 – 1630 cm^{-1}, originating from NaOH which does not react completely during the geopolymerization process. There are also siloxo and sialate bond as mark of geopolymerization occured.

Figure 8 shows the results of FTIR geopolymer based on MgO refractory waste with a composition of 25% and 75% using dry and ambient curing methods. Resume of resulting bonds are covered in Table 4.

Based on Figure 8 and Table 4, it is known that there is stretching –OH and bending H-O-H in the geopolymer similar Al_2O_3 refractory waste-based geopolymer. Stretching –OH is at wavelength number 3700–2300 cm^{-1} and bending H-O-H is at wavelength number 1650 –

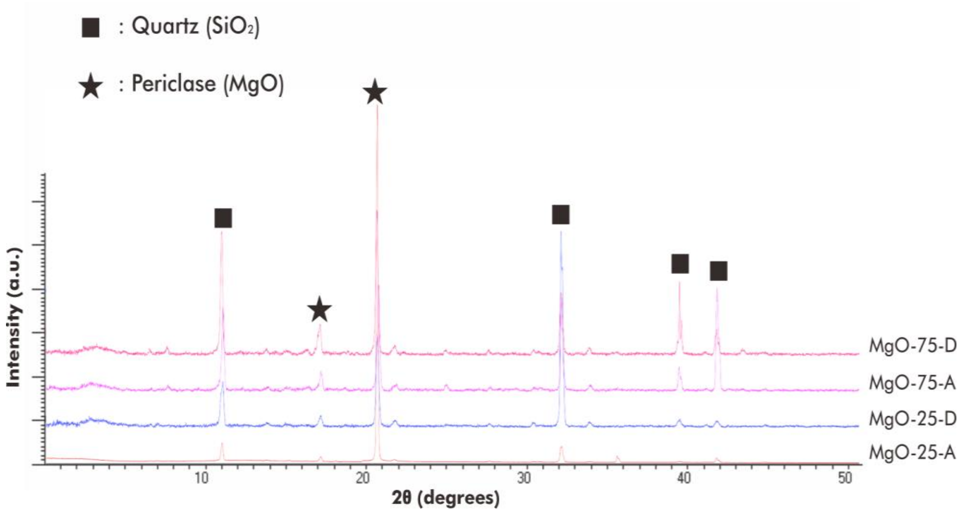

Fig. 6. X-Ray Diffractogram of MgO refractory waste partial replacement for fly ash Geopolymer.

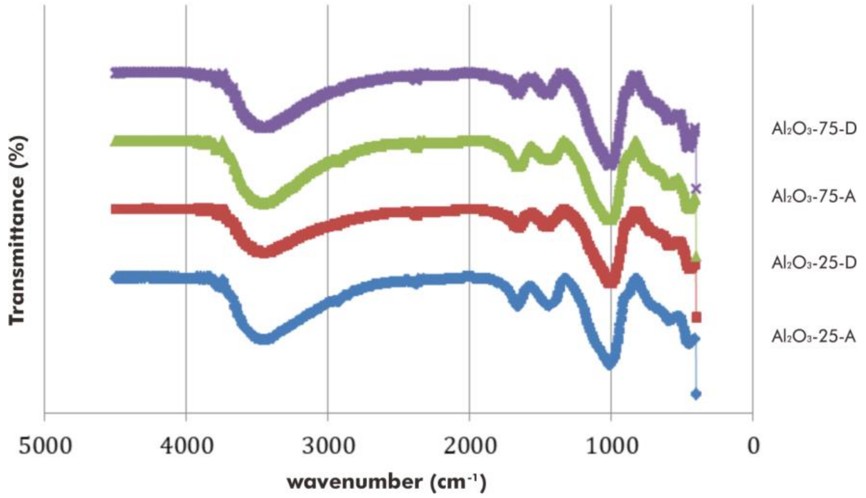

Fig. 7. FTIR spectra of Al_2O_3 refractory waste partial replacement for fly ash Geopolymer.

Table 3.

FTIR resume of Al_2O_3 refractory waste partial replacement Geopolymer (cm^{-1}).

No	Code	Stretching OH-	Bending H-O-H	Stretching O-C-O	Stretching Si-O-Si Si-O-Al	Symetric Vibration Si-O-Al	Bending Vibration Si-O-Si O-Si-O
1	Al_2O_3-25-A	3450.65	1658.78	1440.83	1010.7	590.22	441.7
2	Al_2O_3-25-D	3450.65	1658.78	1448.54	1008.77	586.36	447.49
3	Al_2O_3-75-A	3448.72	1656.85	1440.83	1008.77	588.29	447.49
4	Al_2O_3-75-D	3450.65	1660.71	1442.75	1010.7	590.22	451.27

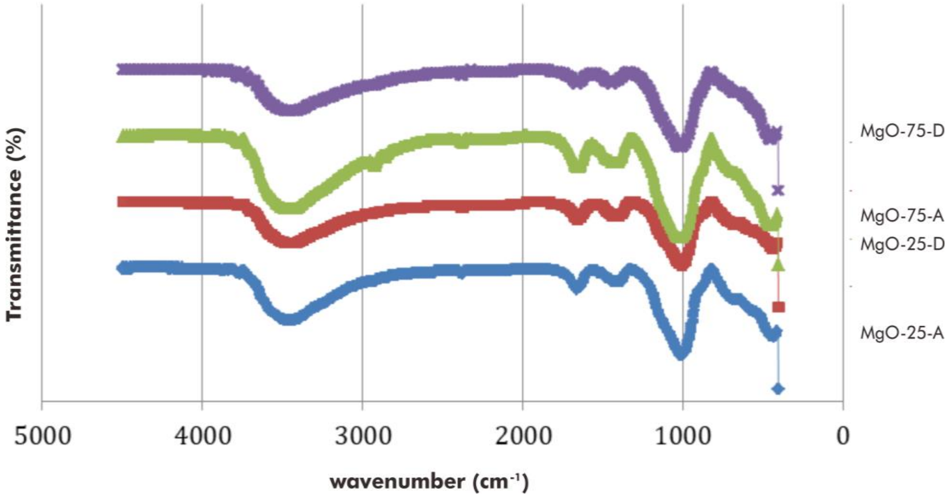

Fig. 8. FTIR spectra of MgO refractory waste partial replacement for fly ash Geopolymer.

Table 4.

FTIR resume of MgO refractory waste partial replacement Geopolymer (cm^{-1}).

No	Code	Stretching OH-	Bending H-O-H	Stretching O-C-O	Stretching Si-O-Si Si-O-Al	Symetric Vibration Si-O-Al	Bending Vibration Si-O-Si O-Si-O
1	MgO-25-A	3450.65	1656.85	1392.61	1006.84	684.73	441.7
2	MgO-25-D	3448.72	1649.14	1415.75	1008.77	682.8	437.84
3	MgO-75-A	3448.72	1649.14	1411.89	1006.84	686.66	439.77
4	MgO-75-D	3448.72	1656.85	1440.83	1010.7	688.59	453.27

1630 cm^{-1}. This shows that there are –OH and H-O-H bonds in the geopolymer, originating from NaOH which does not react completely during the geopolymerization process. When compared to MgO-25-D the compressive strength of MgO-25-A (20.8 MPa) is below MgO-25-D (27.6 MPa) and from the curve it can be seen that the stretching area of –OH in MgO-25-A is wider than in MgO-25-D. This also occurs in specimens MgO-75-A and MgO-75-D. The wider stretching area curve –OH results in lower compressive strength of Geopolymer.

In all four specimens, there are O-C-O stretching vibrations. O-C-O stretching vibrations occur at a wavelength of about 1420 cm^{-1}. This indicates the formation of trona or sodium bicarbonate in all specimens [24]. The O-C-O bond arises due to the reaction between NaOH solution and carbon dioxide (CO_2) from the air to form Na_2CO_3. The effect of the formation of trona or sodium bicarbonate is the formation of porosity which affects the compressive strength of the geopolymer. The FTIR results show that in the four specimens Si-O-Al and Si-O-Si bonds are formed, this proves that in all specimen's sodium alumina silicate has been formed as a result of the dissolution of silica and alumina. The presence of Si-O-Al and Si-O-Si bonds indicates the formation of geopolymer matrix.

Conclusions

From this study, it can be concluded that MgO and Al_2O_3 based refractory waste has different effect on are the compressive strength of fly ash-based geopolymer when used as partial replacement with different curing temperature. The FTIR characterization shown that all geopolymer specimens undergo geopolymerization marked by the presence of siloxo and sialate bonds. The strength of MgO refractory waste based geopolymer is lower than that of Al_2O_3 refractory waste based geopolymer because the addition of MgO inhibits the geopolymerization rate. Moreover, they are more suspectible to capture CO_2 from the environment proven by the presence of stretching O-C-O bonds, which indicated the formation of Na_2CO_3. Resulting geopolymer compounds were generated by X-Ray Diffraction. MgO refractory waste based geopolymer generated Quartz and Periclase compound, while Al_2O_3 refractory waste based geopolymer produces Quartz, Albite, Corundum, and Kyanite compounds. The formation of albite marked the geopolymerization.

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Характеристика вогнетривких відходів на основі MgO та Al_2O_3, як часткова заміна геополімеру на основі золи

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Зелені технології – це методи технології виробництва без шкоди для навколишнього середовища, а одним із екологічних процесів є регенерація та повторне використання відходів виробництва для відповідних продуктів. Геополімер є одним із прикладів екологічно чистих продуктів. Геополімер – це матеріал із синтезованого алюмосилікату та лужного силікату, який утворив полімерну структуру SiO_4 та AlO_4. Геополімер можна застосовувати як будівельний або вогнетривкий матеріал. Алюмосилікатний матеріал, такий як зола-винесення, використовується як прекурсор геополімеру. У цьому дослідженні летюча зола як попередник замінюється вогнетривкими відходами на основі оксиду магнію (MgO) і оксиду алюмінію (Al_2O_3). Потім його активували активатором, що складається з гідроксиду натрію (NaOH) і Na_2SiO_3 (водяне скло). Результати показали, що міцність на стиск вогнетривкого геополімеру на основі відходів Al_2O_3 зазвичай вища, ніж геополімеру на основі вогнетривких відходів MgO. Результат показав, що додавання вогнетривких відходів Al_2O_3 покращує міцність на стиск зразка геополімеру. Для аналізу морфологічної сполуки та зв'язку отриманого геополімеру досліджено XRD та FTIR характеристики. Речовиною, яка міститься в геополімері на основі тугоплавкого MgO та Al_2O_3, є кварц, периклаз, корунд та альбіт. Результати FTIR показують силоксо- та сіалатний зв'язок як доказ того, що геополімеризація відбулася успішно.

Ключові слова: геополімер; зола; вогнетривкі відходи; міцність на стиск; FTIR.