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Effect of Calcination on the Ash from Lokon Volcano and Its Potentially Sustainable Binder Material

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***Abstract*— The need for cement as a housing construction material has continued to increase due to the growing population. This high demand increases carbon dioxide emissions. Hence, it is necessary to optimize the use of natural pozzolan material. Volcanic ash is a natural pozzolan material in North Sulawesi, but its use could be more optimal. This study aimed to determine the effect of calcination on the physical properties of volcanic ash originating from the eruption of Mount Lokon. The calcination was carried out to determine the potential of Lokon ash at different temperatures to assess the structural characteristics, mineral phases, metal oxide composition, functional group bonding, morphology, and its potential as a binder for concrete mixtures. The ash material used comes from sand taken from the Pasahapen River and filtered through a 325-mesh sieve. Lokon ash was calcined at temperatures of 800, 900, and 1000^{o}C to determine the structural and morphological characteristics. At the same time, the effects were examined using an X-ray diffractometer (XRD), Raman spectroscopy, X-ray fluorescence (XRF), Fourier Transform InfraRed (FTIR), and Scanning Electron Microscopy (SEM). The results showed that calcination triggered the formation of hematite in the ash, which will increase its reactivity as a pozzolan material. This process causes the crystallinity of ash minerals to increase, but the ash material produced is predominantly amorphous. Hence, it has excellent potential as a binder material in concrete mixtures.**

***Keywords*—Amorphous; crystallinity; concrete; hematite formation; pozzolan material.**

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I. INTRODUCTION

Population development and global urbanization have increased cement production for construction in various parts of the world. This leaves a severe environmental problem related to carbon dioxide emissions. One source of carbon dioxide is Ordinary Portland Cement (OPC) [1]. The production of 1 ton of OPC can produce CO_2 emissions of 0.94 tons [2]. Furthermore, the intensity of these emissions continues to grow as global demand for OPC is expected to increase by almost 200% in 2050 [3]. Increasing OPC production will increase CO_2 emissions. Therefore, developing natural materials for cement supplements is urgent and relevant. Several natural materials used as cement additives in concrete mixes include agricultural waste such as bamboo, fishery waste such as fish scales, and natural pozzolanic materials such as calcined clay, paper sludge, and various types of ash [4]–[7]. Bamboo fiber in a reinforced concrete mixture provides additional strength compared to conventional concrete [8]. Fish scales waste can act as a biopolymer and aggregate in concrete [5] because they contain chitin, collagen, calcium, and hydroxyapatite [9]. Pozzolans can be defined as materials containing siliceous or siliceous alumina, which, in the presence of water, can react with calcium hydroxide (lime) at room temperature to form compounds with cementitious properties [7]. When mixed with water and lime, pozzolan forms stable compounds with hydraulic cement properties [6]. The use of natural pozzolan as a cement substitute will produce a significant reduction in CO_2 emissions [2].

Natural pozzolan material that is widely used today comes from the results of volcanic eruptions, mainly volcanic ash [10]–[12]. The use of volcanic ash in cement has been studied extensively in the last thirty years. It was concluded that volcanic ash is suitable as a complement to cement in concrete because it has pozzolanic activity. Volcanic ash will be pozzolanic when it reacts with calcium hydroxide released during cement hydration to improve concrete's mechanical properties [1]. According to [13], the mechanical properties of concrete will increase if volcanic ash is used as an OPC supplement with a ratio of 30-50% in volume.

At 30% and 50%, volcanic ash has a participatory role in controlling the kinetics of chemical reactions related to concrete hardening [10]. Meanwhile, according to [14], a volcanic ash ratio of 15% gives good flexibility and tensile strength. At a ratio of 10%, volcanic ash from Mt. Sinabung in North Sumatra, Indonesia, provides nearly the same ultimate compressive and tensile strength as concrete without volcanic ash admixture [15]. Volcanic ash has a high potential as a cement supplement because it can increase the pozzolans by 22% from 28 to 90 days [16].

Volcanic ash has a larger specific surface area than cement [17] with a more porous and finer microstructure, making it more reactive in alkaline media [18]. Its reactivity can be increased by providing mechanical or thermal treatment [19]. Mechanical activation triggers structural changes in the minerals in the ash, while thermal activation through calcination affects the clay minerals [20]. Calcination and grinding of volcanic ash material will make the ash more reactive as a binder in cement composites [21]. The calcination process causes the evaporation of volatile components, entropy changes, atomic structure reorganization, and crystalline phase breakdown, making volcanic ash more amorphous and reactive. Removing the sand fraction or grinding can modify the ash into finer particles. The grinding process can increase the surface area of the material to become more reactive and the amorphization of minerals.

Furthermore, calcination at temperatures above 700°C will increase the density and reactivity and produce a cement construction material with more substantial mechanical properties [22]. Andesitic volcanic ash calcined at 700 and 800°C had a faster fixation of calcium when added to $Ca(OH)_2$ solution than at 900°C [21]. Volcanic ash can act as a binder in cement materials when the SiO_2 + Al_2O_3 composition is at least 70%, and the SiO_2/Al_2O_3 molar ratio is at an optimal value of 3.3 to 4.5 with a 36% minimum amorphous content [21].

North Sulawesi is a volcanic area rich in natural pozzolan material in the form of ash. It is abundant, but its use as a binder material in concrete mixtures has yet to be optimized [21], [22]. Volcanic ash from Lokon Volcano is andesitic with moderate SiO_2 content and can potentially be a sustainable binder [23]. Therefore, calcination was carried out to determine the potential of Lokon ash at different temperatures to assess the structural characteristics, mineral phases, metal oxide composition, functional group bonding, morphology, and its potential as a binder for concrete mixtures.

II. Materials and Method

A. Sampling and Sample Preparation

The ash from Lokon Volcano sampled in this study was filtered from sand from the Pasahapen River area, a flow area for pyroclastic material produced by eruptions (Figure 1). The sand was dried in the sun between 10.00 am to 03.00 pm for three days. The dry sand was sifted through a 120-mesh sieve to protect against unwanted impurities or non-sand materials. The results were then filtered again with a 325 mesh sieve to obtain fine ash ready to be calcined [22], as shown in Figure 2. The calcination process was carried out with furnace equipment at the Chemistry Laboratory of the Faculty of Mathematics and Natural Sciences, University of Sam Ratulangi, Manado. The ash samples were calcined at different temperatures, namely 800°C, 900°C, and 1000°C, for 3 hours, and one uncalcined sample was prepared [24], [25]. Afterward, the four samples were characterized by their structural and mineral phase properties, morphology, chemical composition, and the vibrational mode of the molecular bonds [24], [26].

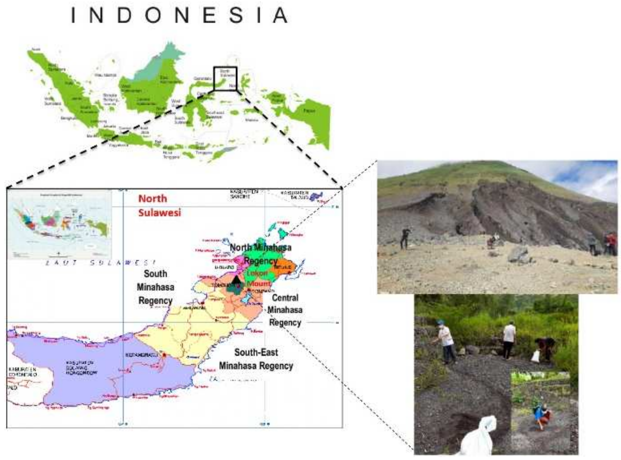

Fig. 1 Lokon sand sampling location

Fig. 2 Sample of Lokon volcanic ash

B. Sample Characterization and Analysis

Lokon's volcanic ash structural properties were characterized using X-Ray Diffractometer (XRD) and Raman spectroscopy equipment [24]. The main oxide composition in the ash was investigated with X-Ray Fluorescence (XRF) equipment. In contrast, the type of molecular bonds in the ash material compounds was assessed using Fourier Transform InfraRed (FTIR) equipment [26]. Furthermore, the morphological characteristics were observed using Scanning Electron Microscopy (SEM) equipment [24]. XRD, XRF, FTIR, and SEM were characterized at the Central Laboratory of the State University of Malang. At the same time, the Raman spectroscopy was performed at the Physical Chemistry Laboratory of Bandung Institute of Technology. XRD spectral analysis was conducted with the Profex 5.0 Software to identify the mineral phase contained in the ash sample [27]. The characterization results using XRD, Raman, FTIR, XRF and SEM were used to analyze the effects of calcination on Mount Lokon volcanic ash.

III. Results and Discussion

A. X-Ray Fluorescence (XRF) Analysis

The chemical composition of the ash was determined using XRF analysis; the results are shown in Table 1. The ash material filtered from Lokon sand was rich in SiO_2, iron oxide (Fe_2O_3), Al_2O_3, and calcium oxide (CaO). Meanwhile, potassium oxide, manganese oxide, and titanium oxide existed to a lesser extent. Magnesium and sodium oxide were not detected because they contain light elements with minimal content. Apart from that, Lokon ash has a comparative advantage because it contains significant amounts of calcium oxide. Calcium oxide, silica, iron oxide, and aluminum oxide in volcanic ash will contribute significantly to the density of the concrete microstructure, thereby increasing its mechanical properties [6].

TABLE I
Main oxide composition in weight percent (wt %) of Lokon ash based XRF analysis

Oxide/ samples	**SiO_2**	**Al_2O_3**	**Fe_2O_3**	**CaO**	**K_2O**	**MnO**	**TiO_2**	**P_2O_5**
Not calcinated	51.9	12	20.2	10.3	2.50	0.33	1.73	-
800^{0}C	50.8	12	20.0	10.4	2.80	0.34	1.67	0.81
900^{0}C	51.0	12	20.7	10.6	2.78	0.35	1.71	0
1000^{0}C	49.7	12	21.6	10.8	2.64	0.36	1.75	0
Average	50.85	12	20.63	10.53	2.68	0.35	1.72	0.045
SiO_2+Al_2O_3	62.85							
SiO_2+Al_2O_3+ Fe_2O_3		83.48						

The total oxide content of SiO_2 and Al_2O_3 from each of the four samples was 62.85%, while the total oxide composition of SiO_2, Al_2O_3 and Fe_2O_3 was 83.48%. These values are close to typical values of 70% (SiO_2 + Al_2O_3), which are suitable for producing alkali-activated binder materials [6]. They also meet the minimum standard value of ASTM C618-15, which is 70% (SiO_2+ Al_2O_3+ Fe_2O_3) for pozzolan material [22], [28]. The content of aluminosilicate materials (SiO_2 + Al_2O_3) in volcanic ash will make volcanic ash easily soluble in alkaline media, increase the kinetics of the geopolymerization reaction, and increase the compressive strength of concrete [22], [29]. The addition of volcanic ash to cement composites causes an active pozzolan reaction between silica/alumina from volcanic ash and calcium hydroxide from the cement that forms calcium silicate hydrate (C-S-H) or calcium aluminate silicate hydrate (C-A-S-H) and which can play a big role in increasing physical, mechanical, durability and microstructure properties of cement composite materials [30]. XRF analysis showed that the composition of SiO_2, Fe_2O_3, and Al_2O_3 did not change significantly when calcined at 800°C, 900°C and 1000°C. Based on the chemical composition data and calcination effect, the Lokon ash sample fulfilled the requirements as an excellent natural pozzolan [31].

B. Raman Analysis

The calcination process can cause a widening of the mineral types in volcanic ash due to the complexity of their chemical composition, making it challenging to interpret the XRD spectra of the calcinated ash samples. Raman analysis was used to help formulate the mineralogy of the ash material to confirm the XRD spectra. The Raman spectra of Lokon volcanic ash in Figure 3 show different peaks. In the samples calcined at 900°C and 1000°C, several spectra were identified. In the samples that were not calcined and those that were calcined at 800°C, only a few spectral peaks appeared because they were still dominated by high amorphous content.

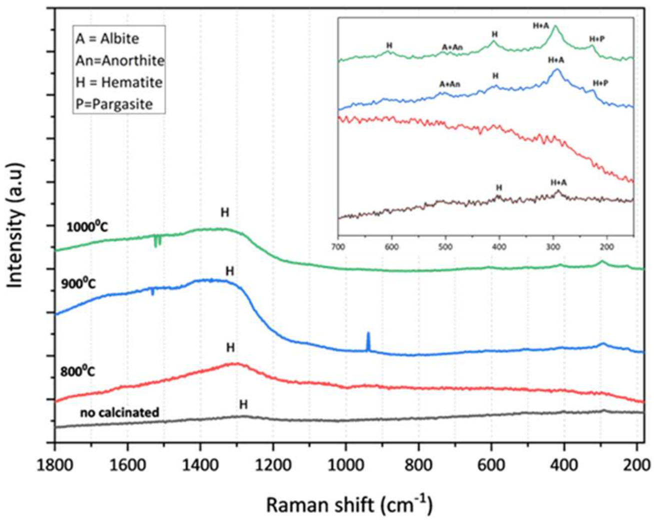

Fig. 3 Raman spectra of not calcinated and calcinated samples

The hematite mineral phase (α-Fe_2O_3) is indicated by several spectral peaks, namely peaks with high intensity and a broad spectrum at 1345 cm^{-1} [32], the spectral peak is located at 606 cm^{-1} [32], and at 411 cm^{-1} [33]. The mineral phases of hematite and albite ($CaAl_2Si_2O_8$) are mixed at the spectral peak of 296 cm^{-1} [33]. Moreover, the mineral phases of hematite and pargasite ($NaCa_2Mg_4Al_3Si_6O_{22}(OH)_2$) are also integrated at the spectral peak of 226 cm^{-1} while for albite and anorthite ($CaAl_2Si_2O_8$) are located at 503 cm^{-1} [25], [27]. Based on the XRF analysis, it can be concluded that the albite and pargasite phases are unlikely to exist in the Lokon ash mineral because Na and Mg oxides were not detected. Raman analysis did not detect any spectral peaks associated with the mineral ilmenite ($FeTiO_3$) phase. However, the ilmenite phase likely exists because the Fe_2O_3 and titanium oxide content was very significant, as shown in Table 1. The characterization results showed that the dominant mineral phase in the ash heated at 900°C and 1000°C is hematite, essential in increasing the reactivity of volcanic ash as a pozzolan material [24], [26].

C. X-Ray Diffraction (XRD) Analysis

Figure 4 describes the crystal structure and types of minerals obtained from the results of XRD analysis with the Profex 5.0 software [27], which was used to investigate the presence of the ilmenite phase in the sample. The results are consistent with the XRF analysis. It appears that there is a crystalline phase of plagioclase in the form of anorthite, as well as the presence of Fe_2O_3 phases in the form of magnetite (Fe_3O_4) and ilmenite [23], [33]. The XRD pattern of uncalcified ash material in the angle range of 2θ around 24° to 35° is similar to that shown in some references as a distinct natural pozzolan area with a small number of crystalline peaks dominated by amorphous forms [34].

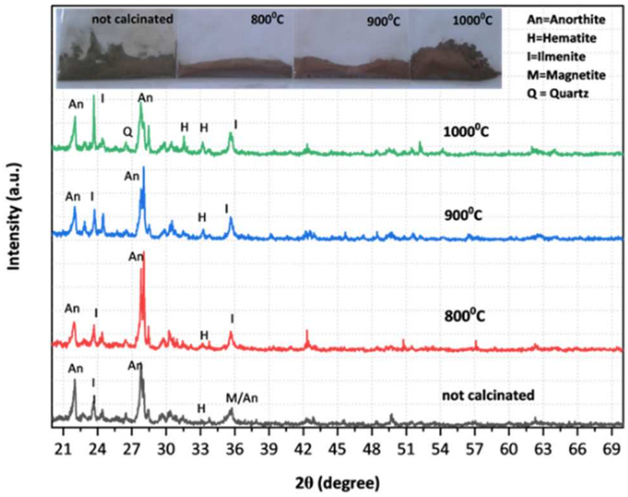

Fig. 4 XRD spectra of Lokon volcanic ash

Heating the ash samples at different temperatures increased the spectral peaks' intensity and made them sharper. A crystallization process also occurred, as demonstrated by the color change from dark grey to slightly reddish starting at 800°C. Increasing the calcination temperature to 900°C and 1000°C caused the grey color to turn redder, specifically at 1000°C, which is associated with the hematite phase [33]. The increase in crystallinity was confirmed, as shown in Figure 5.

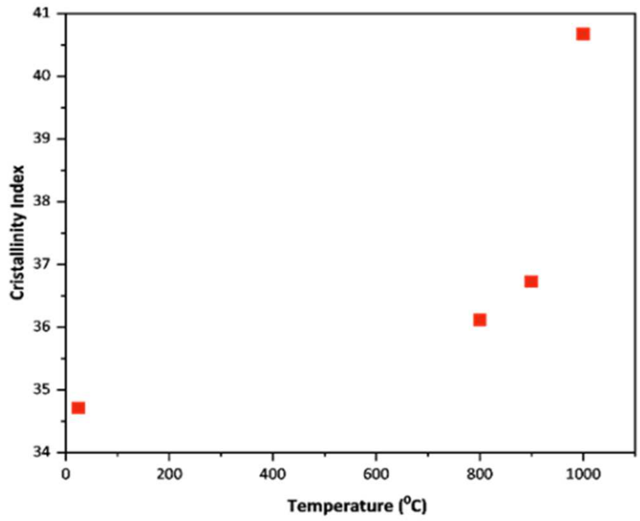

Fig. 5 Crystallinity of Lokon volcanic ash

In the XRD spectra at 1000°C, the spectral peaks around the 2θ angle between 22° and 28° were reduced in intensity compared to 800°C and 900°C. This reduction is related to the recrystallization and the transformation of the mineral phase to hematite. This was confirmed by Raman's analysis, which characterized the spectral peaks of hematite in samples heated at 1000°C. The higher the calcination temperature, the sharper and higher the intensity of the Raman spectra. The presence of hematite peaks in both the XRD and Raman spectra confirmed this. In other words, the results of the XRD analysis are appropriate to those obtained from Raman. XRD and Raman analysis results show that volcanic ash from Mount Lokon is thermally stable up to 800°C. It is related to concrete characteristics [11].

Based on the pattern, sharpness, and intensity of XRD, the ash samples calcined at 900°C and 1000°C can be used as a binder in the concrete mixture due to the increased Fe2O3 (hematite) content. At 1000°C, the crystallinity increased sharply compared to 900°C (Figure 5). Moreover, the particle size of the ash meets ASTM C618 criteria because it is smaller than 45 µm through the sieving process [21].

D. Fourier Transform InfraRed (FTIR) Analysis

FTIR analysis was used to determine the molecular bonding of compounds in ash materials using infrared waves. Figure 6 shows the same pattern, namely the absorption of intense and comprehensive infrared rays at intervals of wave numbers around 1600 cm^{-1} and 800 cm^{-1}, occurring in all samples with the peak intensity at 1450 cm^{-1} [24]. Water molecules trapped in the ash material vibrate at 3304 cm^{-1} for O-H bonds and 1630 cm^{-1} for H-O-H bonds [22], [23].

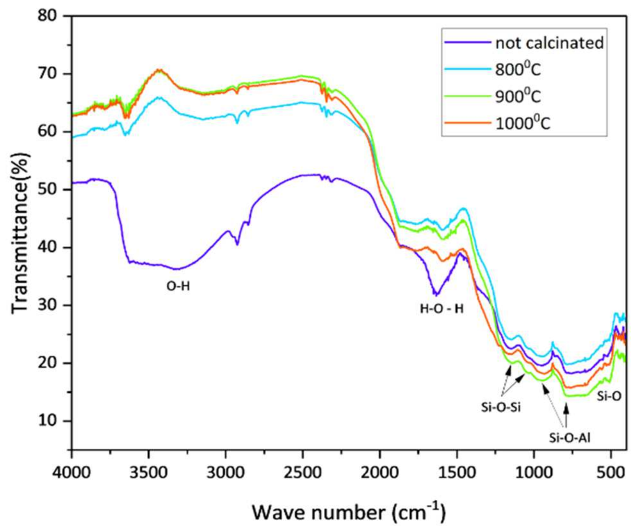

Fig. 6 FTIR spectra of volcanic ash

The quartz mineral vibrated at 557 cm^{-1} with the Si-O mode [22]. Two significant shoulder peaks from the FTIR spectra, namely at 1140 cm^{-1} and 1045 cm^{-1}, are related to the Si-O-Si bond mode [22]. Furthermore, the Si-O-Al bond stretching was found in the bands 929 and 943 cm^{-1}. The Si-O-Si and Si-O-Al bonds are associated with anorthite minerals in the Lokon ash material [23]. Calcination caused the release of water trapped in the O-H mode from its bonds at 3304 cm-1;

hence, the intensity of transmittance in the ash material increased.

FTIR results are consistent with the XRD and Raman analyses. The FTIR peak intensity of the sample calcined at 1000ºC was smaller than 900ºC, specifically in the wave number region of 600-1200 cm^{-1}, which is associated with Si–O–Si and Si-O-Al bonds. This result is consistent with the XRD spectra where the intensity decreased or even disappeared at an angle of 2θ between 22º and 28º but is offset by the emergence of new XRD spectra associated with the hematite phase. The Raman analysis results also confirmed this. The decrease was probably caused by changes in the Si–O–Si or Si-O-Al, where heat treatment and formation of solid solutions (hematite-ilmenite) affect the molecular structure of the anorthite mineral phase associated with the atomic vibration mode [24].

E. Scanning Electron Microscopy (SEM) Analysis

The surface morphological structure of samples that were not calcined or calcined from three types of ash sizes was studied from SEM data. Figure 7 shows that the ash particles from the uncalcined sample consisted of small aggregates and numerous micro- or nano-sized amorphous fragments. The ash samples calcined at 800ºC, 900ºC, and 1000ºC consisted of particles and fragments that began to accumulate one on another. The higher the temperature, the more the ash particles and fragments accumulate and coalesce as a dense matrix with clear particle boundaries. This is due to the formation of a hematite-ilmenite solid solution that can unite particles of all mineral compositions [24], [26]. SEM image shows that the calcination effect will increase the density of ash due to particle accumulation. Based on the physical characteristics above, Lokon volcanic ash can be used as a substitute for cement, which will act as a binder [35], [36].

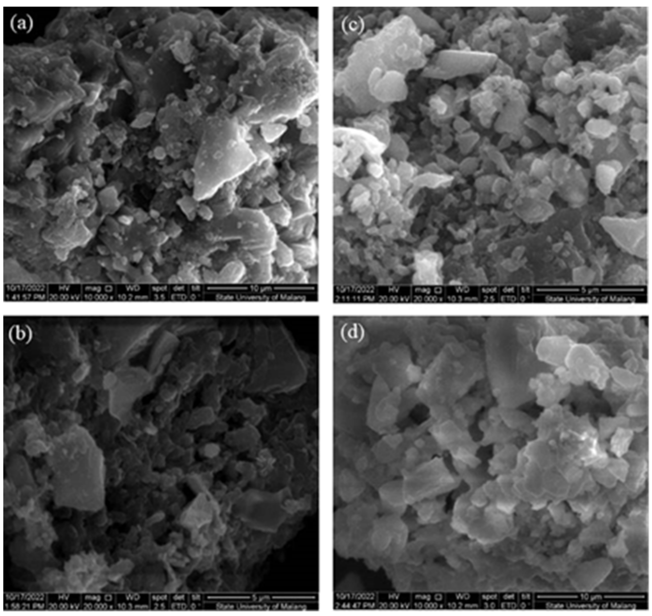

Fig. 7 SEM image of Lokon ash (a) not calcinated, (b) 800ºC, (c) 900ºC, (d) 1000ºC

As an activated alkaline binder, volcanic ash will increase cohesion and mechanical strength in concrete composites [37]. Using volcanic ash as much as 30-50% by volume in the concrete mix has shown compressive strength comparable to concrete from OPC cement, lower CO_2 emissions, and comparable costs [13], [38]. From a thermal, chemical, and microstructural point of view, substituting OPC cement with volcanic ash will control the reaction kinetics and play a role in forming new hydration products in the cement mixture [30], [31].

Lokon volcanic ash has the potential as a substitutional binder for OPC cement because it contains a significant element of iron and has good thermal stability. The significant iron content in the alkaline active material can change the nanostructure of the bonding phase, thereby accelerating the reaction and strengthening the concrete mix bond [39]. The iron content in volcanic ash and its combination with phosphoric acid will speed up the hardening time of concrete and increase its compressive strength [40]. Lokon ash was characterized as an aluminosilicate to form phosphate geopolymers. Phosphate geopolymer has the best thermal stability at 1000⁰C where crystallization of iron (III), phosphate (V), and hematite occurs [41]. Hematite formation has been shown in the results of characterization by Raman spectroscopy. Finally, using volcanic ash from Mount Lokon is very prospective for the cement industry because substituting volcanic ash by 10% in a cement mixture can reduce overall costs by 30% and increase mortars' strength and durability properties [43], [44]. Implementing cement supplement materials, including volcanic ash, will reduce CO2 emissions by 1.7 million tons per year and create around 50,000 jobs [45].

IV. CONCLUSION

Volcanic ash derived from the sand of Mount Lokon fulfills ASTM C618 requirements as a pozzolan material both in terms of particle size and composition of SiO_2, Al_2O_3, and Fe_2O_3. Calcination increased the crystallinity of the ash due to the presence of the hematite phase. Hematite formation at 900ºC and 1000ºC improved the reactivity of Lokon ash as a pozzolan material. The increase in ash crystallinity due to calcination did not change the amorphous percentage significantly beyond the minimum limit of 36%; hence, calcined ash has excellent potential as a natural binder. Further investigations are recommended to implement calcined Lokon ash into the concrete mix. Variations in the composition of ash with cement, sand, and water need to be studied for their effect on the compressive strength of the concrete mixture. This will facilitate using Lokon volcanic ash on a small industrial scale.

NOMENCLATURE

ASTM	American Standar Testing and Materials
FTIR	Fourier Transform InfraRed
OPC	Ordinary Portland Cement
SEM	Scanning Electron Microscopy
XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence

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