

REZISTENȚA LA COMPRESIUNE ȘI CARACTERISTICILE MICROSTRUCTURALE ALE GEOPOLIMERILOR PE BAZĂ DE CENUȘĂ ZBURĂTOARE CU CONȚINUT RIDICAT DE CAUCIUC NATURAL COMPRESSIVE STRENGTH AND MICROSTRUCTURAL CHARACTERISTICS OF FLY ASH BASED GEOPOLYMER WITH HIGH VOLUME FIELD PARA RUBBER LATEX

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This study assessed and investigated the compressive strength and microstructure of geopolymer mortars containing field Para rubber latex (FPRL). Fly ash based geopolymer mortar blends were prepared with FPRL to fly ash ratios 0.10:1, 0.20:1 and 0.30:1 by weight, and with varied heat curing times. The microstructure was characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD), and Fourier transform infrared spectroscopy (FTIR), and additionally compressive strength and density were determined. The results show that geopolymers with FPRL had compressive strengths in the range 10-30 MPa at 28 days of age. Assessed SEM images showed that FPRL gave a highly porous microstructure and correspondingly low density. The geopolymer mortars incorporating FPRL could be used in building walls or in paving blocks.

Keywords: microstructure; geopolymer; mortar; field Para rubber latex; density

1. Introduction

Geopolymers have potential for use in several industrial applications [1]. The geopolymer binders are aluminosilicate inorganic polymers and have expanding applications as construction materials. Several studies have reported that geopolymer binders possess many advantageous properties, specifically good early strength, high ultimate strength, low shrinkage, and excellent durability. However, some geopolymers have low compressive strength depending on the mixture ratio and source or type of materials used [2-4]. Shadnia *et al.* [5] showed that when geopolymer mortars prepared with fly ash and phase change materials the strength decreased with the content of phase change materials. Ban *et al.* [6] studied the effects of wood ash on compressive strength, flexural strength, dynamic modulus, water absorption, and microstructure of class f fly ash based geopolymer mortar blocks cured at ambient temperature. The results revealed that increasing the wood ash content to 50, 60, or 70% gave poor strength mortar blocks. However, such comparatively low strength geopolymer mortar/concrete can still be applied in building construction, as masonry or as paving blocks.

The main product of geopolymerization reactions is alkali aluminosilicate, which contributes to the good mechanical properties and resistance to

chemical attacks in geopolymers. However, the physical and mechanical properties of component phases do not sufficiently explain the behavior of geopolymers, instead microstructural analysis of geopolymers is necessary. The information about the structure of geopolymers obtainable by X-ray diffraction (XRD) is rather limited. Consequently, scanning electron microscopy (SEM) and Fourier transform infrared (FT-IR) were considered for additional information in this study. Some studies [7, 8] have investigated the geopolymer structure by SEM and FT-IR.

In its fresh or field state, FPRL contains also non-rubber substances such as sludge, proteins, and some inorganic materials along with its 30-40% of rubber particles [9]. Generally, FPRL in Thailand has 20-45% rubber particles and 50-75% water and other materials [10]. In 2015, Thailand produced approximately 4,466,000 tons of Para rubber [11]. Thus, the amount of FPRL produced annually in Thailand is of the order 10 to 22 million tons. Prior research [12] indicates that concrete containing natural rubber latex has reduced strength, but only little research is available on the applications of FPRL in construction materials. In particular, compressive strength, density, and microstructure of geopolymers from fly ash containing FPRL are currently not well established. This gap in knowledge can be addressed by investigating the effects of FPRL in geopolymer matrix composites.

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Table 1

Chemical composition of fly ash.

Component	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	SO ₃	K ₂ O	MgO	Na ₂ O	TiO ₂
% by weight	45.3	23.0	9.4	12.5	2.7	2.3	1.7	1.0	0.4

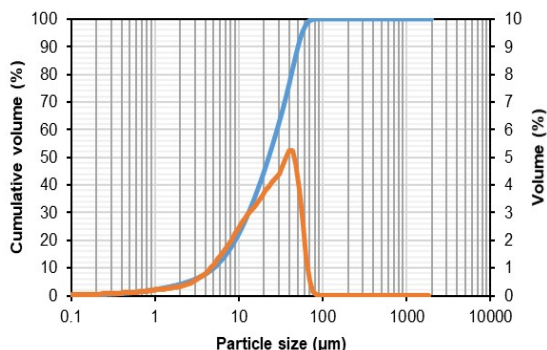


Fig. 1 - Particle size distribution of fly ash.

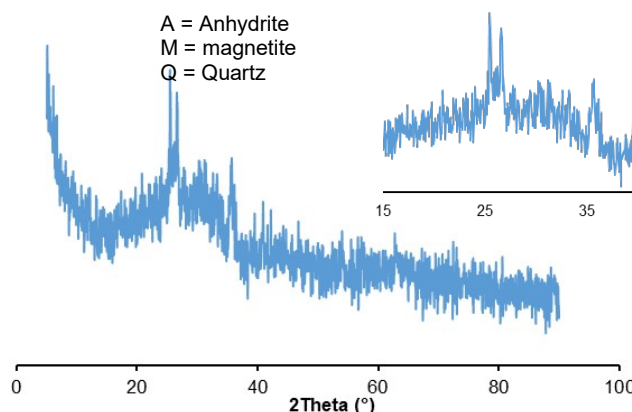


Fig. 2 - X-ray diffraction pattern of fly ash.

The current study focuses on fly ash with FPRL. This work aimed to study the strength and the microstructure of fly ash based geopolymer with FPRL included in the blend, with various heat curing times. Samples were prepared with FPRL contents of 0%, 10%, 20%, and 30%, and were heat cured in an oven at 80 °C for 0.5 (30 min), 1, 2, or 4-h. Measurements were taken after 0 h, 1, 7 and 28 days, following the standard hardening times used in specifying the quality of concrete. The study aimed to analyze the effects on compressive strength, density, and to assess the microstructure using SEM, XRD, and FT-IR techniques.

2. Materials and experimental methods

2.1. Materials

The materials used in this research to produce geopolymer samples were fly ash, field Para rubber latex, sodium silicate (Na₂SiO₃), sodium hydroxide (NaOH), water, and river sand. The chemical composition of fly ash is shown in Table 1 as analyzed using X-ray fluorescence (XRF); it had the main components 45.3% SiO₂, 23.0% Al₂O₃ and 9.4% Fe₂O₃. Based on ASTM C618 [13] this fly ash fell into class F, as the total content of SiO₂ + Al₂O₃ + Fe₂O₃ was greater than 70%. The fly ash was ground in a ball mill and the produced particles size distributions were determined with a Mastersizers Malvern Instrument, with results shown in Figure 1. The main phases included anhydrite (Calcium sulfate; Ca(SO₄)), magnetite (Iron oxide; Fe₂O₄) and quartz (Silicon oxide; SiO₂) as detected by X-ray diffraction (XRD), see Figure 2.

Field Para rubber latex (from clonal variety RRIM 600 of rubber trees) used in this study was collected from southern Thailand. The FPRL is a

suspension with 35-40 % total solids content. The chemical analysis and physical properties of FPRL are shown in Table 2 [14].

The alkaline activators of geopolymerization reactions were prepared of sodium silicate (Na₂O = 14.85%, SiO₂ = 29.45% and H₂O = 55.7%), sodium hydroxide (NaOH) in flakes of 99% purity, and water.

River sand passing No. 4 sieve, with particle size below 4.75 mm, the fineness modulus of 2.59, and specific gravity of 2.56, was used to prepare geopolymer mortars.

Table 2

Chemical analysis and physical properties of field Para rubber latex

Property	Value
Total solids content (%)	38.6
Dry rubber content (%)	35
Water (%)	61.4
pH	6.5-7
Density (g/ml.)	0.975-0.980
Particle size (μm)	0.04-4.0
Mean particle size (μm)	1

2.2. Specimen preparation

Geopolymer mortars were prepared by mixing fly ash, field Para rubber latex, river sand, sodium silicate, sodium hydroxide and water. The mixture proportions are given in Table 3. First, the fly ash and river sand were mixed for about 3 min. Second, the sodium hydroxide and water were mixed in sodium silicate to a homogeneous alkaline solution. Then, the alkaline solution was added to and mixed with the fly ash and river sand from the first step, for another 3 min. A further mixing of about 5 min was done after adding the FPRL. The mortar samples were then poured into acrylic molds and wrapped with polyvinyl, and then

Table 3

Mixed proportion of geopolymer mortars (by weight)

Code label	FA (g)	FPRL (g)	SS (g)	SH (g)	W (g)	RS (g)	Heat curing (h)
C-0.5	227.27	0	54.55	36.36	56.82	625	0.5 (30 min)
10L-0.5		22.73					
20L-0.5		45.45					
30L-0.5		68.18					
C-1	227.27	0	54.55	36.36	56.82	625	1
10L-1		22.73					
20L-1		45.45					
30L-1		68.18					
C-2	227.27	0	54.55	36.36	56.82	625	2
10L-2		22.73					
20L-2		45.45					
30L-2		68.18					
C-4	227.27	0	54.55	36.36	56.82	625	4
10L-4		22.73					
20L-4		45.45					
30L-4		68.18					

Abbreviations used: FA = fly ash; FPRL = field Para rubber latex; SS = sodium silicate; SH = sodium hydroxide; W = water; RS = river sand

cured at 80 °C for 0.5, 1, 2 or 4 h. After heat curing, the molds were removed from the oven. After unwrapping and demolding the samples were stored at ambient temperature until testing.

The geopolymer samples were prepared in 50 mm cube molds to test their compressive strength at the ages 0 h, 1, 7, and 28 days.

2.3. Test procedure

The compressive strengths of geopolymer mortars were determined according to ASTM C 109/C 109M [15], using a universal testing machine at a loading rate of 5.00 mm/min.

The growth of the geopolymerization structures on the paste samples was illustrated using SEM, XRD, and FT-IR. A JMS-5800 LV model scanning electron microscope (JEOL, Japan) was used to identify the microstructure of the geopolymer paste. Powder XRD of geopolymer paste was conducted using an X'Pert MPD X-ray diffractometer (PHILIPS, Netherlands) angles from 5° to 60° (2θ) using the clay and rock 0.4 program. XRD was conducted to identify the notable crystalline phases and to detect the positions of the peaks. FT-IR was performed on the paste samples on the EQUINOX 55 spectrometer (Bruker, Germany) using the KBr pellet technique in 4000-400 cm⁻¹ range.

3. Results and discussion

3.1. Compressive strength

The compressive strength of geopolymer mortars gave mean strengths that were compared between the experimental design parameters. The results across various amounts of FPRL, curing times in the oven, and hardening at ambient temperature are graphically shown in Fig. 3. It can be seen that the compressive strength improves, in general, both with the curing time and with the hardening time. The C-4 case without FPRL achieved high early (0 h) strength, about 87% of its

28-day compressive strength. Hawa *et al.* [16] reported that extended heat curing of metakaolin-based geopolymers could speed up the geopolymerization and give high early strength. However, as Fig. 3a shows, the 28-day compressive strength of C-4 sample (with only fly ash) was astoundingly the highest among samples heat cured for 1 h in the oven. The research progress on the geopolymers heated curing for 1 h has been rarely available. Rovnaník [17] used metakaolin with geopolymer binders while varying the heat curing conditions. He reported that the 28-day compressive strength of geopolymer containing only metakaolin was significantly improved in the samples cured for 1 h. Also, Hawa *et al.* [18] reported that metakaolin is able to reach higher compressive strengths when cured for 1 h as a hot mixture, as confirmed for fly ash based geopolymers in this current study. Thus, whether the geopolymer was prepared with fly-ash or metakaolin, the maximum compressive strength was obtained at the age of 28 days.

As seen in Figures 3b-d, the compressive strength of the geopolymers decreased with FPRL content. This loss of compressive strength suggests decreased as shown in the SEM images of geopolymer pastes in Section 3.2.1. The compressive strength was highest at 28 days of hardening, in all experimental cases, and it clearly decreased with FPRL content. This is because of the water in FPRL, contributing free water in the geopolymer mixes. Xu *et al.* [19] reported that FA based geopolymer composites with the increase of water-to-binder ratio the compressive strength of geopolymer has decreasing. Ban *et al.* [20] synthesized geopolymer from coal fly ash and wood ash with higher water-to-binder ratio gave lower strength at all curing ages. Moreover, some behavior and mechanical properties of geopolymers based on MK and sludge [21, 22] have a similar tendency to these prepared with fly-ash. Yang *et al.* [21] reported that the compressive

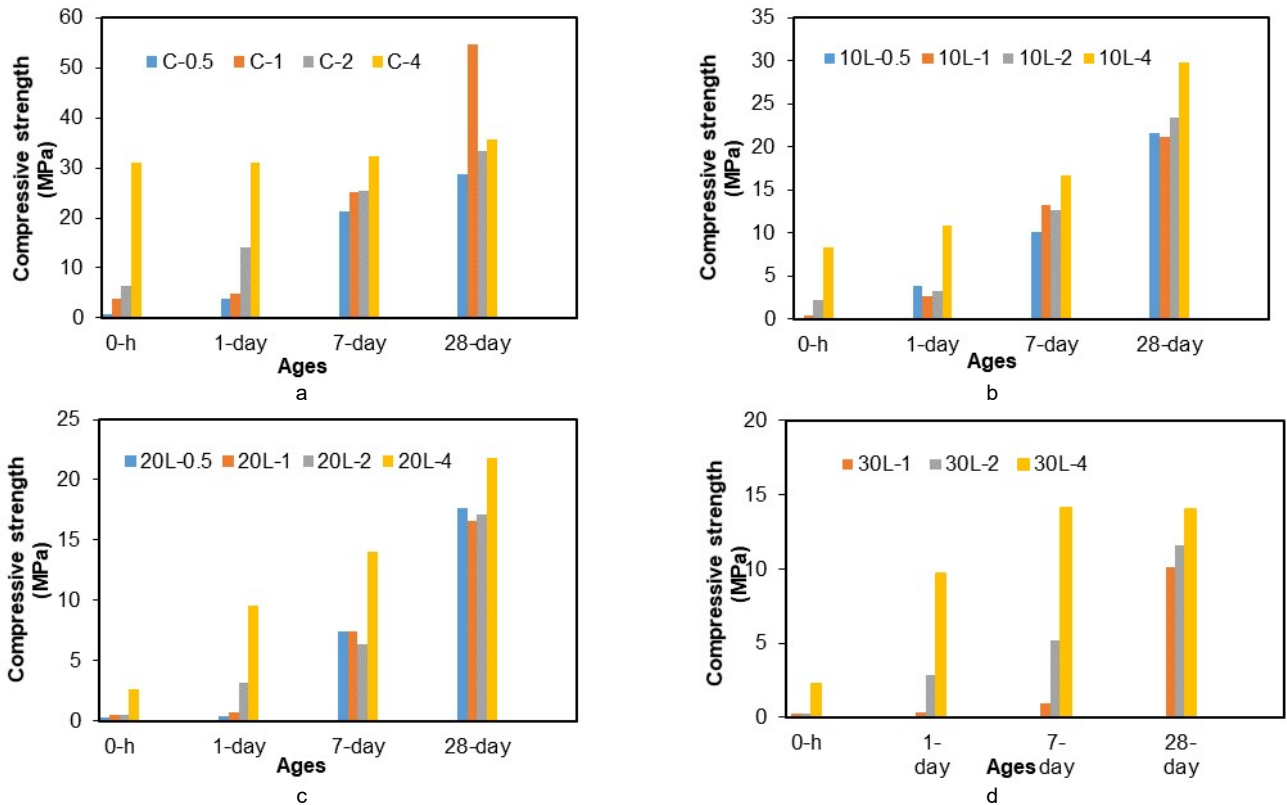


Fig. 3 - Compressive strengths of geopolimer mortars at various hardening times. The heat curing times are shown in the legends. The plots compare the cases (a) without FPRL, (b) 10% FPRL, (c) 20% FPRL, and (d) 30% FPRL.

strength of geopolimer with slag powders is better with the water-binder ratio $w/b = 0.4$ than with $w/b = 0.5$. Chen *et al.* [22] determined the effects of water-binder ratio on geopolimers with reservoir sludge partially replacing metakaolin, and high water-binder ratios reduced the strength. Ye *et al.* [23] produced geopolimers with red mud, and the compressive strength decreased with the water-binder ratio. The average compressive strengths of our geopolimer mortar specimens, with various amounts of FPRL, heat cured at 80 °C for 4 h, are shown in Figure 4. The C-4 had slightly lower early compressive strength than its 28-day strength. In the samples containing FPRL, longer curing accelerated the development of compressive strength.

As expected, it can also be seen in Figure 5 that the 28-day density of geopolimer mortar decreased when its content of FPRL was high. The pores of geopolimer mortars are crucial to its density and compressive strength. Figure 5 show the density against the content of FPRL in the geopolimer mortars. The decrease in density with high FPRL content is simply caused by the FPRL having lower specific gravity than the fly ash.

3.2. Microstructure characterization

3.2.1. Scanning electron microscope (SEM)

The SEM images of geopolimer pastes at the magnifications 500× and 1500×, after heat curing for 4 hours, at 28 days of age (or hardening)

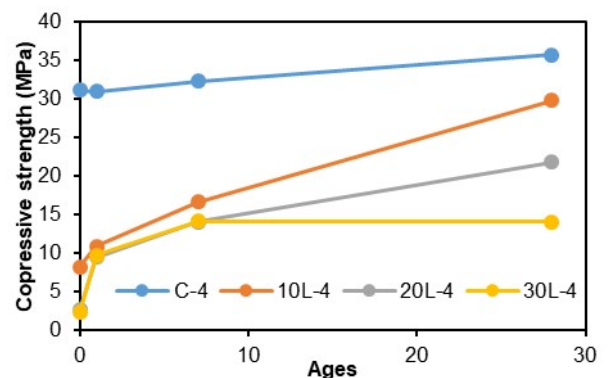


Fig. 4 - Compressive strength development with hardening time for geopolimer mortars with FPRL, after heat curing at 80 °C for 4 h.

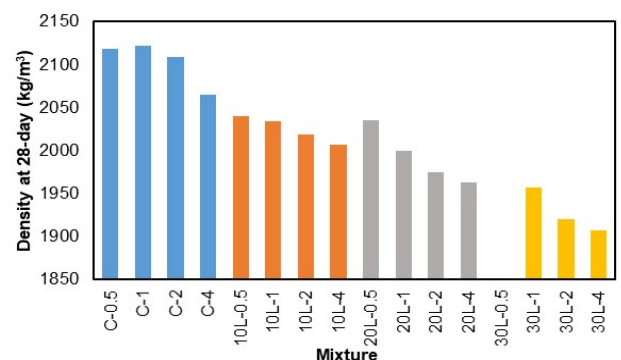


Fig. 5 -Bulk densities of all the experimental geopolimer mortars.

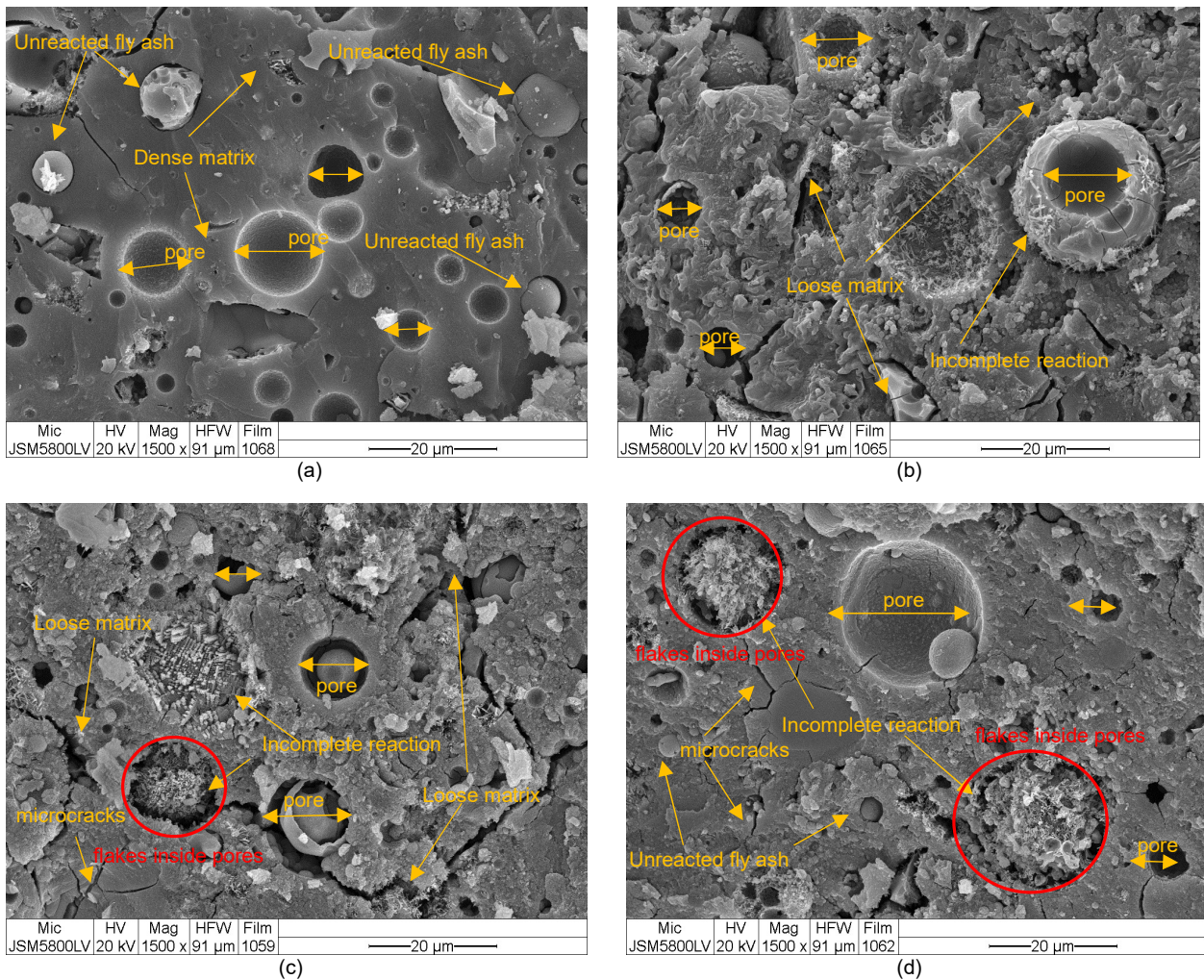


Fig. 6- SEM images of geopolymer pastes; (a) C-4, (b) 10L-4, (c) 20L-4 and (d) 30L-4.

are shown in Figure 6. The effects of various FPRL contents are seen in the SEM images (Fig. 6 for FPRL:FA ratio of 0.0:1, 0.1:1, 0.2:1 and 0.3:1). The images show the pores at the surface, and pore sizes and the matrix can be assessed after the geopolymerization of aluminosilicates. On comparing with the geopolymer blends with FPRL (10L-4, 20L-4 and 30L-4) in Figs. 6b-d, the C-4 had dense compact and homogeneous matrix with small pores (Fig. 6a). The differences are possibly due to higher extent of reactions between the fly ash and the alkaline activator. The compressive strength is reduced due to the decrease in the activator concentration. The activator concentration has an effect on the compressive strength and the microstructure of geopolymers [24, 25], to form aluminosilicate gel, which also would contribute to the higher compressive strength of the C-4 sample. Moreover, microcracks are observed in the samples with FPRL, contributing to their low strength. The spherical surfaces of FA are clearly observed, and with addition of FPRL there were flakes and large pores. The SEM results match the low compressive strength and density of geopolymers with FPRL. The pores in the 10L-4, 20L-4 and 30L-4 cases can be considered

macropores. Clearly both pore size and porosity increased with FPRL content, as shown in Figs. 6b-d. The microstructure was investigated by SEM imaging, showing increased porosity with FPRL content. The trend of result in previous research has been similarly adopted [16]. The geopolymer had high porosity that degraded its compressive strength.

3.2.2. X-ray diffraction (XRD)

The XRD patterns of fly ash based geopolymer pastes aged for 28 days at ambient temperature are shown in Figure 7. The samples presented amorphous phase as a peak between 23 and 37° (2θ). It is clear that the alkali activation of aluminosilicate results in the formation of amorphous aluminosilicate gel, which then gives the good chemical and mechanical properties [26]. The C-4 case had magnetite and quartz from the fly ash. The cases with FPRL also generated other chemical products such as sodalite (sodium aluminum silicate chloride; $\text{Na}_8(\text{Al}_6\text{Si}_6\text{O}_{24})\text{Cl}_2$), shown as broad humps at approximately 14° , 24° , 35° and 42° (2θ). The samples had similar diffraction patterns and did not significantly differ in their splits to amorphous and crystalline phases,

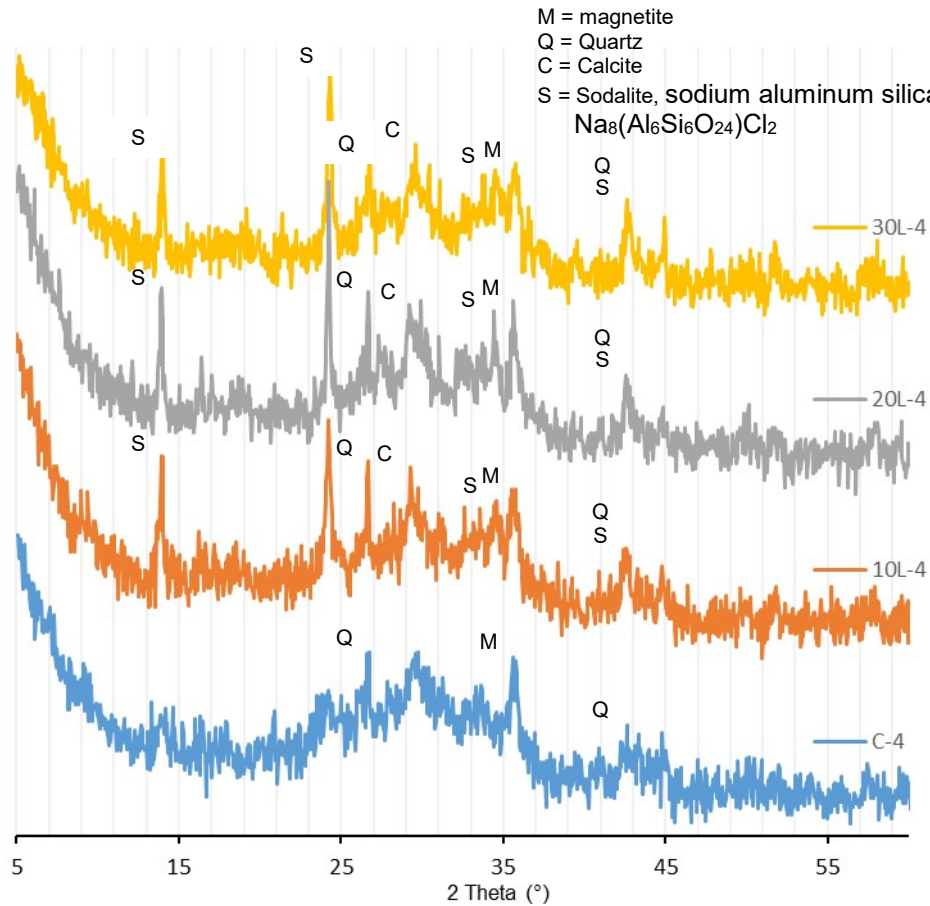


Fig. 7 - XRD pattern of geopolimer pastes with FPRL, heat cured at 80 °C for 4 h.

including the case C-4 for which the peaks of sodalite almost disappeared. The cases with FPRL had obvious new peaks around 29° and 42° (2θ) for calcite and quartz, respectively. Considering the intensity of these crystalline peaks, they did not match the compressive strengths observed in this study. However, previous studies [27, 28] have reported for fly ash based geopolymers that increased crystallinity matched improved compressive strength. In the current case, the loss of compressive strength is dominantly caused by porosity effects, as opposed to chemistry of reaction products (See Figs. 6b-d).

3.2.3. Fourier transform infrared spectroscopy (FT-IR)

The FT-IR spectra are presented across wave numbers 4000-400 cm⁻¹ for the geopolimer pastes aged at ambient temperature for 28 days in Figs. 8a-d. Table 4 indicates a major band for these geopolimer fly ash with FPRL systems, and the main peak shifts with FPRL content. For fly ash, the bands for O-H stretching and H-O-H bending of water molecules are at 3449 cm⁻¹ and 1634 cm⁻¹, respectively. In the geopolimer matrix the band of O-H is slightly changed by the geopolimer, and the H-O-H band shifted to the higher wavenumber 1634 cm⁻¹ by about 10 cm⁻¹.

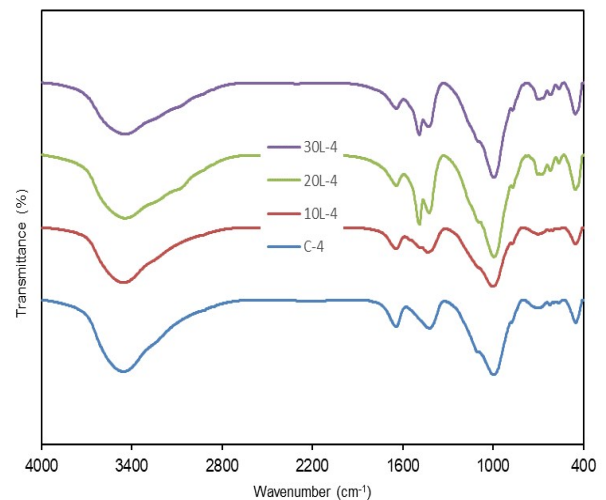


Fig. 8 - FT-IR spectra of geopolimer pastes.

The carbonate in geopolimer matrix is seen in the bands around 1490 cm⁻¹ and 1430cm⁻¹. Mozgawa and Deja [29] studied alkaline activated slag, and the carbonate in the system was consistent with calcite. The results of the present study confirm that in the composition carbonate is in the form of calcite (XRD pattern in Fig. 7).

Table 4

Detailed data about the transmittance peak of geopolymer paste

Functional group	Wavenumber (cm ⁻¹)				
	FA	C-4	10L-4	20L-4	30L-4
O-H	3449	3451	3448	3445	3441
CH ₃ , CH ₂ stretching	2920, 2851	-	-	-	-
H-O-H	1634	1644	1643	1643	1644
CO ₃ ²⁻	-	-	1489	1490	1489
C-H	1473	-	-	-	-
CO ₃ ²⁻	-	1434	1426	1427	1431
Si-O stretching	1028	1002	997	995	996
Si-OH stretching	-	872	871	870	871
Al-O-Si	-	700	706	682	695
Si-O bending	461	452	455	455	456

The most significant transformation is that the bands attributed to asymmetric stretching vibrations of Si-O- at 1,028 cm⁻¹ in the fly ash shift to around 1,000 cm⁻¹ in the geopolymer matrix. The intensity of the bands attributed to stretching vibrations of Si-OH at around 870 cm⁻¹ and vibrations of Al-O-Si at around 700 cm⁻¹ in the fly ash increase significantly in the geopolymer matrix. The FT-IR spectra of the geopolymers show a distinct intensity band between 1300 and 900 cm⁻¹ associated with the Si-O- asymmetric vibrations. This band is often used to determine the degree of geopolymerization [30].

4. Conclusion

The present study investigated geopolymer mortars containing FPRL. The samples were prepared as hot mixtures with four experimental FPRL to fly ash ratios, namely 0.0:1, 0.10:1, 0.20:1 and 0.30:1, and the following conclusions may be deduced.

a) Heat curing for 4 h increased the early compressive strength, especially for the C-4 case with only a slight increase from 4-h to 28-day compressive strength.

b) Heat curing stimulates the geopolymerization reactions; hence, long heat curing times (4 h) gives good early compressive strength in all cases tested.

c) High FPRL content in the geopolymer matrix reduced its density and compressive strength. FPRL in the samples produced small pores.

d) SEM imaging made it evident that with FPRL the geopolymer matrix displayed loose conformations and inhomogeneous phases.

e) Generally, increased crystallinity improves the compressive strength of a geopolymer, but in the current cases the porosity effects on compressive strength were dominant.

f) The FT-IR spectrum of these geopolymers had a distinct intensity band at 1300-900 cm⁻¹, associated with Si-O- asymmetric vibrations. It was observed that the geopolymers with FPRL

contained new reaction products such as calcite, from the modes of CO₃²⁻ ions.

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MANIFESTĂRI ȘTIINȚIFICE / SCIENTIFIC EVENTS

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The key goal of the conference is to enable the transfer of information between specialists in these disparate areas of science and engineering:

- Significantly reduce Greenhouse gas emissions in construction
- Improve the technical performance of materials used in construction, composites and related products
- Use large volumes of industrial (waste) by-products or low-value natural minerals in place of virgin resources
- Effectively immobilize hazardous, toxic and/or radioactive wastes
- Increase resource efficiency by producing cementitious and concrete products with longer service lives.

Contact: <http://www.engconf.org/conferences/materials-science-including-nanotechnology/geopolymers-ii-versatile-materials-offering-high-performance-and-low-emissions/>
