

# Influences of High Temperatures and Environmental Conditions on Mechanical Properties of Geopolymer Mortar based on Fly Ash

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**Abstract** - Fly ash based geopolymer mortar can sustain itself when exposed to considerably high temperature. While Ordinary Portland cement (OPC) to product mortar degrades and degenerates at high temperature, it has been found through different studies that fly ash geopolymer mortar can maintain its desired compressive strength at 400 degrees centigrade. Its strength starts deteriorating once the temperature crosses 400 degrees centigrade. In sum, its strength remains almost constant at higher temperatures. Environmental conditions (water, acid, ice) also influence geopolymer mortar, due to their direct effect on its mechanical, chemical and physical properties. In this paper, basic processes will be described and typical test results will be presented to illustrate the various parameters.

**Keywords** - Fly Ash, High Temperature, Environmental Conditions, Compressive Strength, Geopolymer Mortar.

## I. INTRODUCTION

The manufacture of OPC releases large amount of CO<sub>2</sub> (from 74 % to 81 % of the total CO<sub>2</sub> emissions of concrete) to the atmosphere, because the chemical reaction process creates CO<sub>2</sub> from the calcinations of limestone (calcium carbonate - CaCO<sub>3</sub>) at very high temperatures (about 1450 °C) and silica according to the reaction:  $3CaCO_3 + SiO_2 \rightarrow Ca_3SiO_5 + 3CO_2$

The production of one ton of OPC emits approximately one ton of CO<sub>2</sub> into the atmosphere [1]. About 2.5 billion tons of cement is produced every year, which amounts to almost 0.3 tons for every person on the planet. By 2050, global production is expected to reach 5 billion tons, meaning that approximately 5 billion tons of CO<sub>2</sub> will be released into the atmosphere [2]. Fig. 1 shows the projections for the global demand of the main binder OPC of concrete structures. Therefore, there is a need to find alternative types of binders to produce more environmentally friendly mortar and concrete. Recently, geopolymer has emerged as a promising new material with its environmentally sustainable properties. These properties have attracted much attention due to their excellent fire resistance (up to 1000 °C), mechanical properties and long-term durability, heavy metal ions fixation and acid resistance (including sea water), low shrinkage and low thermal conductivity [1-5]. Potential applications of geopolymer based materials used in many fields of industry includes: automotive and aerospace industries, especially for various applications that require high temperature resistance

and thermal insulation, new ceramics, cements and concrete, asbestos-free materials and high-tech materials [1, 6, 7]. In this work, geopolymer resin was synthesized from shale fly dust burnt in a rotary kiln (for 10 hours at 750 °C) with Si/Al molar ratio of 2.0 with sodium hydroxide (NaOH) and sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>). The purpose of this current research is observe the influence of high temperature on mechanical properties of geopolymer mortar.

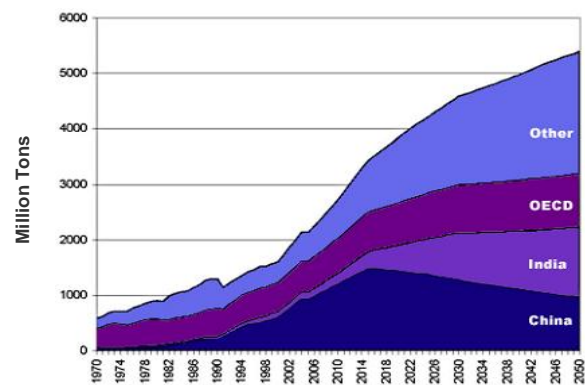


Fig. 1. Global cement demand by region and country [8].

## II. EXPERIMENTAL

### A. Materials

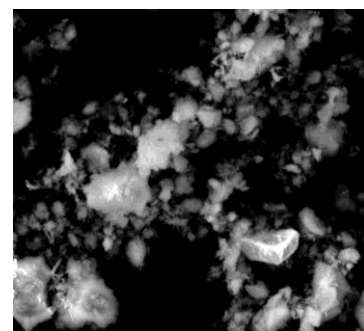


Fig. 2. SEM image with magnification 5000x of powder cement.

In this research, geopolymer material was synthesized from powder cement produced by shale fly dust burnt in a rotary kiln (for 10 hours at 750 °C) with Si/Al molar ratio of

2.0 combinations with sodium hydroxide (NaOH) and sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>) with modulus 1.5. The specific surface area of powder cement was 20.8 m<sup>2</sup>/g and the mean particle size was d<sub>50</sub> = 4.2 μm and d<sub>90</sub> = 9.3 μm. The microstructure of powder cement was analyzed by scanning with an electron microscope (SEM) in Fig. 2 and X-ray diffraction data and determination of LOI (see Table I).

TABLE I. CHEMICAL COMPOSITION OF POWDER CEMENT AS DETERMINED BY XRD

Compound	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	SO <sub>3</sub>	CaO	LOI
[% mass]	41.6	52.6	2.6	1.1	0.8	1.3

**B. Fly ash**

Currently, over 40 percent of fly ash is used annually in a variety of engineering applications [9-11]. Fly ash has been used in several areas, such as: Portland cement concrete, soil and road base stabilization, bricks, flow able fills, grouts, structural fill and asphalt filler, etc [11]. In addition, it is widely used as an additive in the cement, mortar and concrete building industry worldwide [11, 12].

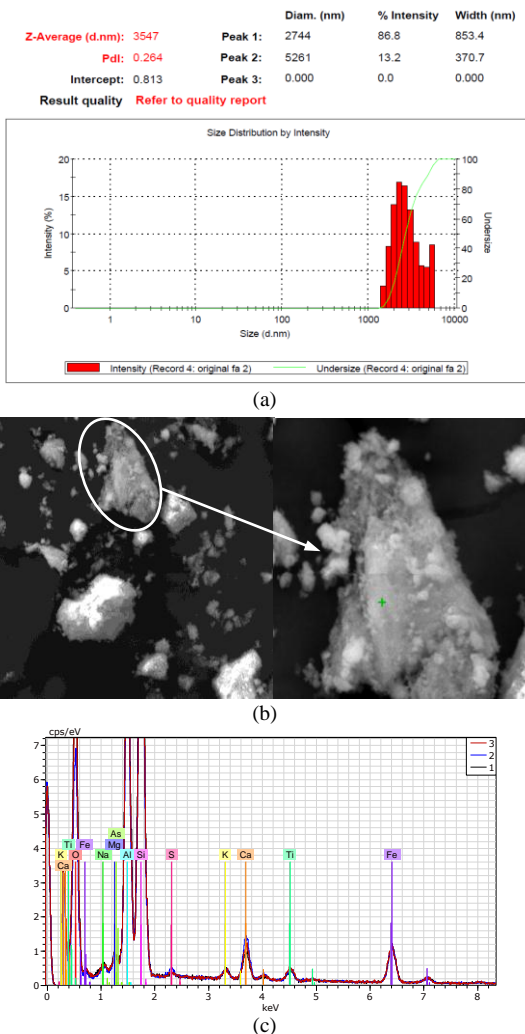


Fig. 3. Particle size distributions (a), SEM photographs (b) and EDX mapping (c) of an individual fly ash at magnification 5000x.

Also, fly ash is a good source material for making geopolymer owing to its high content of silica and alumina [13-15]. The fly ash geopolymer can totally substitute the use of normal Portland cement. Fly ash has many different colors such as brown and light grey to black due to its chemical compositions and contaminants. In this paper, the researchers used the brown color fly ash K6\_LF from sources in the Czech Republic. Fly ash particles are generally sharp, pointed, and with a characteristic particle diameter Z-average about 3547 nm as shown in the Fig. 3a. An Energy Dispersive X-ray Analysis (EDX) (see in Fig. 3c) on TESCAN VEGA 3XM microscope was employed to analysis chemical compositions of fly ash (in Table II).

TABLE II. QUANTITATIVE ELEMENTAL ANALYSIS DATA OF FLY ASH K6\_LF

Element	Atomic [%]	Standard deviation
O	52.81	0.52
Na	1.81	0.10
Mg	0.97	0.06
Al	14.73	0.18
Si	23.97	0.52
S	0.39	0.05
K	0.41	0.04
Ca	1.69	0.29
Ti	0.57	0.06
Fe	2.57	0.11
As	0.09	0.01

**C. Fabrication of geopolymer mortar**

Test specimens were used with eight mixtures of mortar to test high temperature. The details of mixtures are given in Table III. In this study, the researchers used cylinder specimens (Ø50 x 100) mm with accordance AS 1012.9 – 1999 to determine the compressive strength of mortar after heating at high temperature and environmental conditions. The compressive strength of geopolymer mortar is measured on a VEB Werkstoff Prüfmaschinen Leipzig 500 kN in ambient condition. Values are the averages of four separate tests. Data that deviated more than 10 % were eliminated.

TABLE III. COMPOSITION OF FRESH GEOPOLYMER MORTAR K6\_LF MIXES BY ADDING ALKALINE

Mixtures No	Materials			
	Fly ash [%]	Geopolymer Cement [%]	Alkaline [%]	Fine sand [%]
MLF <sup>-</sup> 2	20	39.5	40.5	-
MLF <sup>-</sup> 3	30	33	38	-
MLF <sup>-</sup> 4	40	22	38	-
MLF <sup>-</sup> 6	25	28	38	9
MLF <sup>-</sup> 7	25	23	35	17
MLF <sup>-</sup> 8	25	18	32	25
MLF <sup>-</sup> 9	25	12	33	30
MLF <sup>-</sup> 10	25	8	31	36

**D. Effect of high temperature**

All samples after curing at room temperature for 28 days are heated in the oven ranging from 200 °C to 1000 °C at a heating rate of 5 K/min and with a soak time of 1 hour at the maximum temperature and finally cooled in the furnace with an opening gate for 24 hours. The weight loss and shrinkage of specimen were also investigated.

**E. Effect of environmental conditions**

The researchers used a climate chamber LIEBISCH KB 300 and a freezer to test the effects of moisture (relative humidity) conditions, freeze/thaw and wet/dry on geopolymer mortar.

**Climate chamber test:** The cycles were stopped after 28 days curing (about 120 cycles) with the conditions: distilled water, cyclic changes of temperature and humidity see in Fig. 4. In this paper, the researchers ran trials with mixtures from MLF<sup>-6</sup> to MLF<sup>-10</sup>.

**Freeze-Thaw:** The samples were first saturated with water and then frozen at -15 °C for 24 hours. Next, the samples were removed from the freezer and immediately put into water without thawing. The cycles were stopped after 28 days (sufficient time duration for regular laboratory testing). After 28 days curing at room temperature, the compressive strength against the initial strength.

**Wet-Dry:** The samples were first saturated with water and then dried at 70 °C for 24 hours. Next, the samples were taken from the furnace and immediately put into water without cooling. The cycles were stopped after 28 days (sufficient time duration for regular laboratory testing). The compressive strength and initial strength were compared after 28 days curing at room temperature.

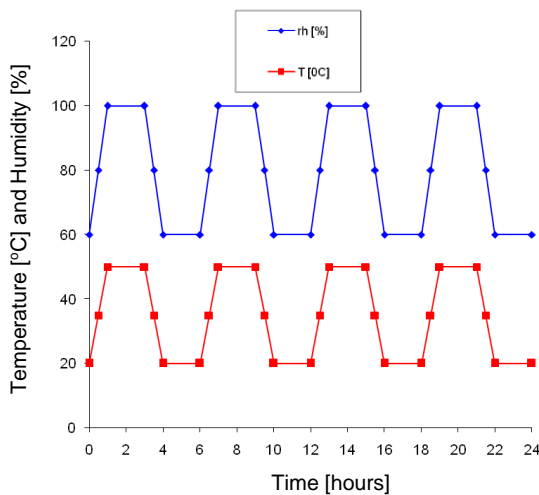


Fig. 4. Cyclic test geopolymer mortar (4 temperatures and 4 humidity cyclic/24 hrs).

**F. Effect of acids**

In this study, the specimens were soaked in sulfuric acid solution with selected concentrations ranging from 1% to 3% with the measured pH at 1.0. The test specimens were immersed in sulfuric acid solution in a container. In each case, three samples were immersed in the sulfuric acid solutions for 28 days. The acid resistance of geopolymer mortar was then evaluated based on the change in compressive strength and the change in mass after acid exposure.

**III. RESULTS**

**A. Effect of high temperature**

The distance cracks were increased and made many branch cracks on the surface of samples when increasing the heating temperature (see in Fig. 5) and up to 1000 °C the adhesion between geopolymer, fly ash and fine aggregate is not good.

The behaviors also look the same with geopolymer concrete. However the cracks of concrete are smaller than mortar causing much coarse aggregate content lead to reduce shrinkage and weight loss. When comparing a macrostructure of sample MLF<sup>-3</sup> with an image of mixture MLF<sup>-10</sup> heated at 800 °C, the cracks occurring in samples MLF<sup>-3</sup> (without fine sand) are bigger than MLF<sup>-10</sup>.

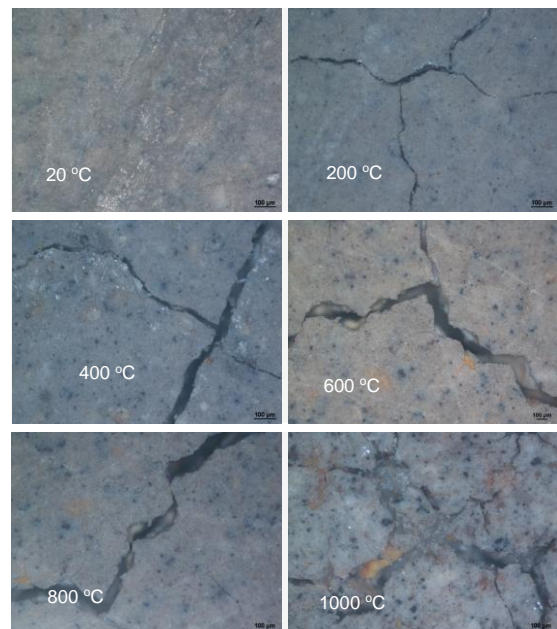


Fig. 5. The surface of samples MLF<sup>-2</sup> after curing at 20 °C and heated from 200 °C to 1000 °C at magnification 500x.

Shrinkages in length and in diameter (Fig. 6) is the consequence of reduction in volume which is primarily caused by loss of water contained in the alkaline and burnt some particles on the surface of samples during the heating process. Percentage of shrinkage of samples was also dependent on temperature and aggregates content. Aggregate plays an important role in affecting shrinkage of concrete. Indeed, most aggregates restrain concrete shrinkage because they are less elastic than the cement paste to which they are bonded. Concretes with higher aggregate contents shrink substantially less than cement-rich mixes, all else being equal [16].



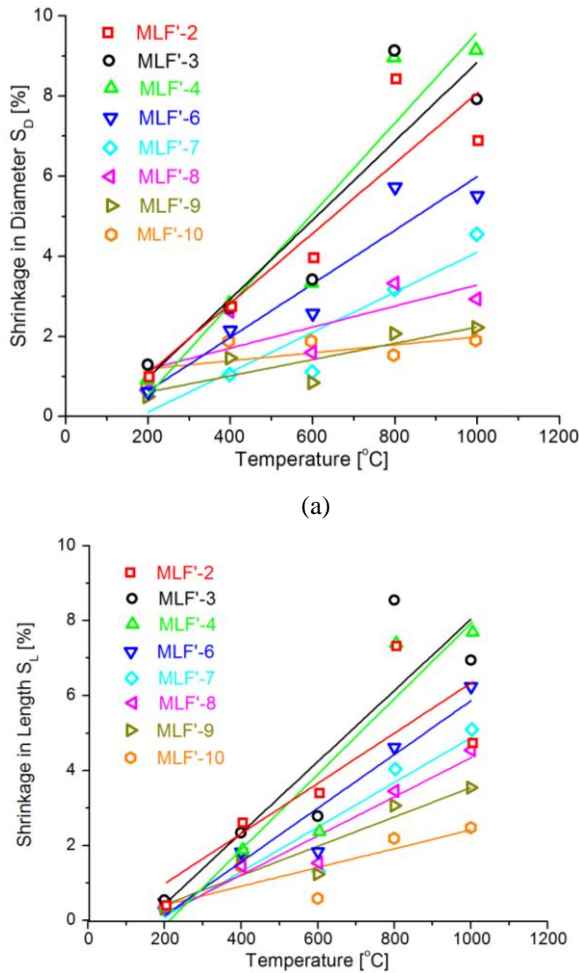


Fig. 6. Shrinkage in Diameter (a) and in Length (b) of mortar at high temperature.

The weight loss, shrinkage and compressive strength of geopolymer mortar were determined during the experiment and the detail results are shown in Table IV. The weight loss of mortar is increased by about 20 % when the temperature increased to 400 °C and remained up to 1000 °C.

Davidovits introduced the concept that the smaller drying shrinkage strain of fly ash-based geopolymer concrete may be explained by the block polymerization. According to this concept, the Si and Al atoms in the fly ash are not entirely dissolved by the alkaline liquid. The polymerization that takes place only on the surface of the atoms is sufficient to form the blocks necessary to produce the geopolymer binder. Therefore, the insides of the atoms are not destroyed and remain stable, so that they can act as micro-aggregates in the system and this could increase the aggregate content in concrete [16-18]. The below Fig. 7 shows the shrinkage of fly ash geopolymer mortar after heated to 800 °C.

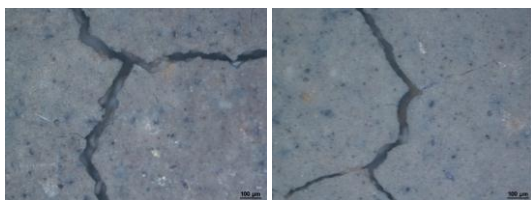


Fig. 7. Influence of sand on the shrinkage performance after heated at 800 °C: 0 % (left) and 50 % (right) at magnification 500x.

Fly ash based geopolymer mortar can sustain itself when exposed to considerably high temperature. Fig. 8 shows that the highest compressive strength is obtained when the temperature is 200 °C. The strength starts dropping once the temperature is over 400 °C. The lowest values of the residual strength were observed in the temperature range of 600 to 800 °C; they were due to the presence of the melt that started forming. While OPC mortar degrades and degenerates at high temperature, it has been found from different studies that fly ash geopolymer mortar can maintain its desired compressive strength even at 400 °C [16], the residual strengths of the OPC concrete are very low, on the order of a few MPa.

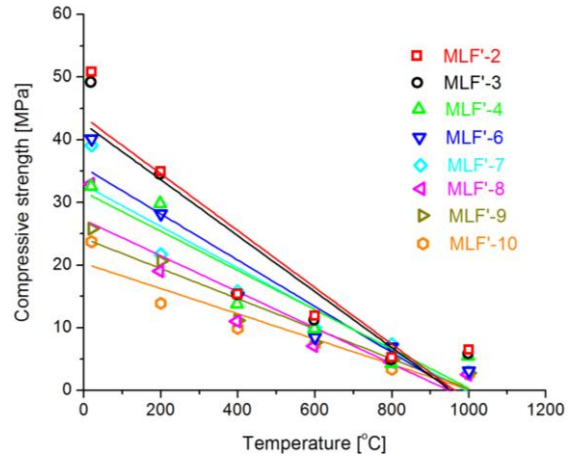


Fig. 8. Influence of high temperature on the compressive strength of geopolymer mortar.

**B. Effect of environmental conditions**

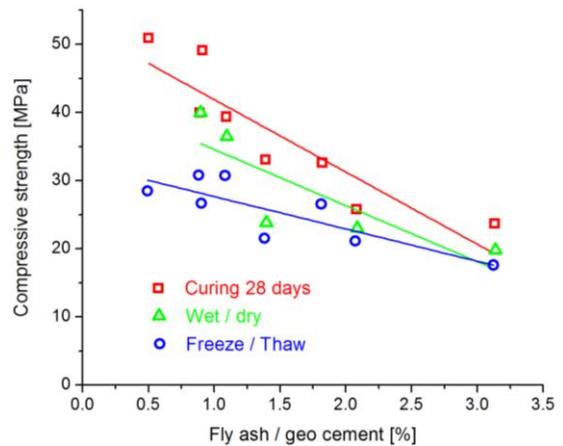


Fig. 9. Compressive strength of geopolymer mortar after freeze/thaw and wet/dry cycle, comparison with initial strength at 28 days.

Next, freeze/thaw and wet/dry tests determined water absorption, weight loss, shrinkage and compressive strength of the geopolymer mortar presented in Tables V and VI. Fig. 9 shows the results obtained from mixtures of MLF'-2 to MLF'-10 samples subjected to the different environments together with the control samples test at 28 days. This figure presents the results of geopolymer mortar for 3 environments: ambient conditions, freeze/thaw, and wet/dry. The effects of freeze/thaw cycling were stronger than wet/dry and the compressive strength of MLF'-7 was reduced about 45 % and

in wet-dry test it was 8 %. It is easy to see that, mixtures (MLF'-2 to 4) without sand are significantly effected by environments on the properties when compared with mixtures MLF'-6 to MLF'-10. The results of the freeze/thaw cycles in Table V show that the weight of samples is increased. That means that geopolymer mortar absorbs water at about 1.5 %. At the end of the analyzing micrograph, it was observed that

the micro cracking was the result of freeze/thaw, wet/dry and humidity conditions on the surface of geopolymer mortar (see Fig. 10).

TABLE IV. SUMMARY OF SOME PROPERTIES OF GEOPOLYMER MORTAR AFTER HEATING AT HIGH TEMPERATURE

Temp [°C]	Properties	Mixtures No							
		MLF'-2	MLF'-3	MLF'-4	MLF'-6	MLF'-7	MLF'-8	MLF'-9	MLF'-10
1000	W <sub>L</sub> [%]	20.96	21.69	19.33	22.51	21.70	20.05	19.20	18.37
	S <sub>D</sub> [%]	6.86	7.88	9.15	5.51	4.54	2.94	2.24	1.88
	S <sub>L</sub> [%]	4.72	6.98	7.69	6.20	5.08	4.54	3.53	2.48
	f <sub>cm</sub> [MPa]	6.57±0.6	5.75±0.5	5.46±0.4	2.92±0.2	3.10±0.4	2.65±0.3	2.74±0.3	2.54±0.1
	[HV]	200±4	151±6	142±7	105±3	131±14	102±18	108±15	124±4
	ρ [kg/m <sup>3</sup> ]	1491	1597	1639	1612	1653	1467	1519	1481
800	W <sub>L</sub> [%]	21.80	20.92	20.31	22.52	21.68	210.4	19.69	20.65
	S <sub>D</sub> [%]	8.40	9.10	8.97	5.73	3.16	3.33	2.09	1.51
	S <sub>L</sub> [%]	7.30	8.58	7.39	4.57	4.02	3.45	3.05	2.19
	f <sub>cm</sub> [MPa]	5.30±0.6	4.79±0.2	4.28±0.2	6.77±0.6	7.54±1.5	4.66±1.3	4.85±1.7	3.39±0.1
	[HV]	201±20	171±8	133±2	202±20	184±14	147±12	129±9	119±9
	ρ [kg/m <sup>3</sup> ]	1590	1628	1687	1566	1586	1455	1473	1503
600	W <sub>L</sub> [%]	20.85	20.24	19.47	22.18	19.54	17.97	19.52	17.89
	S <sub>D</sub> [%]	3.93	3.38	3.33	2.57	1.10	1.60	0.87	1.86
	S <sub>L</sub> [%]	3.38	2.81	2.37	1.79	1.29	1.53	1.22	0.59
	f <sub>cm</sub> [MPa]	12.00±0.3	11.14±0.6	9.85±0.3	8.29±1.0	10.20±0.3	7.22±0.6	7.63±0.2	7.34±0.2
	[HV]	214±8	183±8	164±8	207±21	200±8	188±1	205±8	230±9
	ρ [kg/m <sup>3</sup> ]	1401	1379	1397	1437	1484	1397	1458	1505
400	W <sub>L</sub> [%]	18.97	20.60	18.10	22.43	20.80	20.24	19.78	19.68
	S <sub>D</sub> [%]	2.71	2.67	2.82	2.15	1.04	2.64	1.48	1.86
	S <sub>L</sub> [%]	2.59	2.37	1.89	1.78	1.78	1.44	1.71	1.44
	f <sub>cm</sub> [MPa]	15.48±0.9	15.21±1.4	13.82±1.4	14.82±1.6	15.96±3.9	11.17±1.2	11.18±0.5	9.92±0.4
	[HV]	222±12	210±13	197±4	207±12	292±19	251±9	254±18	273±4
	ρ [kg/m <sup>3</sup> ]	1407	1404	1424	1446	1534	1413	1468	1491
200	W <sub>L</sub> [%]	7.77	8.97	8.57	9.09	7.86	8.61	8.58	9.46
	S <sub>D</sub> [%]	0.96	1.25	0.91	0.61	0.61	0.66	0.52	0.71
	S <sub>L</sub> [%]	0.38	0.58	0.37	0.43	0.24	0.33	0.29	0.35
	f <sub>cm</sub> [MPa]	35.04±1.6	34.40±2.6	29.85±0.2	28.05±0.8	21.85±2.7	19.16±5.1	20.65±4.1	13.88±1.9
	[HV]	324±9	309±17	277±12	245±12	228±9	227±8	234±8	211±8
	ρ [kg/m <sup>3</sup> ]	1472	1470	1451	1598	1660	1551	1605	1620

W<sub>L</sub> - Weight loss, [%]; f<sub>cm</sub> - Compressive strength, [MPa]; S<sub>D</sub> - Shrinkage in diameter, [%]; S<sub>L</sub> - Shrinkage in length, [%]; HV - Hardness, [HV]; ρ - Density, [kg/m<sup>3</sup>]

TABLE V. SUMMARY OF PROPERTIES OF GEOPOLYMER MORTAR AFTER TESTING FREEZE/THAW

Properties	Mixtures No							
	MLF'-2	MLF'-3	MLF'-4	MLF'-6	MLF'-7	MLF'-8	MLF'-9	MLF'-10
W <sub>L</sub> [%]	-1.40	-1.62	-1.51	-1.01	-0.68	-1.06	-0.86	-1.14
S <sub>D</sub> [%]	0.05	0.08	0.68	0.18	0.35	0.45	0.20	0.24
S <sub>L</sub> [%]	0.04	0.22	0.15	0.10	0.23	0.14	0.37	0.08
f <sub>cm</sub> [MPa]	28.43±3.8	26.60±2.4	26.50±2.6	30.72±2.8	30.68±4.5	21.47±5.1	21.09±3.4	17.53±4.3
[HV]	308±8	280±2	278±8	266±9	253±7	246±10	248±10	245±13
ρ [kg/m <sup>3</sup> ]	1609	1588	1593	1777	1808	1711	1730	1768

TABLE VI. SUMMARY OF PROPERTIES OF GEOPOLYMER MORTAR AFTER TESTING WET/DRY

Properties	Mixtures No				
	MLF'-6	MLF'-7	MLF'-8	MLF'-9	MLF'-10
W <sub>L</sub> [%]	12.87	12.03	11.61	11.11	12.17
S <sub>D</sub> [%]	0.51	0.56	0.50	0.40	0.38
S <sub>L</sub> [%]	0.40	0.57	0.48	0.41	0.28
f <sub>cm</sub> [MPa]	39.81±5.4	36.28±3.2	23.64±3.1	22.83±3.4	19.60±1.8
[HV]	285±11	281±4	267±7	262±17	261±2
ρ [kg/m <sup>3</sup> ]	1537	1586	1503	1549	1561

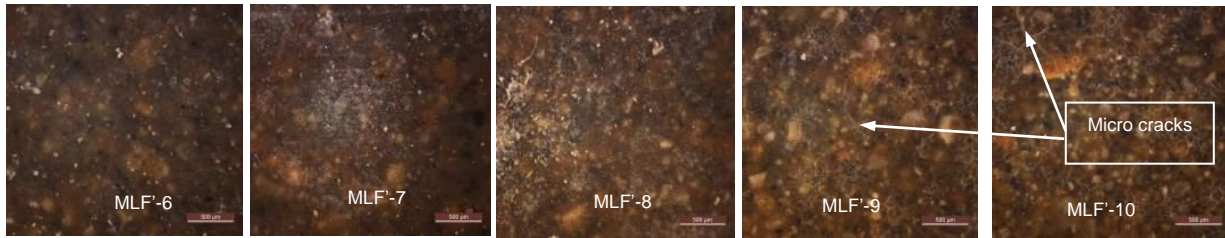


Fig. 10. The photographs of geopolymer samples after testing in a climate chamber.

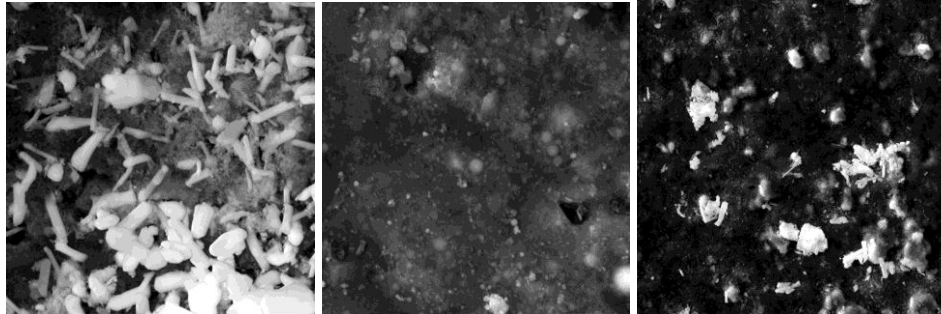


Fig. 11. Effect of 3 % sulfuric acid (left), 5 % chloric acid (middle), 5 % nitric (right) on the surface of geopolymer.

### C. Effect of acids

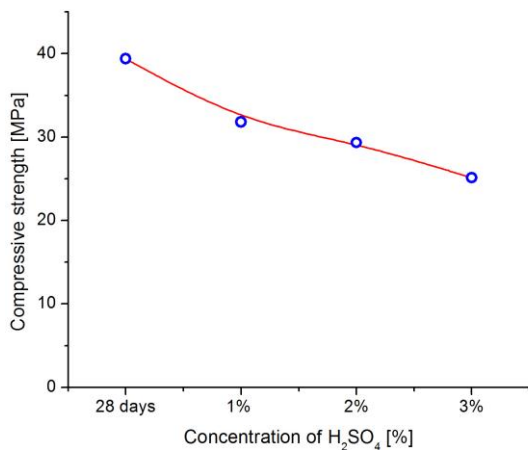


Fig. 12. Compressive strength of MLF'-7 curing ambient temperature at 28 days and immersion in H<sub>2</sub>SO<sub>4</sub> solutions for 28 days.

We can see from Fig. 11 left and right that the appearance of these white zones is probably related to two causes: first, by extracting unstable aluminum, i.e. Al-end units with non-bridging oxygens; second, by formation of a zeolitic structure, which causes strength loss depending on the acid concentration. The process similarly occurs with nitric acid, but there were little effects on the structure of geopolymer mortar when samples were immersed in the chloric-acid solution.

The concentration of H<sub>2</sub>SO<sub>4</sub> solution also significantly effected the compressive strength of mortar. Fig. 12 shows that increasing the concentration reduced the strength and increased the weight loss of the samples.

Fly ash-based geopolymer concrete has been proven in many studies to provide better resistance against aggressive environments. The report of Wallah and Rangan calculated that the geopolymer concrete exposed to 0.5% concentration of H<sub>2</sub>SO<sub>4</sub> acid solution and that the compressive strength decreased about 20% after one year of exposure. This value was about 52% and 65% respectively for geopolymer concrete exposed to 1% and 2% concentrations [19]. Song and his colleagues suggested that the reduction in compressive strength was in the range of 32 to 37% after 56 days of exposure to 10% H<sub>2</sub>SO<sub>4</sub> acid solution [20].

### IV. CONCLUSIONS

The results of this study show that the aggregate are significantly influenced by the strength and shrinkage of geopolymer mortar. And the ratio of fly ash to alkaline liquid also effected the general strength and fire resistance of geopolymer. It was found that the fly ash-based geopolymer displayed an increase in strength after temperature exposure [21]. Moreover, the intrinsic chemistry of the geopolymer binder does not require the retention of water or hydration within gel phases to maintain structural integrity of the binder in fire processing [22]. Therefore, geopolymer mortar can be applied in places or in conjunction with equipment requiring high degrees of fire resistance.

For freeze-thaw and dry-wet tests, mixtures MLF'-6 to MLF'-10 were used. After 28 days, the environment significantly influenced the compressive strength, weight loss, shrinkage, and microstructure of the geopolymer mortar.

The sulfuric acid resistance of fly ash-based geopolymer mortar was studied for mixture MLF'-7. The concentration of sulfuric acid solution was 1 %, 2 % and 3 % for soaking specimens. The sulfuric acid was also effective on the compressive strength, change in mass and microstructure of samples. However, the sulfuric acid resistance of geopolymer mortar was significantly better than that of OPC mortar as reported in earlier studies.

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