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Strength and Permeation Characteristics of Zeolite-based Geopolymer Lightweight Concrete

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ABSTRACT

Geopolymer concrete is a material manufactured by polymerizing sources of aluminates and silicates like fly ash, metakaolin, slag, zeolite, etc. with an alkaline solution. A study has been undertaken to produce lightweight geopolymer concrete by using waste zeolite particles (zeolite molecular sieve) as aluminates and silicates source and at the same time as lightweight medium. In addition, others three geopolymer lightweight concrete mixes were produced by partially replacing the waste zeolite particles (25% of volume) with sources materials (fly ash type F, fly ash type C and waste zeolite powder. Moreover, the impact of this partially replacement on dry density, compressive strength and permeation characteristics of produced geopolymer lightweight mixes was studied. An alkaline solution of sodium silicate and sodium hydroxide was used in all the investigated mixes as an activator. From the findings, a geopolymer lightweight mix suitable for insulation purposes (density of 1610 kg/m^3 and 28 days compressive strength of 5.1 MPa) was successfully produced using waste zeolite molecular sieve. It was found also that the lightweight zeolite particles were uniformly distributed through the produced mixes. Finally, it was found that replacement of 25% of volume of zeolite particles by fly ash (type C) helped in not only enhancing the compressive strength by about 13% but also reducing the water absorption by about 33%.

1. Introduction

Developing new materials is the key existing difficulty for material science. By adjusting material composition, mechanical properties, durability, and chemical resistance of the materials get better. Their pricing and production cost should be unaffected, though, by this very little change. In the previous decades, environmental effect, greenhouse gas emissions, and energy usage during production have attracted more and more focus. Apart from water, Portland cement-based concrete is the most often used building tool. Its low cost and outstanding performance have spurred extensive global use. Conventional concrete does, however, have certain drawbacks like limited chemical resistance against acids and salts, low thermal and fire resistance, particularly at temperatures above 500 °C (Nikolov, Rostovsky, & Nugteren, 2017). Therefore, a need for sustainable building materials, such

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as geopolymer, has been increased.

Geopolymer concrete is an inventive material for building that utilizes no cement as a binder material. This concrete is manufactured by polymerizing materials as sources of large amounts of aluminates (Al) and silicates (Si) (Ma, Awang, & Omar, 2018). An alumino-silicate substance is the primary raw component (precursor) used in the synthesis of geopolymers. Natural sources of precursors include kaolin, zeolite, volcanic ash or natural pozzolans. They may also be thermally treated material, including metakaolin, fly ash, granulated blast furnace slag, calcined shales or other industrial wastes. The alumina-silicate material needs to be activated by another raw material, generally alkaline solution, the so-called activator. Common activators are sodium and potassium hydroxide; sodium and potassium silicate solutions (water glass) Alnahhal, Alengaram, Yusoff, Darvish, Srinivas, & Sumesh, 2022; Nader, Hilal, & Alwan, 2022).

Geopolymerization commences with the dissolution of alumina and silica from the source ingredients upon mixing. Following the preliminary extraction of surface metals like Na+ and Ca_2+ by metal/In proton exchange processes, the alumina-silicates are subjected to assault by OH^- ions. Due to the relative weakness of Al-O bonds compared to Si-O bonds, aluminum will initially dissolve as $Al\ (OH)_4^-$ complexes. Detached Si-tetrahedra will now be more susceptible to OH^- attack, resulting in silicon acids and oligomers, commonly represented as $Si(OH)_3O^-$ in solution, with alkaline cations neutralizing the negative charge (Duxson & Provis, 2008).

Fly ash (FA) is one of the pozzolans most used worldwide. Fly ash in coal-fired electric generating plants results from burning ground-up coal. The features of the coal, burning systems, ash collecting methods, cooling process, and other factors significantly affect the fly ash composition. But as manufacturing flourished and more thermal power plants opened, daily fly ash consumption rose all around (Böke, Birch, Nyale, & Petrik, 2015; Lo, Cui, Memon, & Noguchi, 2016).

Fly ash generally consists of a dry powder predominantly composed of rounded and spherical particles, which exhibit varying colors based on their types and chemical compositions. Fly ash is categorized into F and C classes. The standard employed various parameters to classify it based on its chemical composition and physical properties. Fly ash is categorized as class F if it contains 70% or more of Al_2O_3, SiO_2 and Fe_2O_3. The primary constituent of the amorphous alumina-silicate glass in it is partitioned into quaternary crystalline phases by mullite, quartz, magnetite, and hematite. Such ashes generally include 10% to 20% of calcium (CaO). It was reported that if the total amounts of Al_2O_3, SiO_2 and Fe_2O_3 in fly ash exceed 50% and the calcium concentration above 20%, the fly ash is classified as class C (ASTM C618, 2008; Nemec, Gandel, Jerabek, Sucharda, & Bilek, 2024).

Natural zeolites, according to their chemical makeup, are potential raw materials for geopolymer synthesis (Hilal, Nader, & Kurji, 2024; Xu, Pang, Yu, Huo, & Chen, 2007). Zeolite is a crystalline aluminosilicate material characterized by open three-dimensional frameworks composed of $(Al,Si)O_2$, and it classes based on the SiO_2: Al_2O_3 ratio. The primary zeolite properties include its extensive specific surface area between 600 m^2/g and 800 m^2/g, consistent structure, uniformly distribution of pore sizes and excellent heat conductivity. Furthermore, zeolites possess molecular sieve characteristics and exhibit a high capacity of absorption (Nagrockiene & Girskas, 2016).

Alternative utilization of a quantity of binder components with zeolite improves the mechanical characteristics. As a result, this environmentally friendly mortar has been suggested as an alternative to cement mortar in the sustainable development of construction materials (Erfanimanesh & Sharbatdar, 2020).

Lightweight geopolymer concrete is a specialized form of concrete that possesses a lower density than traditional concrete. Moreover, because to its reduced density, it is facile to manage and convey from one location to another throughout building. Various types of binder and aggregate wastes were utilized as substitutes in its production (Chanda & Guchhait, 2024).

Lightweight geopolymer concretes have garnered interest due to their enhanced durability and less effect on environment. They consist of natural or synthetic lightweight aggregates (fine or course) or entrained air voids, combined with geopolymer binding medium (Masoule, Bahrami, Karimzadeh, Mohasanati, Shoaei, Ameri, & Ozbakkaloglu, 2022).

The aim of this research is to produce geopolymer lightweight concrete by using lightweight zeolite molecular sieve as waste material which is rich in aluminates (Al) and silicates (Si). In addition, others three geopolymer lightweight concrete mixes will be produced by partially replacing the waste zeolite particles with fly ash type F, fly ash type C and powder of zeolite particles. Finally, the impact of this volume partially replacement (25%) on dry density, compressive strength and permeation characteristics of produced lightweight geopolymer mixes will be studied.

2. Experimental details

2.1. Materials Used

Fly ash F with small amount of calcium (5.22%) and fly ash C with larger amount of calcium (28.18%), conforming to ASTM C618 (2008), were the sources of waste substance of Al (aluminates) and Si (silicates). Furthermore, zeolite molecular sieve which also known as aluminosilicate microporous crystal, was employed as another source for aluminates and silicates required to initiate the polymerization process, see Figure (1). Chemical compositions of the aluminates and silicates sources adopted in this study ae listed in Table (1).

Sodium silicate (SS) and sodium hydroxide (SH) solutions were utilized as the activation liquid. Sodium silicate with a viscosity value about 1100 CPS at temperature of 20 °C was utilized at a concentration ratio of 45.9 % and with 2.4 as SiO_2 to Na_2O ratio which is comprising 32.5% SiO_2 and 13.4% Na_2O. On the other hand, making SH involved dissolving 99% pure NaOH in water (Aleem & Arumairaj, 2012). All examined lightweight geopolymer mixes utilized natural sand (≤ 4.75 mm) as filler substance. The sieve analysis of the sand used and the recommended gradations according to the Iraqi Standard Specification No. 45 (1984) are shown in Table (2).

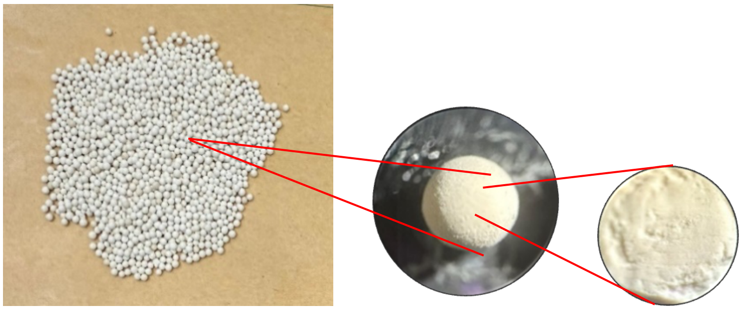


Fig. 1 Waste particles of zeolite molecular sieve used

Table 1 – Chemical compositions of zeolite and fly ash (type F and C) determined by (XRF) (mass %).

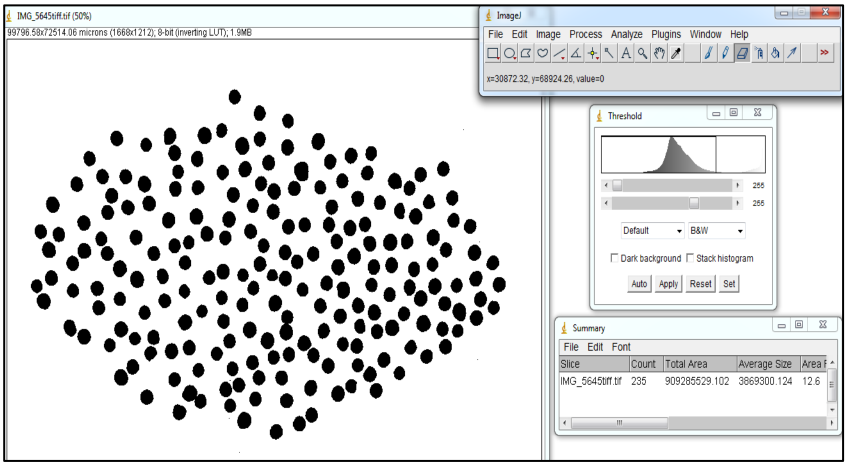
Oxides	**Al_2O_3**	**SiO_2**	**Fe_2O_3**	**CaO**	**MgO**	**K_2O**	**SO_3**	**L.O.I**
Zeolite (%)	13.11	67.76	0.872	3.34	1.2	1.423	-	9.9
Type F fly ash, (%)	28.43	46.88	17.92	5.22	2.27	1.86	0.37	3.21
Type C fly ash, (%)	6.3	31.32	28.62	28.18	0.26	2.48	0.89	-

Table 2 – Sieve analysis of sand used.

Sieve Size	**Recommended sand gradation, Zone 2 (% passing)**	**Sand used (% passing)**
4.75 mm	90- 100	99
2.36 mm	75- 100	83
1.18 mm	55- 90	74
600 µm	35- 59	51
300 µm	8- 30	23
150 µm	0- 10	2

2.2. Particles size distribution

Images were captured for the waste zeolite molecular sieve particles in order to investigate its particle sizes by using ImageJ software as shown in Figure (2). The particle sizes distribution was shown in Figure (3) as frequency (%) and cumulative frequency (%). It can be seen that the zeolite particles sizes are ranging from 1825 to 2825 micrometer with median particles size of 2325 micrometer.



99796.58x72514.06 microns (1668x1212); 8-bit (inverting LUT); 1.9MB

x=30872.32, y=68924.26, value=0

255

255

Default B&W

☐ Dark background ☐ Stack histogram

Slice	Count	Total Area	Average Size	Area F
IMG_5645tiff.tif	235	909285529.102	3869300.124	12.6

Fig. 2 Analysing the zeolite particles using ImageJ software.

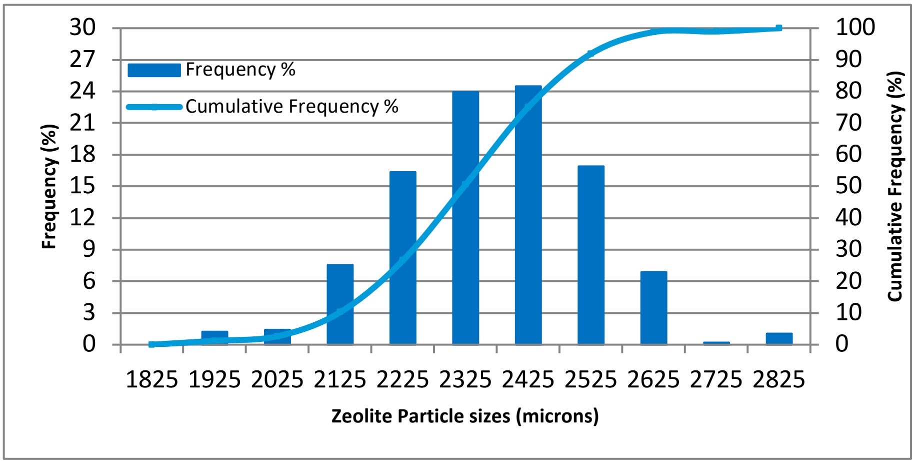


Fig. 3 Zeolite particles **sizes distribution**

2.3. Mix proportions and production

A method of absolute volumes (Obaid & Hilal, 2021) was used to design all examined geopolymer lightweight mixes produced with waste zeolite particles as lightweight substance with 780 kg/m³ density (Hilal et al., 2024). Mixes proportions are listed in Table (3). It should be noted here that the letter z is denoted for the zeolite particles, f is for fly ash type F, c is for fly ash type C, and zp is for the zeolite powder. These sources materials correspondingly replaced the 25% of the zeolite particles volume (0.057 m^3 of 0.23 m^3) in mixes of Mzf, Mzc and Mzzp and used as the amounts listed in Table (3).

The molarity value and the alkaline liquid were selected to be 8 M and 500 kg/m³, respectively. While, the alkaline liquid was adjusted based on the source material investigated in this study to avoid the buoyancy of the waste zeolite particles. In this work, geopolymer mixes studied were generated by first mixing the Al and Si source substance with fine sand then progressively adding the alkaline solution. Then the molds were filled with the geopolymer slurry. For curing, the geopolymer lightweight concrete specimens spent twenty-four hours at 60 ºC in an oven. At last, the samples were taken out of the oven and kept at room temperature until the test period (Hilal and Mahmood, 2018). The mixing, molding and curing stages are shown in Figure (4).

Table 3 – Proportions of investigated zeolite-based geopolymer lightweight mixes.

Consists	**Mixes**			
	Mz	**Mzf**	**Mzc**	**Mzzp**
Sand (kg/m^3)	1036	1036	1036	1036
Zeolite (kg/m^3)	177	123.75	123.75	123.75
Fly ash (F) (kg/m^3)	-	125	-	-
Fly ash (C) (kg/m^3)	-	-	125	-
Zeolite powder (kg/m^3)	-	-	-	125
Alkaline liquid (A) (kg/m^3)	500	500	500	500
Extra solution (kg/m^3)	-	-25	-75	+20
$NaOH/ Na_2SiO_3$	1	1	1	1
Molarity	8	8	8	8

(a) materials mixing (b) produced mix (c) zeolite-based molding

(d) fly ash F-based molding (e) fly ash C-based molding (f) heat curing

Fig. 4 Stages of mixing, molding and curing of investigated mixes

3. Tests

At both ages (7 and 28 days), compressive strength testing was carried out with following the ASTM C513 (2011) on cubical specimens with dimensions of 70 mm^3 and the average of three readings was taken at each age. In addition, water absorption was determined by submerging an 60˚C oven-dried specimens (dry weight) in water (7 days to achieve a constant weight and get a wet weight) and determining their mass change as a percentage of their dry mass (Nambiar & Ramamurthy, 2007).

During a time period of 5 hours, a sorptivity test (capillaries suction performance) was carried out on prismatic specimens of investigated geopolymer lightweight mixes by following the procedures of ASTM C1585 (2013), see Figure (5). It can be seen form this Figure that the low area of specimen sides was sealed with water proof tape to achieve a vertical unidirectional flow. Then, the tray filled with water until the lowest 3 mm of the sealed areas. Wet weight measurements were measured to the nearest 0.01 g during the test over a period of 5 hours (Hilal, Thom, & Dawson, 2014).

The following square root-time equation was adopted to describe the water movement by capillary suction (Lo, Cui, Nadeem, & Li, 2006):

$$\boldsymbol{i = B + St^{0.5}} \tag{1}$$

Where (i) is a cumulative absorbed water volume per water contact surface area (mm^3/mm^2), (t) is time of taking the measurement (min), (B) is the y-intercept at time equals to zero and *(S)* is the sorptivity of mixes obtained from the best linear regression of i versus t$^{0.5}$ curve (mm/min$^{0.5}$).

Fig. 5 Sorptivity test

4. Results and discussion

4.1. Density and particle distribution

As illustrated in Figure (6), four geopolymer lightweight concrete mixes with densities around 1850 kg/m^3 were produced in this research. Due to their porous nature (voids size less than 2 nm in diameter)(Kurji, Mujtaba, & Abbas, 2023), using zeolite particles totally as source substance resulted in manufacturing lightweight mix at density of 1600 kg/m^3, see Figure (1). Moreover, it was reported that adding the waste zeolite as particles resulted in creating very tiny pores (entrapped voids) leading in reducing the total mix weight (Hilal et al., 2024).

For each investigated geopolymer mix, three slices (50 × 50 × 20 mm) were cut, parallel to the casting face, from the middle of three cubical specimens, and used for zeolite particles investigation by using ImageJ software (Hilal, Thom, & Dawson, 2015). The cut slices were first polished with carbide silicon of 40#, 60#, 80#, 120# and 600#, cleaned and oven dried. Then, their surfaces were coated with a permanent marker. The white color of zeolite particles made a good contrast between them and the concrete matrix resulting in good prepared slices for image analysis (Hilal et al., 2015). From the image analysis, it was found a good zeolite particles distribution was achieved and the zeolite particles contents were close to the adding volume (0.23 m^3 for Mz mix and 0.17 m^3 for Mzf, Mzc and Mzzp mixes), see Figure (7). Figure (8) shows the uniform zeolite particles distribution along the height of the Mzf mix cube.

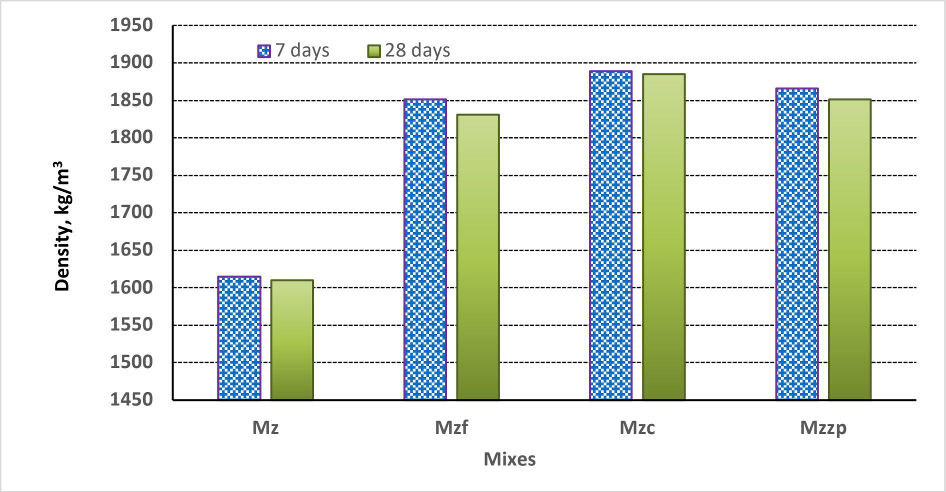
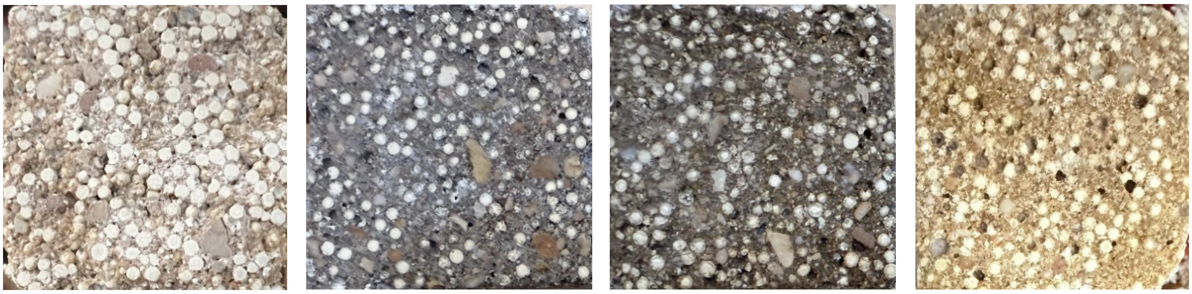


Fig. 6 Dry densities of investigated geopolymer lightweight concrete mixes



(a) Mz mix (b) Mzf mix (c) Mzc mix (d) Mzzp mix

Fig. 7 The zeolite particles content in the different investigated geopolymer mixes [scale: 50× 50 mm]

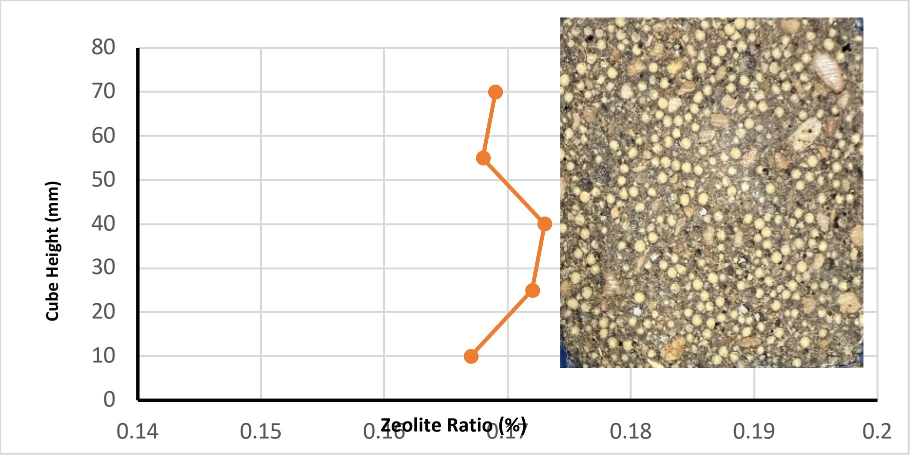


Fig. 8 Zeolite particles content along the specimen height of Mzf mix

4.2. Compressive strength

From Figure (9), it can be seen that the 28 days compressive strength of zeolite-based geopolymer lightweight concrete mix (Mz) is 5.1 MPa. It is well known that in geopolymer material, to enhance the strength, an alkaline liquid to fly ash ratio (Al/FA) should be increased in order to add more (Si) leading to form more Si-O-Si bonds (Al Bakri, Kamarudin, Omar, Norazian, Ruzaidi, & Rafiza, 2011). In this research, in order to improve the Mz mix strength; part of the waste zeolite particles volume (25%) was replaced with source materials as fly ash type F, fly ash type C and zeolite powder as listed in Table (3). Although this replacement ratio led to produce denser geopolymer mixes (around 1850 kg/m^3), these mixes are still in the range of lightweight concrete. It was noticed that using low calcium fly ash in Mzf mix instead of 25% of waste zeolite particles volume helped in achieving higher strength (13% more than that of Mz mix). Replacing zeolite particles by fly ash (C) (Mzc mix) resulted in reducing the compressive strength by about 5.8% in spite of increasing its density. The 28 days compressive strength of the Mzf was higher than that of Mzc mix by about 20% due to the presence of large amounts of Silicates and Aluminates in fly ash (type F) compared to those in fly ash (Type C), see Table (1). Furthermore, it was reported that using of low calcium fly ash (class F) prefers in manufacturing geopolymer concrete more than class C fly ash (high calcium). This is because the latter makes the geopolymer sitting time fast leading to alert its microstructure by interfering the geopolymerization process (Antoni, Wijaya, & Hardjito, 2016). It was found that using of zeolite as powder in Mzzp mix reduced the compressive strength by about 58% of that of Mz mix. This indicates that the waste zeolite powder might be not fine enough to use as Al and Si source in polymerization process (Hilal et al., 2024).

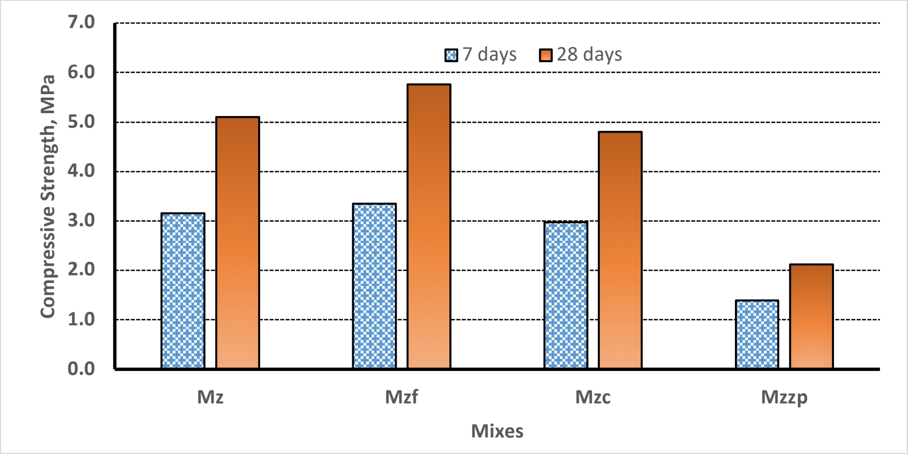


Fig. 9 Compressive strength of investigated mixes

4.3. Permeation properties

Figure (10) shows the water absorption variation due to immersion the investigated geopolymer lightweight concrete mixes in water. It can be observed that the water absorption of Mz mix is higher than those of mixes made with replacing 25% of zeolite particles with powder source as class F, class C and waste zeolite powder. This due to the porous structure of the zeolite particles. Using the above source materials instead of part of waste zeolite particles (25% y volume) resulted in a reduction in water absorption by 33%, 16% and 13% of that of Mz mix, respectively.

With regard to the sorptivity test, it is well known that the higher the very tiny air voids the higher the capillary sorption. It can be seen form Figure (11) that the Mzc mix, which was produced with class C, absorbed less water than those produced with the others source powders. This indicates that compared to Mzf and Mzzp mixes the capillary pores in the flay ash C paste was lower. Noted that, because of its higher CaO, fly ash class C produces not only geopolymer gel as (Si-O-Al) links but also calcium silicate hydrate as (C-S-H) gel resulting in forming paste with less gel and capillary pores (Zahid, Shafiq, Nuruddin, Nikbakht, & Jalal, 2017). By comparing the Mz mix with the others three investigated mixes, it can be found that the very tiny pores (less than 2 nm) in zeolite particles play a role and contribute in increasing the sorption action. This also concluded from the sorptivity values of the investigated geopolymer lightweight concrete mixes, see Figure (12) as example for Mzzp mix. It is reported that with higher sorptivity value, the average length of water movement path decreased; i.e. continued capillary pores.

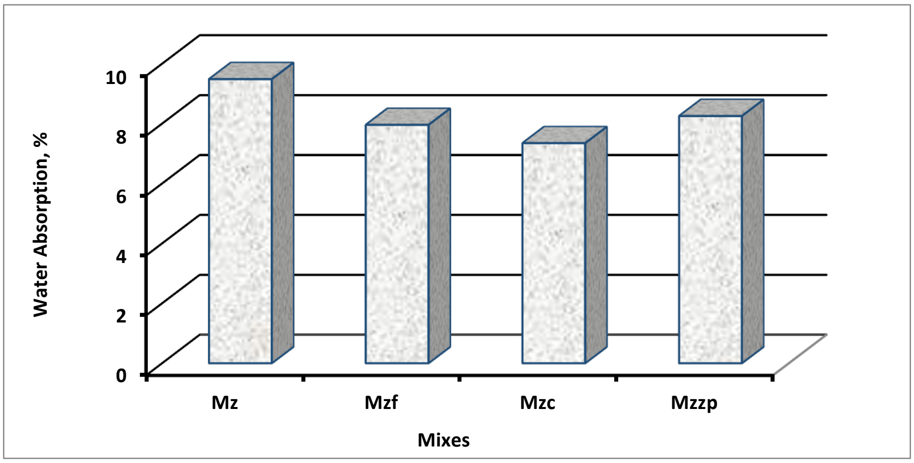


Fig. 10 Water absorption of investigated mixes

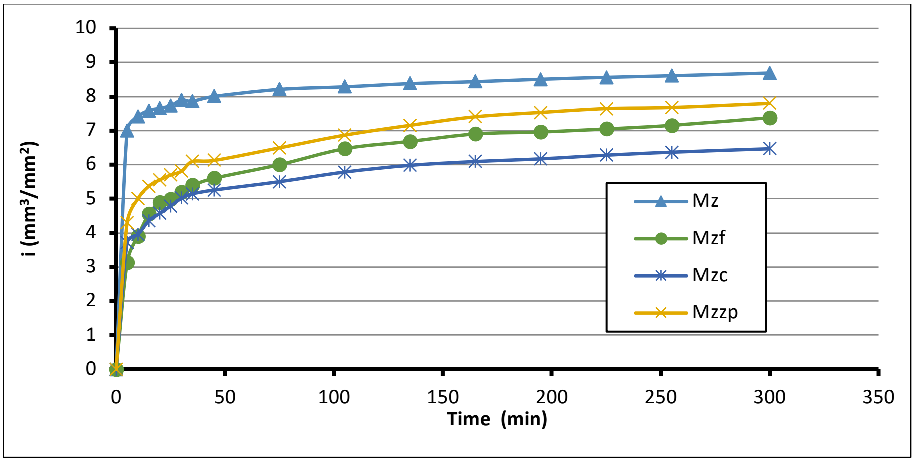


Fig. 11 Water absorbed cumulative volume per area with time

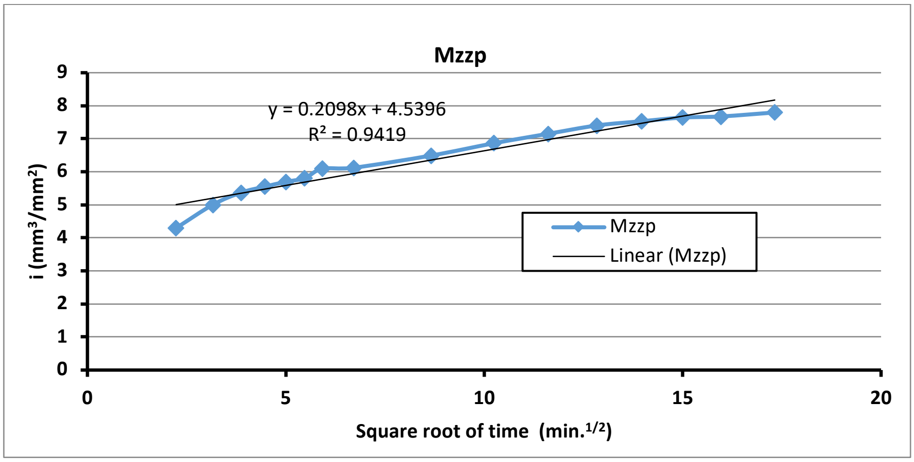


Fig. 12 Water absorbed cumulative volume per area versus square root of time for Mzf mix

5. Conclusion

The strength and permeation properties of zeolite-based geopolymer lightweight concrete were investigated. From this study, geopolymer lightweight mix suitable for insulation purposes (density of 1610 kg/m^3 and 28 days compressive strength of 5.1 MPa) was successfully produced using waste zeolite molecular sieve as silicates and aluminates source. It was found that the lightweight waste zeolite particles were uniformly distributed through the produced mixes.

It was found that the 28 days compressive strength of class F fly ash- based geopolymer lightweight concrete was higher than that of class C fly ash- based geopolymer lightweight concrete by about 20% due to the presence of large quantities of Silicates and Aluminates in class F in comparison with those of class C. However, replacing part of waste zeolite particles (25% by volume) by fly ash (type C) helped in not only enhancing the compressive strength by about 13% but also reducing the water absorption by about 33%. In addition, it was observed that the very tiny pores (less than 2 nm) in zeolite particles played a role and contributed in increasing the sorption action.

Improving the compressive strength of the lightweight waste zeolite-based geopolymer concrete by considering and changing some variables such as the alkaline liquid amount to solid ratio, the calcium hydroxide molarity and curing temperature will be a suggestion for future work.

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