PACE-2021
International Congress on the
Phenomenological Aspects of Civil Engineering
Research Article
20-23 June 2021

Properties of Geopolymer Mortars Obtained from Press Filter Waste and Fly Ash at a Certain Curing Condition

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Keywords

Waste,
Press filter waste,
Geopolymer mortar,
Unit weight,
Compressive strength.

Abstract

While the sugar production process, hydrated lime in the consistency of lime wash is mixed with the raw juice and the colloidal substances in it are precipitated. The excess lime added to the juice is removed from the juice with the help of CO_2 in the carbonation boilers. The sludge part of the sludge taken here is settled in decanters and filtered with the help of press filters. The waste that contains approximately 80% dry matter from press filters is called "Press Filter Waste" (PFW). Study was executed on the recycling of PFW which is released during this process. Geopolymer samples containing fly ash (FA) and PFW were obtained by using alkaline activators. In study, samples were produced with standard sand, FA, Na_2SiO_3, PFW and water (S). Samples were prepared as sand/binder ratio 3.0, H_20/binder ratio 0.19, 0.17 and H_20/Na_2SiO_3 ratio 0.386, 0.341 and were cured at 120 ^{0}C. It has been observed that PFW10FA90S2T samples have the highest compressive strength for 28 days (23.68 MPa), while the PFW30FA70S2T samples have the lowest compressive strength (21.00 MPa). When the 28-day unit weights of the samples were evaluated, it was determined that the PFW10FA90S2T samples have the highest unit weight (1.950 kg/dm^3).

1. Introduction

In global research on construction technologies, many studies especially on the components of concrete have been carried out with the help of the improvements of concrete technologies, [1] nowadays. The cement industry in the world is the most intense construction material produced as a result of the high demand in the construction sector of concrete. Due to such intensive production, the increase in industrial activities (e.g. cement production) over time, it is seen that production waste, including solid, liquid or gas with many different characteristics [2], has emerged. Millions of tons of industrial waste are produced every year; these wastes cause environmental problems due to insufficient storage facility and inadequate safe disposal. This situation may also lead to water and soil pollution [3] around the factories.

Considering the environmental effects of these wastes and the costs affecting production, it is obvious that it imposes a heavy burden and responsibility on the country and the world economy. Although necessary precautions are taken within the framework of legal procedures, some negative conditions arising from the production process may disrupt the environmental balance. For example, with the high energy requirement in the cement production process, this production process is also the source of significant CO_2 emissions in nature. Depending on the cement production technology, it has been determined that CO_2 emission between 0.73 tons and 0.99 tons is exposed [4] in 1-ton cement production.

Concrete; is a composite material consisting of water, aggregate, cement and mineral and/or chemical additives [5]. When water is added to cement, which is a component of concrete, it reacts and creates a binder that holds the other main components of the concrete, together. However, the use of industrial waste materials, such as FA, silica fume, GBFS, which have pozzolanic properties, usually takes place as a substitute for a part of cement. To protect the environment, the way to minimize CO_2 emissions can be accomplished by reducing the percentage of cement used in concrete production. Thus, in recent years, studies have been focused on the use of various waste materials (e.g. FA) with pozzolanic properties in the production of geopolymer cements with the help of alkali activators in accordance with the standards of fresh and hardened concrete properties [6], both in utilization of waste materials and the reduction of costs.

Geopolymer cement has the potential to replace Portland cement [7] in the construction industry. Geopolymer cement mortar; with its rapid resistance, lack of water cure, good mechanical and strength properties, it also prevents the emission of large amounts of CO_2 diffused to the atmosphere [8] during the production of Portland cement.

Industrial waste materials such as FA, GBFS, silica fume and natural pozzolans are used in Portland cement production at certain rates of the weight of Portland cement in the clinker stage or directly in concrete production. While clinker use in cement decreased to 85% in 2003, this rate decreased to 77% in 2010. It is estimated that this rate will decrease to 71% in the future. In America, pozzolanic materials are used more in ready-mixed concrete than clinker. Today, 60% of ready mixed concrete used in America contains pozzolanic material instead of cement [9]. In this context, determining various industrial waste materials with binding properties and utilizing them in concrete technology; has an important place in terms of both recycling and cost. However, the fact that the industry is broad-based and the exposed waste materials have different characteristics has accelerated the studies on the existence of different types of wastes that can be used in building material production.

Within the sugar industry, which is a food production industry, the research conducted within the scope of the utilization of industrial waste (PFW as seen Fig. 1) exposed during sugar production process is of great importance in terms of waste management and recycling. In sugar process annually; approximately 60,000 tons of PFW releases as solid waste. In this way, building materials with different characteristics obtained with the help of PFW, pozzolanic industrial waste materials and alkaline activators; without the use of cement will ultimately reduce the environmental harmful effects and production costs in the long term that exposes from the cement production process and at the same time, this PFW will also be recycled. In addition, it is considered that new special mortars can be produced with the utilization of agricultural waste materials such as PFW; by using as an aggregate, such diverse properties as weight reduction, thermal insulation and fire resistance or workability [10] may be improved.

FA particles are highly contaminating, due to their enrichment in potentially toxic trace elements which condense from the flue gas. Research on the potential applications of these wastes has environmental relevance, in addition to industrial interest. Most of the FA which is produced is disposed of as landfill [11], a practice which is under examination for environmental concerns. Classless FA cannot be used directly because it does not suitable for cement and concrete standards and so causes environmental pollution. FA is one of the most important materials used as the main material of the geopolymer [12] because of its high mechanical performance.

In an experimental study conducted on the thermal cure conditions of geopolymer mortar samples containing FA and alkali activator and the compressive strength values in the process; without using cement, samples were prepared at the rates of sand/FA = 3.00, H_2O/FA = 0.29 and Na_2O/FA= 0.10, and cured at 75 ^{0}C for 4 hrs, 1, 2, 3 and 7 days. The other sample group was cured at 75 ^{0}C for 4 hrs, 1, 2, 3 and 7 days, and then cured under laboratory condition (23±20 ^{0}C and humidity 50%±5) for a total of 28 days. It was determined that the compressive strength of 28-day samples were higher when the geopolymer mortar samples was cured at 75 ^{0}C for 4 hrs, 1, 2, 3 and 7 days and compressive strength [13] were determined, compared to the samples that were kept for up to 28 days. It has been concluded that the repolymerization reaction does not continue in the samples which are kept for a total of 28 days, and therefore the compressive strength increases are slightly or even stopped.

In a study obtaining and light geopolymers containing based ash by alkali activators (Na_2SiO_3 and 98% purity NaOH), two types of curing were applied in the oven at 75 ^{0}C for 20 hrs then cured under laboratory condition at 20 ^{0}C and only cured under laboratory condition at 20 ^{0}C. Some mechanical properties such as unit weight and compressive strength have been investigated. The unit weight of these geopolymers was found to be [14] 1.59 gr/cm^3 and compressive strength was 18.51 MPa. Examining the behaviors of samples prepared with binders such as GBFS, FA and POFA at different temperatures, calcium contents and different activator solutions; It has been determined that increased CaO ratio, reducing workability. High compressive strength was obtained at 60 ^{0}C in samples containing Na_2SiO_3 solution [15]. It has been observed that geopolymers containing high calcium can also be used as repair material. Pozzolanic materials used partially in concrete and mortars instead of cement have a positive effect on durability of concrete. It has been observed that there is a significant amount of amorphous silica in the chemical composition of pozzolans. In its reaction with cement [16], new silicates have emerged as a result of the interaction of free lime and silica within the pozzolan, thereby demonstrating a positive improvement in its mechanical and durability properties.

2. Material and Method

2.1. Material

In the experimental study, mortar mixing water, standard sand, fly ash, press filter waste, alkaline activator (Na_2SiO_3) were used in the production of mortar. The mortar samples obtained were subjected to different curing types, and unit weight and compressive strength values were examined within the framework of fresh and hardened properties.

Press Filter Waste (PFA)

In practice, the chemical analysis of the press filter waste obtained from Eskişehir Sugar Factory is shown in Table 1.

Table 1. Chemical analysis of press filter waste (PFW)

Components	Weight (%)
MgO	2.0242
Al_2O_3	1.2064
SiO_2	3.7480
P_2O_5	0.1948
SO_3	0.2142
K_2O	0.0910
CaO	56.3714
TiO_2	0.0783
Cr_2O_3	0.0392
Fe_2O_3	1.0255
BaO	0.2331
LOI	34.7740

Fly Ash (UK)

The fly ash used in the studies was obtained from the Tunçbilek thermal power plant; Before starting the experiments, the physical and chemical analysis of the fly ash and as seen in Table 2 and Table 3 were determined.

Table 2. Physical analysis of fly ash (UK) (Tunçbilek)

Physical Properties	90 µ (%)	45 µ (%)	Specific Gravity (gr/cm^3)	Blaine (cm^2/gr)
	-	35	2.05	3200

Table 3. Chemical analysis of fly ash (UK) (Tunçbilek)

Components	Weight (%)
SiO_2	52.31
Al_2O_3	16.49
Fe_2O_3	12.38
CaO	5.72
MgO	5.01
SO_3	1.16
K_2O	2.27
lOI	1.13
Free CaO	0.15
Cl^-	0.0106

Standard Sand

While preparing the mixtures in the experimental study, Rilem Cembureau Standard sand was used in accordance with the TSE EN 196-1 standard. The particle size distribution and standard limit values determined as a result of the sieve analysis of the sample standard sand are given in Table 4.

Table 4. Granulometry and limit values of sample standard sand

Property	Grain size, mm					
	0.08	0.16	0.50	1.00	1.60	2.00
Retain, %	99	87	72	34	6	0
Limit, %	99±1	87±5	67±5	33±5	7±5	0

Mixed Water

Eskişehir region water in our laboratory was used as mortar mixing water. The chemical analysis of the mixed water used in the production of mortar is shown in Table 5. In the chemical analysis of the mixed water, it was seen that the water was suitable for the mixed water qualities in TS 1247.

Table 5. Chemical analysis of the mixed water used

Parameters	Unit	Value
pH		7.49 (20 ^{0}C)
Cl-	mg/L	6.53
SO_4	mg/L	91.5
Mg	mg/L	41.5
Ca	mg/L	63.8
Zn	mg/L	0.375
Cu	mg/L	0.092
Fe	mg/L	0.074
NO_3-N	mg/L	4.35
Free ClO_2	mg/L	< 0.05
Total ClO_2	mg/L	< 0.09

Alkaline Activators (Na_2SiO_3)

Sodium silicate was used as activator in certain proportions to the mixtures obtained in the experimental study. This activator was obtained from companies that sell various chemical materials.

2.2. Method

Na_2SiO_3/binder and sand/binder ratios were kept constant in all mixtures in order to examine the changes that will occur in the mortar samples by changing the usage rates of PFW and FA curing at a certain temperature (120 ^{0}C). The ratios that applied and the sample code list used for FA and PFW in study, respectively, are presented in Table 7. Industrial wastes that used for obtaining geopolymer mortar was kept in the oven at 100 ^{0}C for 24 hrs for FA and at 110 ^{0}C for 48 hrs for PFW before starting the experimental studies, and then all passed through a 75 µm sieve. Thus, by reducing the particle diameters of the waste materials used in the geopolymer mortar mixtures, the surface areas were increased and the geopolymer reaction process was made more efficient.

Table 6. Samples prepared with FA and PFW.

	PFW10FA90S1T	PFW10FA90S2T	PFW20FA80S1T	PFW20FA80S2T	PFW30FA70S1T	PFW30FA70S2T
UK, gr	405	405	360	360	315	315
PFW, gr	45	45	90	90	135	135
Sand, gr	1350	1350	1350	1350	1350	1350
Water, gr	85	75	85	75	85	75
Water/Binder	0.19	0.17	0.19	0.17	0.19	0.17
Na_2SiO_3, ml	220	220	220	220	220	220
Curing Temp., ^{0}C	120	120	120	120	120	120

After the mixing process of the geopolymer mortar samples was completed, the mixture was immediately put into the mold. For this purpose, each fresh mixture was filled in two layers in 3 series molds of 40x40x160 mm. Each layer was compressed with 10 to 15 vibrations, and the mortar remaining on the mold after the second layer was taken from the mold with a sawing motion with the help of a trowel and the samples were leveled with a metal gauge to obtain a smooth surface. Each sample has been marked using the sample codes given in Table 6. After the necessary vibration and leveling processes were completed, the fresh mortar samples were poured into molds and wrapped with airproof nylon covers and kept under laboratory conditions (23±2 ^{0}C and humidity 50%±5) without removing the mold for 24 hrs.

After 24 hrs were completed, the nylon covers surrounding the samples were removed and the samples were covered with aluminum foils, this time without removing the mold, in such a way that it completely encloses the mold. After the coating process with aluminum foil was completed, the samples were placed in the oven at a certain temperature (120 ^{0}C) specified in Table 6 and cured for 72 hrs. After 72 hrs of curing process was completed, the mortar samples were taken from the oven and removed from the molds and cured in air (23±20 ^{0}C and humidity 50%±5) under room condition to complete the 7th and 28th day. The samples were experimentalized while they were fresh and hardened after their 7th and 28th days and the results obtained from experimental study were evaluated.

3. Results and Discussion

In order to determine the hardened properties of the produced mortar samples, fresh and hardened unit weight and compressive strength tests were carried out. The unit weight test was carried out on prismatic samples measuring 4x4x16 cm. In order to determine the unit weight according to TS EN 12390-7 (2010), all samples were weighed after curing on a balance with a capacity of 30 kg and a precision of 2 g. Then, the width, length and height of all samples were precisely measured using caliper.

The volumes of the samples whose sizes were determined were calculated. The weight to volume ratio and unit weights (kg/dm^3) of all samples were determined.

3.1. Fresh unit weight

At the stage of determining the fresh unit weights of all samples, a total of 216 fresh mortar samples were weighed while fresh, in order to be able to apply each type of test. Of these 216 fresh mortar samples, 72 of them are mortar samples containing 10%, 72 of them 20% and the other 72 of them 30% PFW content. By taking the average of these data, the changes of fresh unit weights according to the PFW content were collected in the graph. When an evaluation is made in general, it has been determined that the fresh unit weight decreases with the increase of the PFW rate in the mortar samples. For samples containing the same ratio of PFW and having different water content; it has been observed that with the decrease of the amount of water in the sample, the fresh unit weight also decreases.

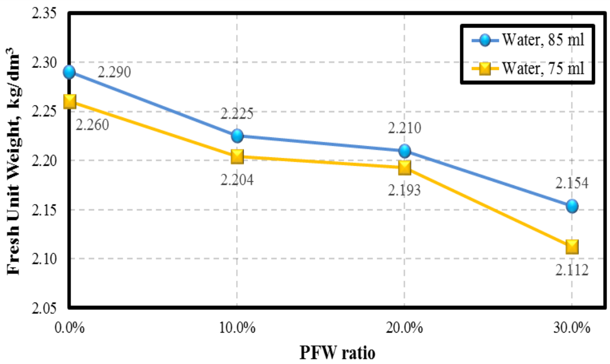

Figure 1. Change of fresh unit weight with PFW ratio.

As seen in Figure 1, the change of fresh unit weight; It was observed that the PFW was inversely proportional to the amount in the sample. In the samples with the same water content and different ratios of PFW and FA content, it was determined that the fresh unit weight decreased with the increase of the PFW ratio. It is seen that the control geopolymer samples containing 100% FA have the highest fresh unit weight. In the geopolymer mortar samples containing 100% FA, according to the water content in different ratios, it was seen that the FA mortar samples containing 85 ml of water had a higher fresh unit weight than the FA mortar samples containing 75 ml of water (2.29 kg/dm^3, 2.26 kg/dm^3).

3.2. Hardened unit weight

The samples containing different ratios of PFW and water were subjected to air curing under laboratory conditions for 24 hrs, and then cured at 120 ^{0}C for 72 hrs. The hardened unit weight of the control samples obtained with 100% FA for 7-day, containing 85 ml and 75 ml of water, was determined as 1.945 kg/dm^3 and 1.953 kg/dm^3, respectively. It is seen that the hard unit weight of the 28-day samples, including the control samples, is higher than the 7-day samples.

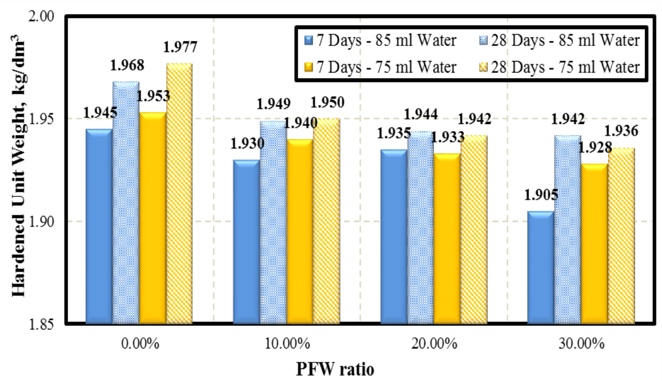

Figure 2. Change of hardened unit weight with PFW ratio cured at 120 ^{0}C.

3.3. Compressive strength

Compressive strength tests were applied on the 7th and 28th days on the samples that different PFW content and water content were cured at 120 ^{0}C. The data obtained are given in Figure 3 for 7 and 28-days strength behavior of all samples

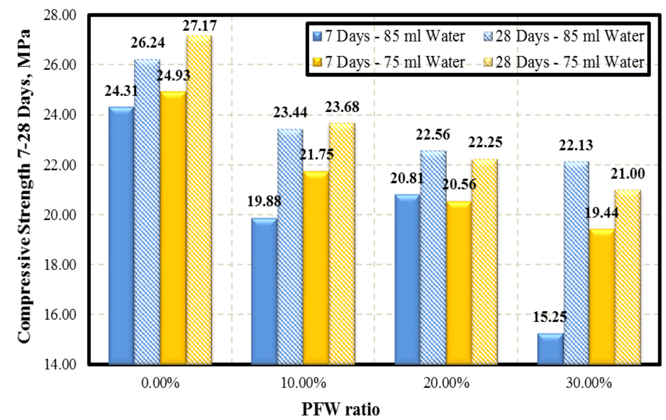

Figure 3. Change of compressive strength with PFW ratio cured at 120 ^{0}C.

As can be seen in Figure 3, it has been determined that the samples containing 75 ml of water for 28 days and 10% PFW have the highest compressive strength, excluding the control samples (23.68 MPa). The lowest compressive strength was observed in 7-day samples containing 85 ml of water and 30% PFW (15.25 MPa). It was determined that with the increase of water content and PFW ratio especially 10% in samples that cured at 120 ^{0}C, there is a decrease in 7 and 28-days compressive strength. When the compressive strength of the control samples with 100% FA and 85 ml water content and the samples with 85 ml water and 30% PFW content are compared, a dramatic decrease of 37.37% is observed. In the study, the highest compressive strength of 28 days samples was obtained at the rate of Si/Al = 7.1 and Na_2O/SiO_2 = 0.24, approximately 32 MPa. The lowest strength (0 MPa) was found as Si/Al = 7.7 and Na_2O/SiO_2 = 0.16 in samples that were cured at room temperature. And the highest strength was detected in samples having the ratio Si/Al = 7.1 and Na_2O/SiO_2 = 0.22 at 75 ^{0}C; cured at room temperature, 50 ^{0}C, 75 ^{0}C and 100 ^{0}C [17]. The 28-day compressive strengths for cement contents of 450 kg/m^3, 550 kg/m^3 and 650 kg/m^3 were 29.4–15.8 MPa, 37.8–19.8 MPa and 44.1–25.4 MPa, respectively, which are low for (SCC) with 10–40% incinerated sugarcane filter cake (ISFC) [18], it was determined. Compressive strengths of all mortars are related to the replacement ratios of limestone with filter mud (FM). When it is less than 20%, the compressive strengths of all mortars are greater than these of the control. The compressive strengths are affected negatively while it is more than 20% [19] in the study was detected. It was observed that the compressive strength of 28 days samples increased, and the flow decreased by the NaOH molarity increased in the fixed weight of Na_2SiO_3, sand, water and FA. Samples containing 9 M NaOH have the highest compressive strength (43.1 MPa), samples with Na_2SiO_3/NaOH 9 M, (400 gr) = 1.00 [20]. It is observed that the compressive strength of the one-part geopolymer paste increases rapidly with a decrease in H_2O/Na_2O ratios with the highest ratio having the lowest strength. It is therefore deduced that the improvement of the water content of the one-part geopolymer with the remaining uninterrupted variables leads to a significant increase in flowability and subsequent decreases [21] in the compressive strength of the one-part geopolymer mixture As in this study Bondar et al indicated that the compressive strength of geopolymer concrete decreased as the ratio of water to geopolymer solids by mass [22] increased. As the H_2O/Na_2O molar ratio increases, the compressive strength of geopolymer concrete [23] decreases.

4. Conclusion

Today, the rapidly developing industrial technology process has brought the industrial waste problem current issue. When the storage costs of these wastes and the negative effects they may have on the environment are evaluated, the recycling of wastes has become obligatory. In order to ensure this recycling, whatever the material is considered as industrial waste, physical and chemical analyzes should be made and materials that can be used and/or applicable for different sectors should be produced. This study was carried out on the usability of geopolymers obtained from PFW and FA, which is a sugar factory waste, as a building material.

- When the fresh unit weights of the samples were evaluated as a result of the experimental studies, the highest fresh unit weight was observed in samples with 85 ml of water and 10% PFW content (2.225 kg/dm^3), while the lowest fresh unit weight was found to be samples containing 75 ml of water and 30% PFW (2.112 kg/dm^3). It is seen that the low PFW unit weight (0.775 kg/dm^3; dry oven at 110 ^{0}C for 24 hrs and passed through a 75 μm sieve) has a direct effect on fresh unit weight. In this context, it was determined that as the fresh unit weight and water content of the samples decreased, the flow amounts decreased, and the viscosity increased.

- It has been observed that the hardened unit weight tends to decrease as the PFW ratio increases in the samples, and also that the hardened unit weight decreases with the increase in the PFW ratio has a negative effect by increasing the water absorption and water absorption percentage. It was determined that the increase in the PFW ratio in the samples and the increase in the void ratio in the porous microstructure decreased the unit weights and consequently the ultrasound pulse velocity.

- As can be seen in Figure 3, it has been determined that the samples containing 75 ml of water for 28 days and 10% PFW have the highest compressive strength, excluding the control samples (23.68 MPa). The lowest compressive strength was observed in 7-day samples containing 85 ml of water and 30% PFW (15.25 MPa). Also, it has seen increase in Na_2O/SiO_2 and SiO_2/Al_2O_3 ratios, the compressive strength of the mortar samples decreases.

With experimental studies to be carried out on this industrial waste containing high CaO content (e.g. PFW) in advanced stages, especially at different activator molarities and different curing temperatures, with different binders; When the mechanical, physical and chemical properties are examined, more effective results [24] are likely to be obtained.

Acknowledgement
The authors thank Eskişehir Osmangazi University, Scientific Research Project Funding (ESOGU BAP) for their financial support [Project number: 2017-1578].

Declaration of Conflict of Interests

The authors declare that there is no conflict of interest. They have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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