An Investigation of Steam Curing Pressure Effect on Pozzolan Additive Autoclaved Aerated Concrete

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***Abstract* – Autoclaved Aerated Concrete (AAC) is a porous light weight concrete obtained by adding a pore-forming material to a mixture made of finely pulverized siliceous aggregate and inorganic binder (lime and/or cement) and hardened by steam cure. In this study fly ash was used instead of siliceous aggregate and experiment samples were obtained by adding 3%, 6%, 9%, 12% silica fume to the cement. Samples were cured under 156^{0}C and 4 bars and 177^{0}C and 8 bars, and were investigated for compressive strength, bulk density and ultrasound pulse velocity to determine their mechanical and physical properties. Microstructure of samples was observed by using SEM and XRD techniques. Samples' bulk density values and compressive strengths are changing between 0.6-0.7 kg/dm^3 and 2.5-4.4 MPa respectively.**

***Keywords* – Aerated concrete, Pozzolan, Cure.**

1. Introduction

Autoclaved aerated concrete (AAC) is a structural material which is commonly used around Europe, particularly as it combines ease of construction with excellent combination of mechanical and thermal properties [1].

AAC is manufactured by steam curing of raw materials consisting of cement, lime and gypsum as binders, fine siliceous material, pore-generating aluminum powder and water [2]. These components are mixed with high amounts of water and molded to produce a cellular green body by H_2 gas generation at atmospheric pressure, and then autoclaved at 200^{0}C under saturated steam pressure for several hours [3,4]. The aluminum powder reacts with calcium hydroxide to form hydrogen gas in the making of aerated concrete, as follows [5].

$$2Al + 3Ca(OH)_2 + 6H_2O \rightarrow 3CaO \cdot Al_2O_3 \cdot 6H_2O + 3H_2$$

In recent years, the preparation of lightweight concrete by incorporation of pozzolanic siliceous material such as silica fume, coal fly ash, and slags has received further attention because of the economical use of naturally occurring raw materials, waste material recycling, and saved energy [6]. Hauser et al. [3] To extend the range of raw materials and lower the production costs, several researchers have investigated the possibility of replacing the traditional raw materials of AAC by industrial waste, such as air-cooled slag, coal bottom ash, efflorescence sand and phosphorus slag [7] and fly ash, silica fume.

Fly ash has pozzolanic activity in the presence of hydrating Portland cement to form compounds possessing cementitious properties [8]. With quartz sand aerated fly ash produced by the generated gas, the concrete is claimed to have better thermal insulation and strength properties.

It is claimed that, aerated concrete made of fly ash has better thermal insulation and strength properties compared to the ones produced from quartz sand. Perhaps due to the fact that fly ash has puzzolanic properties that react with lime which produce new bounds reinforcing the microstructure. Adding dark grey or blackish raw material to production of aerated concrete darkens the color of the final product or turns it into gray [9].

Silica fume is a highly puzzolonic material due to its amorphous and very fine-grained structure and the high amount of SiO_2 it contains [10]. Güçlüer et al. [11] substituted silica sand instead of fly ash and added specific proportions of silica fume to cement and created samples. They subjected those samples to steam cure and achieved similar physical and mechanical properties like commercial aerated concrete. Hauser et. al. [3] replaced cement with fly ash. They obtained maximum compressive strength as 0.7-0.8 Ca/Si ratio in their produced samples. Ünal et. al. [12] states in their study that fly ash and silica fume can be used in aerated concrete production; silica fume enhances the compressive strength of the samples.

This study intended to produce aerated concrete by replacing the main raw material which is silica sand with fly ash and adding 3%, 6%, 9%, 12% amounts of silica fume to cement. Steam curing applied to samples at different compressive strength values and physical and mechanical properties were investigated, samples' microstructures were observed by using SEM and XRD methods.

2. Materials

In this study fly ash obtained from Seyitömer (Kütahya/Turkey) thermal reactor was used. Particle dimensions of fly ash were determined by particle size analyzer (Figure 1). 90% of the fly ash is under 100 µm.

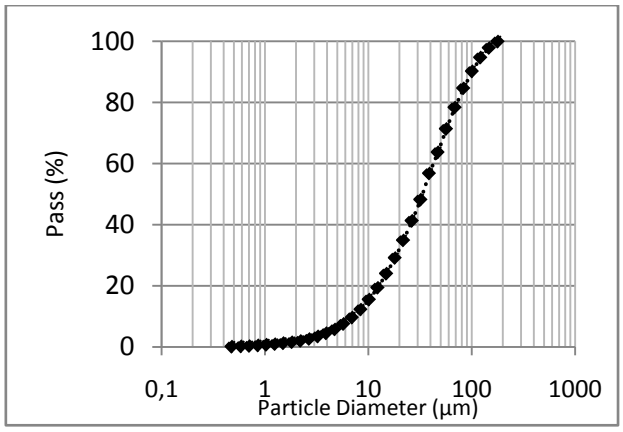

Figure 1.Fly ash particle size distribution

XRD analyze of the fly ash used in the study is shown in the Figure 2.

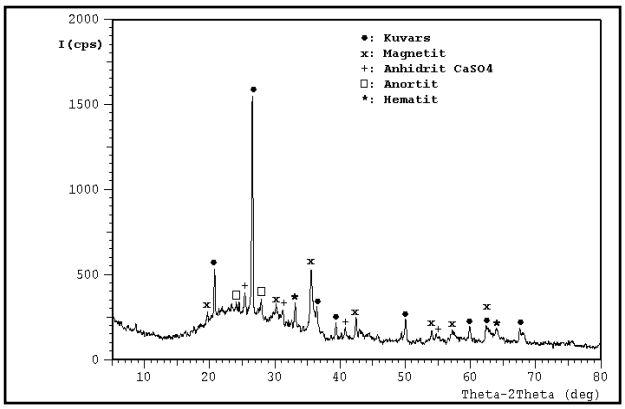

Figure 2.Fly ash XRD analyze

It is observed that ash samples were made of mainly quarts, anhydride (CaSO4), hematite, magnetite and anortite phases. Especially in the analysis of ash samples the peak between $2\Theta = 20^{o}$ and 30^{o} is where the degree of crystalline of the ash is low and points the presence of non-crystalline amorphous structures.

The chemical analysis of the fly ash is given in Table 1. According to this, the total ratio of SiO2 + Al2O3 + Fe2O3 in the fly ash found as 76, 5%.

Table 1. Chemical Structure of the Materials

Oxides	Fly Ash	Silica Fume	Cement
SiO_2	46.30	94.50	20.20
Al_2O_3	19.20	0.88	5.80
Fe_2O_3	11.00	0.70	3.23
CaO	6.34	0.80	64.10
MgO	4.76	1.25	0.44
SO_3	1.54	-	2.66

The CaO amount of the lime used in the study is determined as 96,22%. There are no harmful compounds found in the lime. The particle structure of the lime is very fine grained and 95,4% of the particles were passed from 90 µm. The Blaine finesse and specific gravity values are 3054 cm^2/g and 3.07 gr/cm^3 respectively. Silica fume was obtained from Antalya Etibank Electrometallurgy Factory, and aluminum powder was provided from Antalya YTONG factory.

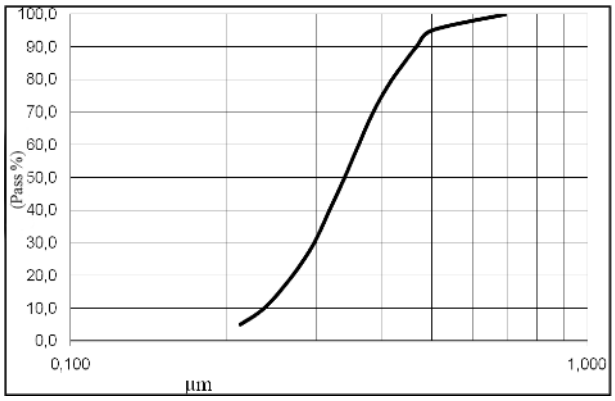

Figure 3.Silica fume particle size distribution

During the production of test samples, five different series were created by adding silica fume to cement with ratios of 3%, 6%, 9%, 12% along control series (Table 2). In the production process firstly fly ash, cement and gypsum were mixed by adding 2/3 of the calculated water in the mixer. After that, lime (CaO), aluminum powder and rest of the water were added and mixed again. Just then fluid aerated concrete mortar was casted without waiting not to lose its homogenous structure. Samples taken from molds were cured under 4 and 8 bars. When the steam curing process was complete physical and mechanical tests and microstructure analysis were applied.

Table 2. Mixture ratios

Mixture Ratios (gr)							
Series Name	Fly Ash	Cement	Silica Fume	Lime	Gypsum	Al Powder	Water
K	1500	800	-	300	300	1	2000
S1	1500	775	25	300	300	1	2000
S2	1500	750	50	300	300	1	2000
S3	1500	725	75	300	300	1	2050
S4	1500	700	100	300	300	1	2150

3. Methods

According to the bulk density experiment results conducted on samples that were steam cured under 4 and 8 bars are shown in Figure 4.

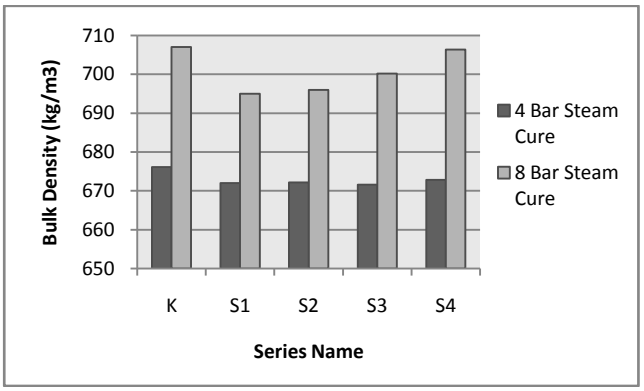

Figure 4. Bulk density values

Samples that were steam cured under 8 bars have greater bulk density compared to the ones steam cured under 4 bars. It is thought that; the gel structure, which is amorphous under 4 bar autoclave curing, has dissolved and passed to the crystalline phase under 8 bar autoclave curing with higher temperature and therefore the samples had more concentrated structure. However, there is an descending in bulk density of the samples containing silica fume, due to the fact that silica fume's specific weight is lighter than the specific weight of the cement.

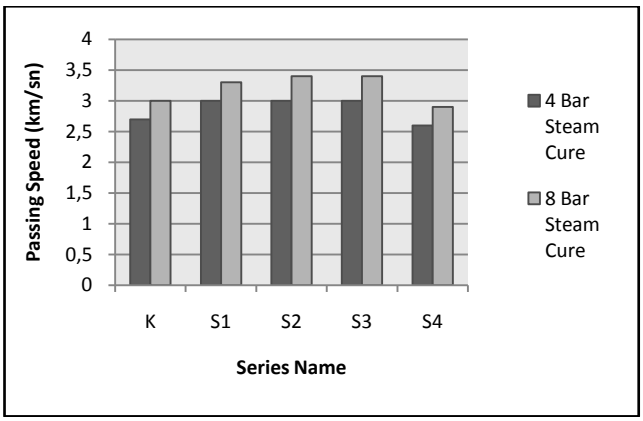

Figure 5. Ultrasonic pulse velocity values

Cement products are hydrated and consuming free water after autoclave curing and new C-S-H phases occur and fill the free spaces which exist in the product. Although it is anticipated that there would be an escalation in the ultrasonic pulse velocity of the new products, the voids occurred due to the effect of aluminum powder create a porous structure by dispersing homogenously in the structure of this hydrated products.

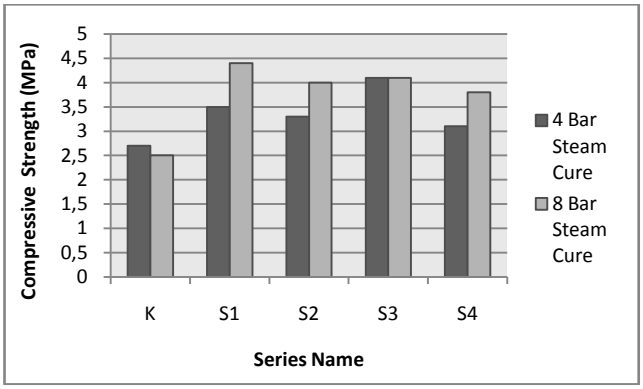

Figure 6. Compressive strength values

The compressive strength values belonging to the samples that were conducted to 4 and 8 bar pressure are given in Figure 6. According to the K-series there is an increase in the series with silica fume. Additional C-S-H gels occurred as a consequence of the reaction formatted between silica fume and free cement created from hydration of cement, which resulted as higher resistance.

Due to the high amount of cement ratio, it is observed that, in the early phases of hydration there is a significant amount of etringit phase, but the amount of this phase descents under autoclave curing conditions (Figure 7). However, the microstructure investigations done after autoclave curing shows the etringit crystals in the structure.

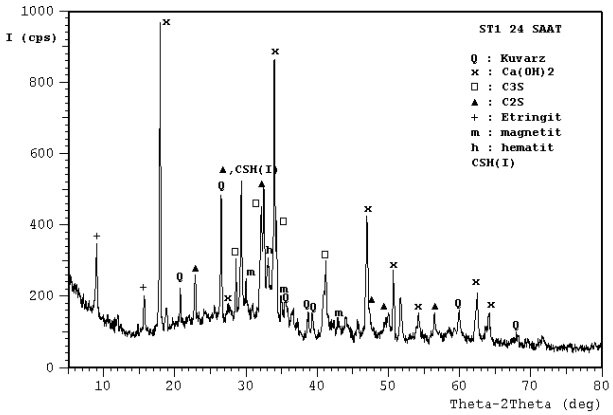

Figure 7. XRD analyze of early age

However, in the advanced hydration period (autoclave conditions) when the etringit phase dissolves under autoclave conditions and turns into new C-S-H phases, it creates major changes in volume and density in the structure which create micro cracks. It is noteworthy that these micro cracks are closer to the gypsum crystals which are rich with sulphate.

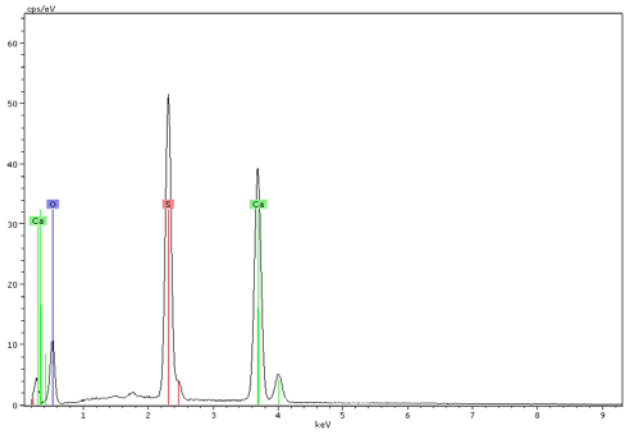

Figure 7. Micro cracks occur after autoclave curing EDX analyze

The deficit in the compressive strength values despite tobermorit plates were observed in the samples that were autoclave cured was an unexpected result. The samples that were autoclave cured. As seen in Figure 8., the tobermorite structure in the aerated concrete samples produced with cement, fly ash, lime components, is different than the tobermorite structure of procured commercial aerated concrete samples.

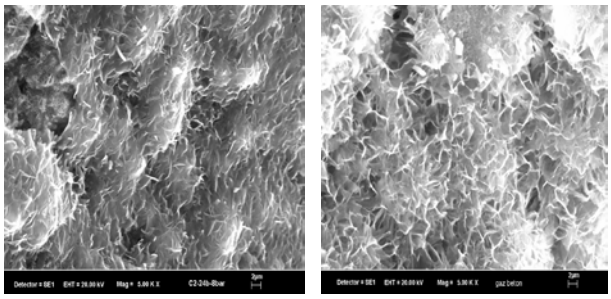

Figure 8. Different tobermorite structures

The crystal C-S-H phase seen in the commercial aerated concrete produced with cement, silica sand, and lime components is named as Normal tobermorite. The tobermorite structure in the aerated concrete produced with cement, fly ash, and lime components is named as Anomalous tobermorite. The reason for this is aluminum, sulphur and alkalis coming from fly ash enters crystal cage of tobermorite. Normal tobermolite structure is not affected from high temperatures (formed under cure conditions) and tobermorite does not lose the water in between its plates. Therefore it doesn't shrink. Anomaly tobermorit loses the water between its plates under high temperatures. Therefore, it significantly shrinks and loses its compressive strength under high temperature and long curing processes. The reduction in compressive strength as a result of the refinements made in hydration conditions can be explained with different types of tobermorite morphologies.

Phases that may occur correspondingly to the components in C-S-H system are given in Figure 9. In XRD observations (Figure 10.) only Xonotlit phase is detected, however some undefined peaks were found. It is thought that those undefined peaks are semi-crystal C-S-H transitions.

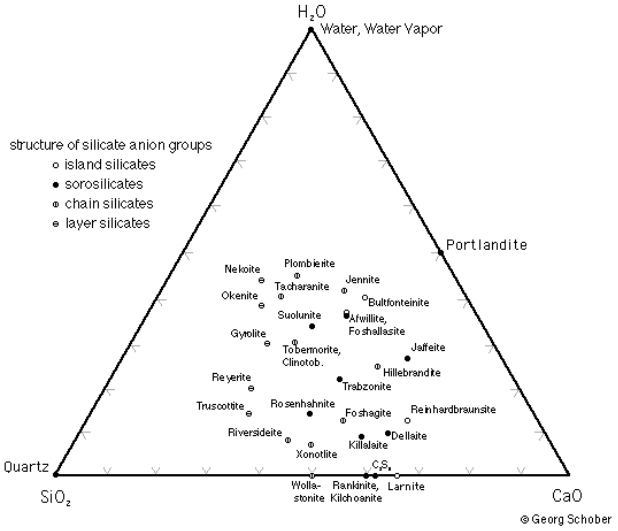

Figure 9. Crystal C-S-H phases and shows the CaO-SiO_2-H_2O system [13]

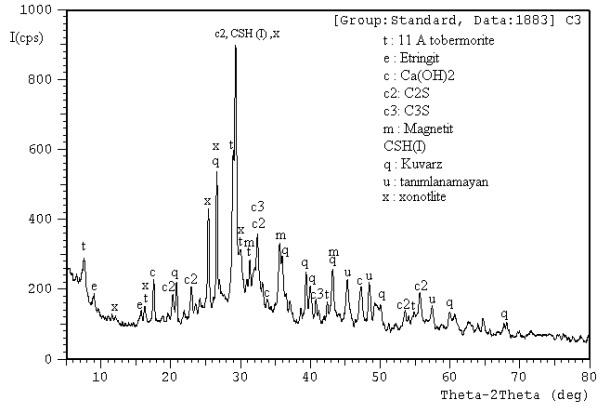

Figure 10. XRD analyze

3. Conclusion

Fly ash-cement-lime system samples were produced, similar physical and mechanical value results with the commercial aerated concrete were obtained.

In the microstructure exploration of silica fume added aerated concrete, structures closer to cement paste were observed. In addition, there were structures who look like weak tobermorit plates and which were seen in specific areas.

When XRD results obtained at the end of autoclave curing are compared to the mineralogical analysis of the commercial aerated concrete samples; it is seen that the tobermorite phase which is important for compressive strength does not evolve well. This result indicated the need for refinement of hydration conditions. This refinement would occur with high pressure autoclave usage. Whereby this may enable the reduction of cement usage and improve the durability of the product. Additionally re-usage of raw materials in production would provide values to the economy of the country.

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