

**COMPARISON OF TEST METHODS ON THE
COMPRESSIVE STRENGTH OF FLY ASH BLENDED CEMENTS**

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HASAN TUNÇ

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**COMPARISON OF TEST METHODS ON THE
COMPRESSIVE STRENGTH OF FLY ASH BLENDED CEMENTS**

submitted by **HASAN TUNÇ** in partial fulfillment of the requirements for the degree of **Master of Science in Civil Engineering Department, Middle East Technical University** by,

Prof. Dr. Canan Özgen
Dean, Graduate School of **Natural and Applied Sciences**

Prof. Dr. Ahmet Cevdet Yalçiner
Head of Department, **Civil Engineering**

Prof. Dr. İsmail Özgür Yaman
Supervisor, **Civil Engineering Dept., METU**

Prof. Dr. Mustafa Tokyay
Co-Supervisor, **Civil Engineering Dept., METU**

Examining Committee Members:

Assoc. Prof. Dr. Sinan Turhan Erdoğan
Civil Engineering Dept., METU

Prof. Dr. İsmail Özgür Yaman
Supervisor, Civil Engineering Dept., METU

Prof. Dr. Mustafa Tokyay
Co-Supervisor, Civil Engineering Dept., METU

Asst. Prof. Dr. Çağla Meral
Civil Engineering Dept., METU

Assoc. Prof. Dr. Mustafa Şahmaran
Civil Engineering Dept., Gazi University

Date:

19.06.2014

I hereby declare that all information in this document has been obtained and presented in accordance with academic rules and ethical conduct. I also declare that, as required by these rules and conduct, I have fully cited and referenced all material and results that are not original to this work.

Name, Last name: Hasan Tunç

Signature:

ABSTRACT

COMPARISON OF TEST METHODS ON THE COMPRESSIVE STRENGTH OF FLY ASH BLENDED CEMENTS

Tunç, Hasan

M.Sc., Department of Civil Engineering

Supervisor: Prof. Dr. İsmail Özgür Yaman

Co-Supervisor: Prof. Dr. Mustafa Tokyay

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In Turkey the compressive strength of cements are determined according to TS EN 196-1. That standard suggests the use of a constant water-cement ratio of 0.50 in preparation of mortar specimens for all types of cements. On the other hand, in USA cement producers use ASTM C 109 to determine the compressive strength of cements, which is based on a constant consistency of cement mortars for blended cements only. The main difference of these methods is the needed amount of water to produce cement mortar.

When a constant w/c is used to prepare mortar specimens as specified by TS EN 196-1, especially for certain blended cements insufficient compaction may occur and compressive strength may not be obtained in a standard repeatable manner. On the other hand, when ASTM C 109 is used, higher amount of water may be used to obtain a constant consistency and the compressive strength can be lower. Therefore, in this study, unlike EN 196-1 and ASTM C 109, a constant water/cementitious by volume method is suggested to prepare mortar specimens to determine their strength.

The aim of this thesis is to demonstrate the variabilities that can be confronted with the determination of compressive strength of fly ash blended cements. For this purpose, portlant cement clinker was replaced with fly ash (FA) at 20 %, 35 %, 55 % replacement levels, by using three different methods which are acquired by the constant water/cementitious by mass (EN 196-1), constant flow (ASTM) and constant water/cementitious by volume methods, 7-day and 28-day compressive strength of mortars were obtained. Then, coefficient of variation (CoV) of the results obtained by three different methods were compared.

After the experimental study, it was observed that a less deviation in the compressive strength results were obtained by constant water/cementitious by volume method and ASTM C109 method. However, for the constant water/cementitious by mass method (EN 196-1), higher deviance in the compressive strength results were obtained. As a result, it was determined that the constant water/cementitious by mass method (EN 196-1) is not proper for fly ash blended cements.

Keywords: Fly Ash, Compressive Strength, Coefficient of Variation, Mortar

ÖZ

TEST YÖNTEMLERİNİN UÇUCU KÜLLÜ ÇİMENTOLARIN BASINÇ DAYANIMLARI ÜZERİNDEN KARŞILAŞTIRILMASI

Tunç, Hasan

Yüksek Lisans, İnşaat Mühendisliği Bölümü
Tez Yöneticisi: Prof. Dr. İsmail Özgür Yaman
Ortak Tez Yöneticisi: Prof. Dr. Mustafa Tokyay

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Türkiye’de basınç dayanımları TS EN 196-1 göre belirlenmektedir. Bu standard bütün çimento tiplerinin hazırlanması için sabit 0,50 su-çimento oranını önermektedir. Diğer taraftan Amerika çimento üreticileri basınç dayanımlarının belirlenmesi için sabit kıvam değerini esas alan ASTM C 109 kullanmaktadır. Bu methodlar arasındaki ana fark harçların hazırlanmasında gerekli olan su miktarıdır.

Katkılı çimento harcı üretmek için TS EN 196-1 kullanıldığı zaman yetersiz sıkıştırma problemleri oluşabilir ve basınç dayanımları standardta istenilen şekilde elde edilemeyebilir. Diğer taraftan ASTM C 109 yöntemi kullanıldığında sabit bir kıvam elde etmek için yüksek su miktarı kullanılabilir ve bu durum basınç dayanımının azalmasına neden olabilir. Belirtilen sebeplerden dolayı, bu tezde TS EN 196-1 ve ASTM C 109 yöntemlerinden farklı olarak dayanım numunelerinin hazırlanması için hacimsel sabit su-çimento oranı önerilmektedir.

Bu tezin amacı uçucu küllü katkılı çimentoların basınç dayanımlarının ölçümünde karşılaşılabilen değişkenlikleri göstermektir. Bu amaç için uçucu kül içeren katkılı çimentolar kullanılmıştır. % 20, % 35, % 55 uçucu kül içeren katkılı çimentolar

hazırlanarak kütlege sabit su/bağlayıcı oranı (TS EN 196-1), sabit yayılma değeri (ASTM) ve hacimce sabit su/bağlayıcı oranı olarak 3 farklı yöntemle göre 7 ve 28 günlük basınç dayanımları elde edilmiştir. Daha sonra, üç yöntemden elde edilen basınç dayanım sonuçlarının varyasyon katsayıları karşılaştırılmıştır.

DeneySEL çalışmalar sonrasında, hacimsel sabit su-çimento oranı ve ASTM C 109 yöntemiyle elde edilen harçların basınç dayanım sonuçlarındaki sapmalar daha düşük olarak gözlemlenmiştir. EN 196-1 yöntemleriyle elde edilen harçların basınç dayanım sonuçlarındaki sapmalar daha fazladır. Sonuç olarak katkıli çimentolar için kütlege sabit su/bağlayıcı oranı yönteminin (TS EN 196-1) kullanılmasının uygun olmadığı sonucuna varılmıştır.

Anahtar Sözcükler: Uçucu Kül, Çimento Basınç Dayanımı, Varyasyon Katsayısı, Harç

To My Family

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LIST OF ABBREVIATIONS

ASTM	: American Society for Testing Materials
C-Flow	: Constant Flow
C-Mass	: Constant Water/Cementitious by Mass
C-Volume	: Constant Water/Cementitious by Volume
CoV	: Coefficient of Variation
EN	: European Norm
FA 20	: 20 % Fly Ash-Blended Cement
FA 35	: 35 % Fly Ash -Blended Cement
FA 55	: 55 % Fly Ash -Blended Cement
METU	: Middle East Technical University
pdf	: Probability Distribution Function
SEM	: Scanning Electron Microscope
TCMA	: Turkish Cement Manufacturers' Association
TS	: Turkish Standard
TSE	: Turkish Standard Institute

CHAPTER 1

INTRODUCTION

1.1. General

Portland cement is the basic ingredient of concrete. Concrete is used in large quantities almost everywhere mankind has a need for infrastructure. Turkey has a large construction industry and has one of the largest cement industries in the world. According to the Turkish Cement Manufacturers' Association (TCMA) Turkey produced 73.8Mt of cement in 2013 as shown in Figure 1.

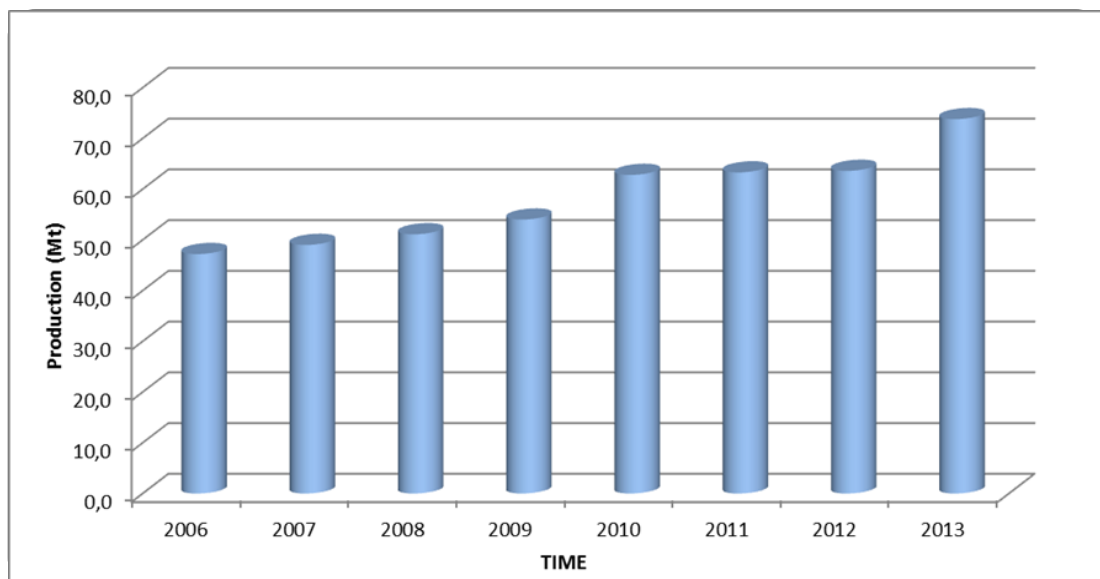


Figure 1 Yearly Cement Production in Turkey [1]

The cement production is an energy intensive industry. Its energy cost represents a significant part of total production costs. Generally, natural resources are used to produce cement. In 2011, the European cement producers consumed an energy equivalent of about 18 Mt of coal, fossil fuel, for the production of 196 Mt of cement [2].

The main constituent of cement is clinker. The clinker granulating stage uses approximately one-third of the energy needed to manufacture one ton of cement [3]. Since these stages consume excessive energy based on natural resources, the cement industry not only needs to care about the environmental sustainability but also to lower the cost of its product.

In order to produce environmentally friendly products, cement producers manufacture blended cements to decrease the clinker content. In blended cement production, part of the clinker is replaced with alternative constituents such as fly ash, natural pozzolan, etc [4].

In Turkey the compressive strength of cements are determined by TS EN 196-1, that is based on constant water/cementitious ratio for all types of cements. On the other hand, USA cement producers use ASTM C 109 standard to determine the compressive strength of cements, which is based on constant flow for blended cement. The main difference between these two methods is the amount of water to produce cement mortar.

1.2. Objective and Scope of the Thesis

The aim of this research is to show the variabilities that can be confronted with the determination of compressive strength of blended cements. For this purpose, fly ash (FA) blended cements were used. Portland cement clinker was replaced with fly ash (FA) at 20 %, 35 %, 55 % replacement levels and cement mortars were prepared by three different methods. The water content of each mortar was changed by using different amounts of water as follows;

- ❖ Constant water/cementitious by mass (EN 196-1)
- ❖ Constant flow (ASTM),
- ❖ Constant water/cementitious by volume

Then, coefficient of variation of the results obtained by three different methods were compared.

This study consists of five chapters:

In Chapter 2, role of the cement in concrete is briefly mentioned. Then, according to TS EN 197-1 cement types are defined. Then general classifications. Moreover, fly ash used in the cement production and effects of fly ash on the properties of cement are briefly explained. Finally, the quality control applications for cements are outlined and two different compressive strength test methods (ASTM C 109 and EN 196-1) are compared.

In Chapter 3, the properties of materials used in the study and the details of mixture preparation are given. Then evaluation procedure of strength test data was presented.

In Chapter 4, the compressive strength test results of cement types FA20, FA35, FA55 are presented. Also, results are statistically analyzed and evaluated for all methods.

In Chapter 5, the conclusions and recommendations for further studies are provided.

CHAPTER 2

THEORETICAL CONSIDERATIONS

2.1. Production of Portland Cement

The clinker is main component of cement. Clinker is produced from raw materials, such as limestone and clay, which are crushed and fed into a rotary kiln. The clinker burning takes place at a material temperature of 1450°C which is needed to form the new compounds. Clinker mainly consists of calcium, silicium, aluminium and iron-oxides. The next step is handled in a cement grinding mill. Gypsum and other additional materials (such as slag, fly ash, natural pozzolanas, etc.) are added to the clinker. All constituents are ground leading to a fine and homogenous powder called cement [5].

In Turkey and European zone, Harmonized EN 197-1 is used, which has 27 different main types of cement according to its chemical composition. Specifically, Turkish harmonized standard (TS EN 197-1) not only contains portland cements but also includes blended cements [6]. However, in USA there are three different types of standards which are ASTM C 150 for Portland cements, ASTM C 595 for blended cements and ASTM C 1157 for hydraulic cements.

2.2 Main Constituents of Cement

Cement and concrete producers have to evolve in the terms of the environment within a sustainable development perspective, which means that more mineral components will be blended with clinker and water/cementitious ratio will be lowered if they want to increase the life cycle of concrete structures and lengthen as much as possible the use of hydraulic binders and aggregates [2].

As the cement types alter, the percentage of these main constituents in cements change. Composition of main constituents of the cements in TS EN 197-1 is given in Table 1.

Table 1 Percentage of Cement Composition [6].

Main types	Notation of the 27 products (types of common cement)	Composition (percentage by mass ^a)													
		Main constituents										Minor additional constituents			
		Clinker	Blast-furnace slag	Silica fume	Pozzolana		Fly ash		Burnt shale	Limestone					
					natural	natural calcined	siliceous	calcareous		L	LL				
K	S	D ^b	P	Q	V	W	T	L	LL						
CEM I	Portland cement	CEM I	95-100	–	–	–	–	–	–	–	–	–	–	0-5	
CEM II	Portland-slag cement	CEM III/A-S	80-94	6-20	–	–	–	–	–	–	–	–	–	–	0-5
		CEM III/B-S	65-79	21-35	–	–	–	–	–	–	–	–	–	–	0-5
	Portland-silica fume cement	CEM III/A-D	90-94	–	6-10	–	–	–	–	–	–	–	–	–	0-5
	Portland-pozzolana cement	CEM III/A-P	80-94	–	–	6-20	–	–	–	–	–	–	–	–	0-5
		CEM III/B-P	65-79	–	–	21-35	–	–	–	–	–	–	–	–	0-5
		CEM III/A-Q	80-94	–	–	–	6-20	–	–	–	–	–	–	–	0-5
		CEM III/B-Q	65-79	–	–	–	21-35	–	–	–	–	–	–	–	0-5
	Portland-fly ash cement	CEM III/A-V	80-94	–	–	–	–	6-20	–	–	–	–	–	–	0-5
		CEM III/B-V	65-79	–	–	–	–	21-35	–	–	–	–	–	–	0-5
		CEM III/A-W	80-94	–	–	–	–	–	6-20	–	–	–	–	–	0-5
		CEM III/B-W	65-79	–	–	–	–	–	21-35	–	–	–	–	–	0-5
	Portland-burnt shale cement	CEM III/A-T	80-94	–	–	–	–	–	–	–	6-20	–	–	–	0-5
		CEM III/B-T	65-79	–	–	–	–	–	–	–	21-35	–	–	–	0-5
	Portland-limestone cement	CEM III/A-L	80-94	–	–	–	–	–	–	–	–	6-20	–	–	0-5
		CEM III/B-L	65-79	–	–	–	–	–	–	–	–	21-35	–	–	0-5
		CEM III/A-LL	80-94	–	–	–	–	–	–	–	–	–	6-20	–	0-5
		CEM III/B-LL	65-79	–	–	–	–	–	–	–	–	–	–	21-35	0-5
	Portland-composite cement ^c	CEM III/A-M	80-88	←-----12-20----->										0-5	
CEM III/B-M		65-79	←-----21-35----->										0-5		
CEM III	Blast furnace cement	CEM III/A	35-64	36-65	–	–	–	–	–	–	–	–	–	–	0-5
		CEM III/B	20-34	66-80	–	–	–	–	–	–	–	–	–	–	0-5
		CEM III/C	5-19	81-95	–	–	–	–	–	–	–	–	–	–	0-5
CEM IV	Pozzolanic cement ^c	CEM IV/A	65-89	–	←-----11-35----->						–	–	–	0-5	
		CEM IV/B	45-64	–	←-----36-55----->						–	–	–	0-5	
CEM V	Composite cement ^c	CEM V/A	40-64	18-30	–	←-----18-30----->			–	–	–	–	–	0-5	
		CEM V/B	20-38	31-49	–	←-----31-49----->			–	–	–	–	–	0-5	

^a The values in the table refer to the sum of the main and minor additional constituents.
^b The proportion of silica fume is limited to 10 %.
^c In Portland-composite cements CEM III/A-M and CEM III/B-M, in pozzolanic cements CEM IV/A and CEM IV/B and in composite cements CEM V/A and CEM V/B the main constituents other than clinker shall be declared by designation of the cement (for examples, see Clause 8).

2.2.1 Fly Ash (FA)

Fly ash is produced in furnaces of coal burning power plants. Fly ashes are very fine predominantly spherical glassy particles collected in the dust collection systems from the exhaust gases of fossil fuel powder [3].

Specifically, in the power plants, coal is first pulverized in grinding mills before being fed into the burning zone of the boiler. In this zone the coal combusts producing heat with temperatures reaching approximately 1500°C.

At this temperature the noncombustible inorganic minerals (such as quartz, calcite, clay minerals) melt in the furnace and fuse together as molten droplets. These particles are carried from the combustion chamber of a furnace by exhaust gas. The droplets cool to form spherical glassy particles which is called fly ash then the fly ash is collected from the exhaust gases by mechanical and electrostatic precipitators [4]. Schematic layout of process can be seen at Figure 2.

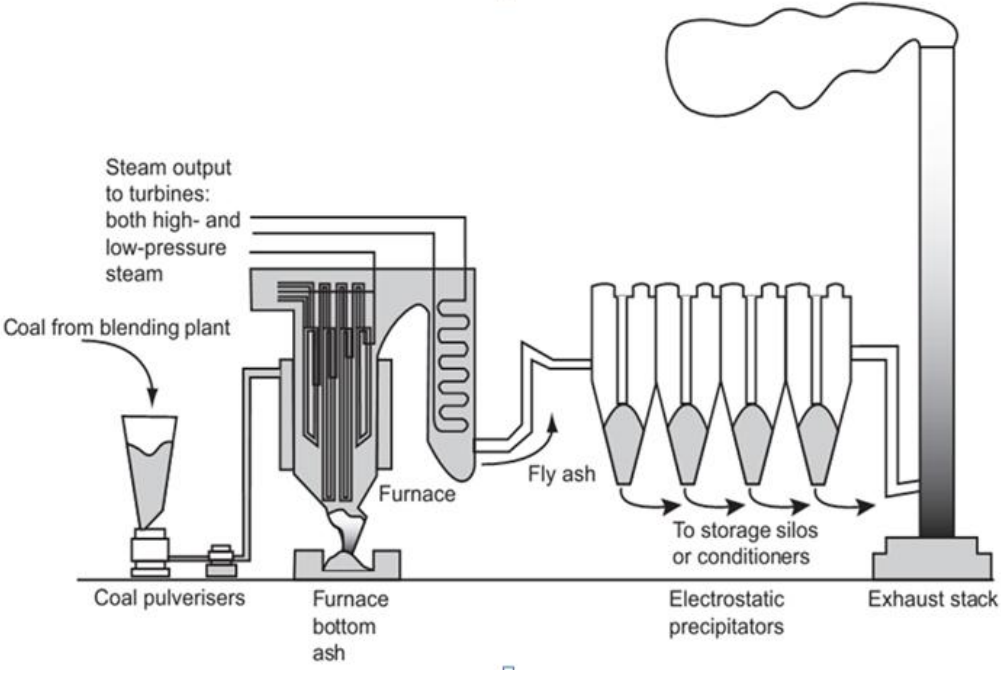


Figure 2 Process of Fly Ash Production [4]

As noted earlier, fly ash is part of coal ash, and is obtained during the combustion of coal in electrical power plants. Depending on the source and properties of the coal being burned, the components of fly ash vary considerably, but all fly ash includes substantial amounts of silicon dioxide (SiO₂) and calcium oxide or lime (CaO) [7].

The classification of fly ashes according to their chemical composition are determined following the ASTM C 618 in USA and EN 197-1 in Europe.

Two classes of fly ash are defined by ASTM C 618: Class F fly and Class C fly ashes. The main difference between these classes is the amount of calcium, silica, alumina, and iron content in the ash [8]. Table 2 shows the chemical composition of fly ash classes. On the other hand, EN 197-1 defines fly ashes into two groups; namely, siliceous and calcareous fly ashes, depending upon the required fly ash content [6].

Table 2 Typical Chemical Composition of Fly Ash [8]

Chemical Composition	Percent of Composition		
	Typical Class C	Typical Class F	Typical Portland Cement
CaO	24	9	64
SiO ₂	40	55	23
Al ₂ O ₃	17	26	4
Fe ₂ O ₃	5	7	2
MgO	5	2	2
SO ₃	3	1	2

In addition, color is one of the important physical properties of fly ash in terms of estimating the lime content qualitatively. It is suggested that lighter color indicate the presence of high calcium oxide and darker colors suggest high organic content [9].

2.2.1.1 Class F Fly Ash

Class F fly ash is normally produced from burning anthracite or bituminous coal. Class F fly ashes meet the chemical requirement Equation 2.1. This type has low-calcium (< 10 % CaO). This class of fly ash has pozzolanic properties [8, 10].

$$\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 \geq 70 \% \quad (2.1)$$

2.2.1.2 Class C Fly Ash

Class C Fly ash is normally produced from lignite or sub-bituminous coal. This class of fly ashes meet the chemical requirement Equation 2.2. Class C fly ashes contain lime contents higher than 10% ($> 10 \% \text{ CaO}$). Moreover, this type of fly ash, in addition to having pozzolanic properties, also has some cementitious properties [8, 10].

$$\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 \geq 50 \% \quad (2.2)$$

2.2.1.3 Fly Ash Chemistry

Chemical constituents of fly ash are generally related on the chemical composition of the coal. However, fly ash that are produced from the same source and which have very similar chemical composition, can have significantly different ash mineralogies depending on the coal combustion technology used. When the maximum temperature of the combustion process is approximately 1200°C and the cooling time is short, the ash produced is mostly glassy phase material. Where boiler design or operation allows a more gradual cooling of the ash particles, crystalline phase calcium compounds are formed [11].

The factors that influence the mineralogy of a fly ash are [12]:

- Chemical composition of the coal.
- Coal combustion process including coal pulverization, combustion, flue gas clean up, and fly ash collection operations.
- Additives used, including oil additives for flame stabilization and corrosion control additives.

2.2.1.4 Effects of FA on Concrete Properties

As mentioned early fly ash is a pozzolanic material with a finely amorphous siliceous or siliceous and aluminous material with varying amounts of calcium. The material reacts with the calcium hydroxide released by the hydration of portland cement to produce various calcium-silicate hydrates (C-S-H) and calcium-aluminate hydrates.

2.2.1.4.1 Workability and Water Demand

The term workability refers to the ease with which fresh concrete can be mixed, placed, molded, consolidated and finished. A well-proportioned fly ash concrete mixture improves workability when compared with a portland cement concrete of the same slump [13], which means fly ash concrete flows and consolidates better than a conventional portland cement concrete when vibrated.

The use of fly ash with a high fineness and low carbon content reduces the water demand of concrete, thus the use of fly ash permit the concrete to be produced at lower water content when compared to a portland cement concrete of the same workability as depicted in Figures 3. Despite the fact that the same amount of water reduction varies generally with the nature of the fly ash and other parameters of the mixture. It is stated by Thomas (2013) that each 10% of fly ash approximately allow a water reduction of at least 3% [14].

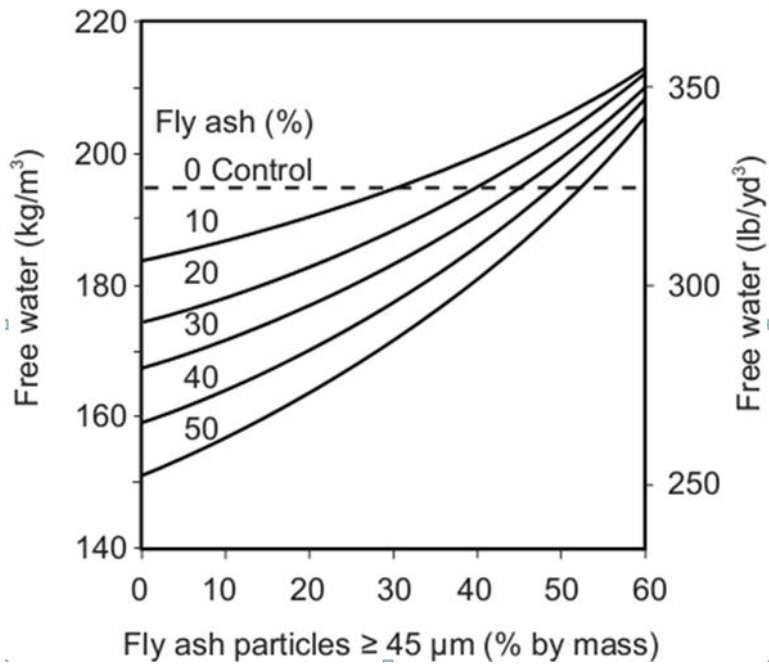


Figure 3 Effect of fly ash fineness on water demand for equal slump [15]

The use of fly ash also improves the cohesiveness and reduces segregation of concrete.

2.2.1.4.2 Strength

A concrete is proportioned to achieve a certain minimum strength at a specified age (typically 28 days). For fly ash blended cements, this can be achieved by selecting the suitable water/cementitious materials ratio. As a matter of course water-to-cementitious materials ratio needed vary depending on the level of fly ash replacement. If the specified strength is required at 28 days or earlier this will usually require lower values of w/cm when using higher levels of fly ash. A lower w/cm can be achieved by a combination of reducing the water content by either taking advantage of the lower demand in the presence of fly ash or by using a water-reducing admixture or both and increasing the total cementitious content of the mix.

Figure 4 states the effect of fly ash replacement for the same water-to-cementitious (w/cm) and strength development of concrete. As the level of replacement increases the early-age strength decreases. However, as seen in Figure 4 long-term strength development is improved when fly ash is used. The age at which strength parity with the control (portland cement) concrete is achieved is greater at higher levels of fly ash. The ultimate strength achieved by the concrete increases with increasing fly ash content, at least with replacement levels up to 50%. Generally, the differences in the early-age strength of portland cement and fly ash concrete are less for fly ash with higher levels of calcium, but this is not always the case [16].

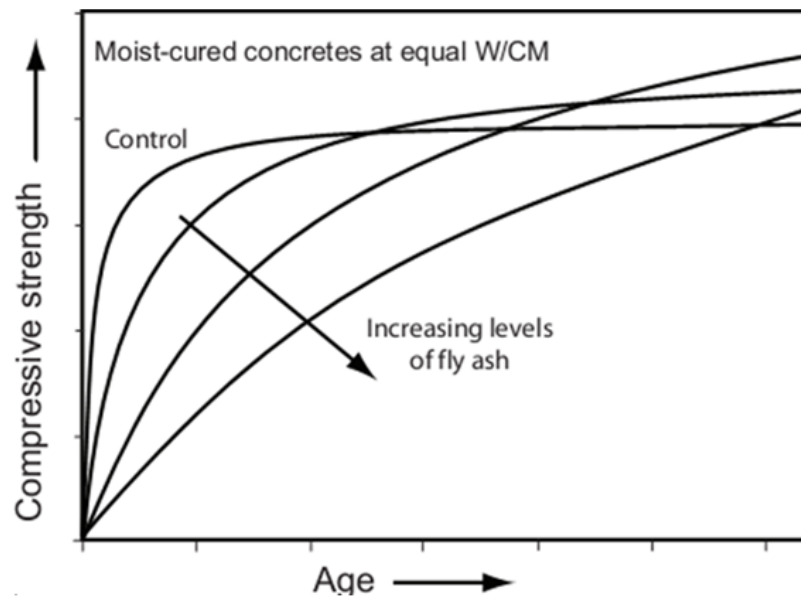


Figure 4 Effect of Fly Ash on Compressive Strength Development [16]

CHAPTER 3

EXPERIMENTAL STUDY

3.1 General

In the preparation of the blended cements, portland cement clinker, fly ash and gypsum are used. Then portland cement clinker was replaced with fly ash at 20 %, 35 %, 55 % replacement levels, namely describing CEM II and CEM IV in EN 197. The blended cements used were prepared by TCMA-R&D laboratory and in order to perform the compressive strength test of fly ash-cements the Construction Materials Laboratory of Turkish Standard Institute (TSE) was chosen. Then, compressive strength of mortars for each fly ash-cement types were obtained by the constant water/cementitious by mass (EN 196-1), constant flow (ASTM) and constant water/cementitious by volume methods.

3.2 Materials

The clinker that used in this study is obtained from Oyak Bolu Cement Plant. Fly ash was taken from Seyitömer power plant. In this research fly ash and clinker were separately ground. The chemical composition of the materials which were used in this study is presented in Table 3. The fineness of the ingredients which are clinker, gypsum and mineral additives (FA) were selected as $3500 \pm 200 \text{ cm}^2/\text{g}$, $4000 \pm 200 \text{ cm}^2/\text{g}$ and $3850 \pm 200 \text{ cm}^2/\text{g}$ respectively. Ingredients which were used in mixture were stated in Table 4. Amount of gypsum was selected as 4 % for 20 % and 35 % fly ash cements and 3 % for 55 % fly ash cements. The blended cements used in study were labeled as FA 20, FA 35, FA 55 according to additive amounts.

All of the cements and CEN Standard Sand conforming TS EN 196-1 were obtained by TCMA-R&D laboratory.

Table 3 Chemical Composition of the Materials

Chemical Composition (%)	Fly Ash (FA)	Clinker (C)	Gypsum (G)
SiO ₂	54.30	20.43	2.26
Al ₂ O ₃	16.80	5.73	0.08
Fe ₂ O ₃	10.50	3.25	0.28
CaO	7.70	65.50	32.16
MgO	4.50	2.67	0.68
SO ₃	1.20	0.42	42.68
Na ₂ O	0.50	0.37	0.34
K ₂ O	1.70	0.58	0.12
Cl ⁻	-	0.0098	-
Loss on Ignition	2.30	1.02	21.54

Table 4 Proportions of Ingredients

Label	Mix Proportions, by mass		
	Clinker (C)	Gypsum (G)	Fly Ash (FA)
FA20	80	4	20
FA35	65	4	35
FA55	45	3	55

Generally, the particles of fly ash are mostly glassy spherical solids, range in size from 1 to 100 microns (0.1mm). The average size is about 20 microns, which is similar to portland cement average particle size [17].]. When fly ash with spherical shape is added into the concrete, the workability of the mix is improved during pouring. This is due to the spherical shape of its particles. Fly ash in the mix allows concrete to flow and pump better than 100% Portland cement concrete. Moreover, the improved workability can be achieved with less water. The amount of water in the mix is decreased in direct proportion to the amount of fly ash added to the mix [18]. Figure 5 shows the surface morphology of typical fly ash particles.

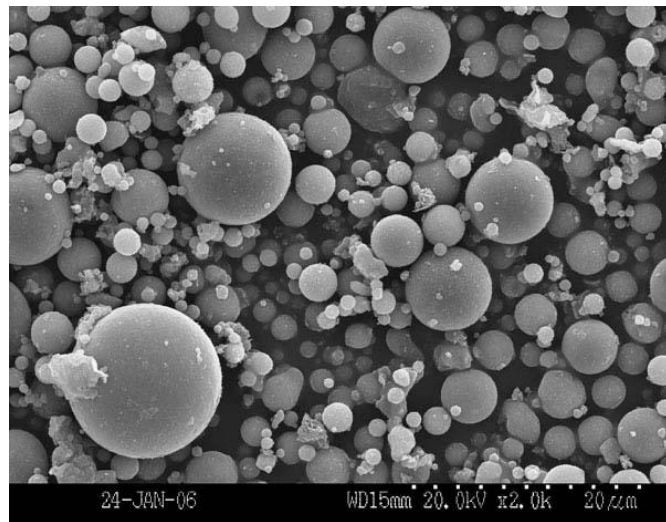
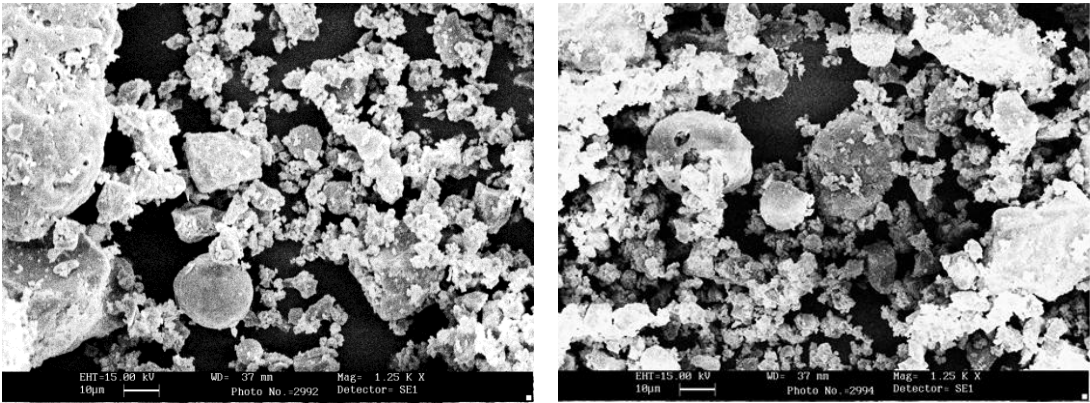


Figure 5 Typical Class F fly ash sample as viewed via SEM at 2000x magnification [19]

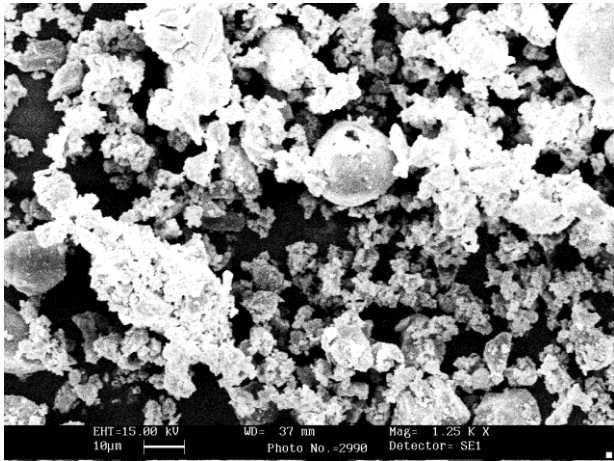
The particle morphology (shape) and the size distribution of fly ash are related on coal origin, the state of combustion of the coal, pulverization conditions (temperature and oxygen level), the uniformity of the combustion process and the type of powder collection system [20].

As can be seen from Figure 6 fly ash particles used in study are not spherical. Fly ash with non-spherical particles dont have the same effect on the workability, they increase water demand in mix because while fly ashes with spherical shape swarm around individual sand grains, and act as “ball bearing” during the flow, non-spherical particles have angular, rugged shapes thus resulting in an increase in water demand [21].



(a) FA 20

(b) FA 35



c) FA 55

Figure 6 SEM of Cements Used In Study at 1250 x Magnification

3.3 Mixture Preparation of Mortars

For the experimental study 3 different blended cements were used and for each blended cements different water/cementitious ratios were calculated and consequently, different mortar mixtures were prepared. Table 5 presents the ingredients used in mixture of the cement mortars.

Table 5 Ingredients Used In Mixture of the Cement Mortars

Cement Type	Method	Amount of Each Ingredient (g)			
		Cement	Water	Sand	w/c
FA 20	C-Mass	450	225	1350	0.50
	C-Volume	450	248	1350	0.55
	C-Flow	450	300	1350	0.67
FA 35	C-Mass	450	225	1350	0.50
	C-Volume	450	265	1350	0.59
	C-Flow	450	350	1350	0.78
FA 55	C-Mass	450	225	1350	0.50
	C-Volume	450	289	1350	0.64
	C-Flow	450	400	1350	0.89

As can be seen from Figure 7, for the same consistency the w/c ratio increases linearly. This is attributed to the non-spherical surface morphology of the fly ash used in this study.

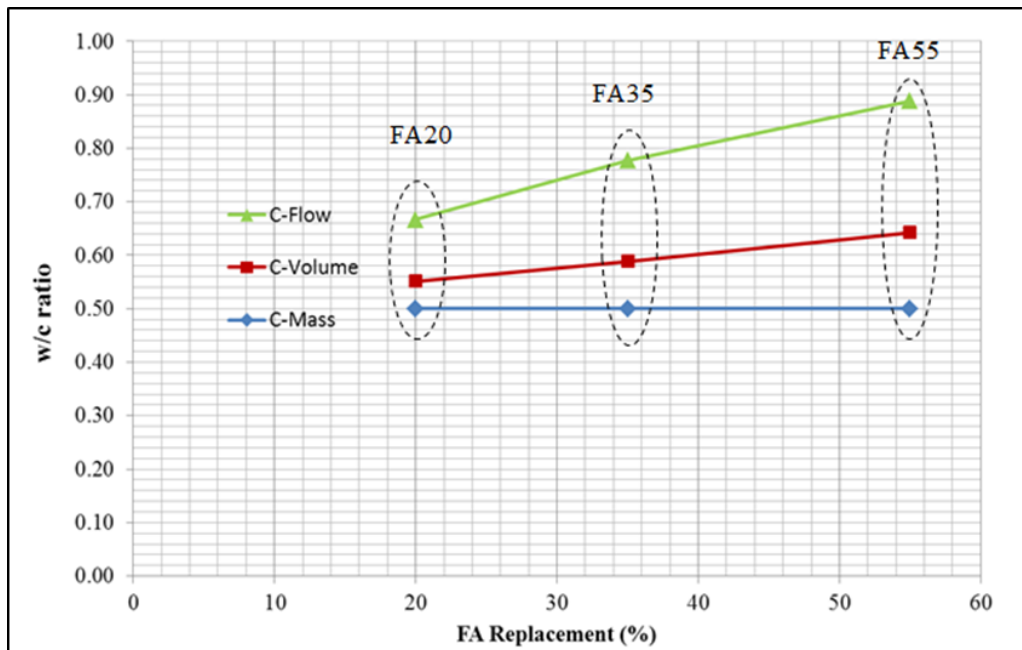


Figure 7 w/c Ratio for Fly Ash-Blended Cements

The water/cementitious ratios used in this study were selected as follows:

- Constant Water/cementitious by mass method: Mortars were prepared according to the TS EN 196-1 standard, which suggests the use of 450 g cement, 1350 g standard sand and 225 g water.
- Constant Flow value method: Mortars were prepared based on the ASTM C109 standard. The amount of water needed was determined by the flow method which is based on 25 drops in 15 seconds and flow value of the mortar will be equal to 110%. Moreover, 450 g cement and 1350 g standard sand were used to follow the TS EN 196-1 standard.
- Constant Water/Cementitious by Volume method: For this method, 450 g cement and 1350 g standard sand were used to follow the TS EN 196-1 standard. Table 7 gives the amount of water needed for volume method. The amount of water needed was calculated by the flowing formula:

Volume Equivalence Formula (3.1)

$$F_w = \frac{p}{(p + c)} \tag{3.1.1}$$

$$F_v = \frac{1}{1 + \frac{G_p}{G_c} \left(\frac{1}{F_w} - 1 \right)} \tag{3.1.2}$$

$$\frac{w}{(c + p)} = \frac{G_c \frac{w}{c}}{G_c (1 - F_v) + G_p F_v} \tag{3.1.3}$$

Where,

w: Water content (by mass)

c: Cement content (by mass)

p: Pozzolan content (by mass)

G_p: Specific gravity of pozzolan

G_c: Specific gravity of cement

F_w: Pozzolan/total binder (by mass)

F_v: Pozzolan/total binder (by volume)

In the formula, specific gravity of pozzolan and cement was given in Table 6.

Table 6 Specific Gravity of Ingredients

Clinker + Gypsum	Fly ash
3.19	2.09

Table 7 Water Content for C-Volume Method

Cement Type	% of Ingredients		Fw	Fv	w/(c+p)	water (g)
	Clinker + Gypsum	Fly ash				
C	105.0	0.0				
FA20	84.0	20.0	0.19	0.27	0.55	248
FA35	68.3	35.0	0.34	0.44	0.59	265
FA55	47.3	55.0	0.54	0.64	0.64	289

3.4 Compressive Strength Test Procedure

In order to obtain compressive strength test results from three different test method three batches of cement mortar were used for each test method and for each batch three prismatic specimens were prepared. Therefore, for each test method total of 9 specimens were used. The compressive strength results were determined from two measurements for 1 specimen, consequently a total of 18 compressive strength results were obtained for each test method.

In order to determine compressive strength test results prismatic specimens of 40x40x1600 mm were used. The calculation of compressive strength was achieved by the flowing formula:

$$\sigma = \frac{F}{A} \quad (3.2)$$

Where, σ is the Compressive strength [N/mm²], F is the maximum force applied [N] and A is the area [mm²] which is equal to 1600 mm².

3.5 Comparison of Test Methods

The determination of compressive strength is carried out according to the TS EN 196-1 which describes exactly the same procedure for all types of cement. Hence, In order to fulfill the experiment, 40 × 40 × 160 mm prism that is described in TS EN 196-1 was used. However ASTM C109 proposes different specimen molds to determine the compressive strength of concrete which is 50 mm cubic mold and ASTM C 109 describes three different methods of specimen preparation for ordinary portland cement, air-entrained portland cements, and blended cements.

The main differences between the two standard methods are the amount of water used and the process of the fresh mortar preparation. Table 8 shows the differences between TS EN 196-1 and ASTM C109.

Table 8 Comparison of ASTM C 109 and EN 196-1.

Standars	EN 196-1	ASTM C 109	
	All Cement	Blended Cement	Portland Cement
Cement	1	1	1
Sand	3	2.75	2.75
Water	0.5	flow	0.485
W/C	0.5	flow	0.485
Mold	40x40x160mm prism	50 mm cube	50 mm cube
Tamping	2 layers & tamping by device	2 layers & hand tamping	2 layers & hand tamping
Loading Rate	(2400 ± 200) N/s	(900-1800) N/s	(900-1800) N/s

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 General

In this study, three different types of blended cements namely, FA20, FA35, FA55 were used. For each type of cement, three different cement mortars were prepared and for each type of mortar three molds were cast and compressive strength of these mortars were tested at 7 and 28 days.

4.2 Compressive Strength Test Results

Three different Fly ash-blended cements labeled as FA20, FA35 and FA55 are separately analyzed.

4.2.1 20 % Fly ash-Blended Cement (FA20)

The results of 7 and 28-day compressive strength test results of FA 20 mortar are given in Table 9.

According to Table 9 the results of C-Mass mortar for 7 day compressive strength vary in between 3.5 MPa and 6.3 MPa. The results for C-Volume 7 day compressive strength vary in between 13.9 MPa and 18.5 MPa and the results of C-Flow mortar for 7 day compressive strength vary in between 14.3 MPa and 17.6 MPa. Difference between upper and lower compressive strength results of C-Mass method is 2.6 MPa, C-Volume method is 4.6 MPa and C-Flow method is 3.3 MPa.

Table 9 Compressive Strength Test Results of FA 20

Compressive Strength (MPa)	C-Mass			C-Volume			C-Flow		
	Mold-1	Mold-2	Mold-3	Mold-1	Mold-2	Mold-3	Mold-1	Mold-2	Mold-3
7-Day	4.9	4.4	5.8	15.3	16.0	15.1	17.1	14.5	15.9
	5.0	4.6	5.6	17.7	15.4	15.0	16.1	15.7	15.7
	4.9	5.4	5.5	17.9	17.2	14.6	17.6	14.3	14.9
	5.1	3.5	6.3	13.9	18.5	14.8	16.1	15.8	16.3
	5.5	4.6	4.6	16.5	17.4	14.9	16.4	16.3	15.1
Mean	4.6	5.5	3.8	14.5	15.0	15.4	15.6	15.5	16.1
CV (%)	5.0	4.7	5.3	16.0	16.6	15.0	16.5	15.4	15.7
	5.9	15.7	17.2	10.4	8.1	1.8	4.5	5.1	3.6
28-Day	10.3	11.2	9.5	25.1	22.8	23.5	24.0	21.7	23.6
	9.6	10.4	11.3	25.9	21.9	20.8	25.9	22.8	24.1
	9.3	9.5	11.7	26.9	20.1	22.4	23.8	22.4	24.3
	9.2	11.5	9.6	26.8	24.7	23.1	24.0	22.6	24.6
	6.8	11.4	14.5	25.9	23.6	23.9	24.5	23.1	23.9
Mean	6.8	11.2	11.2	27.6	18.6	24.0	26.3	22.8	22.6
CV (%)	8.7	10.9	11.3	26.4	22.0	23.0	24.8	22.6	23.9
	17.3	7.1	16.1	3.4	10.3	5.2	4.4	2.1	2.9

According to Table 9 the results of C-Mass mortar for 28 day compressive strength vary in between 6.8 MPa and 14.5 MPa. The results for C-Volume 28 day compressive strength vary in between 18.6 MPa and 27.6 MPa and the results of C-Flow mortar for 28 day compressive strength vary in between 21.7 MPa and 26.3 Mpa. Difference between upper and lower compressive strength results of C-Mass method is 7.7 MPa, C-Volume method is 9.0 MPa and C-Flow method is 4.6 MPa.

Even though the difference between maximum and minimum values are higher for C-Mass test method, strength obtained for this method is also low. Therefore, a better way of intergreting the data is to obtain a dimensional parameter by dividing the variation to the mean value as presented in Figure 8.

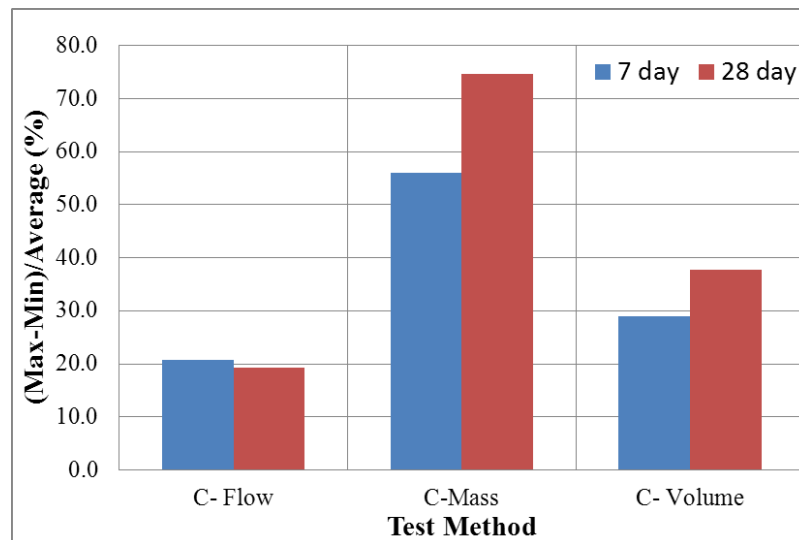


Figure 8 Max-Min Compressive Strength Test Results of FA 20

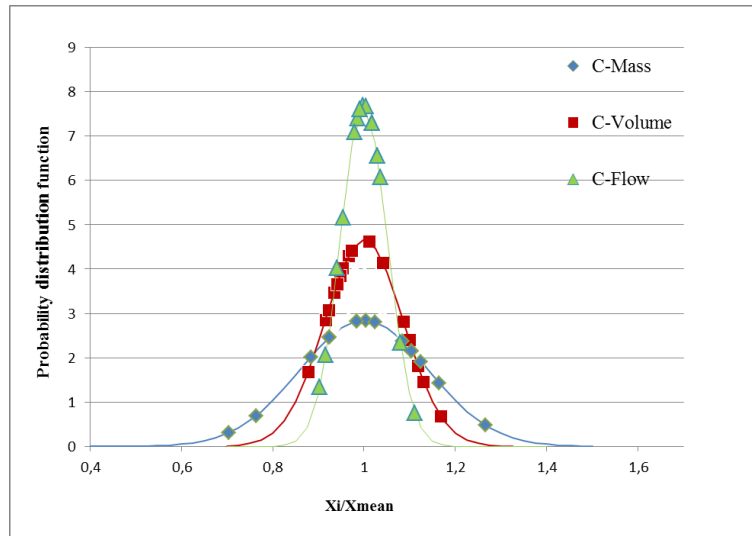
As seen from Figure 8 highest variation in the compressive strength test results are obtained for C-Mass method. On the other hand, lowest variation in the compressive strength test results are obtained for C-Flow method.

Another way to look at these variabilities is to statistically analyze the data coming from the experimental study. For this purpose, the compressive strength test results are assumed to be normally distributed. Then, Probability distribution function(pdf) of the data were obtained and the variation in the results are visually plotted. This is plotted in Figure 9.

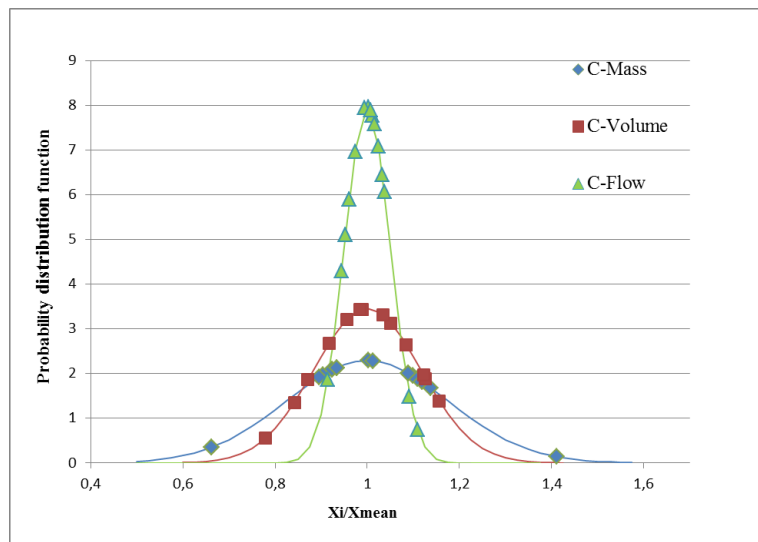
In Figure 9 where, X_i is Individual compressive strength test results (MPa) and X_{mean} is average compressive strength test result for each method (MPa).

Figure 9 (a) shows the probability distribution of 7-day compressive strength results. As observed from Figure 9 (a) the least deviation in the compressive strength results were observed for specimens prepared by C-Flow method. Then C-Volume method has less deviation. However, for the C-Mass method which is prepared by constant water/cementitious, it was observed the highest deviation in the compressive strength results.

Similar observations were determined for the 28-day compressive strength results, as seen from Figure 9 (b).



a) 7-day Compressive Strength



b) 28-day Compressive Strength

Figure 9 Probability Distribution Function (pdf) of FA 20-Blended Cements

As shown in Figure 10, it is generally accepted that for a concrete mix when water/cement ratio decreases, the strength of concrete increases. However, when water/cement ratio decreases from a specific value, concrete cannot be fully compacted because of dry consistency of concrete. Thus at very low water/cement ratios, concrete strength may decrease as shown in Figure 10 [5].

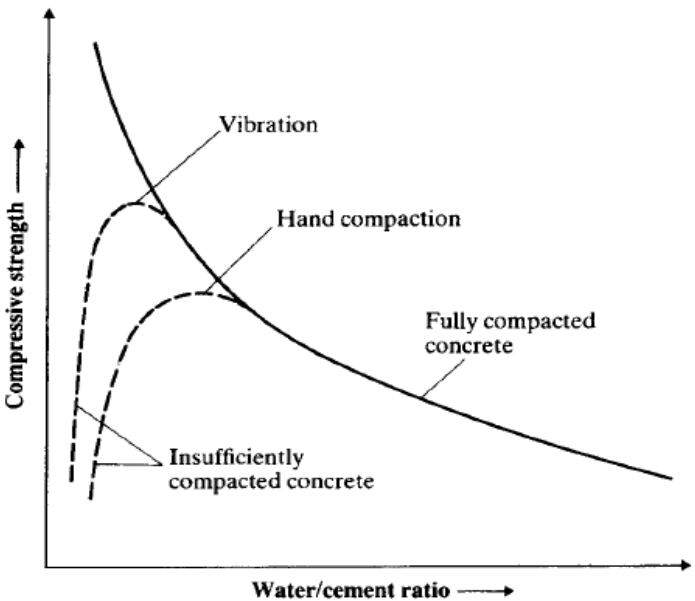


Figure 10 Relation Between Strength and Water/Cement Ratio [5].

In this study as can be seen in Figure 11 as the w/c ratio increase compressive strength, contrary to classic w/c theory, increases. Since fly ashes used in study are not spherical particle they need more water to have a good consistency for full compaction. In C-Mass method mortars were prepared by constant w/c ratio, which is 0.5 and the C-mass method has the lowest compressive strength value, on the other hand in C-flow method mortars were prepared according to constant flow, in spite of an increase in the water content compressive strength increases because of having good workability.

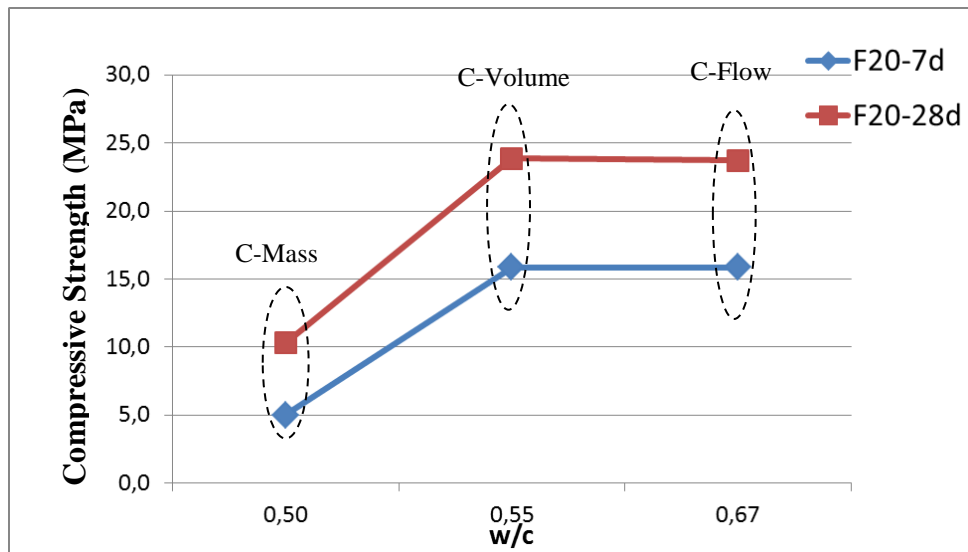


Figure 11 Compressive Strength Test Results of FA 20-Blended Cement

Figure 12 shows the three different pictures of FA 20 mortars. As seen from those figures, the mortar prepared by C-Flow method has a higher consistency than the other two.



a) C-Mass Mortar



b) C-Volume Mortar



c) C-Flow Mortar

Figure 12 Consistency of FA 20 Blended Cement Mortars

In probability theory and statistics, the coefficient of variation (CoV) is a normalized measure of dispersion of a probability distribution or frequency distribution.

As seen in Figure 13, C-Flow method has the lowest coefficient of variation (CoV), C-Volume method has smaller CoV value than C-Mass method. Finally C-Mass method has the highest CoV.

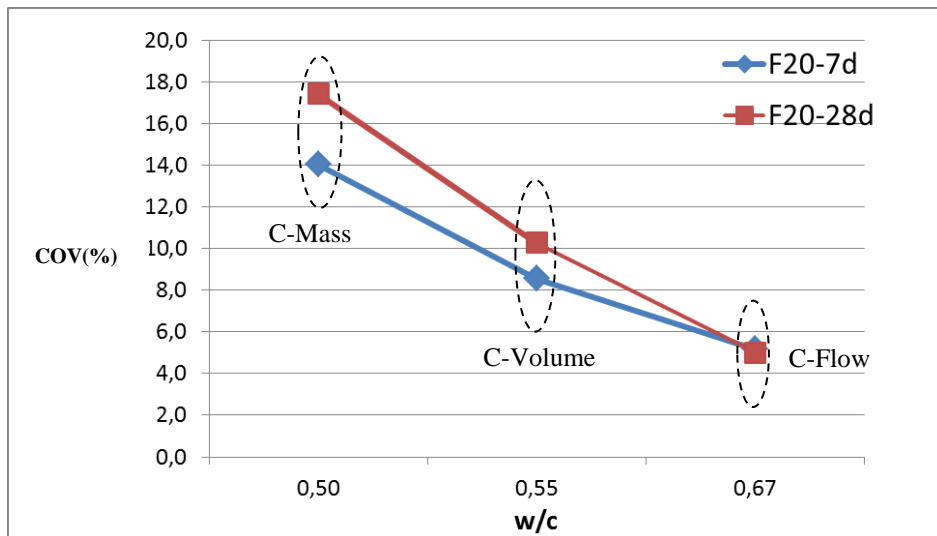


Figure 13 Coefficient of Variation (CoV) of Compressive Strength Test Results for FA 20-Blended Cement

4.2.2 35 % Fly ash-Blended Cement (FA35)

The results of 7 and 28 day compressive strength test results of FA 35 mortars were given in Table 10

According to Table 10 the results of C-Mass mortar for 7 day compressive strength vary in between 1.5 MPa and 2.9 MPa. The results for C-Volume 7 day compressive strength vary in between 3.9 MPa and 6.4 MPa and the results of C-Flow mortar for 7 day compressive strength vary in between 7.3 MPa and 9.1 MPa. Difference between upper and lower compressive strength results of C-Mass method is 0.6 MPa, C-Volume method is 2.5 MPa and C-Flow method is 1.8 MPa.

Table 10 Compressive Strength Test Results of FA 35

Compressive Strength (MPa)	C-Mass			C-Volume			C-Flow		
	Mold-1	Mold-2	Mold-3	Mold-1	Mold-2	Mold-3	Mold-1	Mold-2	Mold-3
7-Day	1.7	1.7	2.5	5.7	4.8	4.4	8.2	8.0	7.6
	1.5	1.9	2.7	6.4	4.4	4.8	8.4	7.7	7.5
	2.9	2.7	2.4	5.4	4.3	4.0	8.5	7.3	8.2
	2.3	2.4	2.5	5.0	5.4	4.8	9.1	7.7	7.8
	2.2	2.2	2.3	6.1	3.9	4.7	8.7	8.0	8.0
	2.2	1.6	2.2	4.7	4.3	4.6	8.4	8.0	7.8
Mean	2.1	2.1	2.4	5.6	4.5	4.6	8.6	7.8	7.8
CV (%)	23.1	20.5	7.2	11.7	11.5	6.8	3.7	3.6	3.3
28-Day	2.5	2.7	2.8	8.2	8.3	7.1	13.3	13.5	15.7
	3.2	2.6	3.2	8.8	8.5	11.3	12.7	13.1	14.4
	2.8	2.7	2.4	9.4	8.7	9.8	13.1	15.0	14.4
	2.8	3.2	2.7	8.1	9.0	8.2	12.6	13.3	14.1
	3.2	2.5	3.0	13.2	11.4	9.5	12.8	14.7	15.4
	2.7	2.5	2.8	9.1	9.5	11.7	13.5	12.8	15.6
Mean	2.9	2.7	2.8	9.5	9.2	9.6	13.0	13.7	14.9
CV (%)	9.8	9.7	9.6	20.0	12.4	18.4	2.8	6.6	4.7

According to Table 10 the results of C-Mass mortar for 28 day compressive strength vary in between 2.4 MPa and 3.2 MPa. The results for C-Volume 28 day compressive strength vary in between 7.1 MPa and 13.2 MPa and the results of C-Flow mortar for 28 day compressive strength vary in between 12.6 MPa and 15.7 MPa. Difference between upper and lower compressive strength results of C-Mass method is 0.8 MPa, C-Volume method is 6.1 MPa and C-Flow method is 3.1 MPa.

As seen in Figure 14, although compressive strength test results are below 3 Mpa for C-Mass, C-Volume method has the highest variation test results according to 28-day compressive strength test results in comparison with C-Mass method. However, for 7-day and compressive strength test results C-Mass method has the highest variation in the compressive strength test results. C-Flow method has the lowest variation in the compressive strength test results.

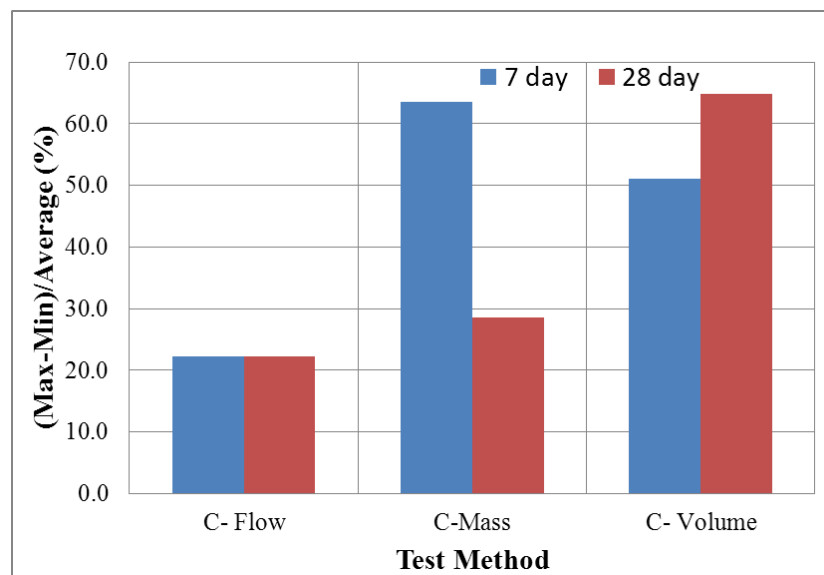
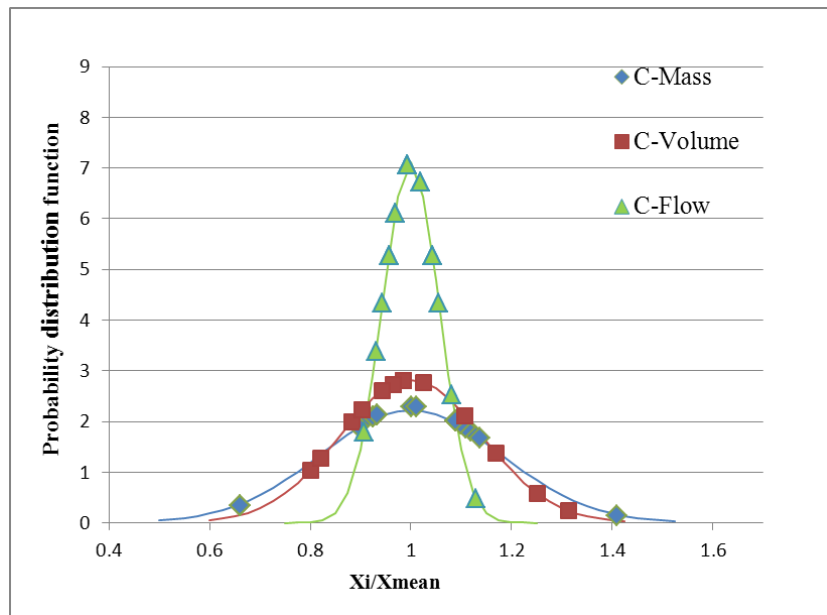


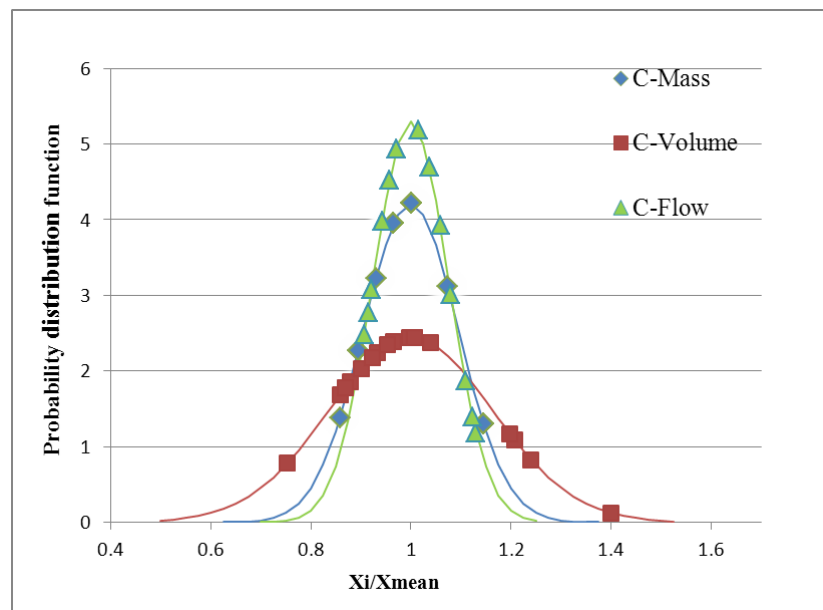
Figure 14 Max-Min Compressive Strength Test Results of FA 35-Blended Cements

Figure 15 (a) shows the probability distribution of 7-day compressive strength results. As can be seen from Figure 15 (a) the least deviation in the compressive strength results were observed for specimens prepared by C-Flow method. Then C-Volume method has less deviation. C-Mass method has the highest deviation in the compressive strength results.

Figure 15 (b) shows the probability distribution of 28-day compressive strength results. Figure 15 (b) shows that C-Flow method has the least deviation in the compressive strength results. On the other hand, C-Volume method has the highest deviance in the compressive strength results.



a) 7-day Compressive Strength



b) 28-day Compressive Strength

Figure 15 Probability Distribution Function (pdf) of FA 35-Blended Cements

Figure 16 shows that; As w/c ratio the increase compressive strength increases. For S35 cement C-Flow mortars have the biggest compressive strength test results, then C-Volume mortars have higher compressive strength test results than C-Mass method and C-mass method has the lowest compressive strength test results because of does not supply enough water to have good workability. Figure 17 shows the three different pictures of FA 35 mortars.

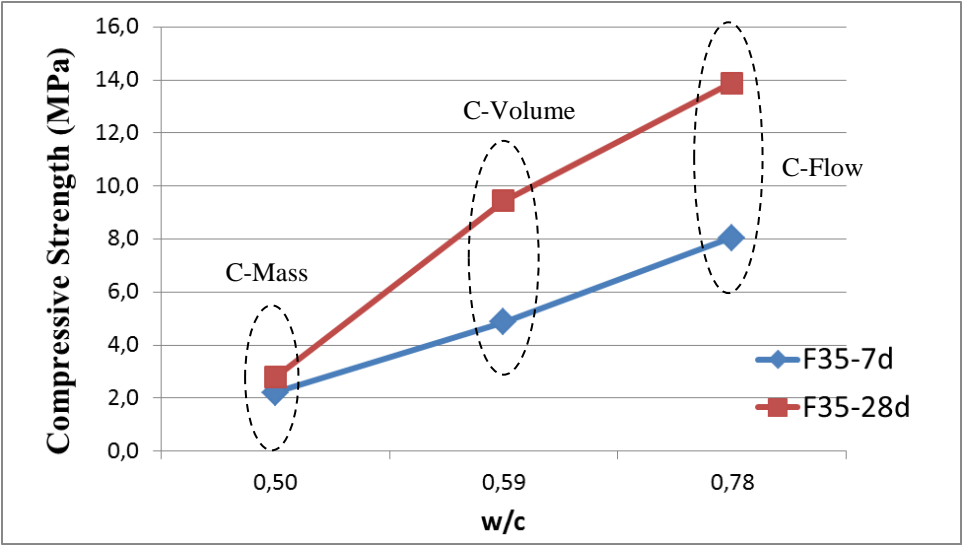


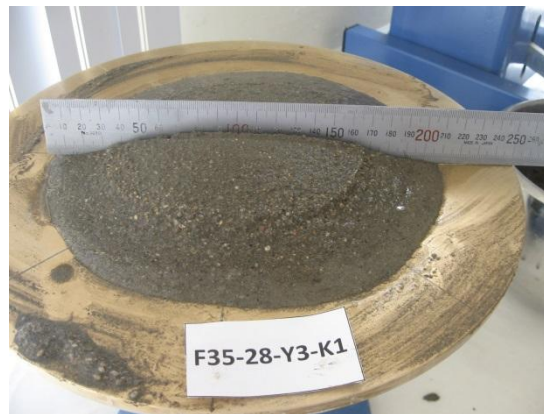
Figure 16 Compressive Strength Test Results of FA 35-Blended Cement



a) C-Mass Mortar



b) C-Volume Mortar



c) C-Flow Mortar

Figure 17 The Consistency of FA 35 Blended Cement Mortars

According to the Figure 18, for 7-days compressive strength test results C-Flow method has the lowest CoV, C-Volume method has smaller CoV value than C-Mass method and C-Mass method has the biggest CoV. For 28-days compressive strength test results, C-Flow method has the lowest CoV then C-Mass method has smaller CoV value and C-Volume method has the biggest CoV.

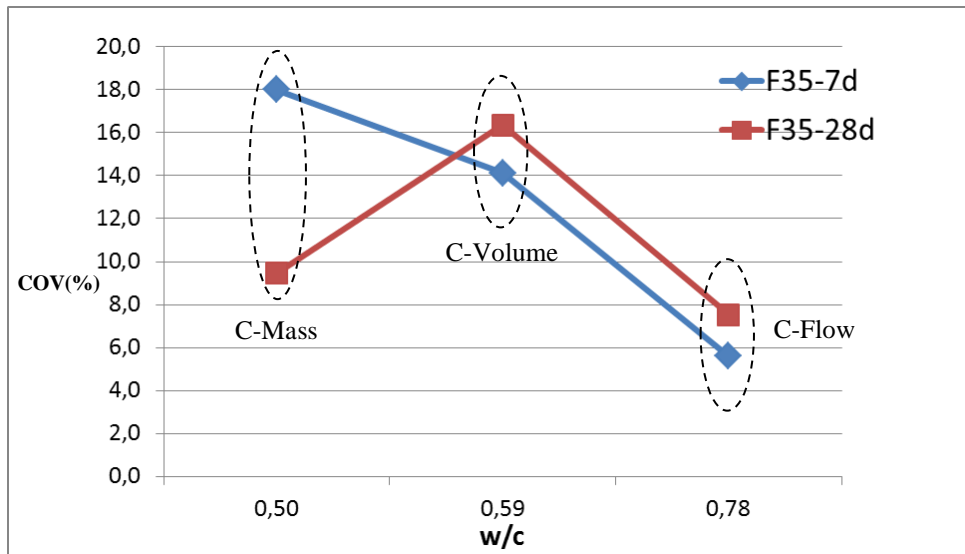


Figure 18 Coefficient of variation (CoV) of Compressive Strength Test Results for FA 35-Blended Cement

4.2.3 55 % Fly ash-Blended Cement (FA55)

The results of 7 and 28 day compressive strength test results of FA 55 mortars were given in Table 11.

According to Table 11 the results of C-Mass mortar for 7 day compressive strength vary in between 0.4 MPa and 0.8 MPa. The results for C-Volume 7 day compressive strength vary in between 1.1 MPa and 2.4 MPa and the results of C-Flow mortar for 7 day compressive strength vary in between 2.8 MPa and 3.9 MPa. Difference between upper and lower compressive strength results of C-Mass method is 0.4 MPa, C-Volume method is 1.5 MPa and C-Flow method is 1.1 MPa.

Table 11 Compressive Strength Test Results of FA 55

Compressive Strength (MPa)	C-Mass			C-Volume			C-Flow		
	Mold-1	Mold-2	Mold-3	Mold-1	Mold-2	Mold-3	Mold-1	Mold-2	Mold-3
7-Day	0.6	0.6	0.7	1.5	1.8	1.1	3.7	3.0	3.6
	0.5	0.5	0.8	1.5	1.8	1.4	3.9	2.8	3.1
	0.6	0.4	0.7	1.4	2.4	1.4	3.8	2.9	3.8
	0.7	0.5	0.5	1.3	2.2	1.2	3.9	2.8	3.7
	0.4	0.7	0.5	2.0	2.2	1.3	3.4	2.9	3.6
	0.6	0.7	0.4	1.6	2.4	1.7	3.6	3.0	3.6
Mean	0.6	0.6	0.6	1.5	2.1	1.4	3.7	2.9	3.6
CV (%)	18.2	21.4	25.8	7.8	12.8	15.4	5.2	3.1	6.8
28-Day	1.3	0.7	0.6	2.4	3.8	2.1	5.5	5.8	7.1
	1.1	0.9	0.6	2.1	3.8	2.5	4.7	5.3	7.4
	1.2	0.6	0.6	2.4	2.5	2.4	5.7	5.9	6.8
	1.1	0.5	0.6	2.2	2.5	2.5	5.6	5.9	7.2
	1.0	0.7	0.4	2.7	3.2	2.8	5.6	5.0	7.2
	1.2	0.8	0.6	3.0	3.0	2.5	5.0	5.3	6.8
Mean	1.2	0.7	0.6	2.5	3.1	2.5	5.4	5.5	7.1
CV (%)	9.1	20.2	14.4	13.5	18.7	9.1	7.5	6.9	3.4

According to Table 11 the results of C-Mass mortar for 28 day compressive strength vary in between 0.4 MPa and 1.3 MPa. The results for C-Volume 28 day compressive strength vary in between 2.1 MPa and 3.8 MPa and the results of C-Flow mortar for 28 day compressive strength vary in between 4.7 MPa and 7.4 MPa. Difference between upper and lower compressive strength results of C-Mass method is 0.7 MPa, C-Volume method is 1.7 MPa and C-Flow method is 2.7 MPa.

As seen in Figure 19, C-Mass method has the highest variation in the 28-day compressive strength test results. On the other hand, C-Volume method has the highest variation in the 7-day compressive strength test results, though compressive strength test results are below 1 Mpa for C-Mass method. As expected, C-Flow method has the lowest variation in the compressive strength test results.

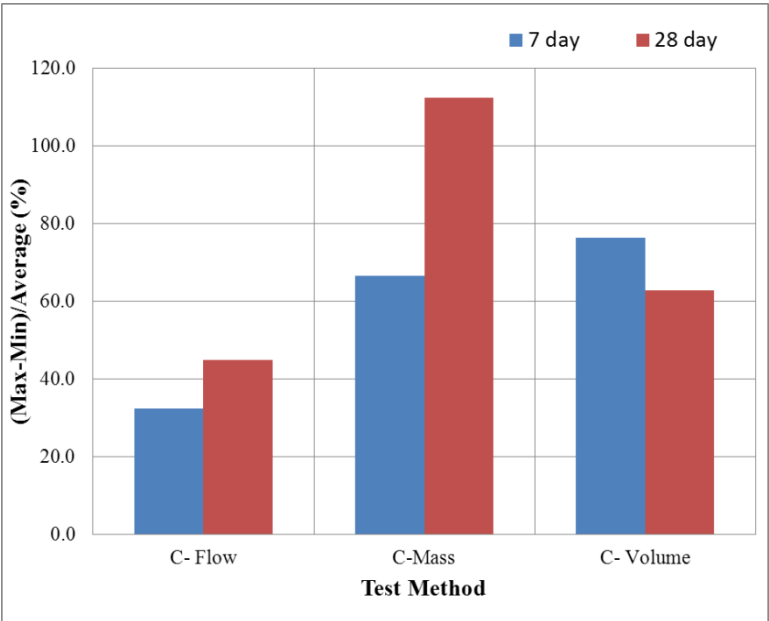
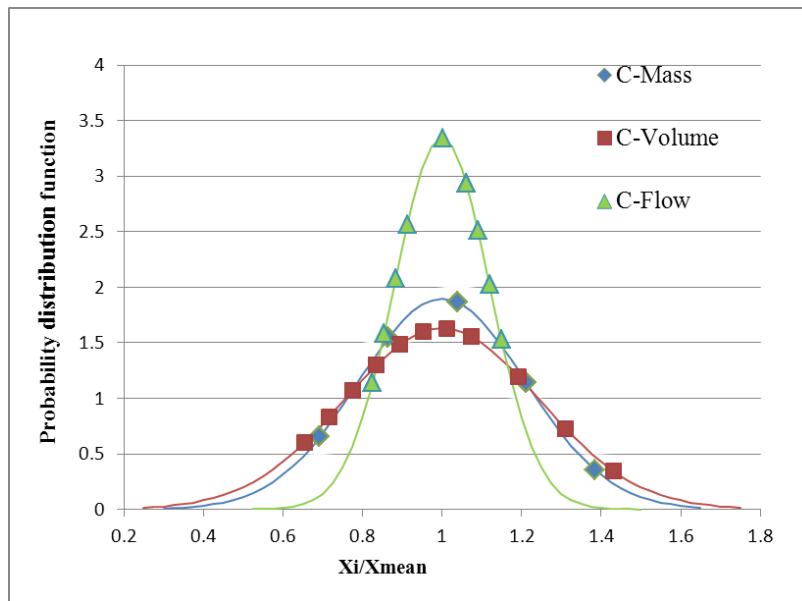


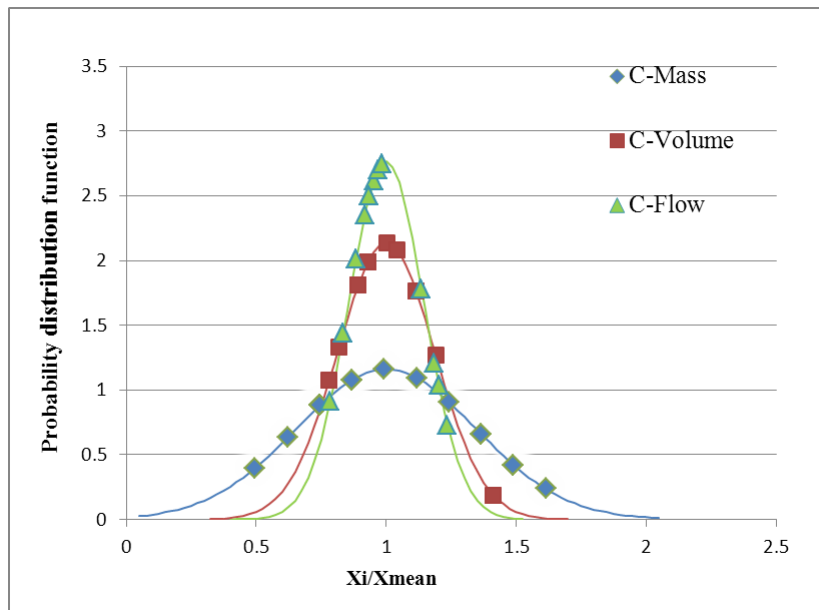
Figure 19 Max-Min Compressive Strength Test Results of FA 55-Blended Cements

Figure 20 (a) shows the probability distribution of 7-day compressive strength results. As can be seen from Figure 20 (a) C-Flow method has the least deviation in the compressive strength results. Then C-Mass method has less deviation. C-Volume method has the highest deviation in the compressive strength results.

Figure 20 (b) shows the probability distribution of 28-day compressive strength results. Figure 20 (b) shows that C-Flow method has the least deviation in the compressive strength results. C-Mass method has the highest deviation in the compressive strength results.



a) 7-day Compressive Strength



b) 28-day Compressive Strength

Figure 20 Probability Distribution Function (pdf) of FA 55-Blended Cements

As given in Figure 21, Compressive Strength Test Results of C-Mass method are below 1 MPa. As can be seen in Figure 22 C-Mass mortars are not normal mortars they had severely compaction problem during experiment especially at 7-day test results because of the constant w/c ratio. Though, in C-volume method it was observed same problem 7-day test results generally, for all methods as the w/c ratio increase compressive strength increases. C-Flow mortars have the highest compressive strength test results, then C-Volume mortars have higher compressive strength test results than C-Mass method and C-mass method has the lowest compressive strength. Figure 22 shows the three different pictures of FA 55 mortars.

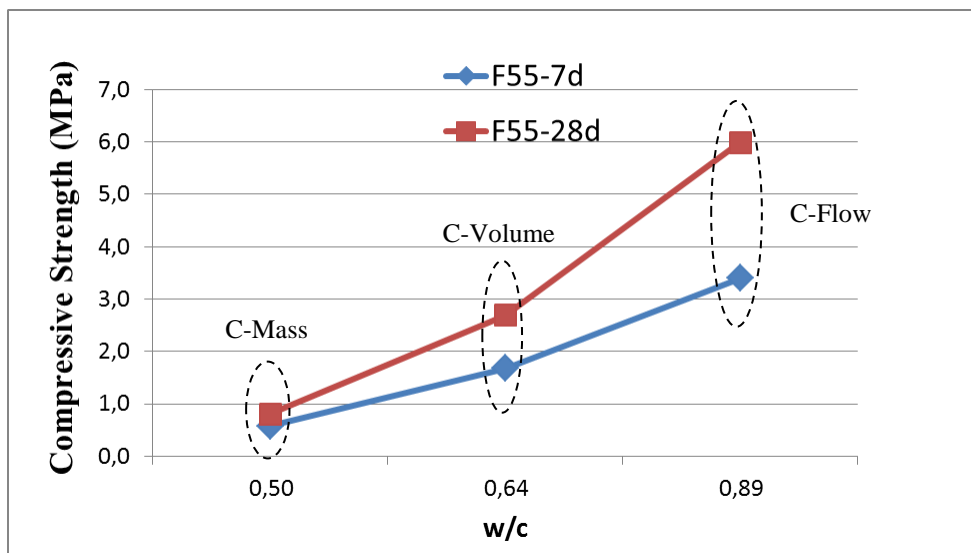


Figure 21 Compressive Strength Test Results of FA 55-Blended Cement



a) C-Mass Mortar



b) C-Volume Mortar



c) C-Flow Mortar

Figure 22 Consistency of FA 55 Blended Cement Mortars

According to Figure 23, for 7-day compressive strength test results C-Flow method has the lowest CoV, C-Mas method has smaller CoV value than C-Volume method and. For 28-days compressive strength test results C-Flow method has the lowest CoV, then C-Volume method has smaller CoV value and finally C-Mass method has the biggest CoV.

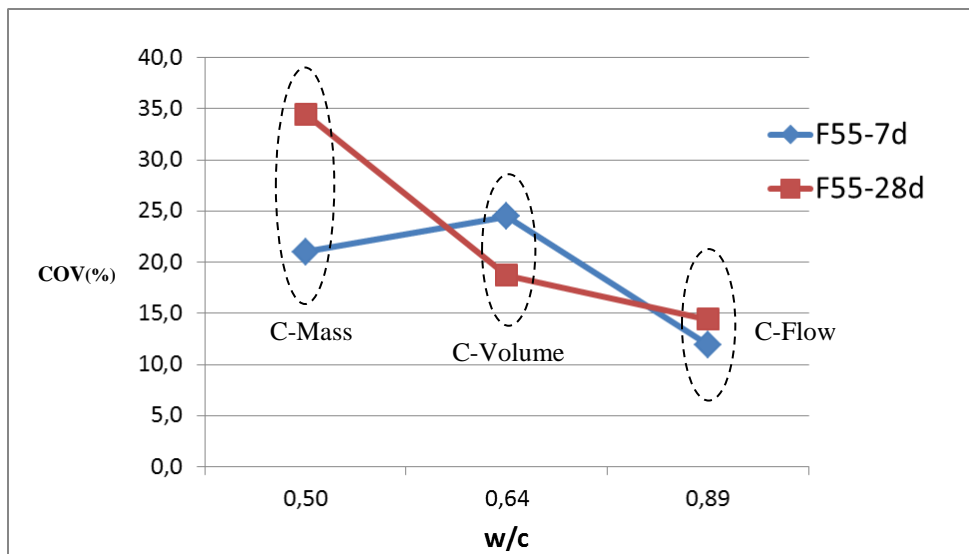


Figure 23 Coefficient of Variance (CoV) of Compressive Strength Test Results for FA 55-Blended Cement

4.3 Comparison of the Compressive Strength Test Results for FA20, FA35 and FA 55 Blended-Cements

As can be seen from Figure 24, for 7-day and 28-day compressive strength test results, generally as the fly ash content increases compressive strength decreases and as the w/c ratio increases compressive strength increases. Mortars need more water to have a good workability, since particle shape of fly ashes used in study are angular, rugged shapes which was shown in Figure 6.

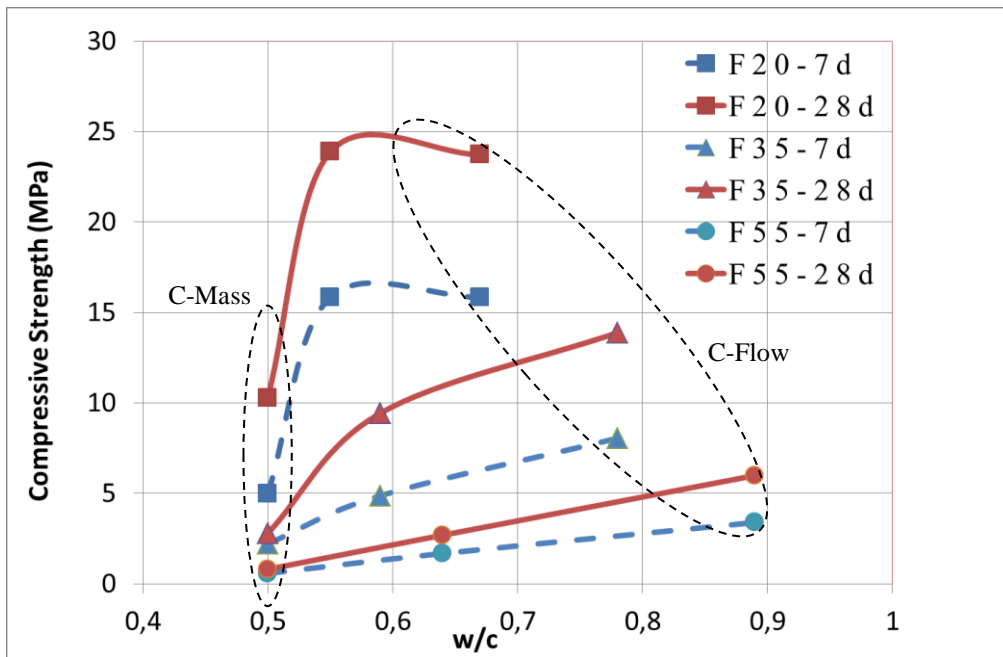


Figure 24 Compressive Strength Test Results of Fly Ash-Blended Cements

These type of fly ash particles increase the demand of water of mixture. Specifically, within same cement type the smallest compressive strength value was observed for C-Mass method prepared by constant w/c ratio because of not having a good consistency. And as expected, the highest compressive strength value was observed for C-Flow method prepared by constant flow, because of having good workability despite of increase in the water content.

4.4 Comparison of Coefficient of Variance for Different Test Methods

According to Figure 25, for FA20 C-Flow method has the lowest variability, C-Mass method has the biggest variability. Then for FA35 C-Flow method has the lowest variability, C-Mass method has the biggest variability. Finally for FA55 C-Flow method has the lowest variability and as expected, C-Mass method has the biggest variability

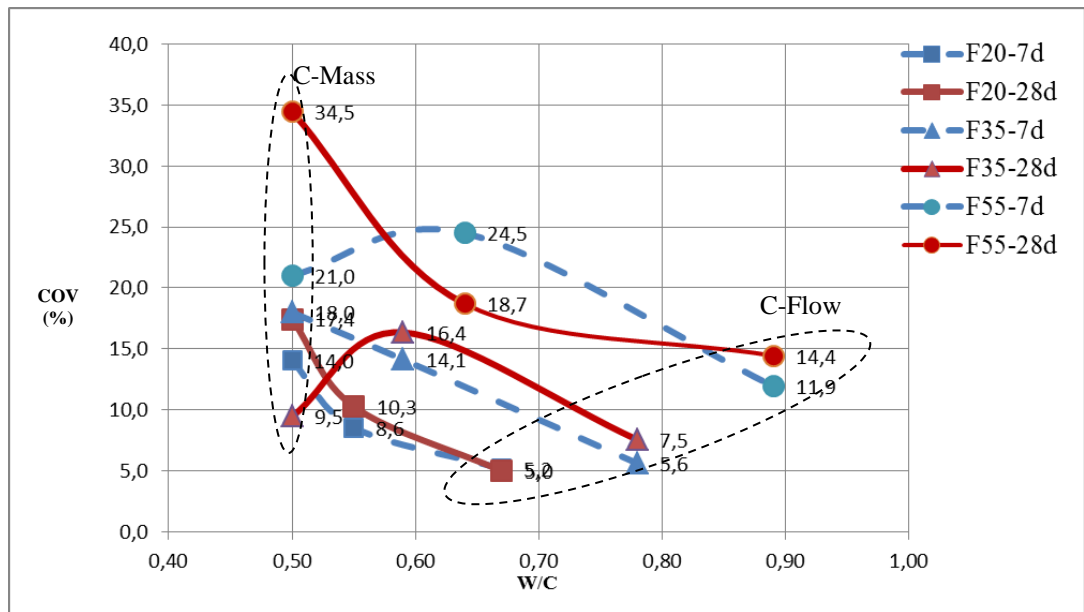


Figure 25 Coefficient of variance (CoV) of Compressive Strength Test Results for Fly ash-Blended Cements

CHAPTER 5

SUMMARY AND CONCLUSIONS

5.1 General

For the determination of compressive strength of blended cements, three different methods which are acquired by the constant water/cementitious by mass (EN 196-1), constant flow (ASTM) and constant water/cementitious by volume methods were used. For this purpose, fly ash (FA) blended cements were chosen. Then, fly ash-blended cements were tested at TSE Construction Materials Laboratory and 7-day and 28-day compressive strength of mortars was obtained. The results obtained from mortar tests were examined. The outcomes of the analyses are as follows:

- 1) According to the experimental test results the fly ash used in this study increased the water demand of the blended cements to bring the mortars to the same consistency which is needed.
- 2) For all cement types highest variability was observed for C-Mass method. This was attributed to the surface morphology of the fly ash.
- 3) According to results of compressive strength, as the w/c ratio increases compressive strength increases. This was attributed to the fact that cement mortars were not fully consolidated. Since fly ashes used in this study are not spherical particle which have angular, rugged shapes, they need more water to have a good workability.

- 4) According to results of three different methods, the constant water/cementitious by mass (TS EN 196-1) method is not an appropriate test method for fly ash blended cements based on the higher variabilities encountered in the tests, since constant water to cement ratio is not appropriate to have a homogenous mix.

5.2 Recommendations for Further Studies

Considering the results obtained from this study, the following recommendations could be made for researchers for future studies.

- 1) The fly ash used in this study has a narrow range in the blended cements. As a result of this study, the studies should be continued with using different blended cement types.
- 2) In this study, only a limited number of specimens were tested. Therefore, it is suggested to increase the number of specimens.
- 3) Better characterization of the fly ash alone would have been helpful for this kind of experimental study.

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