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Investigation of the usage of fly ash containing a high amount of sulfate as a clinker replacement material

Yüksek oranda sülfat içeren uçucu küllerin klinker ikame malzemesi olarak kullanılabilirliğinin incelenmesi

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Abstract

Tons of fly ash are released every year due to the coal burned in thermal power plants that meet 80% of Türkiye's electrical energy needs. Especially considering the low-quality lignite reserves in Türkiye, the recycling rate of the high sulfate fly ash is relatively low. In concrete production, the use of ash containing high SO_3 is not allowed due to the limitation of SO_3 ratios of fly ash by regulations. Herein, the usability of fly ash, which has a high sulfate content due to the low-quality lignite reserves in Türkiye, and is therefore wholly waste, as a clinker replacement material was investigated. Fly ash, which has been used as a clinker replacement material, was obtained from Afşin Elbistan thermal power plant. Binders were obtained by grinding together the high sulfate fly ash, clinker and gypsum at different rates. The setting times of the mixtures in the fresh cement paste were determined, and the compressive strengths of the hardened samples were examined. As a result of the study, it was concluded that although the high sulfate fly ash extended the setting time by approximately 29% compared to the reference specimen, it increased the 28-day compressive strength of the hardened specimens up to 14%

Keywords: Waste, High sulphate fly ash, Clinker, Compressive strength, Cement production.

Öz

Türkiye'nin elektrik enerjisi ihtiyacının %80'ini karşılayan termik santrallerde yakılan kömürlerden dolayı her yıl tonlarca uçucu kül açığa çıkmaktadır. Özellikle ülkemizdeki düşük kaliteli linyit rezervleri düşünüldüğünde, açığa çıkan yüksek sülfat içeriğine sahip uçucu küllerin geri dönüşüm oranı oldukça düşüktür. İnşaat sektöründe ise yönetmelikler tarafından uçucu küllerin SO3 oranlarının sınırlandırılması sebebiyle yüksek SO3 içeren küllerin kullanımlarına izin verilmemektedir. Bu çalışmada, ülkemizdeki düşük kaliteli linyit rezervleri sebebiyle yüksek sülfat içeriğine sahip olan, dolayısıyla tamamıyla atık durumda bulunan uçucu küllerin, klinker ikame malzemesi olarak kullanılabilirliği araştırılmıştır. Klinker ikame malzemesi olarak kullanılacak olan ve yüksek oranda sülfat içeren uçucu kül Afşin Elbistan termik santralinden temin edilmiştir. Yüksek oranda sülfat içeren uçucu kül, klinker ve alçı ile farklı oranlarda birlikte öğütülerek karışımlar elde edilmiştir. Karışımların taze halde priz süreleri tayin edilmiş, sertleşmiş numunelerin ise basınç dayanımları incelenmiştir. Çalışma sonucunda, yüksek sülfat içeren uçucu külün priz bitiş sürelerini referans numuneye göre yaklaşık %29 uzatmasına rağmen, 28 günlük basınç dayanımlarını %14'e kadar artırdıkları tespit edilmiştir.

Anahtar kelimeler: Atık, Yüksek sülfatli uçucu Küller, Klinker, Basınç dayanımı, Çimento üretimi.

1 Introduction

Concrete is the most used building material worldwide. Portland Cement (PC) has continued to be used as the primary binder material of concrete for nearly 100 years. According to the United States Geological Survey (USGS), approximately 4400 million tons of cement were produced worldwide in 2021 [1]. Türkiye is the fourth largest cement producer globally, with 76 Mton productions in 2021 [1]. This high amount of cement production causes much more CO_2 to be released during clinker production and the production of the energy needed to produce clinker. Approximately one million tons of CO_2 are released during one million tons of clinker production (except for energy production) [2]. Researchers aimed to reduce energy costs and the amount of CO2 released by replacing cement with different pozzolanic and mineral additives in the past decades. As a result of these studies, the most preferred replacing materials are wastes produced in other industries, such as silica fume (SF), fly ash (FA), and granulated blast furnace slag (GBFC). While clinker production is reducing, the powders that are harmful to the environment will be recycled by using these wastes as mineral additives in the cement industry. In the meantime, these wastes improve the durability properties of concrete. The FA is the most produced of these wastes. Approximately 780 Mton FA is produced worldwide every year [3]. It is estimated that 10-30% of these FA can be used in the cement and concrete industry [4]. In addition, according to reports by some market research companies, the FA market is expected to reach a market size of US$8.204 billion, with growth at a CAGR of 5.93% from 2020-2027 [5].

The sulfate content of the FA is one of the most critical factors which is affecting its usability in the cement industry. European standards limit the sulfate content of FA that could be used in concrete to 3% max [6]. On the other hand, the most crucial factor affecting the sulfate content of FA is coal quality. Especially high-quality coals (bituminous and sub-bituminous) produce a low amount of SO_3 ashes, while the sulfate content of ashes from low-quality lignite coals can reach up to 15% [7],[8]. In addition, 201 billion tons of 891 billion tons reserves are in the lignite category worldwide [9]. Also, 7813 Mton coals were

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extracted in 2018 worldwide. 5935 Mton of these coals were used by thermal power plants for the same year. 803 Mton were extracted in the lignite category, and 893 million tons were used in 2018 [10]. These high amounts of thermal power plant wastes have been causing devastating environmental pollution.

SO3 amount in FA has been determined as a maximum of 5% in ASTM C618 and a maximum of 3% in European standards, considering factors such as delayed setting, affecting strength development, and causing durability problems in concrete [11],[12]. Considering the factors such as the cost of removal/storage of FA and environmental pollution occurring in landfills, there is a need to investigate the use of FA containing a high amount of SO3 in concrete. Kaladharan and Rajabipour (2022) [11] suggested that using chemical additives could reduce setting time and pore fluid pH problems caused by FA containing high SO3 content. Antiohos et al (2007) [13], on the other hand, stated that when the FA containing high sulfate is replaced by 20% with clinker, the setting time almost does not change. Coal consumption is constantly increasing, especially after the Covid19 pandemic, due to global and regional risks and various problems in the supply of natural gas and oil resources. One of the most striking examples of this is the fact that the use of coal in electricity production has increased by approximately 7% and reached 5350 million tons [14]. It is understood that considering the problems that may be experienced in the supply of fuel resources, studies are needed on the usability of FA containing high SO3 in countries with low-quality coal resources (such as Türkiye).

The primary purpose of this study is to investigate the use of FA, which contains high amounts of sulfate (> 3%), which is formed by burning low-quality lignites in thermal power plants without any pre-treatment [7]. Türkiye is the world's second lignite producer after Germany. The high sulfate FAs (HSFA) used in the study were taken from the Afşin-Elbistan thermal power plant located in the basin where Türkiye's largest lignite reserves (47% of total coal reserves) [15]. In addition, as it is known, gypsum has been added as a source of sulfate to the clinker during the grinding phase to prevent flash setting in cement production. Even if gypsum extraction from the mines is not subjected to heat treatment such as cement, many additional processes are necessary. The HSFA was substituted instead of gypsum and cement in varying amounts in this research. Determination of Vicat standard consistency and setting times tests were carried out on the prepared mixes, and the setting times of the mixes were compared with each other. In addition, compressive strength tests were performed on produced mixtures, and the strength changes were measured.

2 Material and method

2.1 Material

2.1.1 Fly ashes containing high sulfate (HSFA)

The mineral properties of the FA may differ depending on the quality of coal, the degree of combustion of coal, and the humidity. On the other hand, mineral differences of FA cause their color to be different. Unburned carbon particles or a high amount of iron oxide give a darkish color of FA [16]. The color of the FA that comes out with the burning of lignite coal is darker than the FA that is produced by the burning of bituminous coal. The FA also differs in grain sizes and shapes. The grain size of the FA released from bituminous coal is smaller than FA from lignite coal. Furthermore, the fineness of FA is close to the fineness of cement particles. The FA particles may vary between 1-150 μm in diameter and are spherical. However, advanced flue-filter systems in thermal power plants can catch smaller particles [17]-[19].

Türkiye's largest lignite coal reserves are in the Afşin Elbistan/Kahramanmaraş. Lignite coal in this basin has low quality and produces high ash. Firstly, the Afşin-Elbistan A-Type thermal power plant was established in this basin in 1984. Afşin-Elbistan B type thermal power plant was put into use in 2004 [20]. The FA moves upwards together with hot air and is kept in the flue filters during the combustion process of coal. The FA produced at Afşin-Elbistan Power Plant are stored in waste sites and cannot use in the industry due to their high sulfate content. The FA used in this study was obtained from Afşin-Elbistan A-Type Thermal Power Plant and was sieved through a 45-micron sieve before being used. Lastly, XRF test was performed to see the chemical structure of the FA (Table 1).

2.1.2 Clinker

In this study, CEM1 42,5/R-SR5 (Type V) cement clinker produced by Konya Cement was used. The oxide composition of the clinker is given in Table 2.

2.1.3 Gypsum

For centuries, gypsum has been used as a binder in buildings for aesthetic or functional purposes. When the cement is mixed with water, the gypsum covers the clinker particles' surface, thus controlling the reaction temperature and preventing rapid setting. Within the scope of this study, dihydrate obtained from the Konya cement plant was used as gypsum.

2.2 Method

In this study, the mortar specimens with different ratios of materials were produced in order to examine the physical and chemical effects of HSFA, which contains high amounts of sulfate, in cement. In addition, the reference samples with and without gypsum were produced for comparison. The binder ratios of the produced specimens are given in Table 3.

Since cement and gypsum were used together in the N2, N4, N6, N8, N9, N10, and N11 specimens given in Table 3, clinker and gypsum were ground together in the ball mill in these specimens. The grinding process took 80 minutes for each sample. The N1 sample was only produced with clinker. Other samples containing HSFA were prepared by mixing the materials dryly at the rates given in Table 3 and then grinding them together in a ball mill for 80 minutes. The remaining amounts of the ground samples on 45 and 32-micron sieves are given in Table 5. Lastly, dry samples were taken from each mixture, and the XRF results were examined in these specimens (Table 4).

The strength tests of the samples were carried out according to EN 196-1 (2016). 450g binder, 225mL water, and 1350g standard sand were used in the preparation of the specimens. After mixing, the specimens were poured into the 40*40*160 mm molds, and they were kept in a cabinet at 20±2 °C temperature and %99 relative humidity for 24 hours. The samples, which were demolded after 24 hours, were placed in the water-saturated with lime, and they were kept until the 2nd, 7th, and 28th days. The strengths of the samples were determined by taking the average of the compressive strengths of 3 samples produced from each group.

Table 1. Chemical Composition of HSFA.

Oxides	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	Cl
Contents (%)	44.82	17.23	8.15	17.28	2.12	3.59	1.12	0.46	0.08

Table 2. Oxide composition of the clinker.

Oxides	LOI*	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	Cl
Contents (%)	1.75	20.82	3.79	5.65	63.92	1.12	1.45	0.61	0.32	0.024

*LOI:Loss of ognition.

Table 3. Mixing ratios of the specimens.

Specimen Name	Clinker		Gypsum		HSFA	
	Ratio by weight (%)	Weight (g)	Ratio by weight (%)	Weight (g)	Ratio by weight (%)	Weight (g)
N1	100	3000	0	0	0	0
N2	96	2880	4	120	0	0
N3	96	2880	0	0	4	120
N4	94	2820	6	180	0	0
N5	94	2820	0	0	6	180
N6	92	2760	8	240	0	0
N7	92	2760	0	0	8	240
N8	92	2760	4	120	4	120
N9	94	2820	4	120	2	60
N10	90	2700	4	120	6	180
N11	88	2640	4	120	8	240
N12	85	2550	0	0	15	450

Table 4. XRF test results of the specimens.

Compounds (%)	N1	N2	N3	N4	N5	N6	N7	N8	N9	N10	N11	N12	HSFA
LOI	1.75	2.51	1.93	2.84	1.74	2.87	1.92	2.31	2.19	2.23	2.42	2.07	-
SiO_2	20.82	20.53	21.22	20.3	21.54	20.31	21.8	21.14	20.8	21.17	21.53	22.94	44.82
Al_2O_3	3.79	3.5	4.57	3.39	5.19	3.41	5.6	4.61	4.1	4.91	5.43	7.94	17.23
Fe_2O_3	5.65	5.57	5.86	5.54	5.97	5.51	6.04	5.65	5.7	5.81	4.86	6.4	8.15
CaO	63.92	62.1	61.92	61.73	60.3	61.15	59.52	60.49	61.35	59.4	58.1	54.37	17.28
MgO	1.12	1.12	1.2	1.13	1.21	1.25	1.26	1.23	1.18	1.28	1.31	1.32	2.12
SO_3	1.45	2.74	1.4	2.51	1.44	4.26	1.51	2.84	2.88	2.92	2.93	1.53	3.59
K_2O	0.61	0.58	0.63	0.57	0.64	0.56	0.64	0.6	0.59	0.61	0.61	0.68	1.12
Na_2O	0.32	0.32	0.33	0.32	0.34	0.33	0.34	0.34	0.32	0.32	0.35	0.35	0.46
Cl	0.024	0.026	0.026	0.025	0.026	0.022	0.023	0.022	0.024	0.023	0.021	0.018	0.08

*LOI: Loss of Ignition.

Although the main purpose of this study is to investigate the usability of HSFA as an additive in cement, the use of HSFA in cement production instead of gypsum was also examined. For this purpose, different proportions of HSFA were substituted for gypsum and compared with gypsum-containing specimens. Produced specimens and test images are presented in Figure 1.

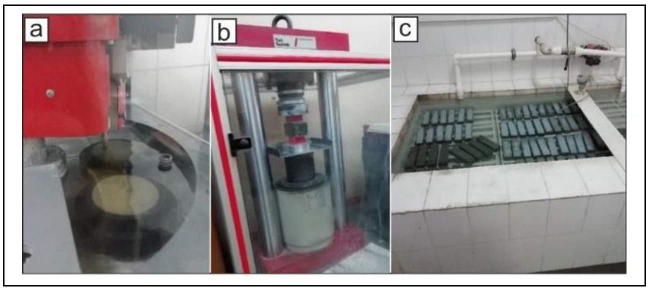

Figure 1(a): Vicat Test, (b): Compressive strength test, (c): Specimens.

3 Results

3.1 Vicat test results

The fineness of the specimens after grinding and the initial and final times of setting have been given in Table 5.

When the setting initial and final times of the specimens were examined, it was observed that the setting time of the N1 sample was faster than all the samples, as expected, because it contained only a clinker. Particularly, the setting times of the specimens containing clinker and HSFA were shorter than the samples containing clinker and gypsum. In the specimens where clinker, gypsum, and HSFA are used together, have been observed that the gypsum in the mixture was more effective on the setting time, while HSFA can affect the system in terms of setting only if it is used in systems at high ratios. The N12 specimen, in which clinker and HSFA were used at a high rate, was the highest open time.

Table 5. Fineness and Vicat test results.

Specimen Name	% Remaining of the sieve		Setting Time (min.)		Open Time (min)
	45 µ	32 µ	Initial	Final	
N1	2.1	17.6	140	210	70
N2	2.5	17.1	185	255	70
N3	2.2	16.6	180	255	75
N4	2.6	18	200	270	70
N5	2	16.1	175	240	65
N6	2.8	18.4	205	250	45
N7	1.8	15.6	180	260	80
N8	2.1	16.0	185	245	60
N9	2.4	17.5	210	270	60
N10	2.4	17.4	210	270	60
N11	2.3	16.7	195	265	70
N12	2.8	17.1	185	270	85

3.2 Compressive strength test results

Compressive strength tests of the specimens were performed at the end of 3, 7, and 28 days according to EN 196-1(2016), and the results were given in Table 6, Figure 2, and Figure 3.

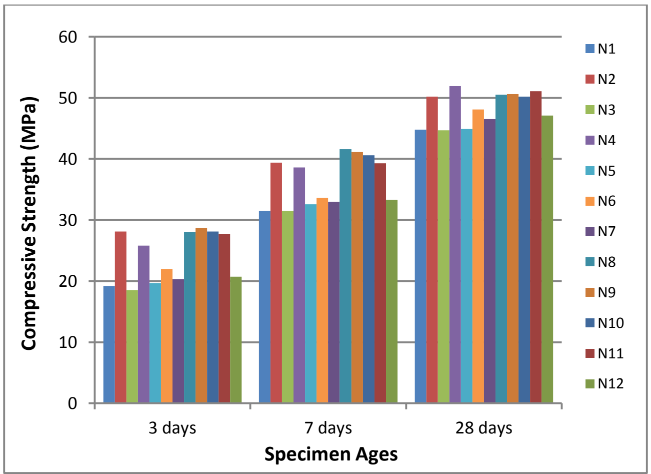

Figure 2. Compressive strength results of the specimens.

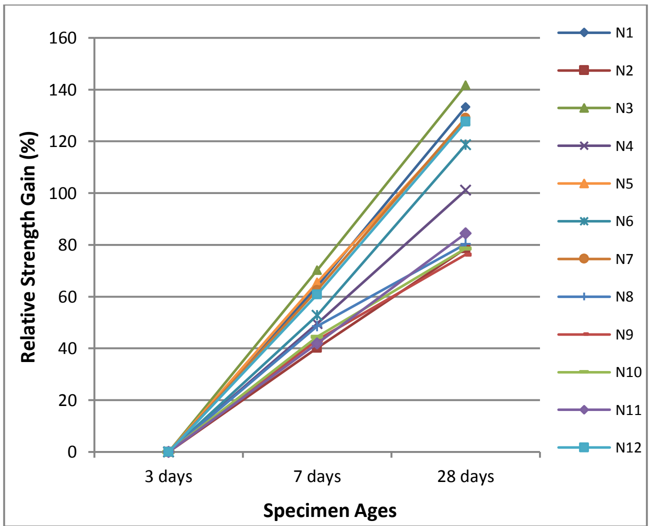

Figure 3. Time-dependent relative strength development of the specimens.

According to the results, the 3, 7, and 28-day strength results of the samples containing only HSFA increased by approximately 3% to 8% compared to the sample containing only clinker. On the other hand, when the compressive strength of the samples containing only gypsum is examined, it is 30% higher on average than the samples containing only clinker. However, although the strength in these samples was higher than the reference, it decreased as the gypsum ratios in the system increased. When the 7 and 28 days strength of the samples containing only gypsum were examined, it was seen that this trend was reversed, and the strength of the samples containing more gypsum was higher, especially at the age of 28. The most important reason for this is that, as can be seen in Table 5, higher rates of gypsum delay the setting, resulting in lower strength at early ages, and higher strength at later ages, as it supports more ettringite formation [21],[22].

The above effects are also confirmed in the samples containing both gypsum and HSFA, and the compressive strengths vary according to the dominance of the components in the system. For instance, while the early ages compressive strength of N9 sample is 4% higher than N11, the 28-day compressive strength of N11 sample is 1% higher than N9 sample. Furthermore, the compressive strength results of the N12, which contained a high amount of HSFA, were consistently higher than the N3, N5, and N7 specimens containing less HSFA. This situation is also due to the formation of secondary C-S-H structures formed, especially in later ages, due to the pozzolanic reaction of the mineral additives used in cementitious systems [23],[24].

The compressive strength development of the specimens containing HSFA was always higher than the specimens containing only gypsum. The main reason for this is that HSFA, which is composed of round and small particles, creates extra area for nucleation of hydration products in the cement matrix. [25],[26]. This extra area also contributes to the growth of the C-S-H structure in the later stages of hydration. [27],[28].

The use of fly ash as an additive in cement plays an essential role in reducing cement use and therefore reducing CO_2 emissions. When fly ash is added as an additive to a cement consisting of clinker and gypsum, the amount of clinker to be used is saved.

Table 6. Compressive strength test results.

Specimen Age	Specimen Name											
	N1	N2	N3	N4	N5	N6	N7	N8	N9	N10	N11	N12
3 days	19.2	28.1	18.5	25.8	19.7	22.0	20.3	28.0	28.7	28.1	27.7	20.7
7 days	31.5	39.4	31.5	38.6	32.6	33.6	33.0	41.6	41.1	40.6	39.3	33.3
28 days	44.8	50.2	44.7	51.9	44.9	48.1	46.5	50.5	50.6	50.2	51.1	47.1

4 Conclusion

In this study, the usability of FAs produced with low-quality lignite reserves in the cement industry was investigated. The following conclusions can be drawn from the results.

- ✓ The use of HSFAs in the clinker without gypsum significantly reduced the compressive strength, especially at early ages, compared to the specimens containing gypsum,
- ✓ The use of HSFA and gypsum prolonged the initial and final setting times of the specimens. Moreover, open times of the specimens containing only HSFA were higher than specimens containing only gypsum. In addition, similar to the specimens containing gypsum, the flash setting was not observed in any mixture in which fly ash was used alone,
- ✓ The compressive strength of the specimens containing HSFA and gypsum was high both at the early age and the 28-day specimens. It is thought that the most important reason for this is that the gypsum supports the formation of ettringite in the first days. Furthermore, the FA has increased the compressive strength of the specimen due to its filling effect,
- ✓ When the relative strength developments of the specimens are examined, it is seen that the strength developments of the specimens containing HSFA on the 7 and 28 days are higher than the other specimens. It is considered that this situation occurs as a result of the pozzolanic activation of fly ash and may become more evident in later ages.

When all these results are examined, it is thought that the use of HSFA in the cement industry can both reduce the amount of gypsum and reduce the amount of clinker even at early ages because of the filling effect of the HSFA. In addition, it is considered that the use of this utterly waste product as a cement additive can, directly and indirectly, provide environmental health.

The findings obtained in this study gave positive results that HSFA could be used as a cement replacement material. However, in future studies, it is recommended to perform various durability tests to determine the long-term effects of HSFA in concrete and perform microstructure analyses to see its effects on hydration products and ettringite formation potential.

5 Ethics committee approval and conflict of ınterest statement

Ethics committee approval is not required for the prepared manuscript and there is no conflict of interest with any person/institution.

6 Author contribution statement

In the study carried out, Murat SAYDAN and Ülkü Sultan KESKİN contributed to the creation of the idea, the preparation of the experimental program and the literature review, the examination of the results of the experiment and the evaluation of the results, and Furkan TÜRK and Hilal SAY contributed to the execution of the experimental methods in accordance with the program.

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