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Reaktif Silis İçeren Agregalarla Yapılan Beton ve Harçlarda Metakaolin Etkisinin Araştırılması

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Özet: Beton ve betonarme elemanların üretiminde uçucu kül (UK), silis dumanı (SD), yüksek fırın cürufu (YFC) ve metakaolin (MK) gibi puzzolanik endüstriyel atıkların kullanımının başarılı bir geçmişi vardır. Yüksek reaktivite metakaolin (MK), saflaştırılmış kaolinitik kilin ısıl işlem ile üretilen bir puzolandır. Bu çalışmada alkali silis içeren agregalarla yapılan beton ve harçlarda, mineral katkı olan metakaolin etkisi; puzolanik aktivite deneyi, beton basınç dayanımı, beton ve harçlarda dayanımı etkileyen alkali silika reaksiyonu deneyleri ile araştırılmıştır.

Puzolanik aktivite deneyi sonucunda 28 günlük değer olarak % 95 (0.95) oranında basınç dayanımı sağlamıştır. Hızlandırılmış harç çubuğu deneyi sonuçlarında metakaolinin belirli oranlarda kullanımıyla beton dayanımını etkileyen alkali silika reaksiyonu (ASR) oluşumu zararsız hale getirilmiştir. Metakaolin katkılı olarak hazırlanan %10,15 ve 20 oranında katkılı numunelerde alkali silika reaksiyonu oluşumu standart değer olan 0,1 sınırının altına indiği görülmüştür. Deney süresi sonunda kontrol numunesi alkali silika reaksiyonu genleşmelerini %0.1 değerinin altına düşüren minimum katkı miktarı kullanılabilecek "emniyetli miktar" olmaktadır.

Basınç dayanımı sonuçlarına göre, metakaolin (MK) kullanımı betonun mekanik özelliklerini olumlu etkilediği, dayanıklılığı arttırdığı, betonda sünme ve geçirimlilik özelliklerini iyileştirdiği tespit edilmiştir. ASR oluşumu ve basınç dayanımı açısından ideal katkı miktarının %10 olması yeterlidir.

Puzzolanik katkıların atık olmaktan çıkması, ekonomiye fayda sağlaması açısından kullanımının yaygınlaşması uygun olacaktır. Doğru karışımlarla hazırlanan betonlarda dayanıklılık yönünden çok yönlü kazanımlar elde edilecek, buda betonarme yapıların servis ömrünü uzatacaktır.

Anahtar Kelimeler: Metakaolin, Alkali Silika Reaksiyonu, Puzolanik Aktivite Deneyi, Genleşme, Endüstriyel Atık

The Investigation of Metakaolin Effect on Concrete and Mortars Made with Agregates Including Reactive Silica

Abstract: In the production of concrete and ferroconcrete elements, the usage of pozzolanic industrial wastes such as fly ash (FA), silica fume (SF), blast furnace slag (GBS) and metakaolin (MK) has a successful background. High- reactivity metakaolin(MK) is a manufactured pozzolan produced by thermal processing of purified kaolinitic clay. In this study, in concrete and mortars made with

aggregates including alkali-silica, the metakaolin effect, pozzolanic activity experiment, concrete pressure strength were investigated by alkali-silica reaction (ASR) experiments affecting the durability in concrete and mortars.

As a result of puzzolanic activity experiment, it is provided pressure strength at the rate of 95% (0.95) for a 28-day value. In the results of Accelerated mortar bar experiment, the formation of alkali silica reaction (ASR) affecting concrete strength was made harmless by the use of metakaolin at certain rates. In the samples with the additive at the rates of 10, 15 and 20%, the formation of alkali-silica reaction (ASR) was seen to fall below the limit of 0,10% that is a standard value. At the end of experiment time, control sample becomes "secure amount" in which minimum additive amount that reduces alkali-silica reaction dilatations below the value of 0.10% can be used.

According to pressure strength results, it was detected that the usage of metakaolin (MK) positively affected the mechanical features of the concrete, increased the strength and improved the creep and permeability properties of the concrete. Ideal additive amount must be 10% in terms of the formation of ASR and pressure strength. Being ceased of the puzzolanic additives to be wastes and spreading its usage in terms of contributing to the economy will be suitable. In the concretes prepared with true mixtures, multi-dimensional acquisitions will be achieved in terms of strength and this will prolong the service durability of concrete structures.

Keywords: Metakaolin, Alkali-Silica Reaction, Puzzolanic Activity Experiment, Expansion, Industrial Waste

1. Introduction

The thing expected from a structure is providing the strength, durability, economy and aesthetics. In the structuring in many countries, concrete and ferroconcrete is commonly used. The concrete being used in the structures encounters many factors that may cause corrosion in its body during service time. Concrete durability is defined as resistance capability of the concrete during service time of the concrete against deteriorating chemical and physical events resulted from air conditions, sulphate conditions and environment conditions where the concrete used. is The strength of hardened concrete is as important as at least its durability. The chemical and physical factors affecting the durability of the concrete are;

1- The dissolution of calcium hydroxide in the concrete and the formation of efflorescence

- 2- The effect of sulphate
- 3- The effect of acid
- 4- Carbonation
- 5- Alkali-silica reaction
- 6- Corrosions of the irons in the concrete
- 7- Freeze-thaw effect
- 8- Exfoliation of concrete surface
- 9- Corrosion in the concrete

The durability of the concrete depends on the quality of concrete and the processes like component materials, mixture rates, manufacturing method, the care and cure of the concrete are effective in the performance.(Yıldırım and Sumer, 2010) The usage of puzzolanic materials in the production of concrete and ferroconcrete has a successful background. Today, among the most common used puzzolans in the World, There are by-products coming from other industry sectors such as fly ash (FA), Blast furnace slag (GBS) and silica fume (SF), rice husk ash. There has been an important usage area of metakaolin in recent years. (Tokyay, 2013)

That metakaolin (MK) produced with the calcination of purified kaolin clay is a white color and it is an alumina silicate with amorphous structure is seen in Figure 1. When pure kaolin clay is cooked at

500-800C, it loses 14% of the water in it and it turns into metakaolin. As a result of the transportation, alumina and silica layers lose their order in their crystal structure, so it gains a reactive structure as kaolin, amorphous and chemical. When kaolin clay is exposed to excessive heat (over 900C) it loses its reactive feature. After a successful heat treatment, amorphous phase metakaolin having puzzolanic feature is obtained. (Changling and others, 1995), (Zhang, and Malhotra, 1995)

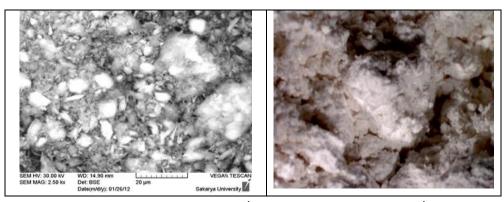


Figure 1. Metakaolin Sem İmage and Sterio Microscope İmage.

The usage of metakaolin (MK) by replacing with cement at appropriate rate in literature, it was reported that mechanical features are positively affected (Poon and others, 2006), it deceases capillary water absorption and permeability (Khatib and Clay, 2004), it increases the durability (Gruber and others, 2001), it is effective in controlng efflorescence and decreases especially the formation of alkali silica reaksion.(Ramlochan and others, 2000) On the other hand, It was reported that with the usage of metakaolin (MK), the drying shrinkage and creep in concrete decreased. (Brooks and Johari, 2001)

According to a research conducted by Jones et al., metakaolin used by replacing partly at the rate of 5-10-15% of cement weight of as a puzzolanic additive apparently reduced gels in alkali-silica reaction (ASR). Besides, metakaolin filled the holes in the concrete and prevented the permeability. It is seen in Figure 2 that fly ash used at the rate of 20% reduced alkali-silica reaction (ASR) at metakaolin level used at the rate of 10%. (Jones and others, 1992), (Walters and Jones, 1991), (Test Report, 1995),

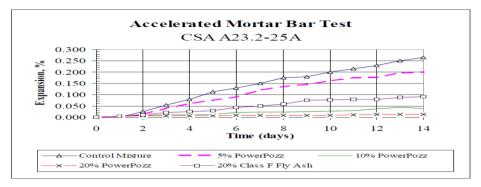


Figure 2. Experiment Graph with the Additive of Metakaolin and of Fly Ash (Jones and others, 1992), (Walters and Jones, 1991), (Test Report, 1995), .

The source of aggregates and in which rate the mineral additives must be is related to alkali content of the cement. For this, it is certainly required to make experimental studies on regional aggregates and to detect the additive rates. That these rates do not give harm to the regional structure of the concrete must be observed.

In this study, in concrete and mortars made with aggregates including alkali-silica, the metakaolin effect, puzzolanic activity experiment, concrete pressure strength were investigated by alkali- silica reaction experiments affecting the durability in concrete and mortars.

2. Experimental Process

2.1 Materials

2.1.1 CEN Standard Sand

Rilem-Cembureau standard sand compatible with TS EN 196-1 was used to prepare cement mortar specimens. Granulometric properties of the sand are presented in Table 1. Unit weight of the sand was found to be 2.01 gr/cm^3 (TS EN 196-1, 2002)

Sieve Mesh Sze (mm)	Material retained on the sieve (%)
0,08	98 ±2
0,16	87 ±2
0,50	67 ±2
1,00	33 ±2
1,60	9 ±2
2,00	0

Table 1. Granulometry Values of Rilem Sand.

2.1.2 Cement and Metakaolin

According to ACI 221, cement equivalent alkali amount $(Na_2O+0.658K_2O)$ should be maximum 0.6% and it is recommended that this limit be 0.4%. Ratio of alkali amount of cement used in the test was $(Na_2O+0.658(K_2O)) = 0.22*0.658*0.46=0.52$ (Na_2O) , which is below the value of 0.6%. (TS-EN 197-1, 2002), (Yıldırım and Sumer, 2014) Alkali levels of cement and mineral additives are presented in Table2[15]. Stereo microscope and SEM images belonging to metakaolin additives are given in Figure 1 and chemical analysis values belonging to cement and metakaolin are given in Table 2.

	CEM I 42.5	Metakaolin (Standard Values)	Metakaolin used
SiO ₂ (%)	19,03	52-54	53, 68
Al ₂ O ₃ (%)	4,30	42-44	42, 44
Fe ₂ O ₃ (%)	4,36	<1-1.4	1,40
CaO (%)	64,24	0,1	0, 60
MgO (%)	1,09	<0,1	0, 73
Na ₂ O (%)	0,22	<0,05	0,00
K ₂ O (%)	0,46	<0,4	0,00
Na ₂ O Equivalent	0,52		
TiO ₂ (%)		<3,0	0, 68
SO ₃ (%)	2,39	<0,1	
Loss on ignition (%)	4,17	<1,0	
Specific Weight (gr/cm ³)	3,17		2,6
45 Micron (>45µm, %)	2,85	D50	D50
Blaine (cm^2/gr)	4289	8600	8600

Table 2. Chemical Analysis Values of Cement and Metakaolin.

2. 2. Puzzolanic Activity Test Experimental Study

The activity of puzzolanic determined by mechanical and chemical tests. (Yıldırım and Sumer, 2014) Mixture ratios of mortar specimens produced for puzzolan activity test is presented in Table 3. The metakaolin that was used as puzzolan was prepared by replacing 20% cement. 6 40*40*160 mm specimens were prepared from each group at this mixture ratio.

	Puzzolan Additive (20%) (g)	Cement (g)	Standard Sand (g)	W/C=0.5 Water (g)	Mean Weight (g)
Reference Specimen		500	1320	250	614
Metakaolin	100	400	1320	250	591

Tablo 3. Puzzolanic Activity Mortar Mixture Amount.

7 day and 28 day compression strength tests were carried out with these specimens. Test results are presented in table 4 and Figure 3.

	7-day results Compressive Strength (N/mm ²)	Percentage of Compressive Strength	28-day results Compressive Strength (N/mm ²)	Percentage of Compressive Strength
Reference Specimen	26,7	1	46,8	1
Concrete with metakaolin additive	24,80	0,93	44,57	0,95

Tablo 4. Puzzolanic Activity Compressive Strength Test Results.

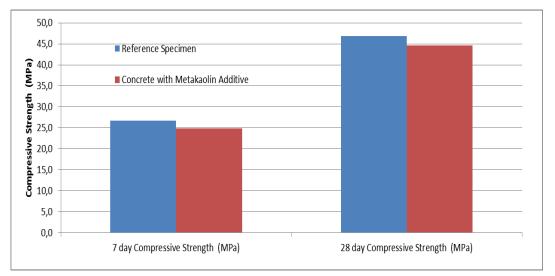


Figure 3. Metakaolin Additive Mortar Puzzolanic Activity Experiment Pressure Strength Graph.

As a standard, ash should have a value of 70% (0.70) in order to be used as puzzolan. Results of puzzolan activity test revealed a compressive strength of 95% (0.95) in 28-day test. Metakaolin will find usage area as an extremely beneficial additive in terms of metakaolin concrete products.

2.3 ASTM-C 1260 Accelerated Mortar Bar Experiment Experimental Study

2.3.1 Materials and Sample Preparation

2.3.1.1 Aggregate

The aggregate we used in experimental study was supplied from a sand gravel quarry near Sakarya River–Geyve town. The aggregate had a thickness of 0-7 mm. Firstly, amount of active silica on the aggregate was determined using chemical analysis method in accordance with ASTM C289, TS 2517 standard. It is presented in Table 5 that it is on harmful zone. (ASTM C-618-12a, 2012), (ASTM C-289-94, 1994)

Table 5. Chemical ASR Report Condu	cted in Accordance with TS 2517.			
NaOH (Consumed) 350 (mmol/L)				
SiO ₂ (Solved)	700 (mmol/L)			
Result	III. Zone (Hazardousaggregate)			

2.3.1.2 Preparation of Specimen Groups

Appropriate aggregate was prepared by sieving the aggregate according to mixture ratios in ASTM-C 227. In our tests, while preparing 5%-10%-15%-20% mortar bars in accordance with ASTM-C 227 standard using metakaolin replacing cement, mortar mixture was prepared in such a way to have an aggregate/cement ratio of 2.25; a spread value of 120-150 mm and W/C ratio of 0.47. Minimum four mortar specimens were prepared from each group in 25×25×285 mm prismatic molds. (ASTM C-227-97, 1994) Material mixture ratios of the control mixture and of the specimens produced by 5%-10%-15%-20% metakaolin replacing cement are presented in Table 6.

Additive	Aggregate	Cement	Mineral	Water		
Ratios	(gr)	(gr)	Additive(gr)	(gr)	W/C	A/C
Reference Specimen	1320	587		276	0.47	2.25
%5 Metakaolin	1320	557.65	29.35	276	0.47	2.25
%10 Metakaolin	1320	528.3	58.7	276	0.47	2.25
%15 Metakaolin	1320	498.95	88.05	276	0.47	2.25
%20 Metakaolin	1320	469.6	117.4	276	0.47	2.25

Table 6. Material Mixture Values of Test Specimens.

Standard values of this method which is known as ASTM-C 1260 accelerated mortar bar test are presented below. (ASTM C1260, 2014)

Expansion percentages at the end of 16-day test are evaluated as follows:

- If the expansions at the end of 16 days are below 0.10%, the aggregates show harmless behavior.
- If the expansions at the end of 16 days are above 0.20%, the aggregates show potentially harmful expansion.
- If the expansions at the end of 16 days are between 0.10%, and 0.20%, the aggregates can show both harmful and harmless behavior at construction site conditions.

If the expansions at the end of 16 days are between the values of 0.10% and 0.20%, test duration for the aggregates had better be continued for 28 days. (ASTM C1260, 2014)

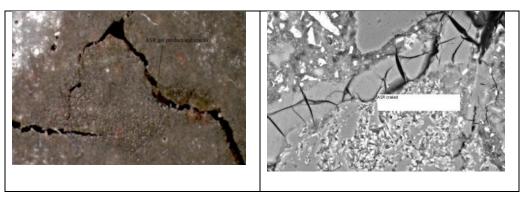


Figure 4. The Images of the Formation of ASR Gel and Cracks in Reference Samples.

In Figure 4, the formation of alkali- silica reaction (ASR) gel and crack occur in reference sample. As it is understood from the images, aggregate used in our experiment include active silica both in chemical analysis result and in reference sample and its extremely harmful in terms of concrete and ferroconcrete products.

Expansion results of control mixture and specimens with metakaolin are presented in Table 7. alkalisilica reaction (ASR) formation graph is presented in Figure 5.

	Reference		Metakaolin	additive ratio	S
	Specimen	MK %5	MK %10	MK %15	MK %20
9.day	0,601921	0,079096	0,047797	0,041469	0,040678
16.day	0,834689	0,094915	0,055254	0,047345	0,044181
28.day	0,920565	0,170847	0,075932	0,063729	0,052994

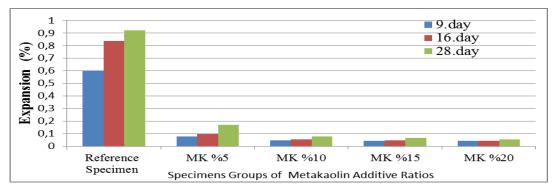


Figure 5. ASR Expansion Values Graph in Metakaolin Additive Mortar Groups.

As a result of ASTMC 1260 Accelerated mortar rod experiment, when maximum standard value was taken to the 0.20, it was seen to reduce under standard value in metakaolin additive mortar types. It was observed that

- It reduced the dilatation around 0.81 in mortar type with 5% additive compared to control mixture.
- It reduced the dilatation around 0.92 in mortar type with 10% additive,
- It reduced the dilatation around 0.93 in mortar type with 15% additive,

• It reduced the dilatation around 0.95 in mortar type with 20% additive.

In reduction of alkali- silica reaction (ASR), the usage of metakaolin was seen to be extremely effective compared to reference sample. According to above mentioned data, alkali-silica reaction that is harmful in terms of the durability of the concrete has been made harmless to certain rates with the usage of metakaolin additive.

In the samples prepared as metakaolin additive at the rates of 10,15 and 20%, the formation of alkalisilica reaction (ASR) was seen to fall below the limit of 0,1 that is a standard value.

At the end of the experiment time, minimum additive amount that reduces control sample dilatations below the value of 0.1% is the "secure amount to be used".

In concretes prepared with true mixtures, multi-dimensional acquisitions in terms of durability will be obtained. This will prolong the service life of ferroconcrete structures.

2.4 Pressure Strength Experiment in the Samples with Metakaolin Additive

Concrete compressive strength is expressed as $f_{c\ cube}$ if cube shaped specimens in compliance with the standard are produced for compressive strength. Compressive strength is determined in specimens of 7 and 28 days. (TS EN 12390-3, 2002)

2.4.1 Materials, Sample Preparation and Experimental study

Fine aggregates exposed to 60% ASR, 40% fine chips and cement and metakaolin, whose analysis values are explained above, were used in compressive strength tests. Concrete mixture groups were prepared in 10×10×10 cm test cubes. Six groups of concrete with a water/cement ratio of 0.45 and 0.60; metakaolin additive ratio of 10% and 20% were prepared for compressive strength test. Material ratios of concrete mixture are presented in Table 8 in unit of m³. Compressive strength values are presented in Table 9 and Figure 6.

			1	1	0	
Additive	0-8 mm	8-13 mm	Cement	Mineral	Water	W/C
Ratios	fine	large	(kg)	Additive	(kg)	
	Aggregate	Aggregate		(Metakaolin)		
	(kg) %60	(kg) %40				
Reference Specimen 1	1040	714	420		189	0.45
MK 1	1040	714	378	42(%10)	189	0.45
MK 2	1040	714	336	84(%20)	189	0.45
Reference Specimen 2	1040	714	250		150	0.60
MK 3	1040	714	225	25(%10)	150	0.60
MK4	1040	714	200	50(%20)	150	0.60

Table 8. Material Mixture Ratios of Concrete Prepared for Compressive Strength.

Table 9. Compressive Strength Values of the Prepared Concrete Groups.

	28 day (Mpa)	90 day (Mpa)	180 day (Mpa)
ReferenceSpecimen 1	56,62	58,3	59,9
MK 1	53,66	57,04	59,8
MK 2	43,2	44,1	45,8
ReferenceSpecimen 2	32,28	36,22	37,6
MK 3	28,98	32,67	34,8
MK4	28,03	31,32	31,5

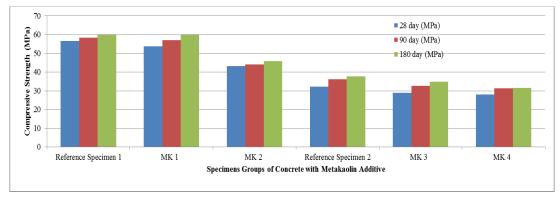


Figure 6. Pressure Strength Results in Concrete Groups with Metakaolin Additive.

According to 180-day pressure strength results, it was seen that

* In concrete group with reference sample 1 and 0.45 of S/C rate, in 10% of metakaolin additive sample, the pressure strength reduced to around 1% and in 20% of additive group, the pressure reduced to around 23% compared to reference sample,

* In concrete group with reference sample 2 and 0.60 of S/C rate, in 10% of metakaolin additive sample, the pressure strength reduced to around 8% and in 20% of additive group, the pressure reduced to around 16% compared to reference sample,

* In concrete group with reference sample 1 and reference number 2, the pressure reduced to around 37% as the S/C rate is getting increased,

* In both sample groups, hardening occurred in normal way and it was observed according to 90 and 180-day pressure strength results that the strength increased but the strength decreased by increasing at the same rates.

3. Results and Discussion

Within the scope of our study, in metakaolin additive mortars and concretes, puzzolanic activity experiment, concrete pressure strength and alkali silica reaction experiments were conducted.

As a result of puzzolanic activity experiment, it was seen to provide pressure strength at the rate of 95% (0.95) as a 28 day value.

As a result of alkali silica reaction experiment results, with the usage of certain rates of metakaolin, it was seen that the formation of alkali- silica reaction (ASR) became harmless. In the samples with the additive at the rates of 10, 15 and 20%, the formation of alkali- silica reaction (ASR) was seen to fall below the limit of 0,1 that is a standard value.

At the end of experiment time, control sample becomes "secure amount" in which minimum additive amount that reduces alkali silica reaction (ASR) dilatations below the value of 0.1% can be used.

According to pressure strength results, it was seen that;

* In concrete group with reference sample 1 and 0.45 of S/C rate, in 10% of metakaolin additive sample, the pressure strength reduced to around 1% and in 20% of additive group, the pressure reduced to around 23% compared to reference sample,

* In concrete group with reference sample 1 and 0.60 of S/C rate, in 10% of metakaolin additive sample, the pressure strength reduced to around 8% and in 20% of additive group, the pressure reduced to around 16% compared to reference sample,

* In concrete group with reference sample 1 and reference number 2, the pressure reduced to around 37% as the W/C rate is getting increased,

* In both sample groups, hardening occurred in normal way and according to 90 and 180 day pressure strength results, it was observed that the durability increased but the durability decreased by increasing at the same rate.

In concrete and mortars, as the advantages of metakaolin usage, it was detected in experimental study content that;

- Strength and performance increased in concretes
- The permeability decreased,
- It reduced the effects of alkali silica reaction (ASR),
- It reduced the processability in concretes,
- It increased shrinking and cracks in concrete,

For alkali silica reaction (ASR) and pressure strength, 10% of Metakolin was enough as an ideal additive amount.

Not regarding mineral additives as wastes will be convenient for the proliferation of its usage in terms of being beneficial for the economy from this aspect.

In concretes prepared with true mixtures, multi-dimensional acquisitions in terms of durability will be obtained. This will prolong the service life of ferroconcrete buildings.

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