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The strength properties of alkali-activated silica fume mortars

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Abstract. In this study, the strength properties of alkali-activated silica fume (SF) mortars were investigated. The crushed limestone sand with maximum size of 0-5 mm and the sodium meta silicate (Na_2SiO_3) used to activate the binders were kept constant in the mortar mixtures. The mortar specimens using the replacement ratios of 0, 25, 50, 75 and 100% SF by weight of cement together with Na_2SiO_3 at a constant rate were produced in addition to the control mortar produced by only cement. Moreover, the mortar specimens using the replacement ratio of 4% titanium dioxide (TiO_2) by weight of cement in the same mixture proportions were produced. The prismatic specimens produced from eleven different mixtures were de-moulded after a day, and the wet or dry cure was applied on the produced specimens at laboratory condition until the specimens were used for flexural strength (f_{fs}) and compressive strength (f_c) measurement at the ages of 7, 28 and 56 days. The f_{fs} and f_c values of mortars applied the wet or dry cure were compared with the results of control mortar. The findings revealed that the f_c results of the alkali activated 50% SF mortars were higher than that of mortar produced with Portland cement only. It was found that the f_{fs} and f_c of alkali-activated SF mortars cured in dry condition was averagely 4% lower than that of alkali-activated SF mortars cured in wet condition.

Keywords: silica fume; alkali activator; mortar; flexural strength; compressive strength

1. Introduction

In recent years, new trends in environmental regulations about disposal or recovery of industrial by-products such as SF, fly ash and granulated blast furnace slag have increased the interest in the utilization of these by-products as a construction material by cement with specific proportions (Han *et al*. 2003). These by-products are used in progressive concrete technology to provide economic concrete production and to improve various properties of concrete like mechanical, durability and impermeability. Especially, SF obtained from reduction of high-purity quartz with carbonaceous materials in electric arc furnaces distinguishes to develop these properties. SF is a material which has an excellent binding property due to its amorphous form, excessive fineness and high silica content (Erdoğan 2003). The chemical effect on the development of these properties of SF is because of the higher surface area of the reaction with calcium hydroxide ($Ca(HO)_2$), and this reaction is due to take place more quickly according to other pozzolans containing amorphous silica. In general, pozzolanic reaction starts at 2^{nd} day and ends 28^{th} day for SF cured at 20°C. A calcium silicate hydrate product which is composed as a result of reaction between the SF and $Ca(OH)_2$ reduces porosity. Because, calcium silicate hydrate products occurred after pozzolanic reaction has less surface area than SF and $Ca(HO)_2$ (Bayasi and Zhou 1993, İlter 2007). Additionally, SF particles provide concretes to reach higher strength, durability and impermeability by filling and reinforcing the aggregate-matrix interface zone (Mazloom *et al*. 2004, Singaram and Kowsik 2016). In addition, SF develops interface zones by filling the voids between the cement particles due to its very fine particle structure (Toutanji *et al*. 2004, Pilvar *et al*. 2016).

Recently, many research works have indicated that it can be possible to produce cementless mortar or concrete by activating the industrial by-products and natural pozzolans with alkaline activator. Particularly, most of these researches have been carried out the activation process on the granulated blast furnace slag and fly ash. In general, the alkalis which are employed as activator are sodium hydroxide (NaOH), sodium metasilicate (Na_2SiO_3), sodium carbonate (Na_2CO_3) and potassium hydroxide (KOH) (Wang *et al*. 1995, Hardjito *et al*. 2004, Pacheco-Torgal *et al*. 2008, Yang and Song 2009). In accordance with the ordinary Portland cement, alkali-activated binders have many advantages such as higher rate of f_c development (Fernandez-Jimenez *et al*. 1999, Bakharev *et al*. 1999, Krizan and Zivanovic 2002, Bernal *et al*. 2011), lower porosity, lower heat of hydration (Shi *et al*. 2006), better resistance to chemical attacks (Roy *et al*. 2000, Bakharev *et al*. 2002, Bakharev *et al*. 2003), better durability to freezing-thawing effect (Puertas *et al*. 2003), stronger aggregate-cement interface zone (Shi *et al*. 1998) and lower permeability. Nevertheless, they have some disadvantages like quick setting (Zivica 2007), lower workability, occurrence of microcrack, high shrinkage (Collins and Sanjayan 1999, Serdar 2013) and efflorescence.

Especially, a lot of investigations have performed to activate granulated blast furnace slag and fly ash with alkalis. Atiş *et al*. 2009 produced cementless mortar specimens with granulated blast furnace slag which is

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Table 1 Chemical properties of cement and silica fume materials

Oxide (%)	SiO_2	CaO	Fe_2O_3	Al_2O_3	SO_3	MgO	Na_2O	K_2O	LOI
Cement	21.20	62.10	2.10	5.90	3.40	2.30	0.40	0.80	1.80
Silica fume	88.75	0.75	0.45	0.90	0.70	4.90	0.35	0.50	2.70

LOI.: Loss on ignition

Table 2 Physical properties of cement

Physical properties		Results
Specific gravity		3.10
Setting time	Initial (min)	130
	Final (min)	215
Fineness	Specific Surface (cm^2/gr)	3310

Table 3 Properties of titanium dioxide

Physical situation	Amorphous odorless white powder
Melting point (^oC)	1800
Fineness	3310
Specific gravity	3.8
Resolution	Insoluble in water
pH	8

Table 4 Gradation of crushed limestone sand and standard limit values

Sieve size (mm)	0.063	0.15	0.25	0.5	1	2	4	5.6	8
Passing from sieve (%)	2.5	10	24.5	42.5	59.5	78.5	98.5	100	100
Limits %	0-3	3-10	8-25	20-50	44-74	69-90	85-98	95-100	100

activated by liquid Na_2SiO_3, NaOH and Na_2CO_3. They performed the f_{fs} and f_c tests on these specimens. Particularly, they obtained the highest f_{fs} and f_c values on the specimens activated with Na_2SiO_3. Bakharev *et al*. 1999 achieved cementless mortar specimens by activating granulated blast furnace slag with liquid NaOH, Na_2SiO_3, Na_2CO_3 and Na_3PO_4. They cured these specimens at 60°C. They observed the highest f_c values on the specimens activated with liquid NaOH. El-Didamony *et al*. 2012 produced granulated blast-furnace slag pastes activated with alkalines NaOH and Na_2SiO_3. They used sea and tap water in their mixes. They reached the maximum f_c values on the pastes activated with liquid NaOH and sea water. Oh *et al*. 2010 created alkali activated geopolymer mortars by activating fly ash with NaOH and Na_2SiO_3 at 75°C. They obtained high f_c values on the geopolymer mortar specimens. Guo *et al*. 2010 used NaOH and Na_2SiO_3 in their mortar specimens to activate class C fly ash at 75^{o}C. Their specimens showed high f_c. Chindaprasirt *et al*. 2007 produced mortars with fly ash which were cured at 75°C approximately 2 days and activated with NaOH and Na_2SiO_3. They performed f_c test on the mortars, and their mortars showed high f_c. Fernandez and Palomo 2005 employed NaOH, NaOH+Na_2CO_3 and NaOH+Na_2SiO_3 in their mortar specimens to activate fly ash, and they cured their specimens at 85^{o}C for 20 hours. They observed the highest f_{fs} values on the specimens activated with NaOH. Conversely, they obtained the highest f_c values on the specimens activated with NaOH+Na_2SiO_3. Khalifeh *et al*. 2014 produced mortar specimens with class C fly ash which was activated with Na_2SiO_3 and NaOH at 87^{o}C and 125^{o}C. They observed the highest f_c values on the specimens activated at 87^{o}C.

In the present work, in addition to control mortar produced with ordinary Portland cement, alkali-activated mortars include 0, 25, 50, 75 and 100% SF and alkali-activated mortars made with 4% TiO_2 in replace of binder with same SF ratios have been produced. Specimens made with these mortars have been exposed to two types of cure conditions: in a water bath at 23±2°C (wet cured) and in an airtight bag (dry cured) at laboratory condition. The unit weight, f_{fs} and f_c values of the specimens exposed to these cure conditions have been investigated. The relationships between the f_{fs} and f_c values of the specimens have been examined. Besides, microstructures of limestone sand, matrix and interface on the thin sections made from crushed limestone aggregates, control mortar and 50% SF mortar were examined by polarized light microscope (PLM).

2. Properties of materials

2.1 Cement

ASTM type I Portland cement that is manufactured in Turkey as CEM I 42.5 R type Portland cement, which corresponds to TS EN 197-1 2012, was utilized in the mortar mixtures. The cement was fabricated at Niğde cement plant of ÇİMSA in Turkey. The chemical and physical properties of the cement are presented in Tables 1-2, respectively.

2.2 Silica fume

Silica fume (SF) was procured from Antalya-Etibank Ferro-Chrome Factory in Turkey. Its chemical properties are presented in Table 1. The unit weight and specific gravity values of SF used in the mortars were 247 kg/m^3 and 2.3, respectively.

2.3 Titanium dioxide

Titanium dioxide (TiO_2) doesn't occur in nature purely and it is manufactured from iron titanium trioxide (Fe_3TiO_3) ore in general. The most widely used white pigment TiO_2 is so white. Areas of utilization of TiO_2 are food, cosmetics and pharmaceutical products and also it is used as a pigment provides whiteness and opacity in paint, plastics and paper industries. The physical and chemical properties of TiO_2 used in the mortars are given in Table 3.

2.4 Crushed limestone sand

The crushed limestone sand, which has particle size of 0-5 mm, used in the mortar mixtures was supplied from local resources. The unit weight of crushed limestone sand

Table 5 Properties of superplasticizer

Structure of material	Polycarboxylic ether based
Color	Ambergris
Density	1.082–1.142 kg/liter
Cl content (%)	< 0.1
Alkali content (%)	< 3

Table 6 Properties of sodium metasilicate

Property	Molar Ratio	Weight Ratio	SiO_2	Na_2O
Values	0.94	0.91	44.94	49.38
Limits	0.91-1.01	0.88-0.98	44.30-46.30	47.90-49.90

used in the mortars was determined as 2.58 kg/m^3. The results of sieve analysis performed on the crushed limestone sand and limits according to TS 802 2009 are given in Table 4.

2.5 Superplasticizer

The high range water reducing admixture was used to obtain adequate workability in the mortar mixtures. This admixture called Glenium 51, provided from BASF-The Chemical Company, complies with the requirements of TS EN 934-2+A1 (2013) and ASTM C 494-99a (2002). The high range water reducing admixture based on polycarboxylic ether has been developed for obtaining mortar and concrete which have high strength and durability. The properties of the admixture, fabricated at +20℃ and 50% relative humidity condition, are given in Table 5.

2.6 Alkali activator

In this investigation, solid sodium metasilicate (Na_2SiO_3) was used to obtain alkali-activated SF mortars. Molecular weight, specific gravity at 20^{o}C, purity and melting point of Na_2SiO_3 are 122.06 gr/mol, 2.40, 57% and 1089^{o}C, respectively. The chemical properties of the Na_2SiO_3 used in the mortars are given in Table 6.

2.7 Experimental study

In this study, eleven different mortar mixtures were prepared including control mixtures. Control mixtures were created as three types: the first one which was made with only ordinary Portland cement as binder, the second made with 6% Na with ordinary Portland cement as binder and the third made with 6% Na, 4% TiO_2 and ordinary Portland cement as binder. As to other mixtures, eight different mixtures prepared with SF which was partially replaced with cement by weight at 25, 50, 75 and 100% proportions providing the constant 6% Na. Besides, half of the eight mixtures also include 4% TiO_2.) Crushed limestone sand-binder ratio of 2.7 was determined for mortar mixtures. Solid Na_2SiO_3 was utilized as alkali-activator with constant amount in the mixtures. The amounts of materials used in the mortar mixtures are given in Table 7. While the mortar mixtures were preparing, the solid alkali-activator Na_2SiO_3 was dissolved in some of the mix water. The crushed limestone sand, cement, binder, water, dissolved alkali-activator and high range water reducing admixture were placed into the mortar mixer, respectively. After placing the crushed limestone sand and binders, mortar mixer was adjusted to slow gauge and run for 30 seconds. While the mixer was running at slow gauge, mix water and dissolved alkali-activator were added to the mixture. After that, high range water reducing admixture was added to mixture, the mixer was run at fast gauge for 30 seconds. Finally, after waiting for 15 seconds, the mixer was run with fast gauge for 60 seconds and mixing process was ended.

Table 7 Amounts of materials used in mortar mixtures (kg/m^3)

Mix code	Cement (kg/m³)	SF (kg/m³)	Water (kg/m³)	LS (kg/m³)	Na_2SiO_3 (kg/m³)	TiO_2 (kg/m³)	SP (kg/m³)	Flow (mm)
C	500	0	250	1350	0	0	0	19
CA	500	0	250	1350	80	0	0	18
25SF	375	125	300	1350	80	0	1.25	11
50SF	250	250	350	1350	80	0	2.70	10
75SF	125	375	375	1350	80	0	16.22	8
100SF	0	500	400	1350	80	0	18.93	8
CAT	500	0	250	1350	80	20	0	18
25SFT	375	125	300	1350	80	20	1.25	12
50SFT	250	250	350	1350	80	20	2.70	11
75SFT	125	375	375	1350	80	20	16.22	9
100SFT	0	500	400	1350	80	20	18.93	8

SF: Silica fume, LS: Limestone sand, SP: Superplasticizer

Firstly, shake table test was performed on the mortar mixtures and the flow diameter values of the mixtures were measured. Then, the mixtures were compacted on vibrator table and placed in the prismatic molds with the dimensions of 40×40×160 mm. The specimens taken out from the molds 24 hours after casting were cured in a water bath at 23±2℃ (wet cured) or in an airtight bag (dry cured) at laboratory condition until the testing day. Before the tests performed, unit weight values of the specimens which have saturated dry surface were measured. The f_{fs} tests performed on the 40×40×160 mm prismatic specimens whose unit weight values were measured. The f_c measurements were obtained by placing 40×40 mm plates to top and bottom of the specimens which were broken into two pieces during the f_{fs} test. Strength measurements were performed according to the TS EN 1015-11/A1 2013 standard.

Microscopic analyses are commonly utilized to investigate the mineralogical composition of minerals and rocks in the areas of petrography and geology. In the present study, microscopic analyses of crushed limestone sand, control mortar and 50% SF mortar were examined by Nikon ECLIPSE E400 Pol type polarized microscope. The crushed limestone sand, the 40×40×50 mm pieces made from control mortar and 50% SF mortar were used for PLM analyses. The crushed limestone sand and these mortar pieces were placed in the acetone to clean the pores, and afterwards they were impregnated with resin in a vacuum desiccator until the micro air bubbles in the piece disappear. The hardened pieces were slivered by a cutting machine to

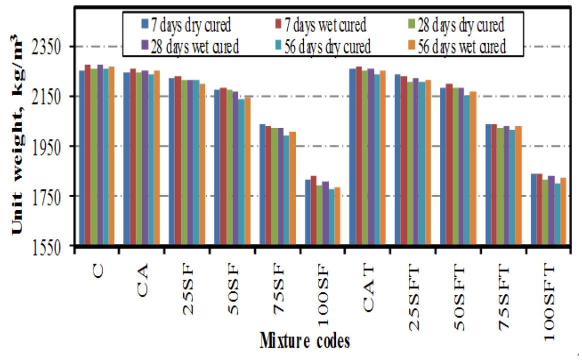


Fig. 1 Hardened unit weight values of mortars

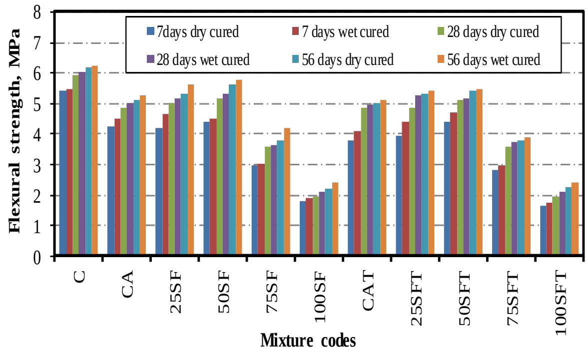


Fig. 2 Flexural strength values of mortars

obtain thin section about 40×40×0.003 mm size. After the section was glued to the glass, they were made even thinner with abrasion for PLM analyses.

3. Results and discussion

3.1 Flow

Flow diameter values of SF mortars activated with Na_2SiO_3 and produced with high range water reducing admixture to obtain adequate workability were measured on the shake test table. The amounts of mix water and high range water reducing admixture were increased with increasing of the amount of SF used as binder. After smoothing the top of the mortar mixtures which were placed into the cone used for shake table test, the cone was removed and 15 shakes were applied for providing the mixtures to be spread on the table. Then, the flow diameters of the mortars were measured from 4 different sides. Arithmetic mean of these 4 values was calculated and so these values were determined as the flow values of mixtures. The flow values of the mixtures are presented in Table 6. Owing to the particle size of SF is very fine compared to particle size of cement, the quantity of high range water reducing admixture and mix water were multiplied. Furthermore, the flow values of mortars were decreased with increasing the amount of SF.

3.2 Unit weight

Unit weight values of hardened mortars were

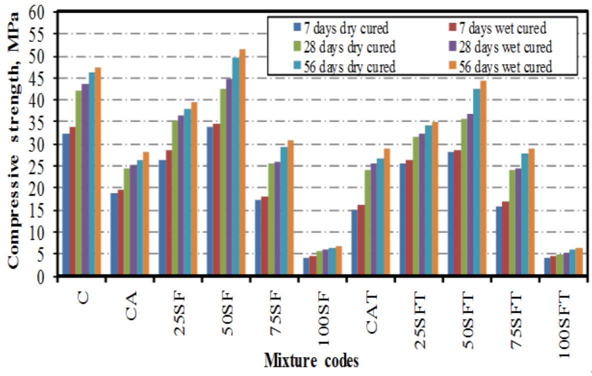


Fig. 3 Compressive strength values of mortars

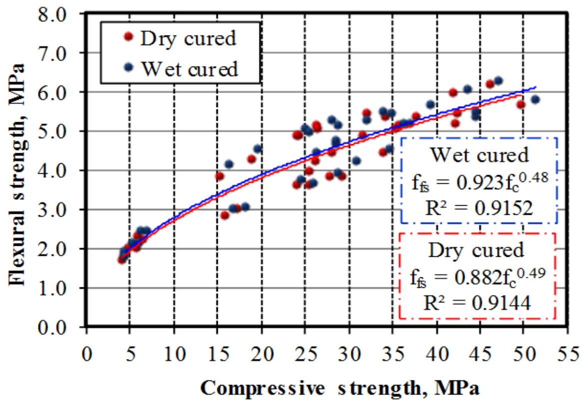
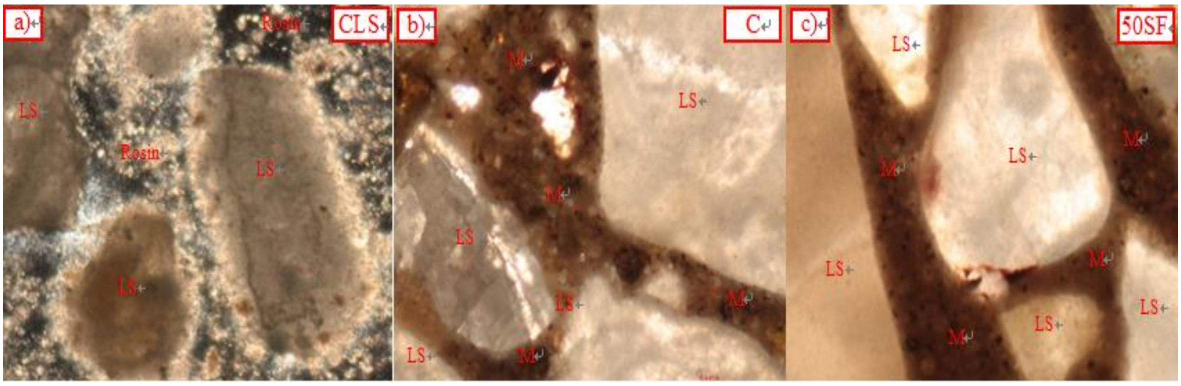


Fig. 4 Relationship between flexural and compressive strength values

determined on the prismatic specimens produced for f_{fs} and f_c tests with the dimensions of 40×40×160 mm. After the specimens pull out from wet cure or dry cure, unit weight values of them were measured in water-saturated dry surface specimens. Firstly, each specimen was weighed separately. Secondly, the means of weight values of three specimens were calculated to determine weight value of each mixture. Finally, unit weight value of each hardened mortar mixture was calculated with dividing the mean weight by the specimen volume. Hardened unit weight values of hardened mortar mixtures at the ages of 7, 28 and 56 days are presented in Fig. 1. When this figure is examined, it can be seen that if the amount of SF replaced with cement increases, unit weight values decrease. The reason of decreasing is that the specific gravity of SF is lower than that of cement. Besides, TiO_2 content has not a significant effect on the specific gravity of mortar mixtures. Because, the amount of TiO_2 replaced with cement is used very little in the mixture. There was no significant effect of cure types on the unit weight values of all the specimens. The unit weight values of the specimens cured in wet condition were more a little bit from specimens cured in dry condition. The unit weight values of the specimens cured in wet condition was slightly higher than the specimens cured in dry condition.

3.3 Flexural strength

In the experimental study, the 40×40×160 mm prismatic specimens were used to determine the flexural strength (f_{fs})



CLS: Crushed limestone sand, LS: Limestone sand, C: control mortar, 50SF: 50% SF mortar, M: matrix

Fig. 5 PLM analyses of (a) crushed limestone sand, (b) control mortar, and (c) 50% SF mortar

values of the mortars activated with Na_2SiO_3. Three specimens belong to each group were processed to breaking the in the three-point flexural test, and the f_{fs} values of each group were calculated by the mean breaking load values of these three specimens. The f_{fs} values of mortars activated with Na_2SiO_3 cured in wet or dry condition at the ages of 7, 28 and 56 days are given in Fig. 2. As seen in this figure, the f_{fs} values of SF and TiO_2 mortars activated with Na_2SiO_3 were lower than that of mortars produced with only cement without activator. According to other mixtures, the mixtures containing TiO_2 had slightly low f_{fs} values. In the mixtures produced with SF as binder, while the ratio of SF content increases, the f_{fs} values decrease. The f_{fs} values have increased with the increase of cure days in the specimens exposed to wet cure or dry cure as expected. Besides, the f_{fs} values of specimens exposed to wet cure are approximately 4% higher than specimens exposed to dry cure. Strength values of SF mortars are lower in dry cure condition according to wet cure condition due to the more water requirement to compose calcium silicate hydrate jell.

3.4 Compressive strength

The specimens divided into two pieces during the flexural tests were employed for determining compressive strength (f_c) values of the mortars activated with Na_2SiO_3. The f_c values of the specimens, which were divided into two pieces belong to each group, were determined by placing 40×40 mm plates on the top and bottom of the specimens. The f_c values of mortars activated with Na_2SiO_3 cured in wet or dry condition at the ages of 7, 28 and 56 days are illustrated in Fig. 3. The f_c values of 50% SF mortars activated with Na_2SiO_3 were higher than that of control mortars produced with only cement without activator and other mortars. The f_c values of other SF mortars activated with Na_2SiO_3 (except for 50% SF) were lower than that of control mortars produced with only cement without activator. Moreover, a decline was observed on the f_c values of mortars prepared with TiO_2 according to the mortars prepared without TiO_2. Consequently, the maximum f_c values were observed on the 50% SF mortars, and minimum f_c values were observed on the 100% SF mortars in all mixtures. The f_c values of specimens cured in wet or dry condition were increased with the increase of cure days as expected. In addition, the f_c values of specimens cured in dry condition were approximately 4% lower than the specimens cured in wet condition as the f_{fs} values. The reason of the reduction can be explained as follows.

Generally, hydration occurs as a result of the reaction between the binder and water. Setting and hardening of binder occur with the calcium silicate hydrate jell which was formed as a result of hydration. The calcium silicate hydrate jell is gained the strength and binding properties of binders. These properties are composed of the attraction force between layers of the calcium silicate hydrate jell. Therefore, there must be enough water in cure condition to gain these properties. The reason why f_c values of wet cured specimens are higher than dry cured specimens is the existence of enough water in the wet condition.

3.5 Correlation between compressive strength and flexural strength

The f_{fs} and f_c values of SF and TiO_2 mortars activated with Na_2SiO_3 cured in wet and dry conditions are correlated as seen in Fig. 4. It can be obviously seen from this figure that there are very good correlations between f_{fs} and f_c values of the specimens exposed to wet cure and dry cure. Equations obtained from the correlations between f_{fs} and f_c values of the specimens exposed to wet or dry cure are given by Eqs. (1)-(2), respectively. These equations and R^2 values are also shown in Fig. 4. The f_{fs} values of alkali-activated mortars cured in wet and dry conditions can be calculated by using the f_c values of these mortars by means of the equations. The R^2 values given in this figure indicate the very good correlations between f_{fs} and f_c values. Consequently, it can say that there is an exponential relationship between f_{fs} and f_c values of the specimens exposed to wet cure or dry cure.

$$f_{fs} = 0.923(f_c)^{0.48} \quad (1)$$

$$f_{fs} = 0.882(f_c)^{0.49} \quad (2)$$

3.6 Polarized light microscope analyses

Microstructures of crushed limestone sand, matrix and interface on the thin sections were examined by PLM as seen in Fig. 5. The impurity, gap and micro-crack were not appeared in the crushed limestone sand (Fig. 5(a), (b) and (c), matrix and interface. The bonding strength between the crushed limestone sand and matrix was very strong as seen from the microscope analyses of control mortar (Fig. 5(b)) and 50% SF mortar (Fig. 5(c)). This bonding strength is very effective on the f_{fs}, f_c, durability and impermeability properties of mortars activated with Na_2SiO_3.

4. Conclusions

Based on the experimental studies of SF and TiO_2 mortars activated with Na_2SiO_3, the following conclusions can be summarized.

• Water requirement of mixtures activated with Na_2SiO_3 increases while the ratio of SF content in the mixtures increases.

• The f_{fs} and f_c values of mortars activated with Na_2SiO_3 cured in wet condition are higher than that of mortars cured in dry condition.

• The f_{fs} values of SF and TiO_2 mortars activated with Na_2SiO_3 are lower than that of mortars produced with only ordinary Portland cement.

• The maximum f_c values are shown in 50% SF mortars activated with Na_2SiO_3. The f_c values of other mortars activated with Na_2SiO_3 are lower than the f_c values of mortars produced with ordinary Portland cement.

• High correlations are observed between the f_{fs} and f_c values of mortars activated with Na_2SiO_3 cured in wet and dry conditions. According to these correlations, the equations are proposed to calculate f_{fs} values of mortars activated with Na_2SiO_3 cured in wet and dry conditions from the f_c values of mortars activated with Na_2SiO_3.

• It can be concluded that using Na_2SiO_3 as activator conduce higher bond strength in the interface zone between the crushed limestone sand and cementitious matrix.

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