Effect of Reactive Metakaolin Prepaird by Thermal Shock on Some Mechnical Properties of Self-Compacting Concrete

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Submission date:- 8/12/2015	Acceptance date:- 12/1/2016	Publication date:-15/7/2019

Abstract

Metakaolin is a very active alumino-silicate pozzolan commonly used recently in many applications of concrete. In this research Metakaolin was used in production of self-compaction concrete with (20%) replacement of cement by weight. It was prepared by burning natural kaolin Clays at different temperatures (700, 750 , 850) Cº using thermal shock method and for periods of time (0.5 , 1 , 2) hours. Fresh self-compacting concrete (SCC) properties, some of mechanical properties were tested at age (28, 56, 90) day. The findings demonstrate that the fresh properties of SCC are affect by the change of temperature and duration of calcining slightly, while the compressive strength enhanced with increasing of temperature and duration of calcining. The best result is obtained at degree of 850 Cº and one-hour calcining duration, while the strength development at (56 day) age was (15.6%) and at (90 day) age was (26.7%). A better enhancement outcome in flexural strength and ultra-sonic plus velocity were at the same temperature and duration of calcining, the development of modulus of rupture at (56 days) was (7%) and at (90 days) it was (15.5 %), while the ultra-sonic plus velocity reached at the age of (28 days) (4.52 km\sec).

Keyword: Self-compacting concrete, Metakaolin, Mechanical properties, Thermal shock calcining.

1- Introduction

Metakaolin (MK) is defined as a reiterated kaolin clay, which is treated with high temprature under neatly controlled situations to make an amorphous aluminosilicate, which is active in concrete. The silicate structure layers in Kaolin are binding together via the Van Der Weal's bond, in which OH- is tied strongly. Metakaolin acts with Ca (OH) 2, which produced during the cement hydration similar to other pozzolans [1].

In ordinary environmental conditions, kaolin is totally stable. It loses about 14% of its weight in bound hydroxyl ions when it heated to temperatures of 650-900 °C. The process of calcination falls down the kaolin structure, so the silica and alumina layers creased and lose their long-term arrangement. The MK will have a highly reactive transition phase due to disorder and Dehydroxylation. It is an amorphous pozzolan, with potential hydraulic characteristics, which is suitable for use as a cementitious material [2]. The process of calcining kaolin may be illustrate by the following equation:

$$Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O \rightarrow Al_2O_3 \cdot 2SiO_2 + 2H_2O\uparrow$$

Kaolin metakaolin

The kaolinite thermal transmutation has shown that the calcining factors like heating rate, temperature, and time, as well as cooling factors (cooling rate and ambient conditions), obviously effect the process of dihydroxylation [3].

The pozzolanic activity is the prime property of the produced metakaolin for the cement-based system uses. It is defined as the material capability to interact with calcium hydroxide in water existence to produce components with cementitious properties. The low dense CH phase and big pores turns into denser C-S-H and smaller pore by Pozzolanic reaction. The following equations shows the pozzolanic reaction [4]:

$$OPC + H_2O \rightarrow CSH + CH \qquad \textit{cement hydration process}$$

$$AS_2 + CH \rightarrow C_2ASH_8 + CSH \qquad \textit{pozzolanic reactivity process}$$

Metakaolin show adequate engineering properties including the pozzolanic activity and the filler effect. The filler effect is prompt; on the other hand, the pozzolanic action effect happens between 3 to 14 days. Compressive and flexural strengths improve by metakaolin addition. Also, it reduces shrinkage, permeability, and efflorescence, however it has a positive effect on durability and chemical attack resistance [5].

European Guidelines define SCC as an innovative concrete, which need no vibration for compaction and placing. It has the ability of flowing under its own weight, totally filling a formwork with attaining complete compaction, although of the crammed reinforcement existence. The resultant concrete is uniform, intensive, and has similar durability and properties as conventional compacted concrete [6].

The using of superplasticizers is necessary in producing SCC to obtain high flowability. Segregation may be occurred; however, it can be avoided by the addition of high percent of powder materials or admixtures for viscosity modifying [7].

2- Objective

The research investigated the effect of calcined temperature degree and duration by the thermal shock on performance of metakaolin in SCC

Experimental Work

1. Materials

1-1 Cement

Ordinary Portland cement (ASTM C150-type I) [8] complies to Iraqi standards (IQS No.5/1984) [9]. It was known as (MASS-BAZIAN) manufactured by mass holding group in Al-Sulaymaniyah city. Table (1) and Table (2) demonstrate the chemical composition and physical properties respectively.

Table (1) Chemical Composition of Cement

Oxide composition	Abbreviation	Content (percent)	Limits of Iraqi Specification No.5/1984
Calcium oxide	CaO	64.91	-
Silicon oxide	SiO_2	20.71	-
Aluminum oxide	Al_2O_3	5.10	-
Ferric oxide	Fe_2O_3	3.23	-
Magnesium oxide	MgO	2.24	≤ 5.0%
Sulphur trioxide	SO_3	2.52	≤ 2.8%
Alkalis	$Na_2O_3 + K_2O$	1.01	-
Loss on Ignition	L.O.I	0.76	≤ 4.0%
Insoluble residue	I.R	0.87	≤1.5%
Tri-calcium Silicate	C_3S	60.71	-
Di-calcium Silicate	C_2S	13.66	-
Tri-calcium Aluminate	C_3A	8.06	-
Tetra-calcium Alumino-Ferrite	C_4AF	9.82	-

Table (2) Physical Properties of Cement

Physical properties	Test results	Limits of Iraqi Specification No.5/1984
Fineness (Blaine method), m^2/kg	361	≥ 230 m^2/kg
Setting time (Vicat’s apparatus) Initial setting time, hrs: min. Final setting time, hrs: min.	 1:15 3:49	 ≥ 45 min ≤ 10 hrs
Compressive strength of mortar 3days, MPa 7days, MPa	 25 31	 ≥ 15 ≥ 23

1-2 Metakaolin

It was prepared by calcining the local material “kaolin clay” using thermal shock at different temperatures (700,750,850) Co and various durations (0.5, 1, and 2) hours to find out the effect of temperature and duration on metakaolin and therefore its effect on properties of SCC. After calcining, metakaolin was gridding in a ball mill for two hours. Table (3) illustrates the chemical composition of metakaolin, which match the requirements of (ASTM C618-05) [10].

***Preparing Metakaolin by thermal shock*

Preparing Metakaolin was done by switching on the burning oven and waiting it to reach the required thermal shock temperature which are (700,750, and 850) Co + 30Co, taking into consideration the temperature drop when opening the oven. The oven is opened and the kaolin sample is put as soon as possible then the sample is left for the specified period (1/2 , 1 , and 2) hr, after that the oven is switched off and the sample is left to cool to room temperature.

Table (3) chemical composition of Metakaolin

Oxides	Content (percent)	Limits of ASTM C618-05
SiO_2	66.24	≥70
Al_2O_3	18.32	
Fe_2O_3	0.14	
Na_2O_3	0.15	-
K_2O	0.20	
CaO	6.03	
MgO	0.87	≤ 5.0%
SO_3	0.24	≤ 4
L.O.I	5.32	≤ 10

1-3 Superplastizer

In current investigation Glenium 51 is used. It is based on modified polycarboxylic ether to increase workability. It suits (ASTM C494-05) type A & F [11]. Table (4) shows the technical description of it.

1-4 Sand

Al-Ukhaider region is the source of natural sand which is used in this research. The specific gravity and SO3 content are 2.60 and 0.07 respectively. Table (5) shows its gradation, which complies to Iraqi specification no.45/1984 [12].

Table (4) Technical description of the Glenium 51#

Technical description	Properties
Appearance	Viscous liquid
Color	Light brown
Relative density	1.1 @ 20 C°
Transport	Not classify as dangerous
pH value	6.6
Labeling	No hazard label required
Dosage*	0.5-1.6 liters per 100 kg of cement
Viscosity	128±30 CPS @ 20 C°

** Dosages outside this range are permissible with trial mixes.*

 # Properties from product catalogue.

Table (5) Grading of fine aggregate used throughout this work

Sieve size (mm)	Cumulative passing %	Limits of Iraqi specification No.45/1984, zone 3
10.0	100	100
4.75	95	90-100
2.36	89	85-100
1.18	81	75-100
0.60	65	60-79
0.30	19	12-40
0.15	7	0-10
Fineness modulus = 2.42		

1-5 Gravel

Natural rounded cleaned coarse aggregate with (10 mm) maximum size was used. Its grading is shown in Table (5) and Fig. (1) Which meet with the Iraqi standards no.45/1984[12]. The SO3 content and specific gravity of coarse aggregate are 0.05 and 2.64 respectively.

1-6 Water

The mixing and curing of samples were done by using potable water.

2. Concrete mixtures

To study the effect of thermal shock at different temperatures and for different periods, 9 concrete mixes were done, all of them have the same proportions which are shown in Table (6). Only metakaolin was changed according to preparation conditions.

Three degrees of temperature were adopted for calcining kaolin by thermal shock (700 , 750 , and 850), each degree is used for preparing three samples of metakaolin, one sample for each duration of calcining, which were (0.5 , 1 , and 2) hour.

Metakaolin replacement was (20%) by the weight of cement (this percent gives the best results as reported in studies) and w/c was 0.45. A pan mixer is adopted in this investigation. The mixer should keep clean, moist and free from surplus water before mixing. Mixing method is start by placing sand and third of water together in the mixer and mix them for 1½ min. then, another third of water with cement and metakaolin is add and mix for 2½ min. later, two thirds of superplasticizer is added to the last third of water and introduced with coarse aggregate to mixer and mixing last for 2 min. after that, the mixture is left to rest for 2 min. Then, the residual part of the superplasticizer is added and for a moreover 2 min. At last, the mixer is stopped to discharge the mixture for testing fresh properties and casting in molds [13]. Table (7) shows the description of mixes.

Table (6) concrete mixes proportions

substance	Quantity
Cement (kg/m^3)	440
Sand (kg/m^3)	790
Gravel (kg/m^3)	773
MK (kg/m^3)	88
Water (kg/m^3)	198
w/c	0.45
Sp (L/m^3)	6.5

Table (7) concrete mixes description

Temperature of calcining	Mix symbol	Description
٧٠٠	A1	burning for ½ hour
	A2	burning for 1 hour
	A3	burning for 2 hours
٧٥0	B1	burning for ½ hour
	B2	burning for 1 hour
	B3	burning for 2 hours
850	C1	burning for ½ hour
	C2	burning for 1 hour
	C3	burning for 2 hours

3. Testing of fresh concrete

Numerous tests have been invented to assess the features of SCC. The tests used in this research are (slump flow, T50 cm, V-funnel, and L-box) which are explained in European guideline for self-compacting concrete [6].

4. Testing of hardened concrete

4-1 Compressive strength test

The compressive strength test was accomplished according to (B.S.1881: part 116:1983) [14]. The capacity of the testing machine is 2000 kN. A total (81 cube) of (100 mm) were tested, the ages of test were (28, 56, and 90) days.

4-2 Flexural strength test

(100×100×400) mm beam were cast and tested to examine the modulus of rapture at age of testing (28, 56, and 90) days. By using the same compression strength machine with a frame made to distributed load into two points put above the sample. The average of three samples was determined for every mix. This test is done according to ASTM C78-05[15].

4-3 Ultra-sonic pulse velocity test

It is quantifying the propagation of velocity of an ultrasonic wave via concrete. The Portable Ultrasonic Non – Destructive Digital Indicating Tester (PUNDIT) (direct way) was used. A standard cube was used after taking them out from curing tanks; the average of the three samples was determined for (28, 56, and 90) day testing age. The specification which governs this test is (B.S 1881: part 203) [16].

3- Results and Descussion

1. Fresh concrete tests

Table (8) shows the results of fresh tests of SSC, all mixes have a zero VSI (visual stability index) according to ASTM C1611 [17], which can be obtained by visual examination of a slump flow patty forthright after it stops flowing. All mixes were identified with the requirements of SCC with good consistency and workability.

The results show that there is a sparse variation in the results of fresh concrete mixtures of all tests; accordingly, thermal shock and calcining time effect slightly the fresh properties of SCC. Figures (2) to (5) show the relations between calcining temperature and parameters of fresh SCC tests.

Table (8) results of fresh self-compacting concrete tests

Mix symbol	Slump flow		V-funnel (sec)	L-box (H_2/H_1) %
	D (mm)	T_{50} (sec)		
limitation	**600 - 800**	**2 – 5**	**5 – 15**	**80 - 100**
A1	750	3.1	8.8	84
A2	748	3.5	9.1	84
A3	745	3.7	9.2	83
B1	752	2.8	8.5	82
B2	747	3.0	8.7	82
B3	745	3.3	9.0	82
C1	748	3.0	8.5	83
C2	744	3.1	9.0	82
C3	744	3.3	9.3	83

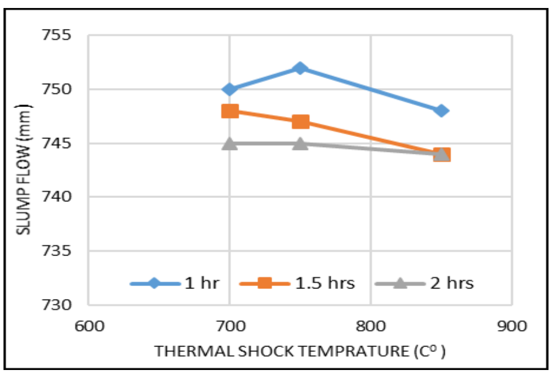


Fig (2) relations between thermal shock Temp. And slump flow diameter

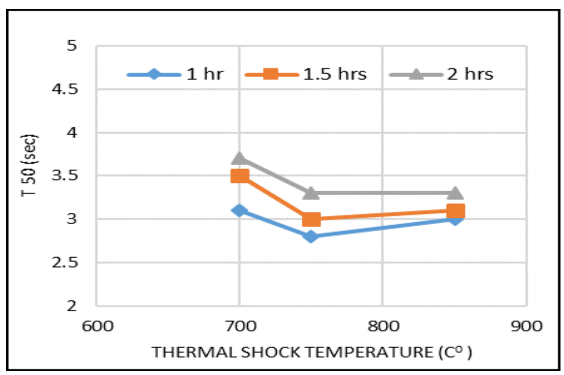
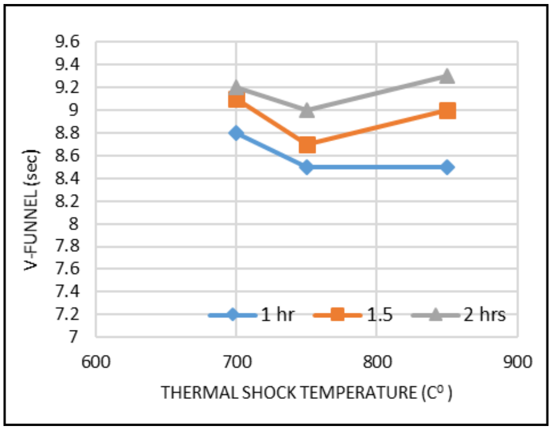


Fig (3) relations between thermal shock temp. and T50cm



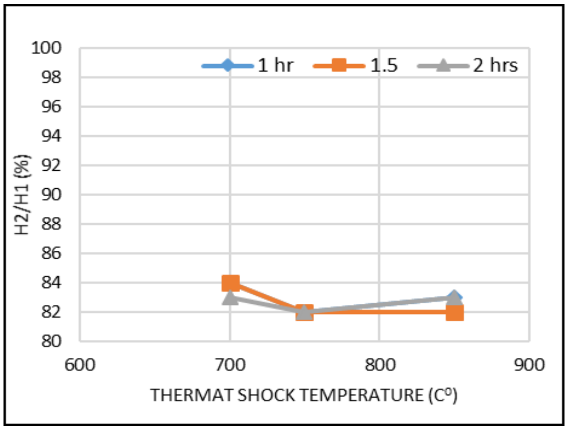


Fig (4) relations between thermal shock temp. and v-funnel time

Fig (5) relations between thermal hock temp. and H2/H1 percent

2. Hardened concrete tests

2-1 Compressive strength test

Table (9) listed the results of compressive strength test; Mix (C2) records the remarkable result in strength and in strength development. With the increase of thermal shock temperature and the duration of calcining, compressive strength increased for all mixes except (C3) mix where the metakaolin was calcining in 850 C and for two hours.

Table (9) results of compressive strength tests

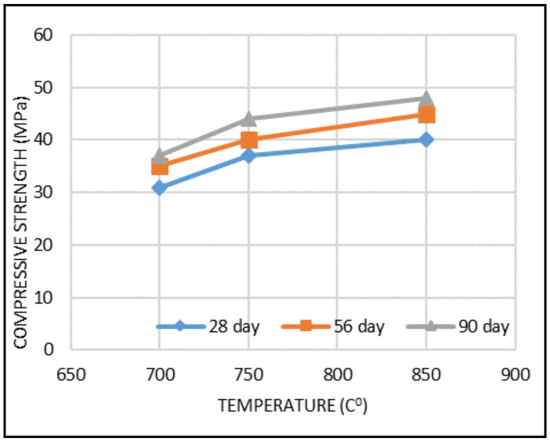
Mix symbol	Compressive strength (Mpa)		
	28 day	56 day	90 day
A1	31	35	37
A2	35	39	43
A3	38	40	43
B1	37	40	44
B2	40	46	50
B3	41	43	46
C1	40	45	48
C2	45	52	57
C3	42	44	46

This decrement in compressive strength may be attributed to the notion that the re-crystallization of metakaolin due to the extended exposure to temperatures above the de-hydroxylation degree and that the mullite ($3Al_2O_3 \cdot 2SiO_2$) formation taken place results in loss of the pozzolanic activity [4]. The activity of lime with calcined kaolin count on the quasi-amorphous nature of the break down structure. Thus, a maximum temperature of calcinations founds for every clay, re-crystallization begins at temperatures above the maximum, while the clay lattice structure is still intact at temperature below the maximum. On other hand the increasing in strength in other mixes belong to the tiered effect of metakaolin by calcining, and the pozzolanic activity with calcium hydroxide emitted from hydration of cement. The filling effect in the voids between cement and other powder particles meliorates the concrete strength [18].

Figs (6), (7) and (8) show the relations between thermal shock temperature and compressive strength for ½ , 1 and 2 hours calcining respectively.

2-2 Modulus of rapture test

Table (10) shows the results of flexural strength test, the modulus of rapture increased with the increasing of thermal shock temperature and the increase of calcined duration. This effect is due to good microstructure of SCC, which leads to the reduce of porosity and to better distributions for pores through the tradition zone. When calcining time of metakaolin increases, it becomes more soft and viscous. While at later age, the pozzolanic reaction appears which releases cement hydration products that fills voids between cement particles. The long and high temperature calcining make metakaolin an inert material and behave just like a filler and this effect is shown in (C3) mix. Figs (9), (10) and (11) show the relations between thermal shock temperature and flexural strength for ½, 1 and 2 hours calcining respectively.



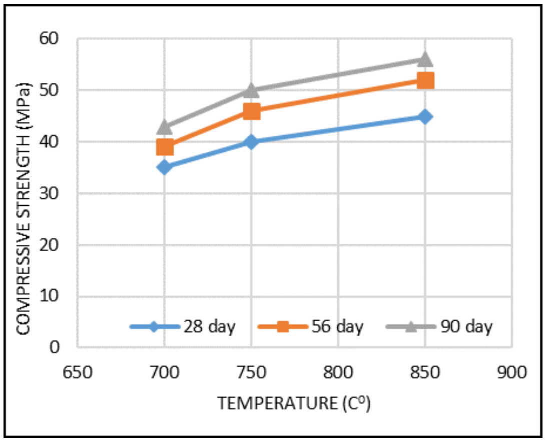


Fig (6) relations between thermal shock temp. and compressive strength for ½ hr calcining

Fig (7) relations between thermal shock temp. and compressive strength for 1 hr calcining

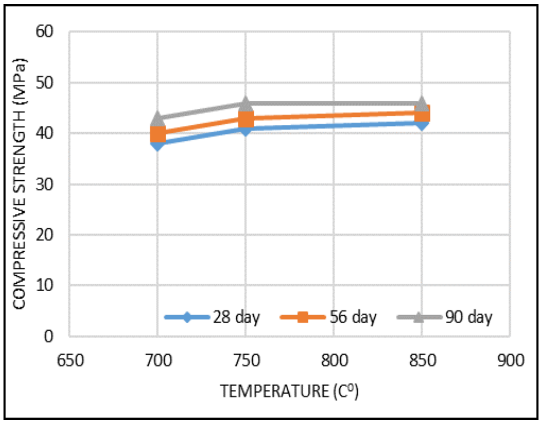


Fig (8) relations between thermal shock temp. and compressive strength for 2 hr calcining

Table (10) results flexural strength tests

Mix symbol	Flexural strength (Mpa)		
	28 day	56 day	90 day
A1	4.06	4.21	4.30
A2	4.69	4.82	5.21
A3	5.17	5.30	5.44
B1	4.81	5.04	5.30
B2	5.64	5.91	6.33
B3	5.82	5.93	6.24
C1	5.42	5.72	6.19
C2	6.26	6.70	7.23
C3	5.10	5.15	6.02

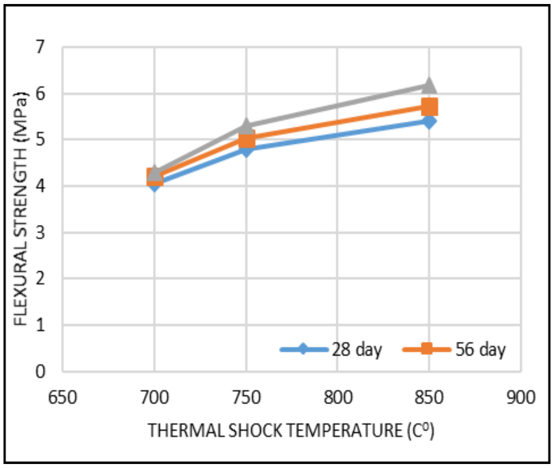


Fig (9) relations between thermal shock temp. and flexural strength for ½ hr calcining

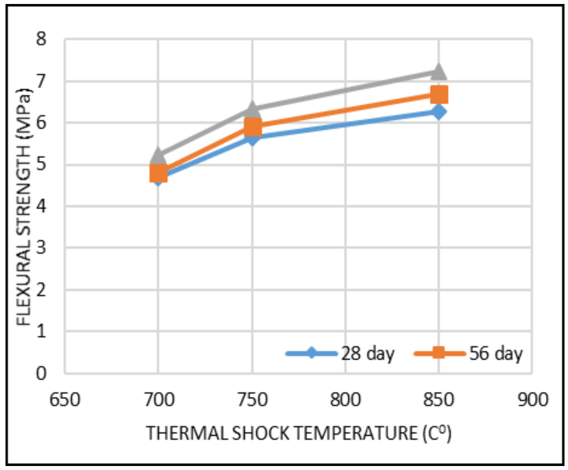


Fig (10) relations between thermal shock temp. and flexural strength for 1 hr calcining

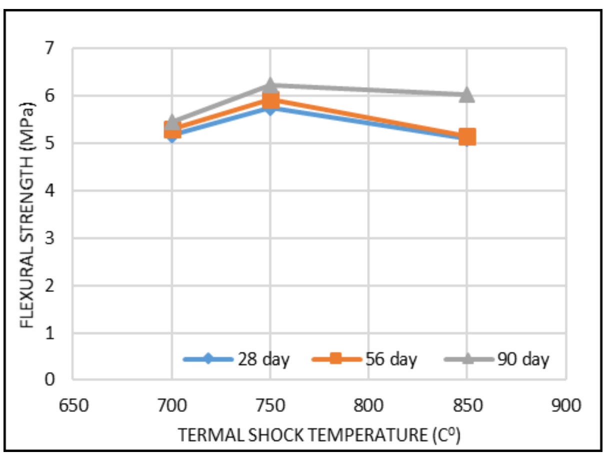


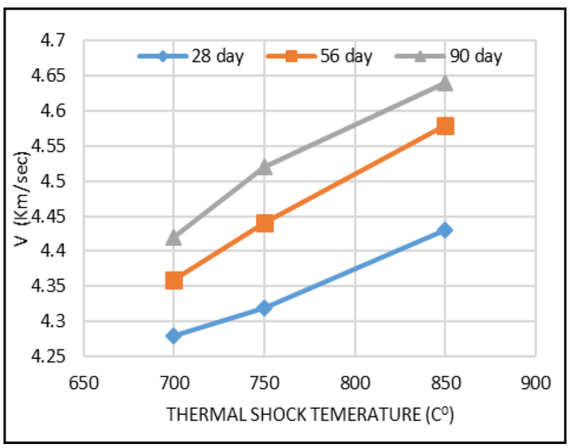
Fig (11) relations between thermal shock temp. and flexural strength for 2 hr calcining

2-3 Ultra sonic plus velocity test

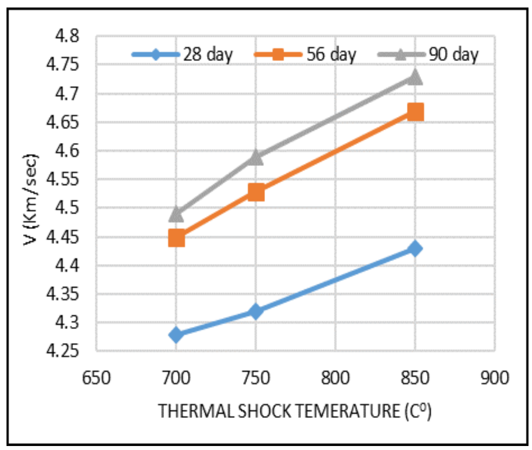
The results of ultra-sonic plus velocity test are shown in Table (11). The velocity increased with exacerbated of thermal shock temperature and duration of calcining because of the pozzolanic reaction of metakaolin. Metakaolin like other filler materials enhances the territory between aggregate and cement-powder paste. The ultra-sonic velocity increased with age of SCC due to continuous hydration of cement and metakaolin, which improves the density of SCC samples. Figs (12), (13), and (14) show the relations between thermal shock temperature and ultra-sonic plus velocity for ½, 1 and 2 hours of calcining respectively.

Table (11) results ultra-sonic plus velocity

Mix symbol	V (Km/sec)		
	28 day	56 day	90 day
A1	4.28	4.36	4.42
A2	4.31	4.47	4.49
A3	4.43	4.48	4.56
B1	4.32	4.44	4.52
B2	4.35	4.53	4.59
B3	4.46	4.53	4.57
C1	4.43	4.58	4.64
C2	4.52	4.67	4.73
C3	4.48	4.55	4.55



Fig(12)relations between thermal shock temp. and ultra-sonic velocity for1/2 hr calcining



Fig(13)relations between thermal shock temp. and ultra-sonic velocity for1 hr calcining

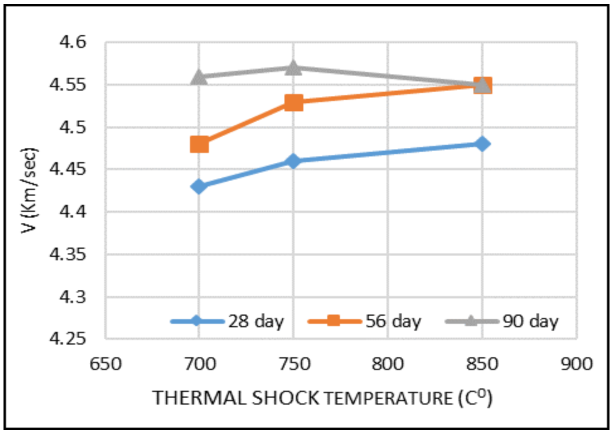


Fig (14) relations between thermal shock temp. and ultra-sonic velocity for 2 hr calcining

Conclusion

1. The fresh properties of SCC are slightly lessen with the increase of thermal shock temperature and duration of calcining.
2. The best compressive strength and strength development occur when calcining kaolin is at 850C° for 1 hour.
3. The behavior of compressive strength was similar to that mentioned in (1) in flexural strength and ultra-sonic plus velocity.
4. The kaolin converted to metakaolin with good pozzolanity effect at 850 C° shock thermal temperature and 1-hour calcining duration.

Conflict of Interests.

- There are no conflicts of interest.

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الخلاصة

يعتبر الميتاكؤولين من المواد البوزولانية (الومينوسيليكات) العالية الفعالية التي شاع استخدامها مؤخراً في العديد من التطبيقات الخرسانية. في هذا البحث استخدم الميتاكؤولين في انتاج الخرسانة ذاتية الرص بنسبة استبدال (٢٠ %) من وزن الاسمنت حيث تم تحضير الميتاكؤولين من اطيان الكاؤولين الطبيعية بحرقها بدرجات حرارة مختلفة (٧٠٠، ٧٥٠، ٨٥٠) درجة سيليزية وباستخدام اسلوب الصدمة الحرارية وبفترات زمنية (٠،٥، ١، ٢) ساعة. تم فحص الخواص الطرية للخرسانة ذاتية الرص ومقاومة الانضغاط والموجات فوق الصوتية ومقاومة الانثناء للأعمار (٢٨، ٥٦، ٩٠) يوم ودراسة تأثير الميتاكؤولين المحضر بالمتغيرات المذكورة اعلاه على هذه الخواص.

اظهرت النتائج ان الخواص الطرية للخرسانة ذاتية الرص تتأثر بصورة طفيفة بتغير درجة الحرارة وفتره الحرق اما فيما يتعلق بمقاومة الخرسانة ذاتية الرص فإنها تزداد مع زيادة درجة حرارة ومدة الحرق وان أفضل نتيجة تم الحصول عليها كان بدرجة حرارة ٨٥٠ م° ومدة حرق مقدارها ساعة، حيث بلغ تطور المقاومة بعمر ٥٦ يوم (١٥،٦ %) وبعمر ٩٠ يوم (٢٦،٧ %). كذلك أفضل نتائج في مقاومة الانثناء والموجات فوق الصوتية تم الحصول عليها في نفس درجة الحرارة ومده الحرق حيث بلغ التطور في مقاومة الانثناء بعمر ٥٦ يوم (٧ %) وبعمر ٩٠ يوم (١٥،٥%)، وبلغت سرعة الموجات الصوتية بعمر ٢٨ يوم (٤،٥٢) كم/ثا.

الكلمات الدالة: الخرسانية الذاتية الرص، الميتاكاؤولين، الخواص الميكانيكية، الحرق بالصدمة الحرارية.