Investigate about the Iraqi Attapulgite Clay as a Mineral Admixture for Concrete

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ABSTRACT

Tremendous research efforts have been directed to investigate the possibility of using the Iraqi Attapulgite clay as a mineral admixture to improve some properties of concrete. To investigate about this clay as a pozzolan, many experimental work need to be done to find the suitable conditions of temperature and time of calcinations. To study the influence of calcinations temperature, different samples of Attapulgite were prepared.

The samples were grinded to fineness 2109 m^2/ kg , then burned to (550 , 600 , 650 , 700 , 750 , 775 and 800)˚C at 1/2 hour , respectively . According to the American Standard Specifications, the strength activity index was conducted on the cubic specimens with dimensions (50 * 50 * 50) mm. The results showed that the optimum calcinations temperature was 750 ˚C. The Attapulgite samples were prepared at (1/2 , 1 , 1 1/2 , 2) hours , respectively and the temperature was 750 ˚C , then strength activity index was conducted . The results showed that the optimum calcinations time was 1/2 hour.

Keywords: Concrete, Mineral admixture, Attapulgite

التحري عن أطيان الأتابلكايت العراقية كمضاف معدني للخرسانة

الخلاصة

بذلت جهود بحثية كبيرة للتحري عن إمكانية استخدام أطيان الاتابلكايت العراقية كمضاف معدني لتحسين بعض خصائص الخرسانة . وللتحري عن مدى صلاحية هذه الأطيان وملاءمتها كبوزولانا ، لابد من القيام بتجارب مختبرية لإيجاد الظروف المناسبة من درجة حرارة وزمن حرق لإنتاج اتابلكايت عالي الفعالية . لدراسة تأثير درجة حرارة الحرق تم تحضير نماذج مختلفة من الاتابلكايت وحرقها بدرجات حرارة (550 ، 600 ، 650 ،700 ، 750 ،775 و 800) °سيلسيوس على التوالي ولمدة 1/2 ساعة وبعدها تم إجراء فحص مؤشر الفعالية لمكعبات من مونة الاسمنت بإبعاد (50 * 50* 50) ملم طبقا للمواصفات الامريكية القياسية . وقد أظهرت النتائج بان درجة حرارة الحرق المثلى هي 750 °سيلسيوس .

بعدها تم تحضير نماذج من الاتابلكايت المحروق بدرجة 750 °سيلسيوس وبأوقات مختلفة من أزمنة الحرق (1/2 ،1 ، 1 1/2 ، 2) ساعة على التوالي ومن ثم إجراء فحص مؤشر الفعالية لهذه الخلطات . أظهرت النتائج بان افضل زمن للحرق هو 1/2 ساعة

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INTRODUCTION:

The construction industry has taken considerable strides for word over last two or three decades with regard to many materials, in particular pozzolansmaterials. Most of pozzolans used in the world today are by products from other industries , such as coal fly-ash ,blast furnace slag , rice husk ash , silica fume…... etc. As such, there has been relatively an investigation done with regard to manufacture a new pozzolanic material. Attapulgite is a fibrous clay mineral as kind of crystalloid hydrous Magnesium – Aluminum silicate mineral and have chemically absorbed water with in its structure. Attapulgites form at the surface of the earth with low temperature of clay environments , hence they are classified as clays Velde 1995[1], the needle of Attapulgite are formed by the structural linkage of tetrahedral – octahedral strips . The chemical form of Attapulgite introduced by Carrol 1970 [2]as: $\mathbf{Si_8Mg_5O_{20}(OH)_2(OH_2)_4.4H_2O}$

In Al-Najaf and Karbala regions, Attapulgite dominates clay mineral Mudstones of the Injana formation. Injana formation (late Miocene – Pliocene) are exposed in Al-Najaf region (Tar Al-Najaf) as bluish green and gray clay stone, 0.5 m thick with plants remains [3]. From the raw material which is available in the country in large amounts, after grinding Attapulgite in a high fineness , then burning it in a high temperature between (550 to 800)˚C , the optimum burning temperature will be determined through the test of S.A.I . Neville and Brooks [4] showed the mechanism of pozzolanic reaction in concrete , where if the pozzolanic admixtures presence in concrete , it will cause to strengthening the transition zone between the cement paste and aggregate which contains the voids more than cement paste (higher porosity) . Where the pozzolanic admixtures works as a filler of this voids , because it has a high fineness and produces the additional gel by its reaction with $Ca(OH)_2$. This is the important agent in strengthening the transition zone between cement paste and aggregate. Pozzolanic materials react with calcium hydroxide which is generated by the hydration of main compounds of cement (C_3S and C_2S).

Aitcin [5] showed that the use of calcinated clays, in addition to Vander Wales bonds and same chemical bonding at later ages, contributes to the transition zone strength. The additional products from gel will reduce the size and total volume of voids, especially the transition zone. A study was made by Al-Hadithi [6] to convert the local kaolin clays to high reactivity metakaolin (HRM). Several tests were made to determine the most suitable burning temperature to produce the HRM. The suitable percentage of HRM as a partial replacement by weight of cement was investigated. The results demonstrated that the optimum burning temperature to convert the kaolin clay in to HRM is 700˚C, and the most suitable percentage of HRM as a partial replacement by weight of cement is 8%. Al–Naeemi [7] showed in her research about the effect of addition (The QatarianAttapulgite) on the mechanical properties of Portland cement, using this mineral after grinding it in a limit of fineness, burning it at 600˚C and using water to cementitious ratio 0.4. The percentages of replacement were 5 % , 10 % , 15 % , 20 % , 25 % , 30 % , 35 % , 40 % , 45 %, 50 % , 70 % and 80 % by weight of cement .The author suggests that the suitable working ratios for production of blended cement using ordinary Portland cement and the Qatarianattapulgite clay activated at 600˚C are 10-35% burnt clay**.**

Experimental Work

Different mortar mixes were prepared for this research through the selection of materials available locally. The Attapulgite was used as a mineral pozzolanic admixture so as to improve the properties of concrete later.

Materials:

Cement:

Ordinary Portland cement manufactured by AL - Mass cement factory was used in the investigations. It was stored in air-tight plastic containers to avoid the effect of atmospheric conditions and to maintain constant quality. The chemical composition and physical properties of the cement are shown in Tables 1 and 2, respectively. The results are conformed to the Iraqi specifications No. 5/1984 [8].

Table 1 * Chemical composition and main compounds of the cement

Oxide Composition	Abbreviation	Percentage byweight	Limit of Iraqi Specification No. 5/1984
Lime	CaO	61.89	-
Silica	SiO_2	21.37	-
Alumina	Al_2O_3	4.60	-
Iron Oxide	Fe_2O_3	3.35	-
Sulphate	SO_3	2.42	≤ 2.8 %
Magnesia	MgO	3.05	≤ 5.0 %
Potash	K_2O	0.36	-
Soda	Na_2O	0.27	-
Loss on ignition	L.O.I.	2.16	≤ 4.0 %
Insoluble residue	I.R.	0.60	≤ 1.5 %
Main Compounds (Bogue′s equations)[4]			
Tricalcium Silicate	C_3S	46.88	-
Dicalcium Silicate	C_2S	26.17	-
Tricalcium Aluminate	C_3A	6.53	> 5.0 %
Tetracalcium Aluminate -Ferrite	C_4AF	10.18	-

* Chemical test of cement was carried out by the State Company of Geological Survey and Mining

Table 2 * Physical properties of cement

Physical properties	Test results	Limits of Iraqi Specification No. 5/1985
Specific surface area (Blaine method) (m^2/ kg)	321	≥ 230
Soundness (Le–Chateliermethod)(mm)	0.5	< 10
Setting time (Vicat′s method)		
Initial setting (hrs : min)	1 : 55	≥ 1 hr
Final setting (hrs : min)	2 : 24	≤ 10 hrs

Compressive strength (MPa) **3 days** **7 days**	23 29	≥ 15 ≥ 23

*Physical tests of cement were made by Structures Laboratory in University of Qadisiyah

Standard Sand:

Standard sand used in the strength activity index test of mortar conformed to the properties of standard sand [9] .

1- Maximum limit passing from sieve (600μm) is 10%.

2- Minimum limits passing from sieve (850 μm) is 98%.

3- Maximum limit of the loss of the hot hydrochloricpanning is0.25%.

4-The source of standard sand user is north of Hussainiyat quarry.

High Reactivity Attapulgite (HRA):

The raw material for the production of active Attapulgite is Attapulgite clays. Attapulgite from Tar AL-Najaf (Injana) region in AL- Najaf province was used to produce high reactivity Attapulgite.

Preparation of local (HRA):

The raw material of Attapulgite contains rocks grinded in the Building Research Center/Ministry of Science and Technology (in AL- Jadiriyah - Baghdad) in a manner of storming and transforming it to the powder that has a high fineness for the purpose of making the most of their effectiveness. Table 3 shows the chemical analysis of Attapulgiteaftergrinding, and Table 4 gives physical properties of Attapulgite.

Table 3 * Chemical analysis of Attapulgite after grinding

Oxide Composition	Oxide content (%)
SiO_2	51.8
Al_2O_3	8.99
Fe_2O_3	4.88
TiO_2	0.58
CaO	7.11
MgO	5.52
SO_3	0.68
Na_2O	1.01
K_2O	1.8
L.O.I	18.65

*Chemical analysis of Attapulgite after grinding was carried out by the State Company of Geological Survey and Mining

Table 4 Physical properties of Attapulgite

Property	Description
Specific gravity	2.34

*** Fineness (by Blaine method m^2/ kg)**	2109
Color	Bluish green

*Fineness of theAttapulgite was determined by Blaine air permeability method in accordance with ASTM C204-84 [22]

Determination of the Suitable Calcinations Temperature of Attapulgite:

When the Attapulgite burns, a certain degree of temperature turns to activate pozzolanic material. The pozzolanicity of calcined clay is achieved by driving of the water molecules. This process results in the formation of amorphous material which is reactive with lime [10]. The investigation about the suitable calcinations temperature is by using a laboratory oven of a maximum temperature 1200˚C. The rate of temperature was (2˚C/ min), and when the oven temperature reaches to the required degree, the sample stills for $\frac{1}{2}$ hour in this temperature. Then, the cooling phase of the model starts gradually by opening the oven door very slightly to allow heat exchange with the laboratory temperature to the next day[11]. The Attapulgite samples burned to the temperatures ranged between (550-800) ˚C.When heat is firstly applied, the absorbed water is removed and, as the temperature increased, the interlayer and hydrated water removed too [12]. Table 5 shows the chemical analysis of Attapulgite after burning.

Table 5 * Chemical analysis of Attapulgite after burning in 750˚C

Oxide Composition	Oxide content (%)
SiO_2	60.48
Al_2O_3	13. 95
Fe_2O_3	6.07
TiO_2	0.74
CaO	8.46
MgO	5.92
Na_2O	1.2
SO_3	0.45
K_2O	2.47
L.O.I	0.1

*Chemical analysis of Attapulgite after burning was carried by the State Company of Geological Survey and Mining

The reactivity of the calcined clay with lime depends on the amorphous nature of the collapsed structure. Therefore, an optimum calcinations temperature exists for each clay, at temperature beyond the optimum, re-crystallization begins, while at temperature below the optimum, the clay lattice structure is still intact [13]. The results of chemical analysis of Attapulgite after burning refers to increase in the SiO_2 percent and other oxides percentage. This increase is because of the absence of L.O.I after burning, where the L.O.I brews of water within clays and feldspar and CO_2 within calcite and dolomite [14]. The X-Ray diffraction analysis of Attapulgite after burning, indicates that the peak of Palygoriskite still exists in spite of, its crawl toward of theright hand of diffraction angle axis. Where the increase in 2ɵ in the diffraction angle axis refers to presence of the Illite mineral which has a high thermal resistance reaching to 900˚C , while the Palygoriskite mineral destroyed below 800˚C [15].

Strength Activity Index of High ReactiveAttapulgite:

Control and test mixture were prepared for strength activity index, for all mixes, the cement or cementations materials (C+ HRA) to fine Aggregate ratio is 1: 2.75 mix proportions as described in ASTM C311 – 02 [16] for the test mixture at (550 , 600 , 650 , 700 , 750 ,775 & 800)˚C .

Due to ASTM C311 – 02 [16] , cementations material contained 20 % HRA (for each sample) as a partial replacement by weight of cement , and water for the control mix was 242 ml and all mortars for the test mixture , water selected or required for flow was ± 5 of control mix, as shown in Table 6. Mixing of mortar was carried out by a small laboratory mortar mixer due to ASTMC109/C109 M-02 [17]. The test method for flow of cementations mortar is due to ASTMC1437-01[18]and ASTM C230/C230M-03[19].

For each type of mortar, three 50 mm cube specimens were molded. After 24 hours from casting, the specimens were demolded carefully, marked and immersed in tap water until the time of testing.

According to ASTM C311– 02[16] , the strength activity index (S.A.I) , Table 7 , with Portland cement was determined as follows :

$$S.A.I = A / B * 100 \qquad \ldots(1)$$

Where:

A : Average compressive strength of test mixture cubes (MPa) .

B : Average compressive strength of control mix cubes (MPa) .

$$\text{Water requirements\%} = (Y/242) * 100 \qquad \ldots(2)$$

Where:

Y : water requirements for the test mixture ± 5 of control mix flow .

From the results of strength activity index of test of samples, the optimum calcinations temperature of the Attapulgite is 750˚C. Table 8 shows the physical requirements of pozzolan, ASTM C618 – 01 [20].

Determination of the Suitable Time of Calcinations:

The Attapulgite samples were burnt in a controlled temperature oven. Its temperature increased gradually until reaching to 750˚C, then left for a period of ($\frac{1}{2}$, 1, $1\frac{1}{2}$ and 2 hours) in that temperature. The suitable time of calcinations limited by S.A.I test is due to ASTM C311– 02 [16].The requirements of S.A.I shown in Table9, and the results of compressive strength and S.A.I shown in Tables 10.

Table(6) *Strength activity index mixes requirements for Tested Mortar

Sample of mixes	Water content (ml)	w/c or w/cm	Cement content (gm)	HRA content(gm) 20 %by weight of cement	Standard Sand content (gm)	Flow (%)
Control mix	242	0.484	500	-	1375	34.6
Test mix** (HRA/550˚C/1/2hr)	253	0.506	400	100	1375	37.69
Test mix (HRA/600˚C /1/2hr)	252	0.504	400	100	1375	38.46
Test mix (HRA/650˚C /1/2 hr)	254	0.509	400	100	1375	38.90
Test mix (HRA/700˚C /1/2 hr)	255	0.510	400	100	1375	36.5
Test mix (HRA/750˚C /1/2 hr)	254	0.509	400	100	1375	38.1
Test mix (HRA/775˚C /1/2 hr)	255	0.510	400	100	1375	35.95
Test mix (HRA/800˚C /1/2 hr)	256	0.512	400	100	1375	39.12

*****All mixes requirements are according to ASTM C311–02[16]

**Test mix(HRA/ Calcinations temperature (˚C) / Calcinations time (hr))

Table(7) Effect of calcinations temperature on the compressive strength, density, S.A.I and water requirements

Sample of mixes	Density (gm /cm^3)	Compressive strength (MPa) at 7 days	w/c or w/cm	S.A.I %	Water * requirements (%)
Control mix	2.18	16.80	0.484	100	100
Test mix* (HRA/ 550˚C /1/2 hr)	2.15	12	0.506	71.42	104.5
Test mix (HRA/600˚C/1/2 hr)	2.18	14.13	0.504	84.11	104.1
Test mix (HRA/650˚C/1/2 hr)	2.17	14.40	0.509	85.71	104.95
Test mix (HRA/700˚C/1/2 hr)	2.18	14.87	0.510	88.51	105.37
Test mix (HRA/750˚C/1/2 hr)	2.20	19.40	0.509	115.48	105
Test mix (HRA/775˚C/1/2 hr)	2.18	14	0.510	83.33	105.37

Test mix (HRA/800˚C/1/2 hr)	2.16	13.40	0.512	79.76	105.78

*Test mix (HRA / Calcinations temperature (˚C) / Calcinations time (hr))
* Water requirements calculated according to ASTM C311– 02 [16]

Table (8) Physical requirements of pozzolan class N

Property	Pozzolan Class N	Attapulgite(HRA)
S.A.I with Portland cement at 7 days , min percent	75	115.48
Water requirement , max percent	115	105
***Autoclave expansion or contraction , max %**	0.8	0.1
Density , max variation from average	5	1

*The Autoclave test were conducted in accordance with ASTM C151- 00 [23]

Table (9) * Strength activity index mixes requirements

Sample of mixes	Water content (ml)	w/c or w/cm	Cement content (gm)	HRA content (gm)	Standard Sand content (gm)	Flow (%)
Control mix	242	0.484	500	-	1375	34.6
Test mix ** **(HRA/ 750˚C/1/2hr)**	254	0.509	400	100	1375	38.1
Test mix (HRA/ 750˚C/ 1hr)	252	0.504	400	100	1375	38.4
Test mix (HRA/ 750˚C/11/2hr)	250	0.50	400	100	1375	39.6
Test mix (HRA/ 750˚C/ 2hr)	251	0.502	400	100	1375	36.5

***All mixes requirements are according to ASTM C311–02 [16]
**Test mix (HRA / Calcinations temperature (˚C) / Calcinations time (hr))

Table (10) *Effect of time of calcinations on the compressive strength and density

Sample of mixes	Density (gm/cm^3)	Compressive Strength (MPa) at 7 days	w/c or w/cm to give flow ± 5 from flow of control mix	S.A.I % by using HRA by weight of % 20) (cement (1:2.75) mix proportion at age7days
Control mix	2.18	16.80	0.484	100
Test mix* (HRA/ 750˚C/1/2hr)	2.20	19.40	0.509	115.48
Test mix (HRA/ 750˚C/ 1hr)	2.18	14.78	0.504	76.18

Test mix (HRA/ 750˚C/11/2hr)	2.16	12.73	0.50	75.77
Test mix (HRA/ 750˚C/ 2hr)	2.15	11.70	0.502	69.64

*Test mix (HRA / Calcinations temperature (˚C) / Calcinations time (hr))

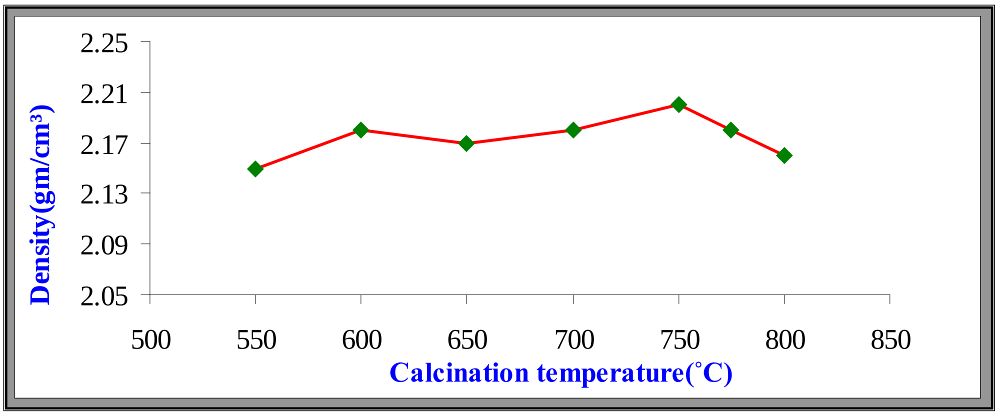
Results and Discussion:

Suitable Calcinations Temperature:

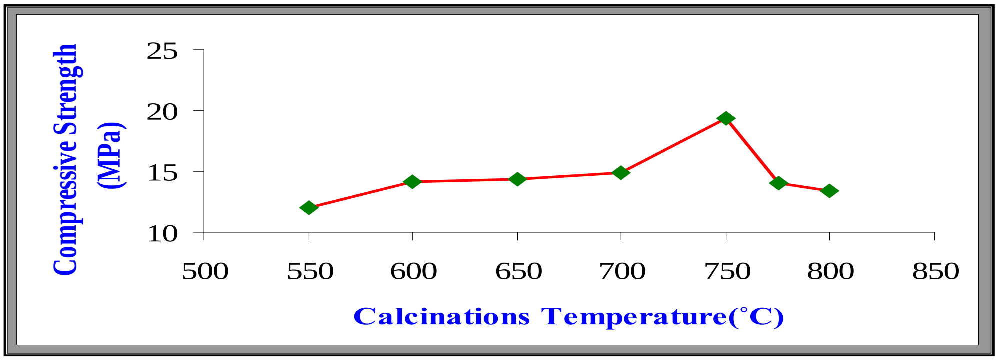
The effect of calcinations temperature on the results of compressive strength of mortar cubes by replacing of HRA with 20% by weight of cement and mix proportion (1: 2.75) is shown in Figs 1 through 3. It is indicated that the optimum calcinations temperature to re-crystallization of the residual amorphous matrix to form high temperature of silicates and oxides of the Attapulgite is 750˚C , where strength activity index (S.A.I) mainly depends on compressive strength. The compressive strength depends on the reactivity of calcined clay with lime which depends on amorphous nature of the collapsed structure [21].

Suitable Time of Calcinations:

Apart from the temperature of calcinations, it is well established that the time of calcinations temperature also influences on the reactivity of the pozzolana. The Attapulgite clay samples were burnt in a controlled temperature furnace so that the temperature increased gradually until reached to 750˚C and left for a period of ($\frac{1}{2}$, 1, 1$\frac{1}{2}$, 2 hours). The results of compressive strength of the mortar cubes at 7days show that thesuitabletimeof calcinationsis $\frac{1}{2}$hour,which has ahigher compressive strength , density and strength activity index (S.A.I). The effect of time of calcinations on the density , compressive strength and strength activity index (S.A.I) at age 7 days is shown in Figs 4 through 6 , respectively.



Figure(1) Effect of calcinations temperature on the density of HRA



Figure(2)Effect of calcinations temperature on the compressivestrength of HRA

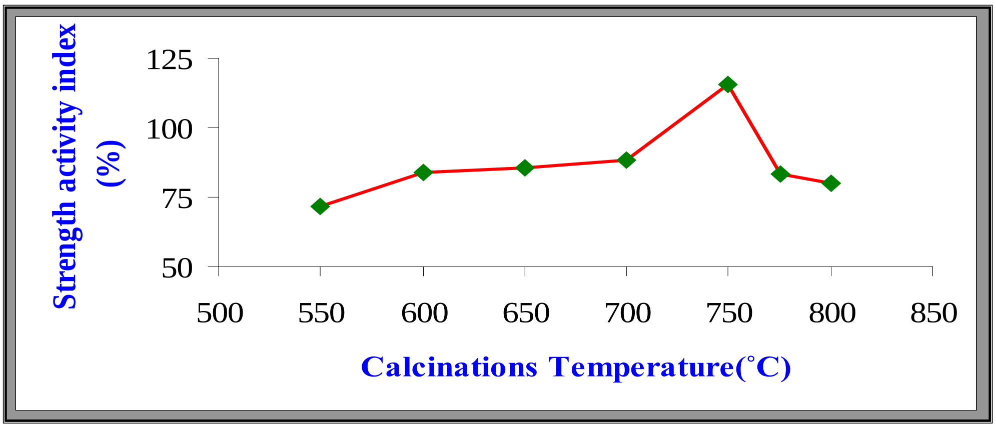


Figure (3) Effect of calcinations temperature on the strength activity index of HRA

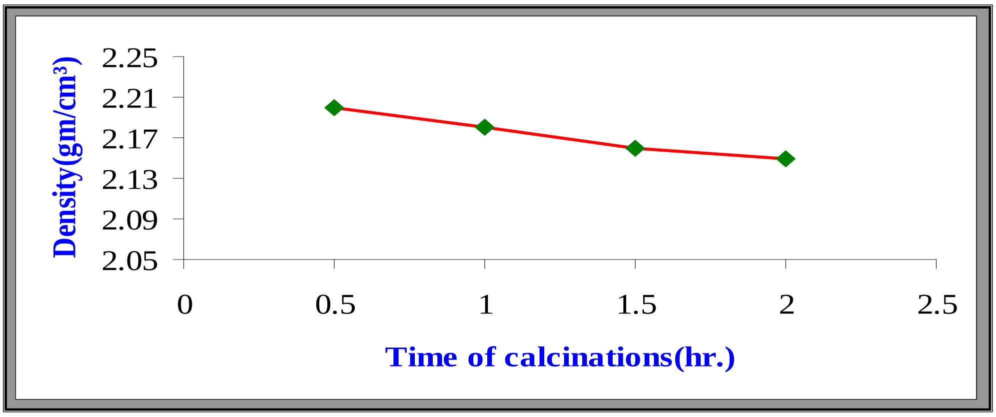


Figure (4)Effect of time of calcinations on the density of HRA

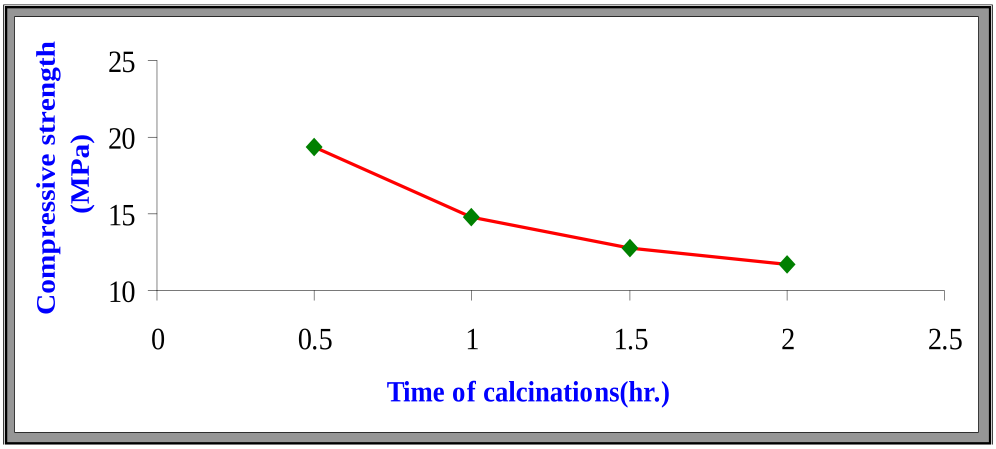


Figure (5)Effect of time of calcinations on the compressive strength of HRA

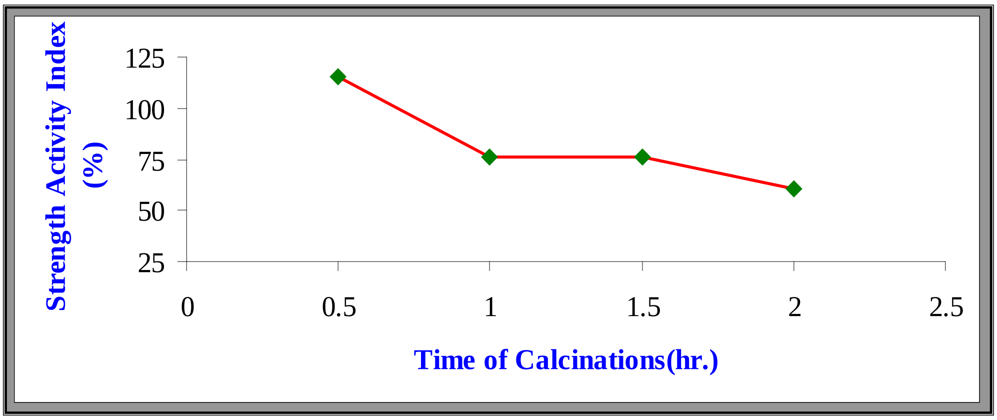


Figure (6) Effect of time of calcinations on the strength activity index of HRA

Conclusions:

Based on the materials, mix proportions, method of testing and the results of these investigations, the following conclusions can be drawn:

1-It is possible to use the Iraqi Attapulgite clay as a pozzolanafterconvertingitto high reactiveAttapulgite.

2-It is possible to prepare high reactivity Attapulgite (HRA) as a pozzolan by burning Attapulgite at the optimum calcination temperature of 750˚C.

3-The optimum time of calcinations is $\frac{1}{2}$ hour .

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