Iraqi bentonite as a natural pozzolan for sustainable concrete

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Research

Abstract

Iraqi high-calcium bentonite was used for the first time to prepare natural pozzolan. Twenty-four calcination programs were studied. The optimum calcination program is 800°C for 90min. The calcined bentonite meets the requirements of the Iraqi specification of pozzolanic materials. The incorporation of calcined bentonite as a part of cementitious binder extends both initial and final setting time in proportion to the percentage of calcined bentonite. In concrete, the calcined bentonite reduces workability and early strength in proportion to the percentage of Portland cement replacement, while promotes the later strength at 5–15% replacement. The retardation effect of calcined bentonite makes it suitable for concrete production in hot climate territories and for mass concrete. Moreover, the relatively low embodied energy and low carbon dioxide emission of calcined bentonite enhance the sustainability of concrete.

1. Introduction

The sustainability of concrete can improve by utilizing pozzolanic materials as a partial replacement of Portland cement [1]. Natural pozzolan is one of the types of pozzolanic materials when used as a part of the cementitious binder; it develops some of the fresh properties, mechanical properties, and/or durability of concrete [2], [3]. Given the absence of naturally formed pozzolans in Iraq and the rarity in industries producing pozzolanic materials as waste, the available alternative is to utilize mineral resources in obtaining natural pozzolan. Iraqi bentonite represents a promising source for natural pozzolan production; its validity for this purpose has not previously been investigated.

Bentonite is a clay that consists dominantly of smectite minerals [4]. In addition to smectite minerals, bentonite may contain multiple species of clay minerals such as palygorskite, kaolinite, illite, or others, and may contain non-clay minerals such as quartzite, gypsum, feldspar, or others [4], [5]. The unique property of smectite minerals is the potentiality to swell due to water absorption [6]. In most cases, bentonite contains a high percentage of montmorillonite mineral, a member of the smectite group. Therefore, bentonite may define as clay consists mostly of montmorillonite mineral [7].

There are numerous allegations about the pozzolanicity of raw bentonite [8]–[14], but these allegations are not supported by conclusive evidence of the occurrence of the pozzolanic reaction between bentonite components and calcium hydroxide resulting from Portland cement hydration [15]. On the contrary, precise examinations have shown that only minor changes in montmorillonite structure occur in highly alkaline environments [16], and a small fraction of montmorillonite may contribute to pozzolanic reaction [17]. On the other hand, the pozzolanicity of both calcined montmorillonite (CM) and calcined bentonite (CB) has been confirmed in several studies [17]–[23].

CB modifies the concrete properties in plastic and hardened states due to both pozzolanic and physical actions. In plastic state, CB incorporation causes a linearly increase in water demand to achieve standard consistency of the paste [23], [24], extends both initial and final setting time [23]–[25], reduces workability

of concrete [26], and increases the dosage of superplasticizer required for self-compacted mortar [27] and concrete [28]. In a hardened state, CB reduces the rate of strength gain due to the low pozzolanic reactivity of CM [21], [29]–[31]. Therefore, CB incorporation leads to a reduction in compressive strength of concrete at early ages, i.e., 21 days or less [8], [9], [25], [26] but the reductions in concrete strength tend to disappear at later ages, i.e., 28–90 days [25], [26], [28]. The most important effect of CB is enhancing the durability of concrete against alkalis-silica reaction [25], sulfates attack [32], acids attack [26], and chlorides penetration [25], [28].

In Iraq, the petroleum industries are the largest consumer of the bentonite. These industries require high swelling bentonite, i.e., sodium bentonite while the bentonite deposits in Iraq contain only low swelling bentonite, i.e., calcium bentonite [33], [34]. Therefore, Iraqi calcium bentonite shall be processed to convert it to high swelling bentonite before using it in the extraction of crude oil. Calcium content is the criterion in determining the validity of calcium bentonite to process and thus converting to high swelling bentonite [35]. On this basis, Iraqi bentonite is classified into two classes [33]–[37]: high grade or low-calcium bentonite in which CaO ≤ 5%, and low grade or high-calcium bentonite in which CaO > 5%. High-calcium bentonite requires complex processes to be converted into high swelling bentonite [33], [36], [37]. Because of this, the producing of the high swelling bentonite from the high-calcium bentonite is not economically feasible, and the utilization of Iraqi bentonite is limited to the low-calcium bentonite only, while high-calcium bentonite considered as useless material. Therefore, the production of pozzolanic material from Iraqi high-calcium bentonite opens up prospects for exploiting mineral resources that are not yet exploited.

2. Materials And Methods

2.1. Materials

2.1.1. Raw bentonite

Bentonite stones were obtained from the Wadi Bashira region in the Western Desert of Iraq. The coordinates of this region are 32° 59' 48.2" N and 39° 44' 53.3" E. Wadi Bashira deposit represents the most important montmorillonitic smectites deposits in Iraq, as it contains reverse of more than 300×10^6 tons [38]. This deposit contains both high-calcium and low-calcium bentonite. The chemical composition of RB is recorded in Table 1.

Table 1
chemical composition of materials.

	OPC	RB	CB (80090)
CaO	63.4	7.4	8.7
SiO_2	21.0	57.6	63.2
Al_2O_3	5.0	13.6	15.6
Fe_2O_3	3.8	6.3	7.6
MgO	2.3	3.2	3.7
Na_2O	0.2	1.1	1.2
SO_3	2.5	1.3	1.6
K_2O	0.5	0.5	0.6
P_2O_5		0.87	0.9
LOI	1.6		
IR	0.3		

2.1.2. Portland cement

Ordinary Portland cement (OPC) conforms to IQS 5/2019 type IQS 5-CEM I 32.5R. The chemical composition of OPC is listed in Table 1, and its physical and mechanical properties are listed in Table 2.

Table 2
physical and mechanical properties of OPC

Property	Result	IQS 5/2019 limit
Compressive strength @ 2 days	19 MPa	10 MPa lower limit.
Compressive strength @ 28 days	34.1 MPa	32.5 MPa lower limit.
Initial setting time	70 min.	45 min. lower limit.
Final setting time	235 min.	600 min. upper limit.
Soundness	0.04%	0.8% upper limit.
Fineness	317 m^2/kg	250 m^2/kg lower limit.

2.1.3. Aggregate

Standard sand meets the requirements of the Iraqi code of practice No. 1/198/2012 was used for making mortar for the pozzolanic activity index test. For concrete production, round graded gravel 14 – 5 mm, and

natural sand in zone IV according to IQS 45/1984 were used.

2.2. Methods

2.2.1. Experimental program

The experimental program included the following sequential steps:

1. Identify the mineral composition of the RB by the XRD technique.
2. Calcination the RB at 700, 750, 800, 850, 900, and 950°C for 30, 60, 90, and 120 min. for each calcination temperature. The nomenclature of each experiment is shown in Table 3. The samples were heating at a rate of 30−40°C/min. until reach to the calcination temperature then kept at this temperature ± 10°C for the calcination period then were removed from the furnace immediately after the calcination was completed, and leave for cooling at ambient temperature. The purpose of fast cooling is to prevent phases' transformations that may occur during slow cooling.
3. Recognize the modifications on crystal phases due to each calcination experiment by XRD technique.
4. Select the probable experiments in which disappear the peaks of clay minerals without formation new crystal phases.
5. Analyse the results of XRD of the probable examinations by quantitative XRD (QXRD) for the determination of the residuals of clay minerals.
6. Decide the optimum calcination program that confirms the maximum pozzolanic activity index at 28 days.
7. Verification of conformity of CB prepared by the optimum calcination program to the requirements of IQS 1748/1992 (Pozzolanic materials for use as a mineral admixture in Portland cement concrete).
8. Examine the effects of partial replacement of OPC by CB at 5, 10, 15, 20, 25, and 30% replacement levels on the properties of the cementitious binder: water requirement, initial and final setting times.
9. Study the effects of partial replacement of OPC by CB at 5, 10, 15, 20, 25, and 30% replacement levels on the properties of concrete: slump and compressive strengths at 7 and 28 days. The details of concrete mixtures are shown in Table 4.

Table 3
calcination programs.

	700°C	750°C	800°C	850°C	900°C	950°C
30 min.	70030	75030	80030	85030	90030	95030
60 min.	70060	75060	80060	85060	90060	95060
90 min.	70090	75090	80090	85090	90090	95090
120 min.	700120	750120	800120	850120	900120	950120

Table 4
details of concrete mixtures.

Mixture	OPC	CB	Sand	Gravel	Water
C100B0	450	0	650	1000	210
C95B5	427.5	22.5	650	1000	210
C90B10	405	45	650	1000	210
C85B15	382.5	67.5	650	1000	210
C80B20	360	90	650	1000	210
C75B25	337.5	112.5	650	1000	210
C70B30	315	135	650	1000	210

2.2.2. Testes

2.2.2.1. XRD and QXRD

XRD patterns for RB and CB were performed using ADX-2700 diffractometer (manufactured by Angstrom Advanced Inc., Boston, USA), the range of 2θ is 3−80°, the rate of scanning is 0.05°/sec., the type of radiation is CuKα$_1$, i.e., λ = 1.54056Å, the voltage is 40kV, and the current is 30mA.

The QXRD of probable experiments were carried out using DIFFRAC.DQUANT software from Bruker UK Limited.

2.2.2.2. Properties of cementitious binder

Testes of water required to achieve standard consistency, initial setting time, and final setting time were carried out according to the Iraqi code of practice 1/198/2012.

2.2.2.3. Properties of CB as natural pozzolan

The testes and requirements of the CB as a pozzolanic material were carried out according to IQS 1748/1992.

2.2.2.4. Testes of concrete

Concrete mixtures were prepared according to Iraqi code of practice 248/1991. Slump tests were carried out according to Iraqi code of practice 354/1992, and compressive strength according to Iraqi code of practice 348/1992. All concrete specimens are 100mm cubes. The changes in compressive strength due to cement substitutions are calculated by Eq. (1):

$\Delta f = [(f_b − f_{co})/f_{co}] \times 100$ …………… (1)

Where: Δf is the change in the strength due to cement replacement by CB, f_b is the strength of CB containing mixture, and f_{co} is the strength of control mixture.

3. Results And Discussions

3.1. Physical effects of calcination

The calcination process causes the powder of RB to turn from a yellow color to a red color. The color of CB gets darker with the increase in the calcination temperature. Figure 1 illustrates the changes in bentonite color due to different calcination programs. The red color is attributed to the formation of hematite Fe_2O_3, and the dark color is attributed to the formation of magnetite Fe_3O_4 [39].

The bentonite powder tends to agglomerate with an increase in calcination temperature, and the formed lumps harden at higher temperatures. Table 5 elucidates the status of CB after calcination and Fig. 2 shows the statuses of some experiments. The formation of lumps is an indication to start the sintering process. The formation of lumps makes it necessary to re-grind the bentonite before using it as a pozzolanic material.

Table 5
status of CB after calcination.

experiment	status
70030, 70060, 70090, 700120, 75030, 75060, 75090, 750120, and 80030.	Powder
80060, 80090, and 85030.	Mixture of friable lumps and powder
800120, 85060, 85090, 850120, 90030, 90060, 90090, 900120, 95030, 95060, 95090, and 950120.	Hard lumps

The calcination process leads to a weight loss of RB by about 18−22%. The major of weight loss is attributed to release of the free water, the adsorbed water molecules from the surfaces of clay minerals, and the hydroxyl groups from the structure of clay minerals. The minor of weight loss is attributed to the decarbonation of dolomite and the removal of water molecules from gypsum structure.

3.2. XRD and QXRD

The result of the XRD test of RB is shown in Fig. 3; and the Figs. 4−8 show the XRD pattern of each experiment in comparison with XRD pattern of RB. RB consists of Ca-montmorillonite as a major clay mineral, kaolinite and palygorskite as minor clay minerals, and quartz, dolomite, and gypsum as minor non-clay minerals; Table 6 shows the crystal properties of clay minerals in RB. The calcination process causes structural changes in the montmorillonite mineral. The peak of 001 plane (2θ 5.8°) has shifted to the right by (4−5° 2θ) in all experiments at 700°C and 750°C, and in 80030 and 85030 experiments which mean the reduction in basal spacing from 15Å to 9−10Å due to removing water molecules layers. The

maximum shifting to 14.8° 2θ was occurred in the 80060 experiment in which the basal spacing has shrunk to 6Å; the shifting of 001 peak associates with reductions in the intensity which indicates that the strains are non-uniform [40], i.e., degrade the crystallinity. For the same experiments, the peak of 100 plane (2θ 19.8°) preserves its location and intensity, which elucidates that the structural changes in the c-axis do not mean the destruction of the montmorillonite crystals [19]. In the experiments 80090, 800120, and 85060, the peaks of the montmorillonite and other clay minerals disappear. That is strong evidence of the transformation of the crystalline phases of the clay minerals into amorphous phases. This conclusion is confirmed by QXRD which shows the absence of clay minerals in the experiments 80090, 800120, and 85060 as shown in Figs. 10−12. Despite the absence of the peaks of the clay minerals in the 85090 and 850120 experiments and all experiments at 900°C and 950°C but this absence is associated with the appearance of new peaks. The new peaks indicate the formation of highly crystalline inert phases such as cristobalite, spinel and/or magnesium aluminum silicate ($MgAl_2Si_4O_{12}$) [19]. The results obtained are compatible with previous studies, which showed that the amorphization temperature of montmorillonite mineral lays in the range 800−900°C [41]. Based on XRD and QXRD results, the experiments 80090, 800120, and 85060 are the probable experiment for adapting as an optimum calcination program.

Table 6
clay minerals in RB

Mineral	hkl	d (Å)	2θ
Ca-montmorillonite	001	15	5.8
	005	3	29.3
	100	4.5	19.8
	300	1.5	62.1
Palygorskite	040	4.5	19.8
	101	4.3	20.8
	231	3.3	26.5
Kaolinite	020	4.5	19.8
	111	3.4	26.5
	-223	1.8	50.1
	203	1.5	60.1
	-3-31	1.5	62.1

3.3. Pozzolanic activity index

The PAIs of probable experiments at both 7 and 28 days are listed in Table 7. At the age of 7 days, all PAIs were less than 100% which indicate the low reactivity of CB. At the age of 28 days, all probable

experiments developed PAI equal or more than 100%. The maximum 28 days PAI (108.3%) is that of 80090 experiment. Therefore, the 80090 experiment is the optimum calcination program.

Table 7
PAI for probable experiments

	f_{7d}	PAI_{7d}	f_{28d}	PAI_{28d}
Control	34.3	100%	36	100%
80090	26.6	77.6%	39	108.3%
800120	31.3	91.3%	35.8	99.4%
85060	26	75.8%	38.3	106.5%

3.4. Requirements of pozzolanic materials

CB prepared according to optimum calcination program (80090) meets the mandatory requirements of IQS 1748/1992. The results of tests were reported in Table 8.

Table 8
properties of CB (80090)

Chemical properties		
Property	Result	IQS 1748/1992 limits
Σ ($SiO_2 + Al_2O_3 + Fe_2O_3$)	82.4%	70.0% min.
SO_3	1.5%	4.0% max.
Moisture content	0.4%	3.0% max.
Loss on ignition	0.4%	10.0% max
Physical properties		
Property	Result	IQS 1748/1992 limits
Amount retained on 45μm	0%	34% max.
Strength activity index @ 28 days	108.3%	75% min.
Water requirement	99%	115% max.
Soundness	0.05%	0.8% max.

3.5. Properties of cementitious binder

Portland cement replacement by CB causes increase in water required to achieve standard consistency of cementitious binder. Figure 13 shows the linear relation between w/cm and the percentage of cement

replacement. The amorphous nature of CB, the platy shape of CB particles, and relatively low specific gravity are the possible causes of the increase in water demand.

Both initial and final setting times extend linearly with the percentage of cement substitution as shown in Fig. 14. The reduction in C_3A and C_3S contents, the low reactivity of CB components, the presence of phosphates in CB, and higher water content are the reasons of the delay in both initial and final setting time. Similar observations were reported by Darweesh and Nagieb [23].

3.6. Concrete workability

CB is characterized by its lower specific gravity compared to the Portland cement, so the volume of CB required to replace a specific weight of cement is greater than the volume of the substituted cement, and this increase in the volume of cementitious binder requires an increase in the mixing water to maintain the same workability. In the case of fixation the amount of mixing water, an increase in the volume of the cementitious binder leads to a decrease in concrete workability. Figure 15 shows the effect of CB on the slump of concrete. It is noticeable that the slump value decreases as the CB content increases. The effect of CB in reducing the slump has previously been reported by Ahmad et al. [9].

3.7. Compressive strength

CB is characterized by its low reactivity because most of it is the low reactive CM [31], in addition, it contains percentages of inert minerals such as quartz, and compounds that slow down the hydration of cement such as phosphates. At the age of 7 days, the compressive strength of the control mixture was 28.1 MPa, the changes in strength were negative for all mixtures, and the value of the strength change proportioned to the cement substitution. The reduced effect of CB is attributed to its low reactivity. It is worth noting that the reduction percentage is less than the percentage of cement replacement in all mixtures. This observation may be evidence of little pozzolanic reactivity of CB at early ages and/or physical actions of CB incorporation in the promoting of cement hydration.

At the age of 28 days, the compressive strength of control mixture was 33.3 MPa. The slow pozzolanic reaction leaded to overcoming the negative changes in compressive strength and its reversal into positive changes for replacement levels 5–15%; while the replacement levels 20–30% caused negative changes in compressive strength by 5–10% only. Figure 15 shows the changes in compressive strength at 7 and 28 days.

4. Sustainability Improvement

The utilization of CB as a partial replacement of OPC in concrete production leads to improve the sustainability of concrete due to the following reasons:

1. Reduce CO_2 emission resulting from the decarbonation of the calcite mineral due to the Iraqi bentonite containing a small percentage of this mineral not exceeding 10% [35] compared to the raw materials in the cement industry in which the calcite percentage exceeds 60%.

2. Reducing energy consumption, as the temperature required to prepare CB is 800°C compared to the temperature required for cement manufacturing, which is about 1400°C.
3. Reducing the resource consumption of the cement industry and the exploitation of untapped resources.
4. Reducing the measures for concrete cooling in hot climates and the measures of the control of cement hydration heat in the mass concrete.
5. At 5–15% replacement, reducing OPC content in concrete without any reduction in compressive strength which leads to reduce the environmental impact of concrete production.

5. Conclusions

1. Iraqi high-calcium bentonite is suitable for the preparation of natural pozzolan by the method of calcination.
2. The optimum calcination program is 800°C for 90min.
3. Pozzolan prepared from Iraqi bentonite is characterized by its slow reactivity, that is, it has a retarder effect on the initial and final setting time, as it reduces the rate of the strength gain.
4. There is no significant reduction in 28 days compressive strength at 5–20% cement replacement by CB. Moreover, 10% strength gain was reported due to 5% cement replacement.
5. Cement replacement at 25–30% causes 5–10% reduction in 28 days compressive strength.
6. Bentonite has a reducing effect on concrete workability and the amount of reduction is proportional to the replacement level.
7. CB improves the sustainability of concrete.

Declarations

Availability of data and materials

The datasets supporting the conclusions of this article are included within the article and its additional files.

Competing interests

The authors declare they have no competing interests.

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Authors' contributions

AA, QJ, and WA contributed to writing the manuscript. All authors read and approved the final manuscript.

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Authors' information

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Figures
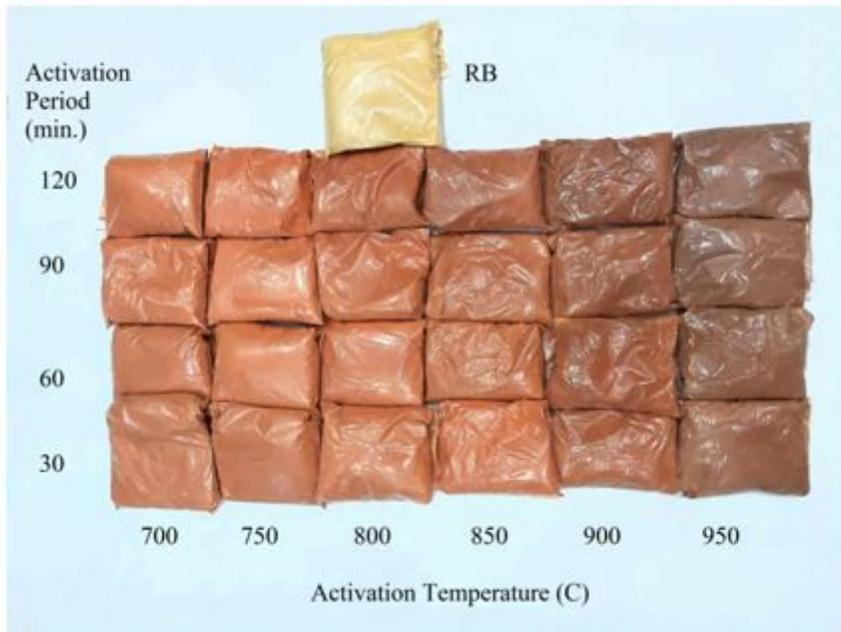
Figure 1

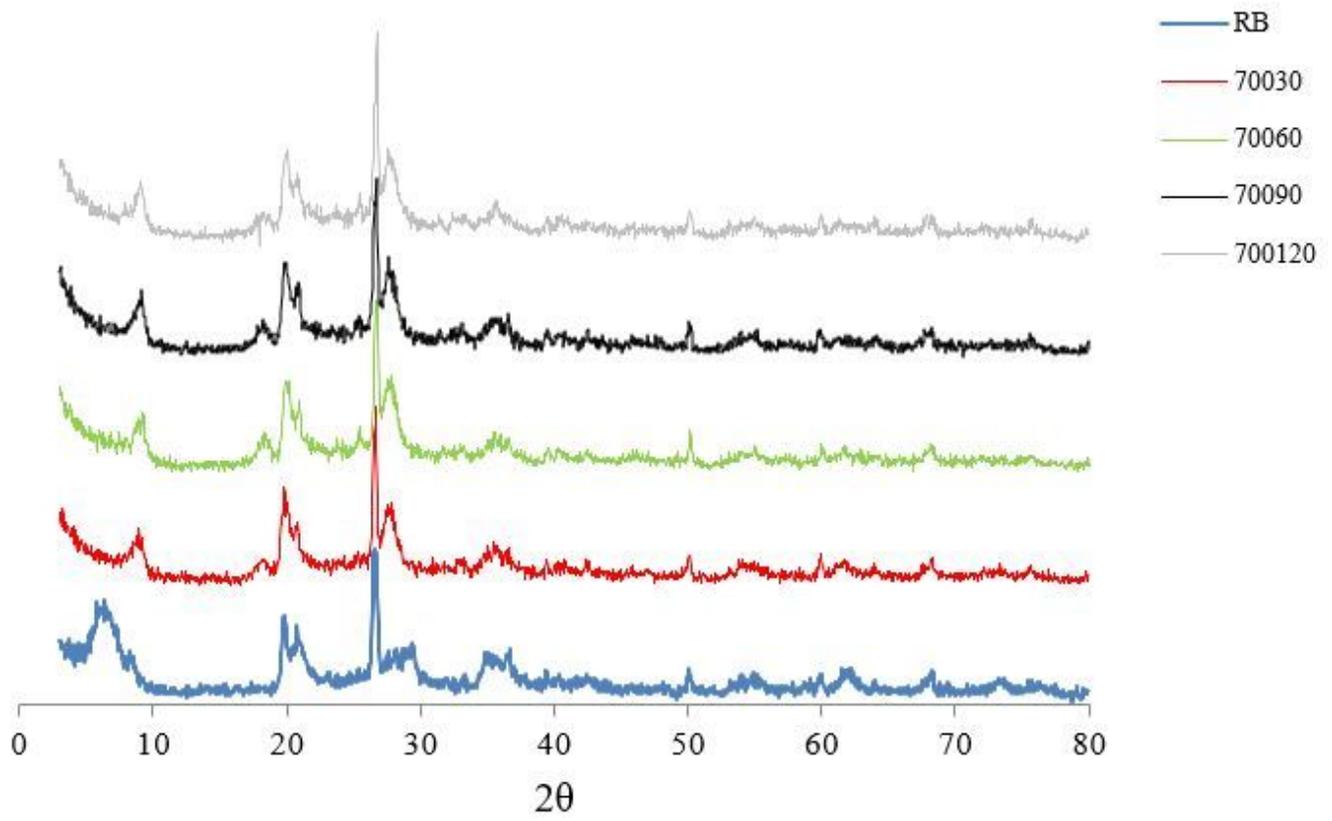colour of bentonite due to calcination programs.

Figure 2

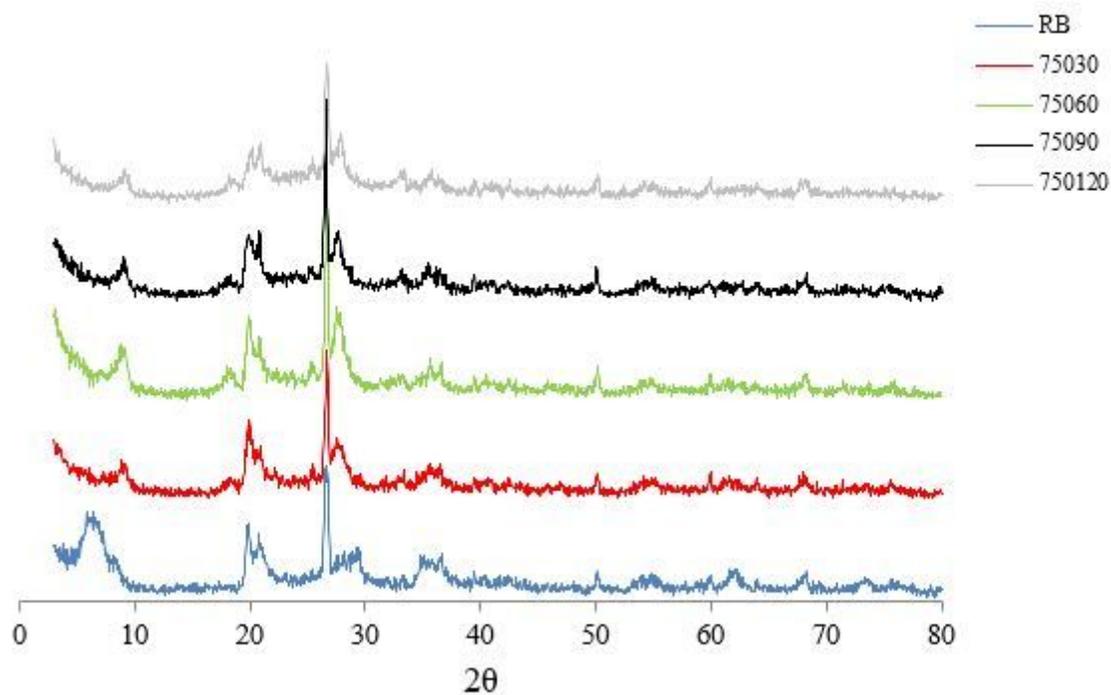status of some experiments.
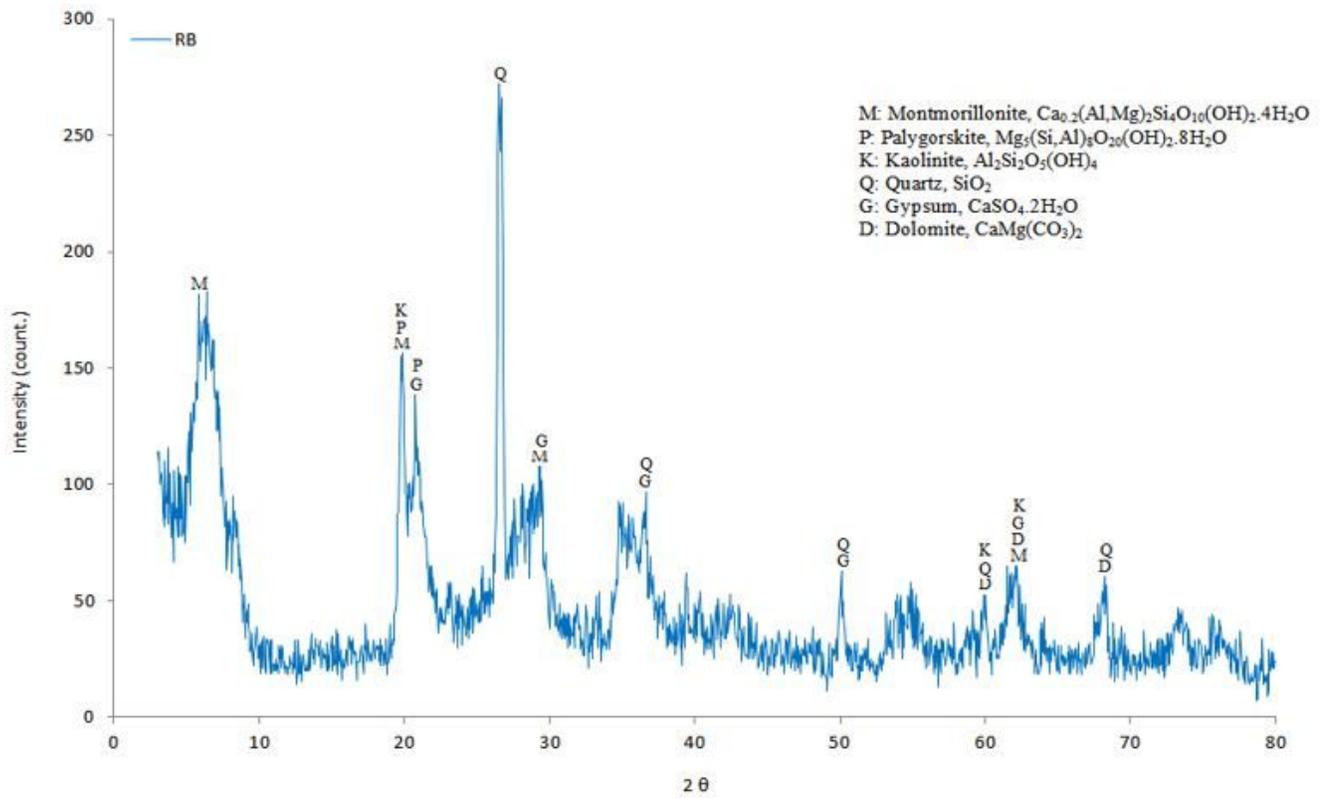
Figure 3

XRD pattern of RB.

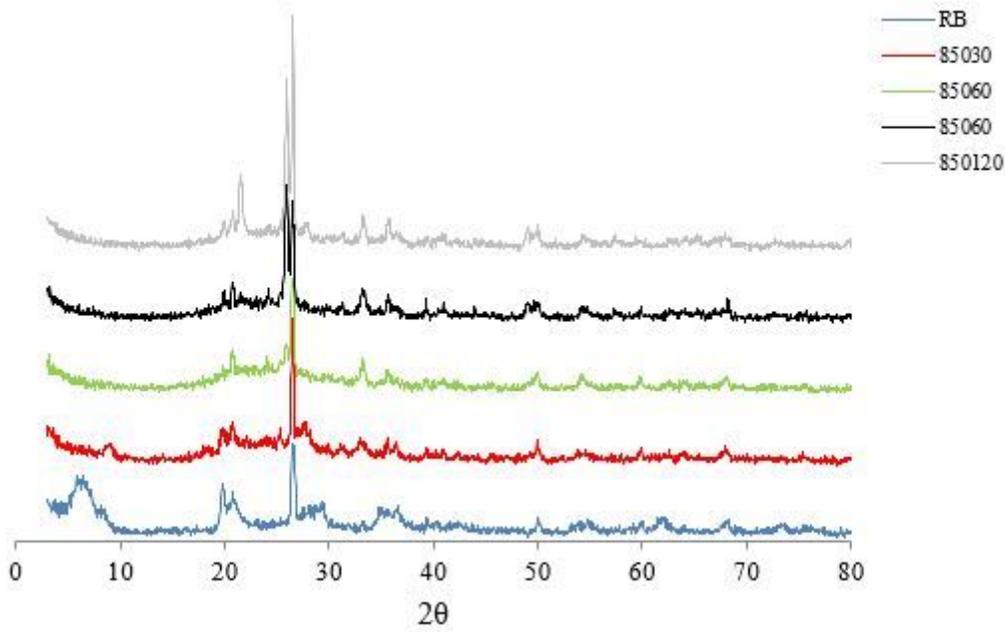**Figure 4**

XRD patterns of RB and CB at 700°C.

Figure 5
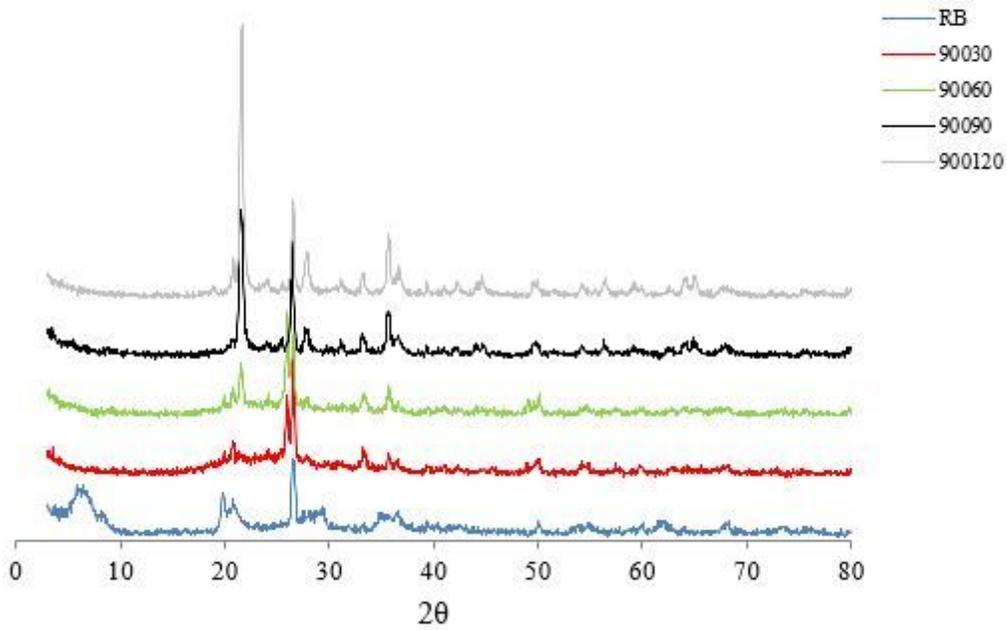
XRD patterns of RB and CB at 750°C.

Image not available with this version

Figure 6

Figure 7

XRD patterns of RB and CB at 850°C.

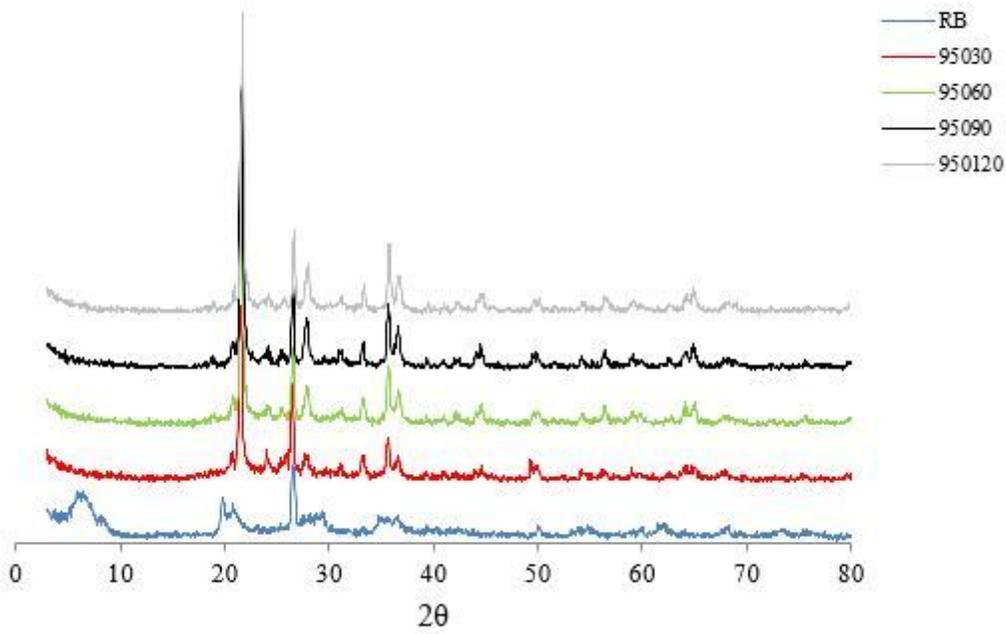**Figure 8**

XRD patterns of RB and CB at 900°C.
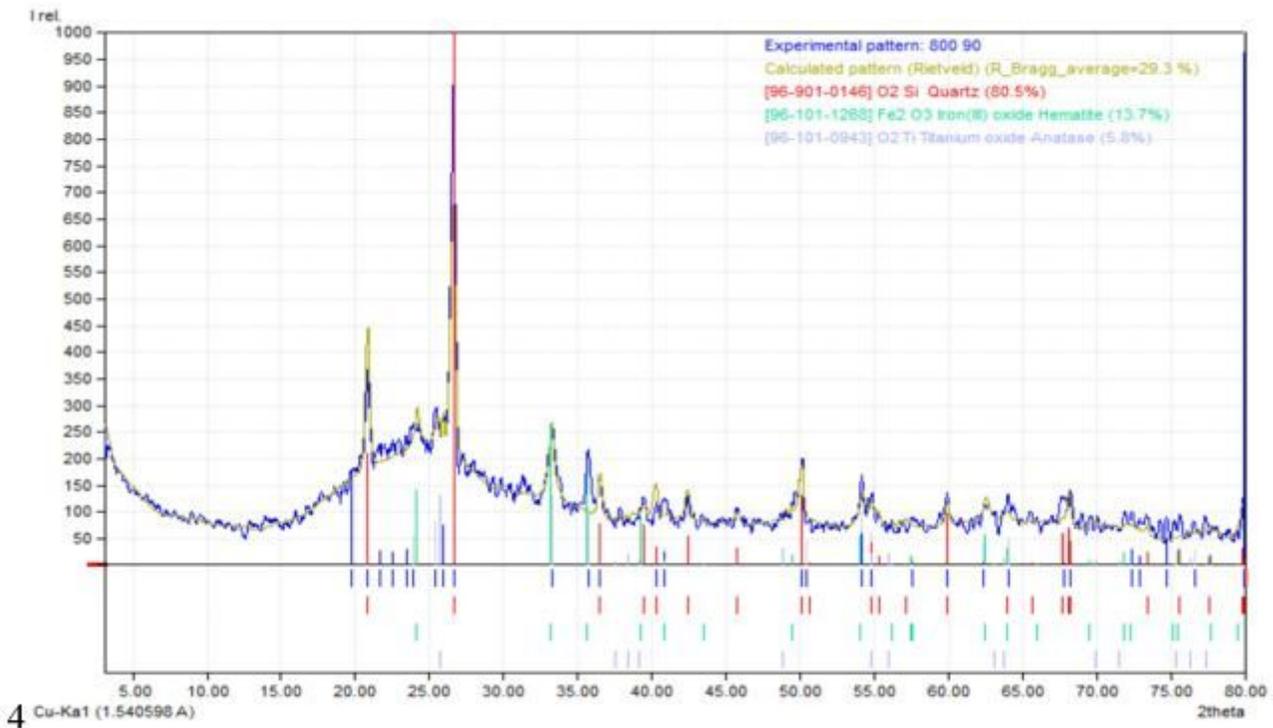
Figure 9
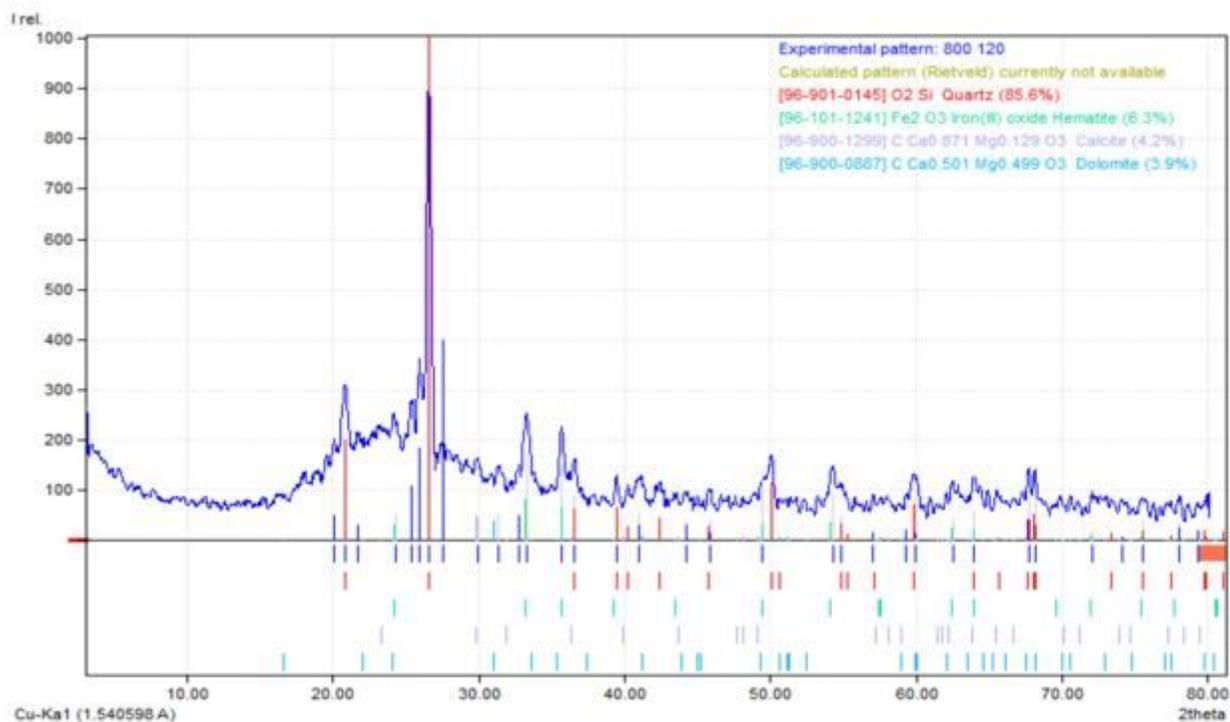
XRD patterns of RB and CB at 950°C.

Figure 10

QXRD for 80090 experiment.

Figure 11

QXRD for 800120 experiment.
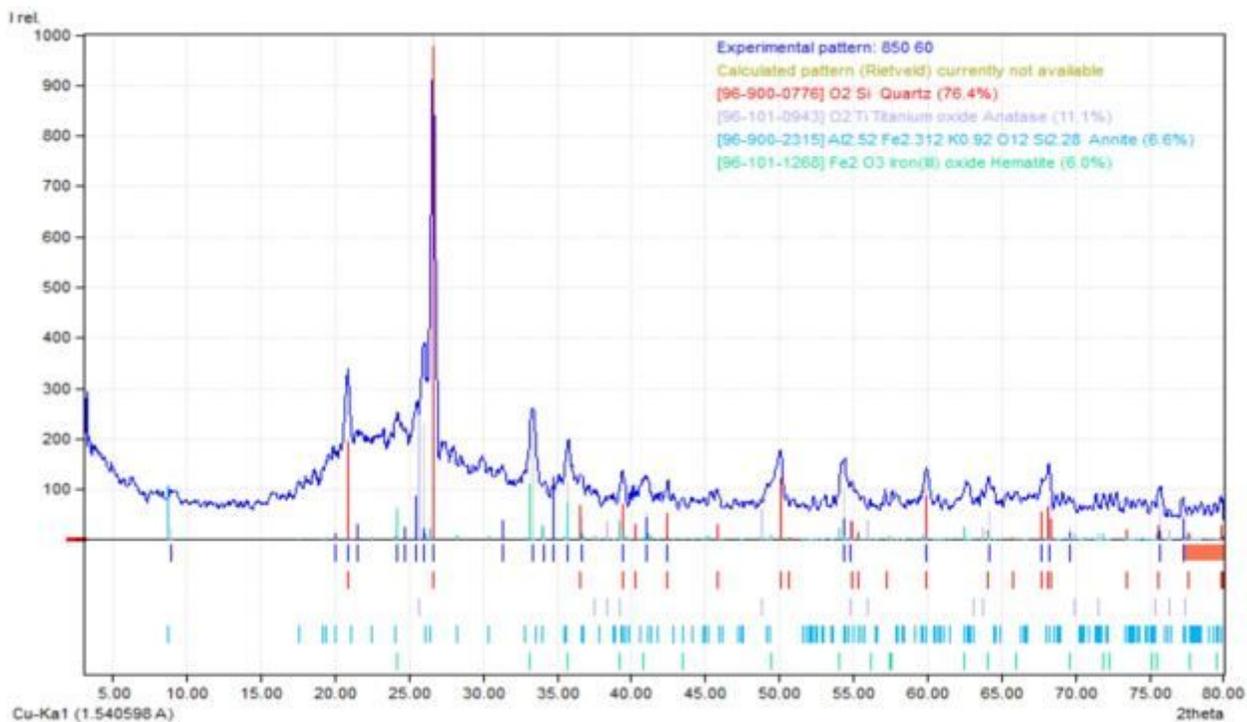
Figure 12

QXRD for 85060 experiment.
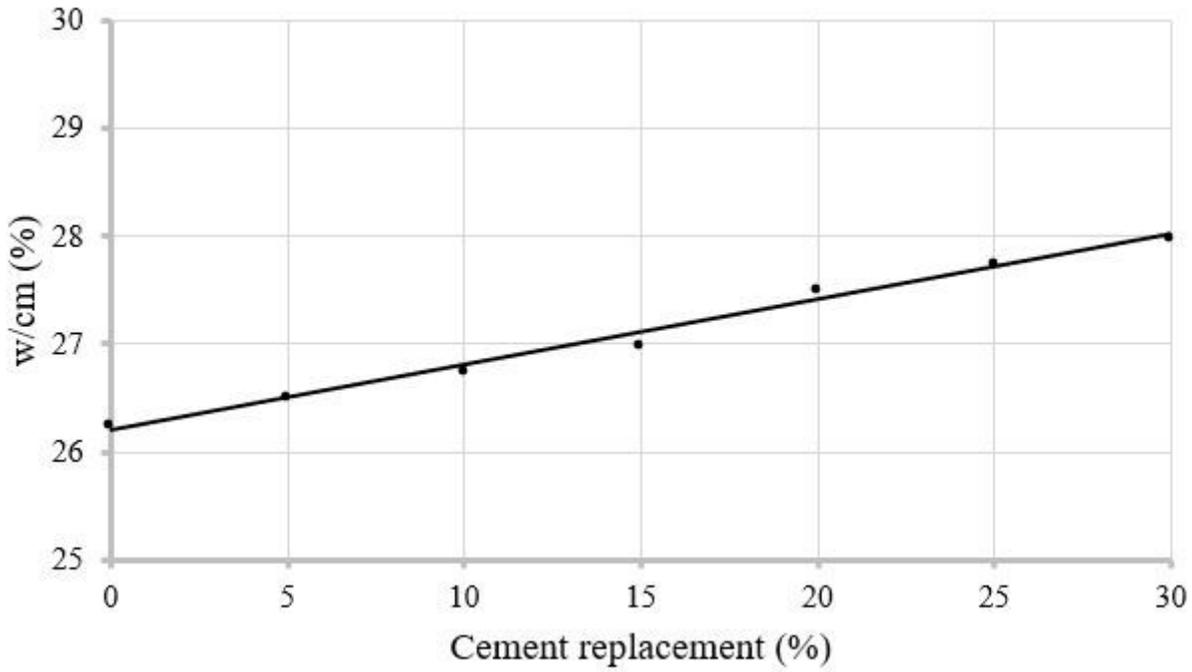
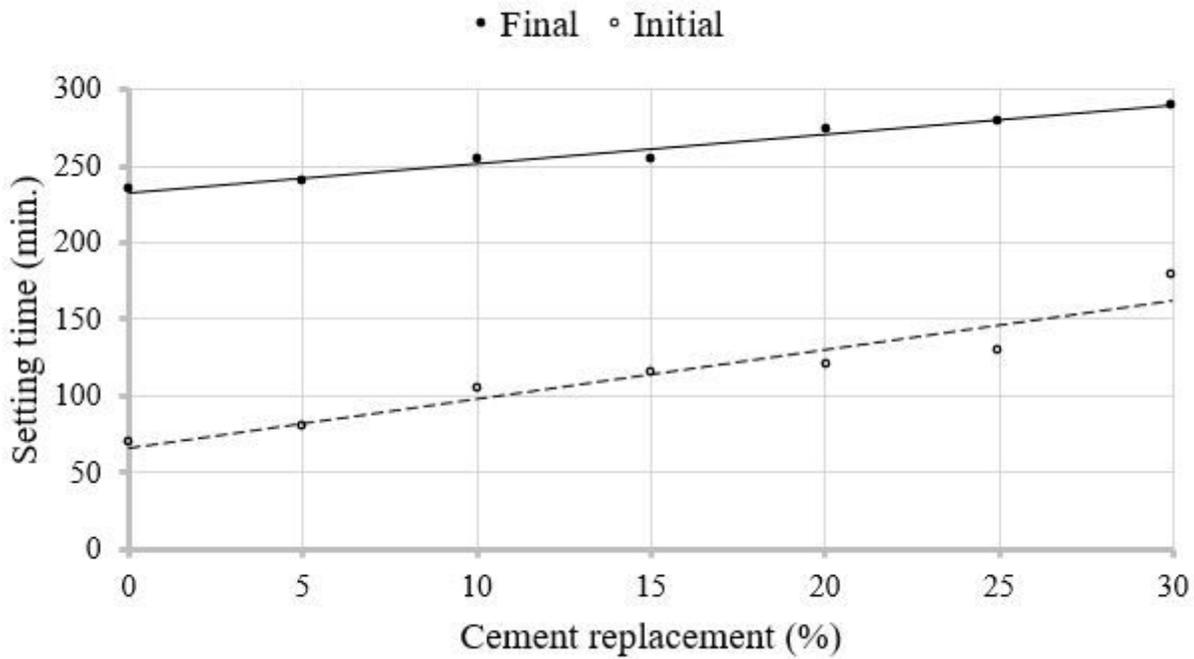**Figure 13**

the relation between w/cm and cement replacement.

Figure 14

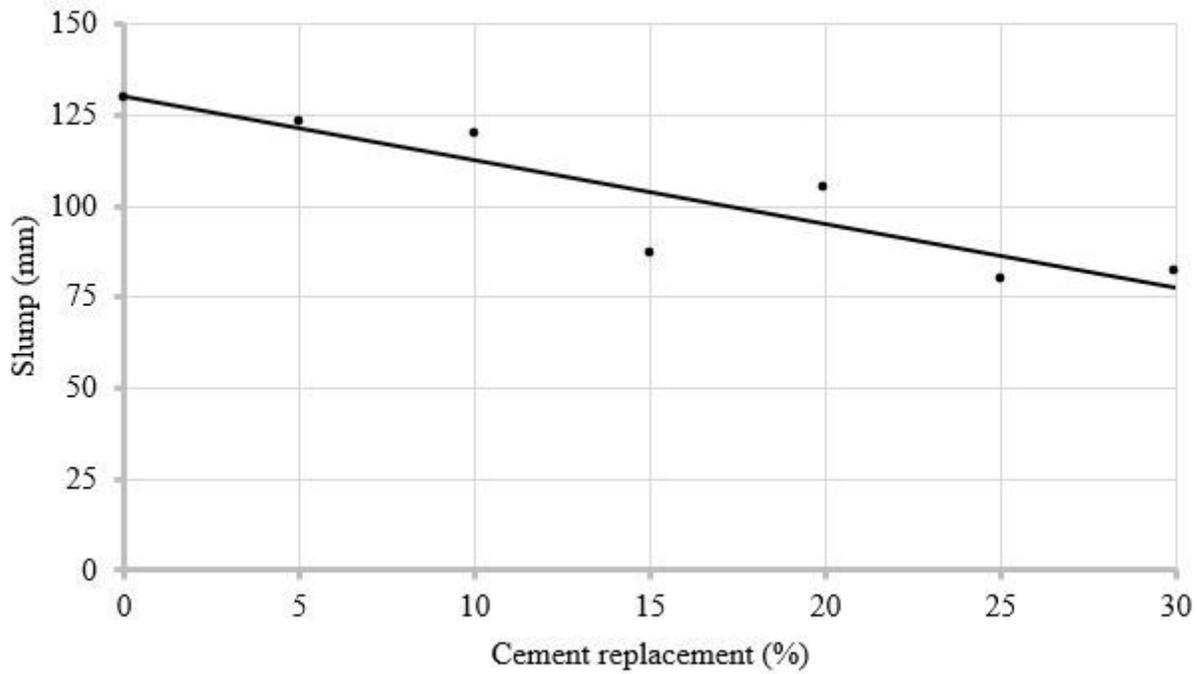effect of CB on the setting time of cementitious binder.

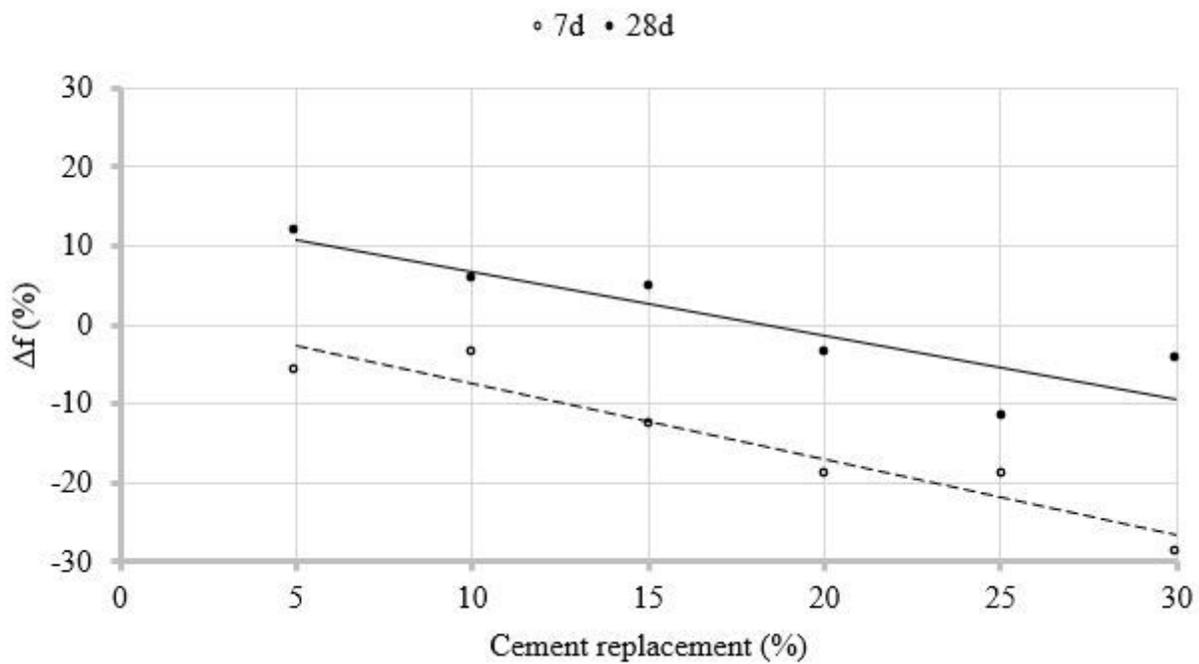

Figure 15

effect of CB on the slump of concrete.

Figure 16

changes in compressive strength due to cement replacement.