Influence of The Activated Qatari Attapulgite Clay Admixture on The Mechanical Properties and Hydration Kinetics of Ordinary Portland Cement

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تأثير إضافة طفلة الاتابولوجيت القطرية المنشطة على الخواص الميكانيكية وكيناتيكية التأدرت للاسمنت البورتلاندي العادي

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تعد الاسمنتات المخلوطة أنواع من الاسمنت لها مواصفات تقنية ذات أهمية ملموسة وتحتوي مواد إضافية عدا تلك المتواجدة بالاسمنت البورتلاندي، لان مثل هذه الاضافات للاسمنت البورتلاندي تزيد من مقاومة الاسمنت ضد الكبريتات والكلوريدات، والبحث الحالي يقدم دراسة مختبرية توفر فرصة فريدة لتقديم محاولة فعالة تعالج مشكلة تدهور الخرسانة في قطر ومنطقة الخليج العربي، وتزودنا بحل للمشكلة بواسطة إنتاج الاسمنت المخلوط من خلال تتبع الخواص الميكانيكية وكيناتيكية التأدرت للاسمنت المخلوط المنتج، ولقد قدم المؤلف ولأول مرة في قطر مخاليط للاسمنت المخلوط مستخدماً بوزولانا قطرية منشطة أصيلة، حيث ثبت أن نسبة ولقد قدم البوزولانا المصنعة تمثل التركيب الأمثل للاسمنت المخلوط المحتوي على الطفلة المنشطة.

- حرق طين الاتابولوجيت القطري عند درجة حرارة تبلغ ٢٠٠°م للحصول على البوزولانا المصنعة.
- تم تحضير عجائن مخاليط الاسمنت البورتلاندي العادي والبوزولانا باستخدام نسبة ماء/صلب= ٤,٠ عند درجة حرارة الغرفة، ولفترات زمنية مختلفة.
 - تم دراسة خواص التأدرت للعجائن المتصلدة بالنسبة لقيم قوى التحمل للضغط الميكانيكي (مقاومة الانضغاط)، وقيم النسب المئوية لكل من، الماء المتحد كيميائياً، والجير الطليق عند أزمنة مختلفة من تفاعل التأدرت. ومن النتائج يمكن الحكم على قابلية الاستخدام لتلك المخاليط.

ABSTRACT

Blended cements are types of cements containing additives other than those used in Portland cement, which have considerable technological interest, because such addition increases the chemical resistance to sulfate and chloride attack. The present investigation represents a laboratory study, which provides a unique opportunity to introduce an effective practical attempt to deal with the problem of concrete deterioration in Qatar and the Arabian Gulf region, and to provide a solution to the problem by studying the production of blended cements. The mechanical properties and the hydration characteristics of the produced blended cements are also taken into account. The author produced for the first time in Qatar, mixes of blended cement using an original Qatari artificial pozzolana. The pozzolana was made by firing attapulgite clay at 600°C. Portland cement/artificial pozzolana pastes were made using an initial water/solid ratio of 0.40 and hydrated at room temperature for various time intervals. The hydration characteristics of the hardened pastes were studied and related to their compressive strength values, combined water and free lime contents. The results could be useful as a prediction of the performance characteristics of the mixes.

INTRODUCTION

In the Arabian Gulf countries, concrete durability is currently of major concern as even recent concrete construction (10 to 15 years) has shown an alarming degree of deterioration. The Gulf environment is extremely aggressive and is characterized by severe geomorphic climatic conditions, severe ground and ambient salinity, and high temperature-humidity regimes. Coupled with these difficult natural conditions are the problems of aggregates, inadequate specifications, inappropriate construction practices and lack of skilled manpower, all of which accentuate concrete deterioration. Thus the deterioration is said to be in a complex way. Portland cement concrete is the world's most versatile and economic building material. The raw materials from which Portland cement is produced, sand, stone, and water that constitute the remainder of the basic concrete mixture ingredients are among the most common mineral materials in earth's crust. However, the outwardly appearing simplicity of concrete belies the extremely complex physio-chemical nature of materials. This coupled with wide variability in the properties of the major ingredients (the aggregates) and virtual insitu batching procedures involved in its production. Stated another way, the durability of concrete is function of the characteristics and proportions of the cement, aggregate, water, and admixtures used, mixing, placing, finishing, and curing of the concrete. The types of concrete distress in the Arabian Gulf region, in general decreasing order of importance are; corrosion of reinforcement with attendant spalling of the concrete cover, salt weathering due to crystallization pressures brought on by capillary transport of soluble salts (principally chlorides and sulfates) and evaporation at exterior surface of the concrete, common sulfate attacks due to the sulfates present in the soils, ground water, and aggregates of the region, drying shrinkage and thermal-gradient-induced cracking due to the environmental conditions of the region, and aggregate-cement reactivity(principally alkali-silica). Reinforcement corrosion and salt weathering are considered to be the most common and most serious form of concrete deterioration in the Arabian Gulf region (1). Recommendations to minimize deterioration from this and other causes concentrate mainly on practices where certain measures have to be taken to ensure good quality concrete and that the concrete is as effective as possible in protecting the reinforcement against aggressive salts entering the concrete from its surface (2). In our

attempt toward solving the concrete deterioration problem in Oatar and the Arabian Gulf, we represent this study. The use of blended Portland Cements containing pozzolana is increasing worldwide because, when compared to Portland cements, blended cements require considerably less energy for production. Also, blended Portland cements are capable of certain performance characteristics that at times are considered more desirable than those of ASTM type 1 or Portland cements are. Notable among such characteristics are the lower heat of hydration, higher ultimate strength, and better long-time durability to chemical attacks. "Pozzolana" is defined as natural or artificial solids involving constituents that react with CaO or Ca(OH), and forms new binding compounds under the presence of water at ordinary temperature(3). "Constituents" we say, means minerals, crystals, noncrystalline materials, glasses and "artificial" means chemical or physical treatment for natural materials such as clay and shales and certain siliceous rocks. Burnt clay is one of the pozzolana in this meaning. Heating clays, at temperatures corresponding to those of thermal destruction of minerals, which characterized them, bring about rupture in the lattice and consequently the formation of a mixture of amorphous silica and alumina in the stoichiometric proportion of the original minerals. Since the instability of the system, reactivity with lime and loosing of plasticity become noticeable. Cement pastes with appropriate proportion can offer mechanical strengths of the same magnitude as those obtained using pozzolanas or fly ashes (4).

In Portland pozzolan cements, the lime for the pozzolanic reaction is produced by the hydration of the anhydrous calcium silicates present in the Portland cement. The exact mechanism by which pozzolanic reaction contributes to the strength and chemical durability of mortars and concrete is not fully understood and still an open field for more investigations. The crystalline hydrates formed in the reaction between lime and pozzolana in the presence of water were summarized by Massazza (5). A mechanism for the pozzolanic reaction was proposed with reference to the structure of feldspar (6). The validity of the test for the pozzolanic cement has been also confirmed (7) by Massazza, etc and had reviewed several publications in the area of cement based on pozzolanic additions. There are many studies that have tried to connect the strength of pozzolanic cement with the character of pozzolana. Battaglino and Shippan (9) stated that there existed correlation between the strength of pozzolanic cement and the change with time of specific surface area and quantity of adsorbed CaO by pozzolana, and concluded that pozzolana, which was rich in amorphous silica was desirable. Stein and Stevels (10) investigated the influence of amorphous silica on hydration of C₂S using C₂S, amorphous silica, quartz and Portland cement. Their test brought them to the following conclusion: Amorphous silica accelerates the rate of hydration of alite in cement pastes, not only the hydration of alite but also the consumption of sulfate by the formation of ettringite, is accelerated by the addition of amorphous silica. Lukas (11) investigated the rate of hydration of each clinker compound, using the specimens prepared of 30% of fly ash and 70% Portland Cement with W/C ratio of 0.4. Fly ash accelerates the early hydration of C,S. Though the rate of hydration of C,A in cement is not so fast as that of pure C,A, fly ash accelerates its hydration. The decrease of Ca(OH), in paste at later age is attributed to the absorption by fly ash over than the amount of Ca(OH), released by hydration. Takemoto, Uchikawa, Ogawa and Yasui (12) studied the hydration of the (6:4) mixture of C₂S and pozzolana with (0.4) of water solid ratio. They concluded that the addition of pozzolana and fly ash to C₃S accelerated the initial hydration within an hour. Degree of hydration of C,S after the induction period was increased by the addition of pozzolana. Pozzolanas consisting mainly of opal and allophane were more reactive with C₂S than that consisting mainly of volcanic glass and fly ash. Summarizing the above mentioned previous works: Additions of pozzolanas accelerate the hydration of C,S. S.A Abo-El-Enein investigated the use of cement kiln dust (14) either in the production of blended cements or in the manufacture of autoclaved building products. He concluded that cement kiln dust (CKD) can be used in the production of blended cements with certain optimum constitutions. The studies of Uchikawa and Uchida (13) indicated that there is no essential difference between the hydration in system pozzolana-cement and that in the system Pozzolana-Ca(OH), and pozzolanic cement compounds. The degree of pozzolanic reaction was expressed in term of the difference between the amount of estimated Ca(OH), which might be formed in the case when no pozzolanic reaction occurs and that formed actually on pozzolanic cement paste.

The mineralogical studies of Qatarian clays indicated the presence of attapulgite (Palygorskite) in shales and clays of the lower and upper Dammam Formations in Qatar. It was found that it may well indicate a possibility for future mining and production of a marketable materials. Clay deposits are formed principally by direct deposition of fine-grained suspended material such as mud, volcanic ash. In some instances, however they precipitate as result of chemical action such as the case for aragonite marls and palygorskite, the later is being the principal clay mineral of the attapulgite Midra Shale. A prerequisite for the formation of palygorskite is the mixing of waters, one containing silica and alumina in solution of fine suspended colloids, and another containing magnesium ions. These conditions would have existed in the past in the Gulf where less dense sea water or continental derived water from the head of the gulf would flowed across the top of the more dense, highly saline, brines and reactions responsible for the formation of reformed silicates would have resulted from the mixing off and reaction between these two water masses. Neo-formation of palygorskite probably predominates as it is favored by alkaline or hyper-saline conditions. In Qatar it would seem to have been deposited under hyper-saline conditions for the clay still retains a proportion of sodium chloride (15).

This study represents an investigation on Portland-Pozzolan Cements produced by blending various proportions of an artificial attapulgite pozzolan with Portland cement. The Portland-Pozzolanic cements containing different proportions of burnt attapulgite clay were tested at various ages for compressive strength, free lime, and combined water determinations. This study is a part of a series of studies investigating the production of blended cements in Oatar.

EXPERIMENTAL

The starting materials used were ordinary Portland cement, for which the chemical analysis is shown in table (1) and Qatarian (W.H.Q) clay. The materials were supplied by Qatar National Cement Company. The clay is the attapulgite located on Qatar map, in Jleaha, south area number (44), greenish in colour. The chemical oxide composition of the material is given in table (2). The attapulgite clay was dried at 110°C for 48 hours. Then crushed and passed completely through a 1 mm B.S sieve. The crushed clay was fired at 600°C for a soaking period of 4 hours and then quenched in air. The burnt clay then subjected to a ball mill grinding for 20 minutes, and then passed through a 75 (m B.S sieve (16), the process of grinding and sieving were repeated until the sample passed completely through the sieve. Different mixtures

were prepared by mixing the Portland cement with the thermally treated clay at 600°C. Twelve mixes of different ratios of burnt clay/Portland cement were used namely 5/95, 10/90, 15/85, 20/80, 25/75, 30/70, 35/65, 40/60, 45/55, 50/50, 70/30, and 80/20 by weight. The amount of cement was placed on a smooth surface, and a crater was formed in the center. The amount of mixing water was poured into the crater with a continuous vigorous mixing for four minutes then completed the mixing operation. At the end of mixing, the paste was directly poured in cubic moulds 1x1x1 inch. The paste was compacted and pressed until homogenous specimens were obtained. The moulds were manually vibrated for two minutes to remove any air bubbles. The surface of the paste was smoothed by a spatula. Immediately, after moulding, the moulds were cured at 100% relative humidity at room temperature for 24 hours, then demoulded and cured under moist cotton in 100% relative humidity at room temperature up to 180 days. The cement pastes were made by using a water/solid ratio of 0.40 by weight. The specimens were cured for various hydration periods of 1, 3, 7, 14, 28, 90 and 180 days. After each time interval of hydration the compressive strength tests were determined and the hydration reaction of the hardened cement paste was stopped (17-19) then the samples were dried and kept in a dessicator. Compressive strength measurements were carried out on the fresh specimens of the hardened cement pastes after curing for various ages. Non-evaporable water (Wn) and free lime contents determinations were done on the dried specimens. Compressive strength measurements were determined using two cubic specimens of the hardened cement pastes cured at 100% relative humidity up to 180 days. The compressive strengths test machine used in this study was of the Ton-Industrie type. Two cubes from each mix having the same age were used for compressive strengths measurements. The results are expressed in N/mm≈. Kinetics of hydration were studied by the determination of non-evaporatable(chemically combined)

water (Wn) and free lime contents in the hardened cement

pastes.

The non-evaporatable water content (Wn) was determined by igniting a certain weight of the dried sample at 1000(C for one hour. The drying process was carried out after crushing the fresh specimen. The stopping solution was prepared as a 1:1 mixture by volume of methyl alcohol and acetone. A representative sample, of about 10g was ground in an alumina mortar and mixed with 100 ml of the stopping solution for one hour with continuous and throughl stirring, the stopping solution, then filtered with 50 ml of fresh methanol. Then the samples were dried at 105°C for 24 hours and kept in the desiccator. The chemically combined water (Wn) was regarded as the ignition loss of the dried sample. Duplicated experiments were carried out in order to guarantee good reproducibility of the results. Chemically combined water was calculated on the ignited weight basis. The free CaO content was determined by the following method. 0.5g sample was taken in 40 ml of a glycerol/ethanol mixture (1:5 v/v), together with a small amount of anhydrous barium chloride (0.5g) as a catalyst, and phenol-phthalein as an indicator. This mixture was kept in a conical flask, fitted with an air reflux, heated on a hot plate for 30 minutes (the colour becomes pink). The contents of the flask were titrated with a standardised alcoholic ammonium acetate solution until the pink colour just disappeared. Heated again and if the pink colour persists, the titration with ammonium acetate solution was continued until the end point noted by the addition of a drop and the colour changes from pink to colourless.

RESULTS & DISCUSSION

3.1. OPC and Attapulgite Chemical Composition

The chemical composition, the phase composition and the physical tests of ordinary Portland cement are given in Table (1); the chemical composition of attapulgite clay is given in Table (2).

Table (1)
Chemical Analysis of the Ordinary Portland Cement

Chemical	SiO ₂	Fe ₂ O ₃	Al ₂ O ₃	CaO	MgO	SO ₃	Cl-	Loss On Ignition			
Analysis	20.72	3.02	4.43	63.18	2.99	2.68	Nil	1.50			
Free CaO				·····	0.60	·	·	-			
SR	2.69										
AR		1.47									
LSF		0.94									
LCF		0.93									
C3S		55.51									
C2S		17.61									
C3A		6.64									
C4AF		9.18									
	Specific Surface Area							337 m²/Kg			
	Setting Tir	ne Initial		160 minutes							
Physical	Setting Tir	ne Final		200 minutes							
Test Results	Le Chatlie	r Expansion		1 mm							
	Compressi	ive Strength		34.8 MN/mm ²							
	Compressi	ve Strength		53.1 MN/mm ²							

Table (2) Chemical Analysis of Attapulgite Clay

	SiO ₂	Fe ₂ O ₃	Al ₂ O ₃	CaO	MgO	SO ₃	Cl-	Loss On Ignition
	48.60	5.25	12.81	10.60	4.07	0.93	0.20	11.70
Chem.		K ₂ O		Al ₂ O ₃		CaO	TiO ₃	MnO
Analysis		3.77		1.32		0.10	0.67	0.04

3.2. Mechanical Properties

The compressive strength results of the hardened Portland cement/burnt clay pastes, investigated in the present study, indicate some interesting characteristics, which could be summarized as follows: The compressive strength tests were carried out on the hardened pastes made of Portland cement and artificial pozzolana obtained by firing the attapulgite clay at 600°C. The compressive strength results for the hardened pastes are graphically represented as a function of the hydration age and shown in Fig. (1 a&b). The compressive strength values are also given in Table (3).

Table (3)

Compressive Strength Values (N/mm⁻) for Different Mixes of Clay Fired at 600BC / Ordinary Portland Cement

Mix Proportions	1 Day	3 Days	7 Days	14 Days	28 Days	90 Days	180 Days
5 / 95 %	24.00	26.40	36.80	29.60	40.96	45.60	49.28
10 / 90 %	35.04	40.00	27.20	49.92	50.40	49.60	58.40
15 / 85 %	20.64	28.80	29.60	31.68	32.48	46.40	51.20
20 / 80 %	14.40	32.00	36.16	35.04	32.00	38.08	40.00
25 / 75 %	25.92	32.48	17.76	31.68	33.28	61.12	70.93
30 / 70 %	14.40	25.28	26.88	27.04	28.32	41.92	42.72
35 / 65 %	20.64	28.00	33.12	37.28	23.36	50.48	40.00
40 / 60 %	12.00	21.12	24.80	24.80	34.88	37.44	51.52
45/ 55 %	37.76	13.92	27.36	28.48	31.20	32.16	55.04
50 / 50 %	10.72	19.20	29.84	35.92	30.56	32.32	35.20
70 / 30 %	5.28	8.320	15.68	24.64	16.96	23.36	24.00
80 / 20 %	0.00	4.13	14.72	9.60	7.36	13.76	15.36
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For all the hardened pastes, the compressive strengths were in a continuous increase with the age of hydration during the early stages of hydration. The free lime released, as a result of hydration of Portland cement undergoes a reaction with the pozzolana to give calcium silicate hydrates which contribute strongly to the compressive strength of these cured specimens (22, 23, 24), and the formation and the later stabilization of the initial hydration products, which act as binding centers between the unhydrated cement, and burnt clay grains. Comparing the results obtained for the compressive strengths of Portland cement/artificial pozzolana pastes in this study and that of the neat Portland cement pastes, it was obvious that the early strength values were lower in the case of the pozzolanic cement pastes than those of the neat Portland cement pastes. During the intermediate stages of hydration (7 Days), however, certain pastes, especially those prepared of Portland cement/burnt clay mixtures, which contain poor clay quantities, such as 5, 10, 15, and 25%, showed some regression in compressive strength. The regression in the strength values are mainly attributed to the crystallization of the initially formed hydrates, having stronger binding forces and/or their transformation into other hydration products having weaker binding forces. The early strength values are mainly due to the

reduction in compressive strength that is observed, a result which might be associated with the transformation of the stable initial hydrates into other hydrates having a metastable character at the time of their formation. The stabilization and later accumulation of these second hydrates, having a relatively low lime content, accounted for the consequent development in compressive strength at the later ages of hydration. The interaction between the initially formed high lime hydrates and the unhydrated parts of pozzolana lead to the formation of low lime hydrates when only minor amounts of free lime are detected (24). On the other hand, richer mixtures, which contained 50, 70 and 80% pozzolana, showed the same behaviour at the later age of hydration (28 Days). Other ratios were transitional between both behaviours, in the time that the 5% acted similar to the former behaviour at hydration age of 14 days, the 20 and 35% followed the later one at 28 days too. Other than the previous mentioned ages, the compressive strength data of the hardened pastes indicated a continuous increment at most stages of hydration process from one day up to 180 days of hydration. The strength curves obtained for the pastes made of 30, 40, 45 % of burnt clay at 600°C, indicated this behaviour very well.

3.3 Hydration Kinetics

The hydration kinetics of the various Portland cement/artificial pozzolana pastes were studied mainly by determining the chemically-combined (non-evaporable) water (Wn %), as well as the free lime (CaO) contents at various ages of hydration.

3.3.1 Combined Water Content

The combined water content of cement pastes has been used as a measure of the degree of hydration (24). The results of non-evaporable (chemically-combined) water contents, (Wn %) are given in Table (4) at various ages of hydration for the hardened pastes made from the pozzolanic cements obtained by burning attapulgite clay at 600°C. The Wn values are also graphically represented as a function of age of hydration in Fig (2 a&b). For all of the hardened Portland cement/burnt clay pastes, there appeared a continuous increment in the combined water content with increasing age of hydration up to the final

stages of the hydration process (180 Days), and this is due to the reactions which takes place during these stages. During the early stages of hydration, the hydraulic reactivity of the artificial pozzolana made of burnt clay depends primirly on the burning temperature. It was found that the hydraulic reactivity of the clay burnt at 600°C is high. Obviously, a rapid hydraulic interactions between Portland cement and artificial pozzolana takes place after 1 day of hydration at room temperature leading to noticeable amounts of combined water contents for almost all pastes investigated. Later, the 'Wn' contents increase gradually with age of hydration up to the final stages of hydration. The hydraulic reactivity of the artificial pozzolana increases with increasing proportions of Portland cement in the solid mixture. Therefore, the hydraulic reactivity of the various Portland cement/pozzolana mixtures increase in the order, "Rich in Pozzolana < Rich in Portland Cement."

Table (4)
Combined Water Content (%) for Different Mixes of Clay Fired at 600(C / Ordinary Portland Cement

Mix Proportions	1 Day	3 Days	7 Days	14 Days	28 Days	90 Days	180 Days
5 / 95 %	16.01	18.03	18.55	18.69	19.21	24.85	30.0
10 / 90 %	22.44	33.73	37.26	38.97	40.00	43.00	46.00
15 / 85 %	15.11	21.61	20.06	20.36	17.68	26.59	29.00
20 / 80 %	8.85	17.18	24.72	29.57	20.20	27.46	32.0
25 / 75 %	14.60	18.42	17.24	17.64	20.04	24.60	19.89
30 / 70 %	36.36	41.13	33.37	41.74	40.51	42.00	44.00
35 / 65 %	14.69	19.78	18.86	20.96	20.78	24.53	27.00
40 / 60 %	18.00	20.32	31.77	19.25	19.01	19.17	20.16
45 / 55 %	12.80	18.49	18.58	20.92	19.08	19.76	21.42
50 / 50 %	13.90	19.25	20.85	23.00	28.20	28.31	29.10
70 / 30 %	13.65	23.15	23.90	24.60	20.10	21.30	22.10
80 / 20 %	13.20	16.70	17.15	15.45	24.50	27.65	29.00

3.3.2 Free Lime Content

The results of free lime (CaO %) content of the hardened Portland Cement/pozzolana pastes are given in Table (5). They reflect negligible amounts at all ages of the hydration reactions; therefore, the free lime released as a result of clinker hydration is almost consumed by the hydration reaction of the pozzolana exists in the blended cement (24).

Table (5)
Free Lime Content (%) for Different Mixes of Clay Fired at 600C / Ordinary Portland Cement

Mix Proportions	1 Day	3 Days	7 Days	14 Days	28 Days	90 Days	180 Days
5 / 95 %	3.90	3.23	7.20	0.0	6.20	5.32	3.31
10 / 90 %	0.00	0.00	0.00	0.00	0.34	0.52	0.83
15 / 85 %	0.39	0.23	0.15	1.23	0.91	0.32	0.95
20 / 80 %	0.00	0.00	7.32	9.27	5.31	4.92	7.90
25 / 75 %	4.03	6.94	4.15	4.55	5.21	6.03	3.21
30 / 70 %	0.00	0.00	0.00	0.00	0.13	0.00	0.00
35 / 65 %	3.43	3.14	5.32	6.02	5.32	2.30	0.74
40 / 60 %	0.00	0.00	0.49	0.34	0.00	0.49	0.53
45 / 55 %	3.11	6.29	6.03	5.20	3.47	4.23	4.10
50 / 50 %	0.00	0.00	0.00	0.00	0.00	0.00	0.00
70 / 30 %	0.00	0.00	0.00	0.00	0.00	0.00	0.00
80 / 20 %	0.00	0.00	0.00	0.00	0.00	0.00	0.00

Therefore, the complete consumption of all of the free lime released as a result of Portland Cement hydration indicates a higher hydraulic reactivity of the burnt clay. Evidently, the free lime contents given in Table (5) are underestimated values due to the increased sailinity of the pozzolanic cement mixtures which affect the extraction of free lime by the solvent extraction method used in this study.

CONCLUSIONS

From the results of this investigation the following conclusions could be derived:

- Mixtures of Qatarian burnt attapulgite clay and Qatarian ordinary Portland cement can be used in the production of blended cements with certain optimum conditions.
- Activation of the Qatarian attapulgite clay by grinding up to 75 (m and igniting at 600°C is suitable for the production of building products containing Qatarian attapulgite clay.
- 3. The author suggests that the suitable working ratios for the production of blended cement using ordinary Portland cement and the Qatarian attapulgite clay activated at 600°C are 10 35 % burnt clay.

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