EVALUATION OF Al-AMIJ AND AL-HUSSAINIYAT CLAYSTONES (IRAQI WESTERN DESERT) FOR THE PRODUCTION OF POZZOLANA

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ABSTRACT

Red kaolinitic claystone from Al-Amij and Al-Hussainiyat regions in the Western Desert of Iraq were evaluated for the production of artificial pozzolana as cement admixtures. Both claystones were calcined in the temperature range of (500 – 900)º C at 100º C interval, for 45 and 60 min duration. The chemical and mineralogical properties of both raw materials and calcined samples were studied; they display useful information that helps to predict the preparation of pozzolana from the claystone. The influence of the calcination temperature and time on pozzolanic activity index were found (as in ASTM C311) and discussed. The results obtained showed that the burning temperature and the duration of burning have significant effect on the activity of the produced pozzolana (calcined claystone). It was also found that the activity index of the pozzolana produced from Al-Amij claystone was higher than that produced from Al-Hussainiyat claystone.

تقييم أطيان العامج والحسينيات (الصحراء الغربية العراقية) لإنتاج البوزولانة

عبد الوهاب عبد الرزاق العجيل، فراس فيصل عبد الحميد، داليا خالد الدهان،
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المستخلص

لقد تم تقييم أطيان الكاؤولين الحمراء من منطقتي العامج والحسينيات الواقعتين في الصحراء الغربية العراقية لإنتاج البوزولانة كإضافات سمنتية، حيث تم حرق (كلسنة) هذه الأطيان بدرجات حرارة تتراوح مابين (500 – 900) درجة مئوية وبزمن استبقاء 45 و 60 دقيقة. كما وتم دراسة الخواص الكيميائية والمعدنية لكل من المواد الأولية والمواد الناتجة عن عملية الحرق، وقد وجد بأن هذه الدراسة لها فائدة في توقعات تحضير البوزولانة من هذه الأطيان. كما تم دراسة ومناقشة تأثير درجة حرارة الكلسنة والزمن على مؤشر فعالية البوزولانة وفق المواصفات الأمريكية المعتمدة (ASTM C311). بينت النتائج المستحصلة بأن درجة حرارة الحرق وزمن الاستبقاء لهما تأثير واضح على البوزولانة المنتجة، كما بينت النتائج بأن فعالية البوزولانة المنتجة من أطيان العامج أعلى من مثيلتها المنتجة من أطيان الحسينيات.

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INTRODUCTION

According to the ASTM C618, a pozzolana is defined as: "a siliceous or siliceous and aluminous material, which in itself possesses little or no cementitious value, but will, in finely divided form and in the presence of moisture, chemically reacts with calcium hydroxide at ordinary temperature to form compounds possessing cementitious properties".

The pozzolanas have to be rich in reactive silica (SiO_2) or alumina (Al_2O_3) plus silica (Bensted and Barnes, 2002). According to ASTM C618 the requirements of chemical composition of natural pozzolana are approximately 70% for the sum of SiO_2, Al_2O_3 and Fe_2O_3 of the total compounds that constitute the material, whereas, Al-Rawas *et al.* (2001) claimed that, the sum of these oxides should be greater than 50%. A wide variety of siliceous or aluminous materials, both natural and artificial may be pozzolanic, and historically the most widely used of these are volcanic ash and calcined clays (Al-Rawas *et al.*, 2001 and Howtopedia, 2007).

Calcined clays, however, seem to have the greatest overall potential as artificial pozzolanic material; due to the availability of clays (particularly kaolinitic clays) in large quantities in almost all regions of the world. The heating (calcination) process of the clay drives water from the mineral kaolinite (Al_2O_3. $2SiO_2.2H_2O$), the main constituent of kaolin clay, and collapses the material structure, resulting in an amorphous Metakaolin phase (Al_2O_3. $2SiO_2$.). The process is known as dehydroxlation (Grim, 1968), and can be presented simply by the equation:

$$\underset{\text{Kaolin}}{2(Al_2O_3.2SiO_2.2H_2O)} \xrightarrow{\text{Heat}} \underset{\text{Metakaolin}}{2Al_2O_3.4SiO_2} + 4H_2O\uparrow$$

Mineralogically; clays are varied a lot and the calcining temperature (to convert kaolinite to amorphous metakaolin phase) is crucial and affects the pozzolanic reactivity of the resultant product. It was claimed (Kakali *et al.*, 2001 and Shavarzman *et al.*, 2003) that the calcination temperature and time, significantly influence the dehydroxlation process and hence Metakaolin formation. Grim (1962), however, stated that, with calcination, the pozzolanic properties of kaolinite clays undergo remarkable changes, both physical and chemical, and he demonstrated that, calcination between 540º C and 870º C is highly effective. Also, it was stated that, calcining temperature that produces active pozzolana is usually in the range of (600 – 800)º C, and it can approaches 900º C (Sabir *et al.*, 2001; Paul *et al.*, 2001 and Castillo *et al.*, 2010). Thereafter, the increase in temperature resulted in recrystallization of kaolinite with the beginning of the nucleation of mullite phase ($Al_6Si_2O_{13}$) and the reactivity would be expected to decrease sharply (Grim, 1968). Thereupon, the calcining temperature and period of calcination together with the chemical composition should be optimized for determining the pozzolanic activity of the clay.

Pozzolanas are commonly used as an admixture to Portland cement to improve concrete characteristics, such as durability, compressive strength, chemical resistance (sulfate resistance), hydrated heat, workability and reduce its cost (Barger *et al.*, 2001 and Chakchouk *et al.*, 2006). A lot of researches have been performed over the years on using calcined clays as a pozzolanic material for mortar and concrete. Al-Rawas *et al.* (2001) and Al-Rawas and Hago (2006) investigated the potential of clay from five locations (Al-Khod, Soor Al-Haboos, Al-Fulaij, Al-Hamra, and Al-Awabi) in Northern Oman for the production of artificial pozzolana. The clays were calcined at a temperature range of (740 – 800)º C for 1 hour. The results showed that, the pozzolana produced from the calcination of Al-Fulaij clay has the higher strength than the other clays. Chakchouk *et al*. (2006) and Samet *et al*. (2007) studied the activity of clays from different locations in Tunisia (Mednine, Zarmdine, Gabs, Tabarka); the clays were calcined at (600, 700 and 800)º C. It was found that, clays rich in kaolinite and calcined at 700º C present the best pozzolanic activity. A recent study performed by Ul-Amin (2010) on clay from Nowshera District, Pakistan as a pozzolanic material in high strength Portland cement indicated that the thermal treatment of the clay can increase its pozzolanic activity. The optimal results obtained were with calcined clay at 600º C. The study carried out by Biljana *et al*. (2010) on clay from Serbia indicated nearly the same results. Subsequently, the objective of this paper is to view the evaluation of Al-Amij and Al-Hussainiyat (Fig.1) colored kaolinitic claystone from the Western Desert of Iraq for the preparation of pozzolanic material.

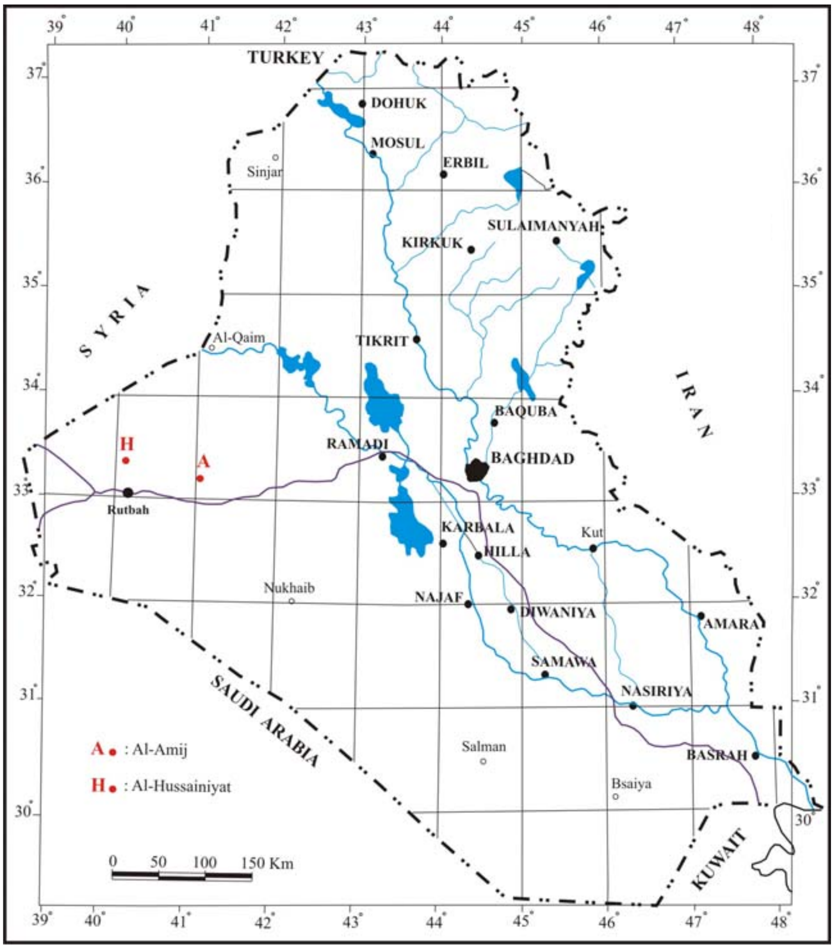

Fig.1: Location map of the claystone deposits

MATERIALS AND METHODS

The kaolinitic claystones (Al-Amij and Al-Hussainiyat), which are located in the Western Desert of Iraq near Al-Rutbah town (Fig.1) are known in the lower parts of the Hussainiyat Formation (Early Jurassic), along Wadi Hussainiyat and in Amij Formation (Middle Jurassic) at Wadi Amij. Both claystones are of low grade and red in color due to the presence of appreciable amount of iron oxide (Mahdi *et al*., 1990 and Mahdi and Al-Delaimi, 1999). Those authors indicated that, kaolinite is the dominant clay mineral in these claystones deposits, associated with goethite, hematite, quartz, and calcite; as well as trace of gypsum.

Ordinary Portland cement and sand were used also to study the Pozzolanic activity. The cement was brought from Tasluja Cement Factory, Sulaimaniyah town, while the sand was brought from Al-Ukhaider region, central part of Iraq. Raw claystone samples from Al-Amij and Al-Hussainiyat deposits were crushed to pass 20 mesh (850 micron); ASTM sieves, and then split into multiple identical samples, using laboratory rifle splitter prior to experiments. The chemical and mineralogical analyses of the claystones were characterized in the Central Laboratories of Iraq Geological Survey. The chemical composition was determined by X-ray fluorescence type Shimadzu-1800, whereas the minerals present in the samples were identified by X-ray diffraction (XRD), using Shimadzu-7000 diffractometer. The generator settings were 40 KV and 30 mA, and the wave length (λ) was 1.54 Å. A scanning rate of 2Ө/min from 3º to 60º was considered.

Sand sample was sieved and predominantly graded between 30 mesh (600 micron) sieve and 100 mesh (150 micron) sieve according to the ASTM C 778 (Table 1). Each sand fraction was washed with HCl and then carefully washed with water to remove the acid and clays.

The physical properties of the ordinary Portland cement were tested in the Building Research Center (Table 2) and found in accordance with the requirements of the ASTM C 150. The graded sand also coincides with the specification given in ASTM C 778. The density of the used cement was measured according to ASTM C188 test procedure.

Table 1: Grain size characteristics of Al-Ukhaider sand

ASTM Mesh/mm	Al-Ukhaider sand % passing	ASTM C 778 requirements
16/ 1.18	100	100
30/ 0.600	98	96 to 100
40/ 0.425	70	65 to 75
50/ 0.300	25	20 to 30
100/ 0.150	2	0 to 4

Table 2: The physical properties of ordinary Portland cement compared with ASTM C 150

Physical properties (unit)	Tasluja cement	ASTM C 150
Fineness (m^2/Kg)	302	280
Setting time		
Initial (min)	95	not less than 45
Final (min)	255	not more than 375
Compressive strength (MPa)		
3 days	15	12
7 days	23	19
28 days	28	28
Soundness (%)	0.12	0.8
Density (g/cm^3)	3.2	–

▪ **Clay Calcination (Pozzolana Preparation)**

The main important process for production of high reactivity pozzolana from kaolin clay is calcination. To optimize the range of calcination temperature and time; of the claystones (Al-Amij and Al-Hussainiyat), which should produce material that possess the highest pozzolanic activity, claystone samples from both regions were calcined at a temperature range of (500 – 900)º C at 100º C interval for different times (45 and 60 minutes). The calcination was carried out from ambient in a controlled laboratory electrical rotary furnace (Herrmann-Moritz furnace 14 – 82). The calcined materials were, then ground to pass 325 mesh (– 45 micron) sieve, and a specimen from each calcined sample was taken for XRD examination. Also specimens were collected for chemical analysis and pozzolana physical properties determination. The fineness, however of the calcined clay (prepared pozzolana) was determined in accordance with the procedure given in the test method of ASTM C 430. It indicates the amount of the sample retained when wet sifted on 325 mesh sieve. The tested samples were completely passed through this mesh. The densities of the prepared pozzolana were measured according to ASTM C188, the results show that all the produced pozzolana from Al-Amij claystone has a density of 2.7 g/cm^3, while that produced from Al-Hussainiyat claystone was of 2.9 g/cm^3.

▪ **Pozzolanic Activity**

The strength activity indices of the prepared pozzolanas (calcined claystone) were evaluated, according to the procedures outlined in ASTM C311 and specified by ASTM C618. The tests were conducted in the Building Research Center. In this test, the 7 days and 28 days compressive strength of cement-pozzolana mixture was compared to that without pozzolana. Samples were prepared for each calcination temperature and duration for each claystone site. The mixture (pozzolana + cement) should provide 75% of the strength of the control (cement only) at 7 d and 28 d, according to ASTM 618 specification. The water requirement and compressive strength for the strength activity indices were determined according to the procedure given in ASTM C109.

RESULTS AND DISCUSSION

▪ **Raw Materials Characteristics**

The chemical analysis of Al-Amij and Al-Hussainiyat raw claystone samples used in this work is given in Table (3). The results indicate that both Al-Amij and Al-Hussainiyat claystones have silica (SiO_2) contains of 45% and 42%, alumina (Al_2O_3) of 28% and 24% with about 7% and 13% (Fe_2O_3), respectively.

According to ASTM C618 (98), the criteria required for good pozzolanic activity from claystone, the total of silica, alumina and iron oxide content should be greater than 70%. Therefore, from the chemical point of view, it is most likely that Al-Amij and Al-Hussainiyat claystones can produce good pozzolanas, if they are properly calcined. The mineralogical characteristic of the studied claystone samples are presented in XRD pattern (Fig.2). This result indicates that, the claystones are rich in kaolinite, and it is the main and only clay mineral present in the claystones. Besides, hematite, quartz, goethite and anatase are also identified as non clay mineral impurities. The peak intensity of the hematite (H) and goethite (G) in Al-Hussainiyat claystone sample appear to be more intense than that of Al-Amij claystone, which is a reflection of its high iron oxide content (Table 3). However, it was demonstrated, that claystones rich in kaolinite are capable for producing a high activity pozzolanic materials (Ayub *et al*., 1988; Al-Rawas *et al*., 2001, and Chakchouk *et al*., 2006). Therefore, it can be expected that Al-Amij and Al-Hussainiyat claystones would show good pozzolanic activity after proper heat treatment.

Table 3: Chemical composition of the studied raw claystones
(Al-Amij and Al-Hussainiyat) (in wt%)

Claystone	SiO_2	Fe_2O_3	Al_2O_3	TiO_2	CaO	MgO	SO_3	L.O.I	Na_2O	K_2O	Cl^-
Al-Amij	45.32	7.81	28.07	1.34	1.44	0.92	0.29	12.65	0.62	0.81	0.65
Al-Hussainiyat	42.77	13.6	24.53	1.59	0.86	1.88	0.21	12.53	0.34	0.54	0.15

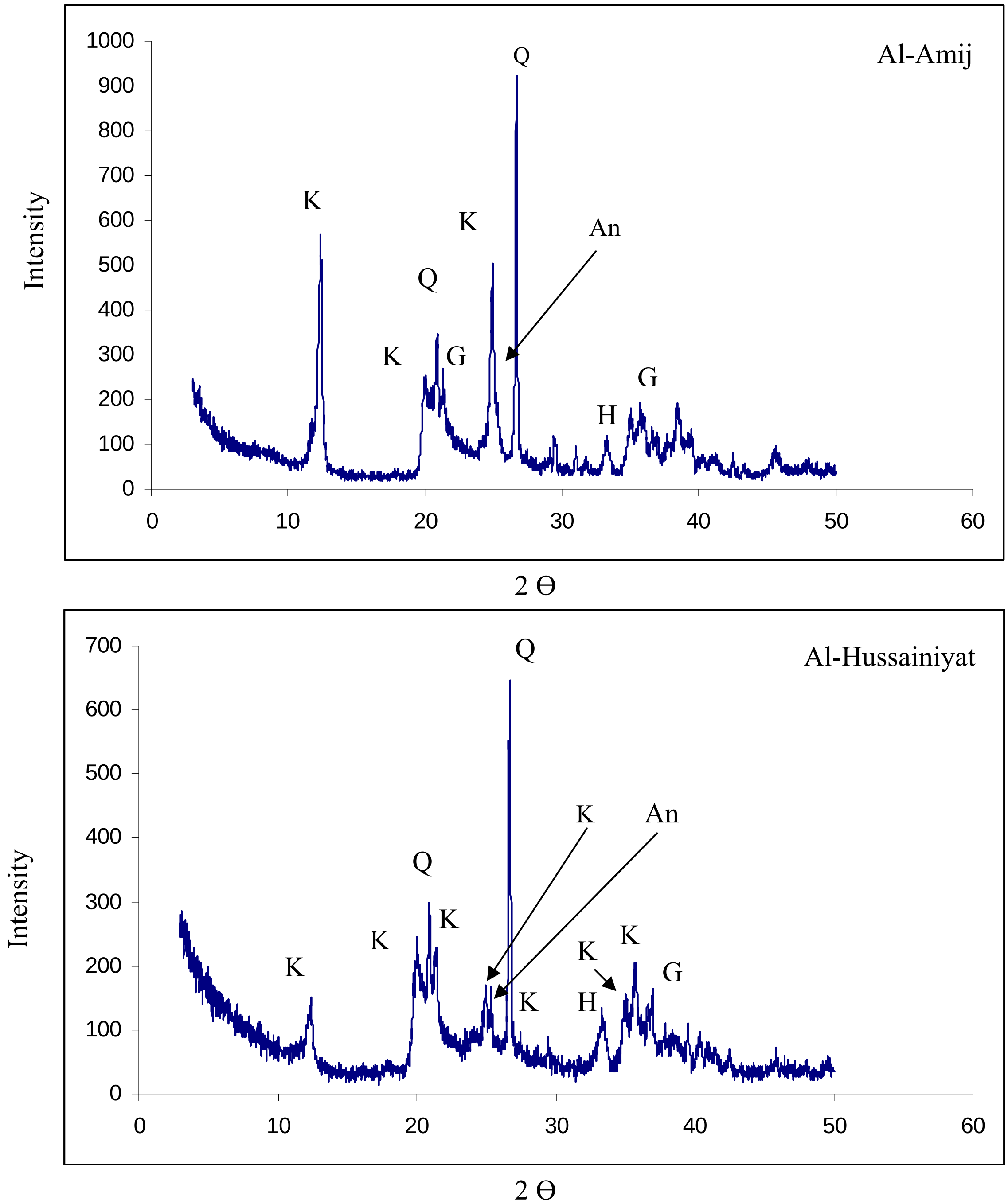

Fig.2: XRD pattern of Al-Amij and Al-Hussainiyat raw claystones,
(K: Kaolinite, Q: Quartz, An: Anatase, G: Goethite, and H: Hematite)

- **Chemical and Mineralogical Characteristics of the Calcined Claystone (Prepared Pozzolana)**

The chemical compositions of the calcined Al-Amij and Al-Hussainiyat claystones are given in Table (4). The results display that the ∑ of SiO_2 + Fe_2O_3 + Al_2O_3 of Al-Amij calcined claystone are in the range of about (88 – 91) %, whereas that of Al-Hussainiyat claystone are in the range of about (89 – 92) %, which conform the requirements (70% min.) given by the ASTM C618. Also the result indicated that the SO_3% does not exceed 0.95% in Al-Amij and 0.44% in Al-Hussainiyat calcined claystones, which are far below the maximum (4%) permissible limit; stated by ASTM 618 specification. Furthermore, the maximum value of the L.O.I in both calcined claystones obtained was of about 4%, yet again it is below the allowed level (10% max.) prescribed in the ASTM C618 (Table 4). According to these results, the calcined claystones; from both sites chemically match the chemical requirements of the pozzolana materials; as specified by the ASTM C 618 (type N). The mineralogical characteristics of the calcined Al-Amij claystone samples with respect to different temperatures (500, 600, 700, 800 and 900º C) for various times (45 and 60 min.) are presented by the XRD patterns (Figs.3 and 4), respectively, whereas those of Al-Hussainiyat claystone are shown in Figs. (5 and 6). In regard to Al-Amij claystone, the pattern of Fig. (3) shows that the main peaks at 2θ 12.41º and 24.9º corresponding to kaolinite initially present in the raw material (Fig.2) remain clear and not affected in the samples heated at 500º C for all duration tested. The same is true for Al-Hussainiyat claystone (Fig.5). Yet, this peak disappeared as the samples heated above 500º C (Figs.3, 4, 5 and 6), collapses of the clay structure occur, resulting in an amorphous phase (Metakaolin) due to the dehydroxlation of the kaolinite (Grim, 1962 and Biljana *et al*., 2010). The presence of a dome, however, between 20º and 30º 2θ confirms the transformation of the kaolinite to the amorphous phase. For Al-Hussainiyat samples calcined at 600º C (Fig.5), it can be seen that a small peak of the 2θ 12.41º appeared at calcination time of 45 min, and disappeared as the time raises to 60 min. Figures. (5 and 6), however display that the characteristic peaks for kaolinite 12.41º and 24.9º 2θ disappeared in all samples heated at (700, 800 and 900)º C for the times (45 and 60) min. On the other hand, the peaks at approximately 20.21˚ 2θ were persisted all the way through the temperature range (500 – 800)º C, and then disappeared after heat treatment at 900º C. Generally, all samples except that heated at 500º C, show a broad band in the range of 20º to 30º 2θ, consistent with the presence of amorphous state of the Metakaolin phase (Chakchouk *et al*., 2006 and De Geulierrez *et al*., 2008).

- **The Pozzolanic Activity**

The test results following the ASTM standards are shown in Table (5) for water requirements, compressive strength and strength activity indices at 7 days and 28 days (d). The result, shows that the water requirements for the strength activity index of the prepared pozzolana (calcined clay) from both claystone sites are in the range of (260 – 262) milliliter (ml), which is higher by about (18 – 20) ml than that (242 ml) of the control (cement). This, however, is in accordance with the requirements established in the ASTM C618 specification (not more than 115% of the water requirements of the control). The results in Table (5) indicated that, all the activity indices of the samples tested at both 7 days and 28 days were well above the 75% level prescribed in ASTM C618. The calcined claystone samples appear to be practically active in combination with the Portland cement. However, it was claimed (Shavarzman *et al*., 2003) that the burning or calcination temperature of the clay as well as the residence time affects the pozzolanic activity of the resultant product. Figure (7) shows the relation between strength activity index (28 days) and calcination temperature (600, 700, and 900º C) at duration of (45 and 60) min for both claystone regions. The data in this figure shows a well consistency, the activity increases slightly as the temperature rises to 700º C and then significantly decreases with increase in calcination temperature. This decrease in reactivity is due to the formation of less reactive Metakaolin and occurrence of some form of recrystallization in the calcined materials. The results in general, indicated that the highest pozzolana reactivity was achieved, when the claystones from both sites (Al-Amij and Al-Hussainiyat) were calcined at 700º C for (45 and 60) min, respectively. It is also found (Fig.3) that the pozzolana prepared from Al-Amij claystone has higher activity from that of Al-Hussainiyat claystone. According to the test results (Table 5) and in regard to Al-Amij site, it can be seen that, the reactivity (114%) at 600º C for 45 min is slightly lower than that (120%) obtained at 700º C for 60 min (the same behavior occurred for Al-Hussainiyat claystone). Therefore, it can be stated that the optimum calcination temperature from technical economical standpoint to obtain a high pozzolana activity, is 600º C for duration of 45 min for Al-Amij claystone and 60 min for Al-Hussainiyat claystone.

Table 4: The chemical composition of the calcined claystones (Al-Amij and Al-Hussainiyat)

Material	Calcined sample (code)	Calcination condition temp/ time (ºC/ min)	Wt%					∑ SiO_2 + Al_2O_3 + Fe_2O_3
			SiO_2	Al_2O_3	Fe_2O_3	SO_3	L.O.I	
Al-Amij claystone	A1	600/ 45	50.62	30.95	8.5	0.75	2.6	90.1
	A2	600/ 60	49.28	30.01	8.6	0.74	3.44	87.89
	A3	700/ 45	50.56	31.2	8.7	0.33	2.27	90.46
	A4	700/ 60	49.42	30.7	8.5	0.77	4.08	88.62
	A5	900/ 45	50.35	31.2	8.4	0.95	2.59	90
	A6	900/ 60	50.8	31.3	8.6	0.92	2.15	90.6
Al-Hussainiyat claystone	H1	600/ 45	47.5	26.67	15.11	0.35	2.51	89.58
	H2	600/ 60	46.7	26.78	14.9	0.17	3.08	88.5
	H3	700/ 45	47.36	27.07	15.0	0.32	2.81	89.43
	H4	700/ 60	47.0	27.0	15	0.15	3.13	89
	H5	900/ 45	47.9	27.8	15.34	0.44	1.78	91
	H6	900/ 60	49.0	28	15.4	0.27	0.67	92.4
ASTM C 618 specification						**4.0% max.**	**10.0% max.**	**70% min.**

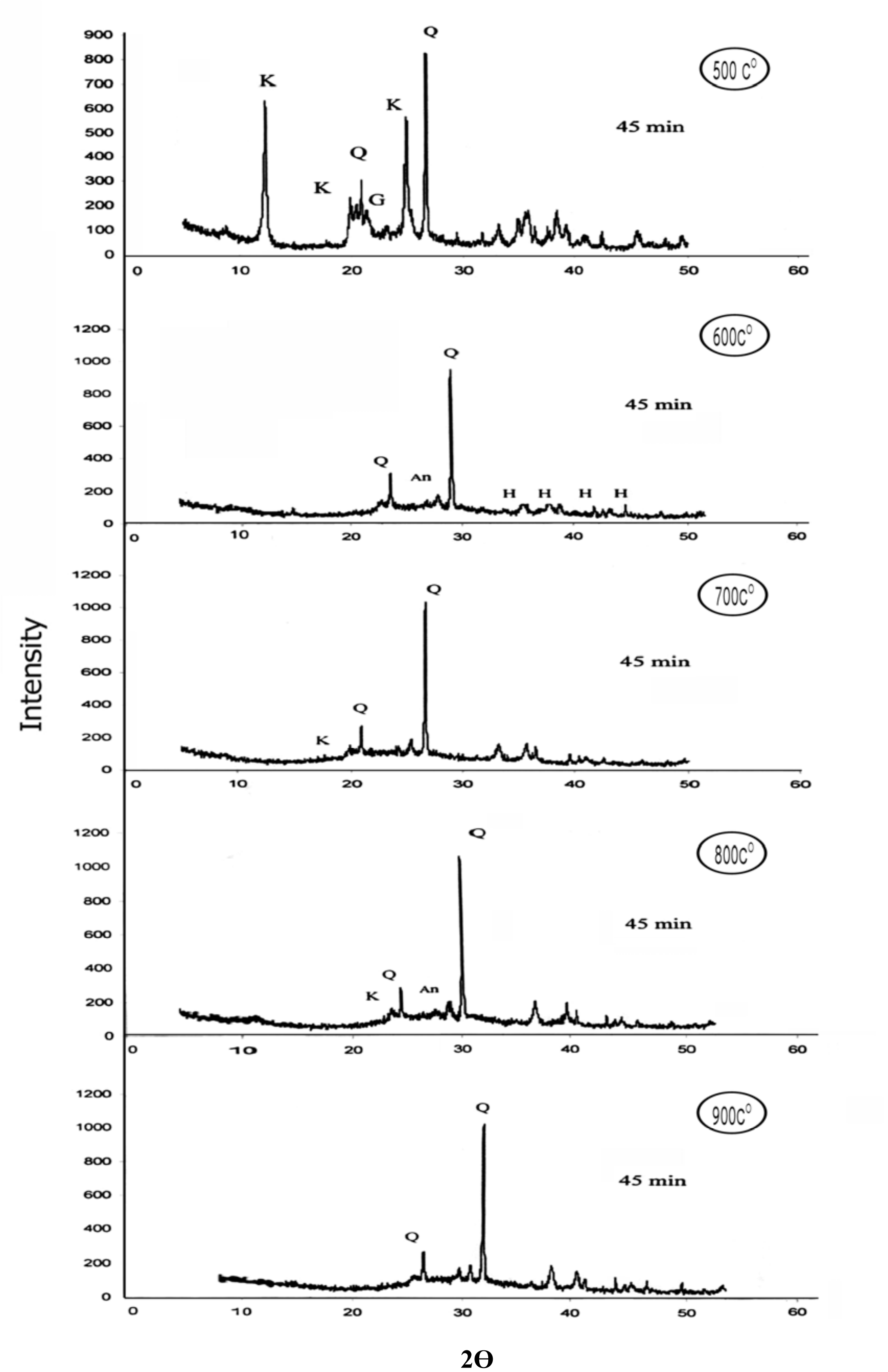

Fig.3: XRD Patterns of calcined Al-Amij claystone at different temperatures for 45 min (K: Kaolinite, Q: Quartz, An: Anatase, G: Goethite, and H: Hematite)

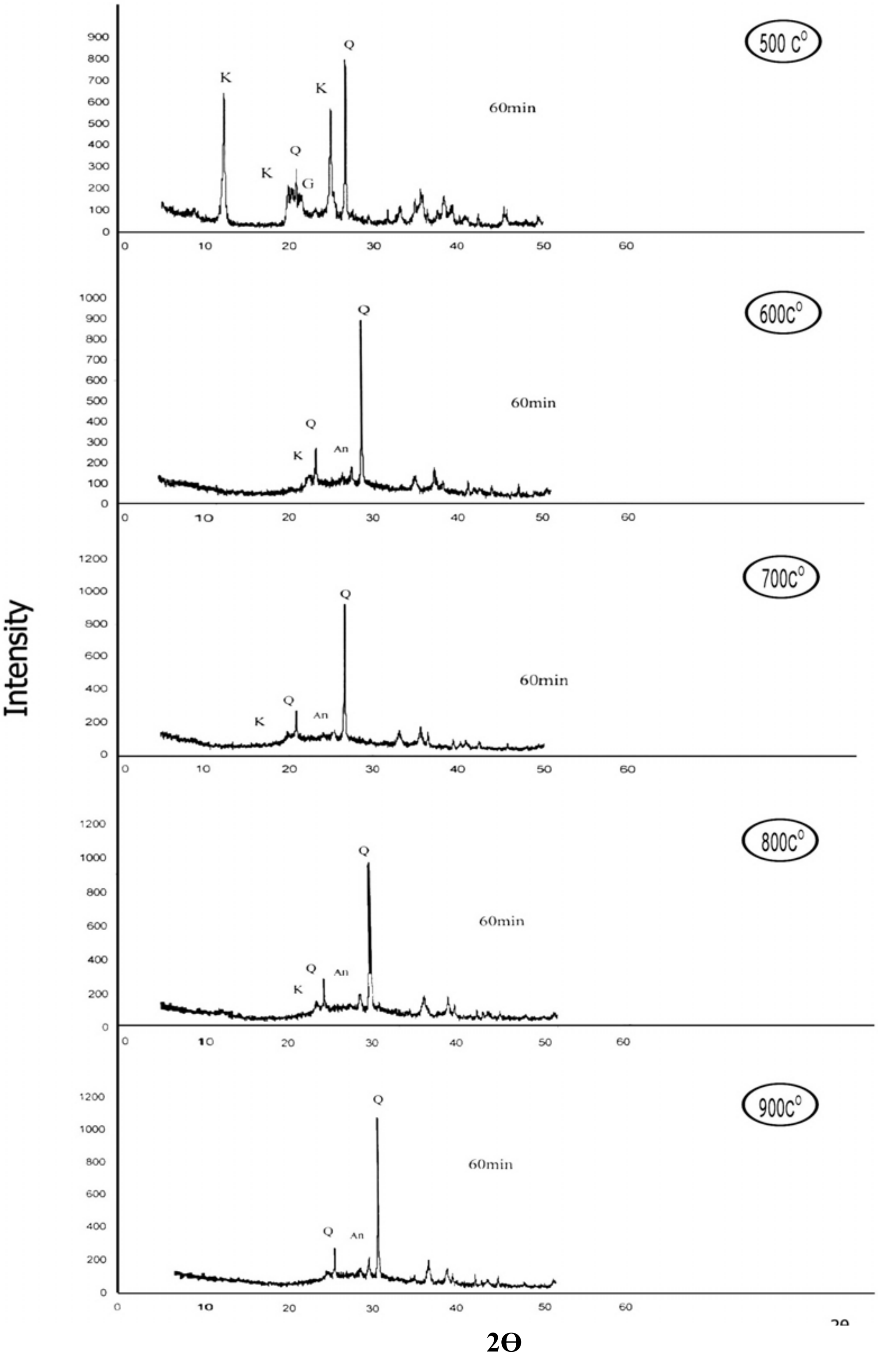

Fig.4: XRD Patterns of calcined Al-Amij claystone at different temperatures for 60 min (K: Kaolinite, Q: Quartz, An: Anatase, G: Goethite, and H: Hematite)

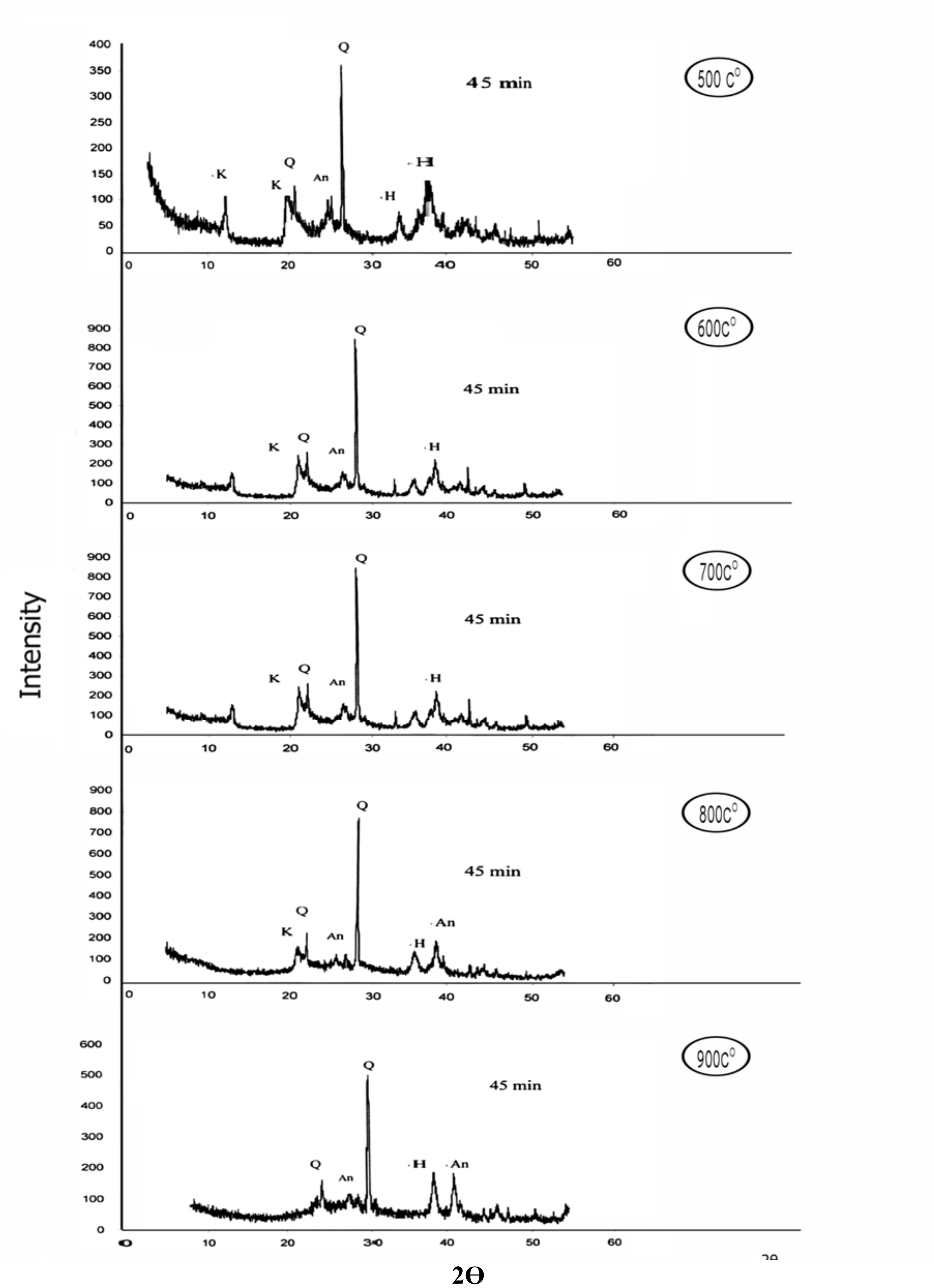

Fig.5: XRD Patterns of calcined Al-Hussainiyat claystone at different temperatures for 45 min
(K: Kaolinite, Q: Quartz, An: Anatase, G: Goethite, and H: Hematite)

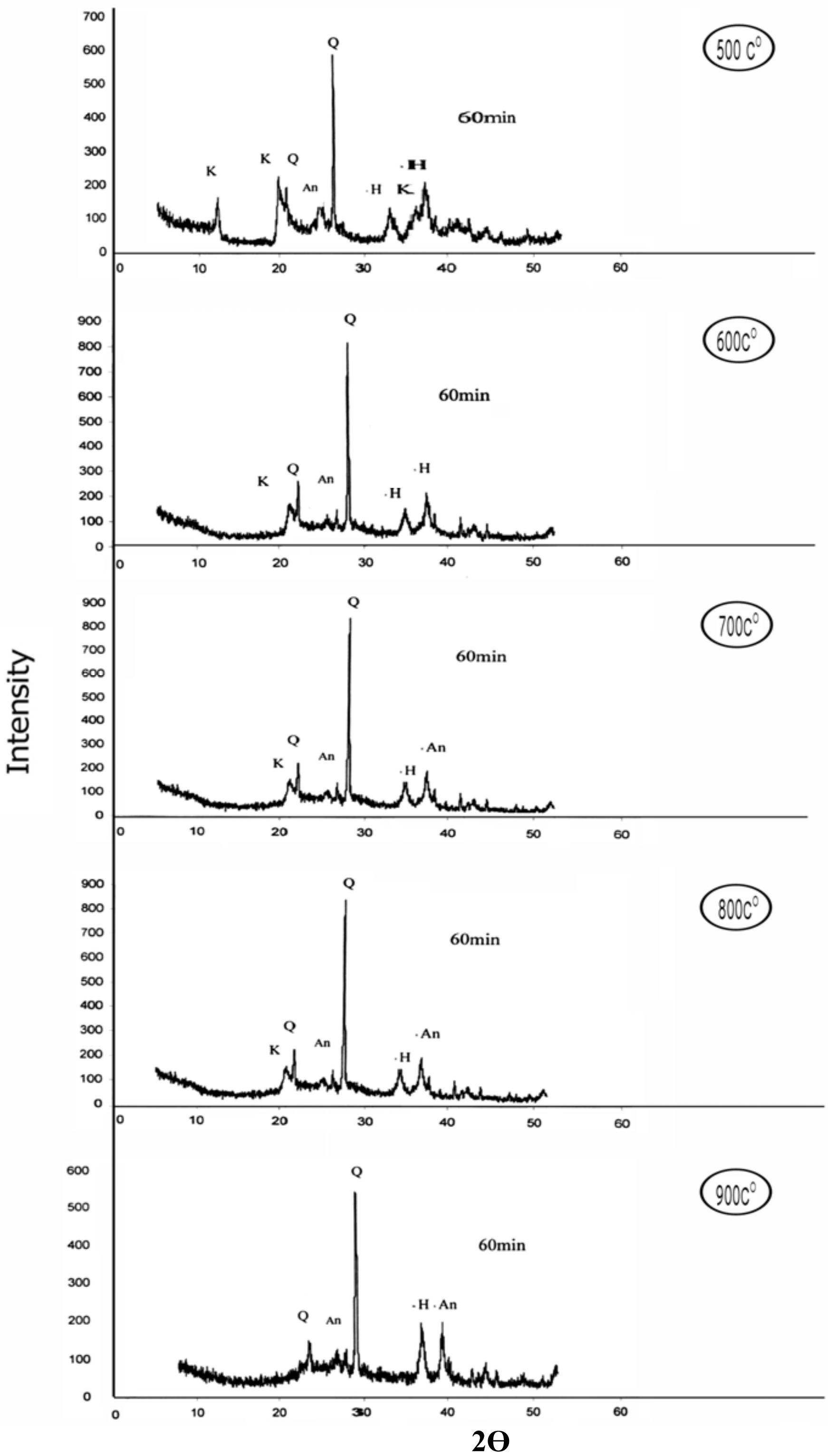

Fig.6: XRD Patterns of calcined Al-Hussainiyat claystone at different temperatures for 60 min (K: Kaolinite, Q: Quartz, An: Anatase, G: Goethite, and H: Hematite)

Table 5: Results of prepared pozzolana (calcined claystone) from Al-Amij and Al-Hussainiyat

Sample Code	Water requirement (ml)	Compressive strength (MPa)		Activity Index (%)	
		7 d	28 d	7 d	28 d
A1	262	28.4	31.8	142	114
A2	260	26.4	28	132	100
A3	260	28.6	33.6	143	120
A4	260	25.8	29.2	129	104
A5	262	24	31	120	111
A6	262	20.6	24.2	103	86
H1	262	24	24.6	120	88
H2	262	21	27	105	100
H3	260	22.2	25.2	111	90
H4	260	26	28	130	100
H5	260	16	23	80	82
H6	262	17.7	23.3	88.5	83
Control (cement)	242	20	28	100	100
ASTM C618	**115% of the control**			**75**	**75**

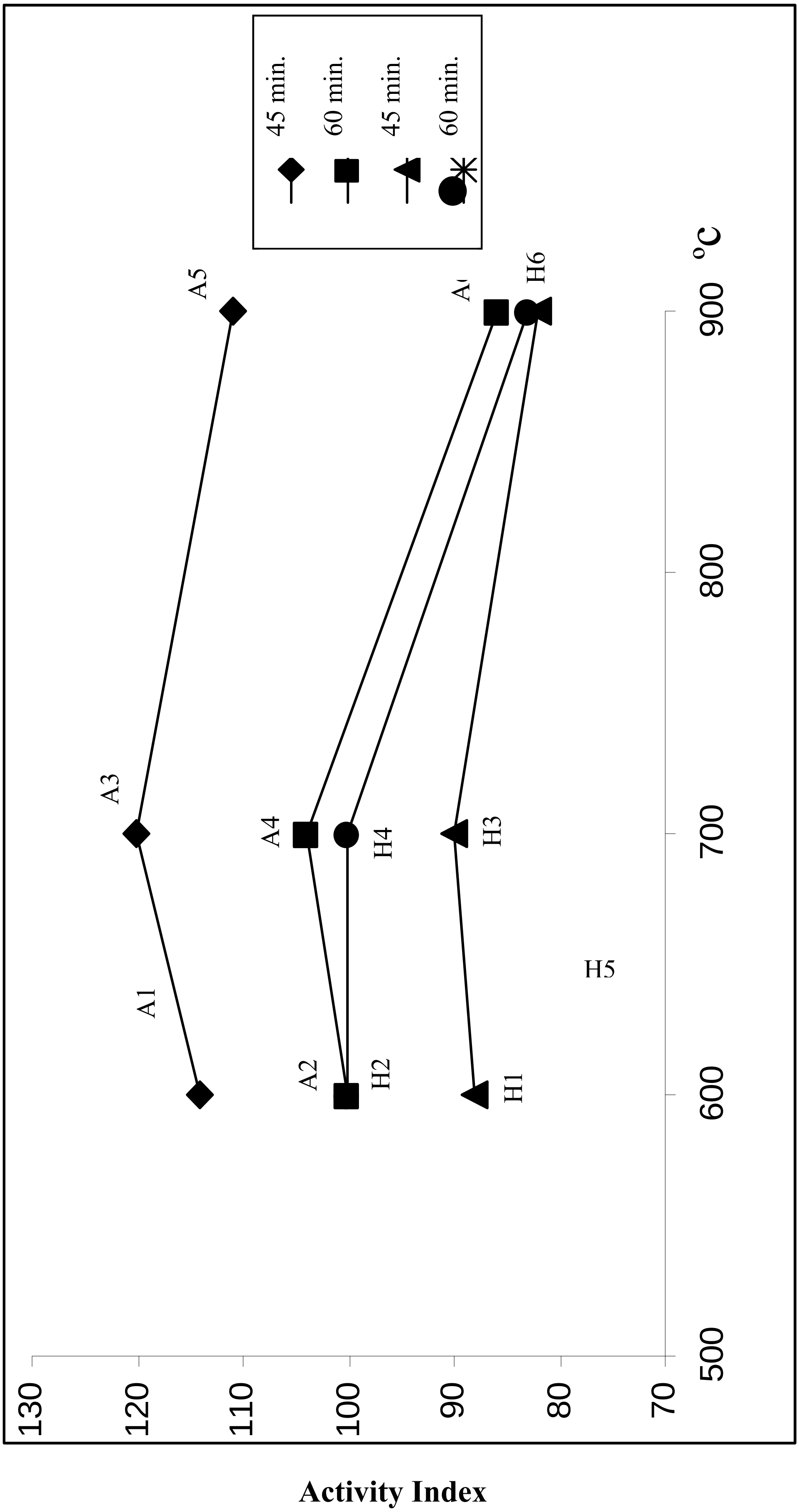

Fig.7: The relation between Activity index and Calcinations temperature for Al-Amij and Al-Hussainiyat claystones

CONCLUSIONS

According to the test results, the following conclusion can be stated:

- The claystones from Al-Amij and Al-Hussainiyat regions are quite good for the production of pozzolana material.
- The produced pozzolana is chemically and physically conformed to the requirements of ASTM C 618 type (N).
- The kaolinitic claystone from Al-Amij is much better than that of Al-Hussainiyat for producing more active pozzolana needed for manufacturing the Portland pozzolana cement.
- Economically, calcination at 600º C for 45 min is well enough to produce active pozzolana.

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