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Utilization of Metakaolin as an Inhibitor of Alkali Silica Reaction in Cement Mortars containing chert and silicified limestone aggregates

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Abstract
The following work aims at investigating the utilization of meta-kaolin and calcined brown smectite rich clays as inhibitors of alkali silica reaction (ASR) and improving the compressive strength in Type 1 ordinary Portland cement mortars including sand size chert or silicified limestone. Mortars were cured and tested at 56 days age. The compressive strength values increased to 40 MPa for the sample with 10% of Metakaolin-and 3.64% of chert content. The compressive strength values increased to 30 MPa when calcined brown clay was used instead of Meta-kaolin at the same proportion of chert.. Maximum compressive strength values of 36 Mpa and 30 Mpa were achieved when metakaolin and calcined brown clay were added to the reference mortars at a range of 5-10% and 8% respectively. Compressive strength was varied according to reactive aggregate type and percent in addition to the calcined brown clay and meta-kaolin content in the tested mortars. Scanning electron Microscope images (SEM) have indicated the absence of cracks usually related to swelling silica gel due to the consumption of the gel through its reaction with kaolin and calcined brown clay.
Keywords: chert, silicified limestone, metakaolin, alkali silica reaction

1. Introduction

Alkali silica reaction is a deleterious reaction that takes place in concrete under some circumstances. ASR is activated if alkalinity index of cement (Na_2Oe) is more than 0.6 and high microcrystalline silica (chert aggregates or silicified limestone) are used for concrete production.
Equivalent alkalis index in Portland cement expressed as:

$Na_2Oe = Na_2O\% + 0.658 \times K_2O\ \%)$

According to (ASTM C 150), the specified maximum alkalinity index for type 1 OPC is 0.6 by weight of cement. ASR output is a silica gel. The gel is characterized by its swelling ability due to the reaction nature in which the output gel has a higher volume than the input of (cement alkali_aggregate silica) reactive ingredients. Internal tensile stresses are created due to the swelling activity that leads to concrete cracking.

The addition of pozzolanic material will help to rapidly tie up alkalis and inhibit their mobilization. Bektas (2004) studied Alkali reactivity of mortars containing chert and incorporating moderate-calcium fly ash and concluded that the expansion decreased as the fly-ash amount increased. Kakodkar et al. (1997) reported that the addition of Class C fly ash at any level caused a decrease in the expansions of mortar formed with highly reactive aggregates.

Different mineral admixtures as metakaolin, silica fume, or artificial zeolites can be used to inhibit ASR (Malvar et al., 2002). The microstructure characteristics of concrete including pore structure and interfacial transition zone (ITZ) were highly affected when metakaolin is added to concrete (Ping Duan et al., 2013).

Alkalis consumption takes place through alkali pozzolanic reaction to produce extra calcium silicate hydrate (C-S-H) when high calcium ash is added to mortars (Abdul Hadi et al., 2008; Abdul Hadi et al., 2009; Abdul Hadi et al., 2014).
Through utilization of natural and industrial mineral admixtures as cement substitutes, strength was increased by adding some siliceous additives to sand cement mortars as reported by (Maaitah, et al., 2015). The capillary pores were decreased and the ASR was inhibited through immobility of alkali ions. At early ages of concrete, expansion is inhibited by adding fine grained pozzolanic materials to the Ca-rich environment. The formed alkali-silicate gel quickly reacts and converts to C-S-H, (http://www.understanding-cement.com).

Investigation of stress in high performance concrete with different metakaolin content revealed an increase of compressive strength with increasing metakaolin from 5% up to 30% (Qian and Li., 2001).

Calcined clay as pozzolanic material has an influence on ASR in mortar bars and causes a significant reduction in expansion at a replacement level of 25% (Sarfo-Ansah, J., et al., 2014).

Compressive strength increased with increasing curing temperature when Metakaolin prepared from calcinations of kaolin was added to ordinary Portland cement and cement mortars, (Potgieter and Vermaak, 2006).

The presence of chert and/or silicified limestone aggregates in concrete is responsible for the deleterious ASR reaction between hydrated cement alkalis and microcrystalline silica. Alkali silica gel with swelling characteristics is the product of the reaction. Cracks in concrete leads to decrease compressive strength and durability as a result of internal tensile stresses caused by swelling pressure of the gel. Quarries around Amman, both active and, abandoned utilize indurated limestone to produce varying size aggregates for use in concrete production. The best formation for extracting the limestone is the Wadi Es Sir Formation known as A7 (Parker, 1970) and the Massive Limestone Unit, (Bender, 1974). Wadi Es Sir Formation consists of around 90 m of various types of limestone (Abed, 1982).

The Wadi Es Sir Formation is overlain by about 20m of soft, white chalk known also as the B1 Formation (Parker, 1970). It forms the base of the Belqa Group (Bender, 1974; Powell, 1989). The Ghudran Formation is followed upwards by the Amman Formation (B2a) known as Silicified Limestone Amman (Bender, 1974) or Silicified Limestone Formation (ASL) (Parker, 1970) or the Silicified Limestone (Powell, 1989; Abed, 2000).

The Amman Formation in this area is around 65 m thick and consists of alternating beds of chert and various types of limestone in the order of same magnitude **(Fig.1).** Because of the high chert content in the Amman Formation, the limestone horizons are not suitable for quarrying. However, blocks or debris of chert and silicified limestone might fall down to the Wadi Es Sir Limestone quarries and get mixed with the limestone.

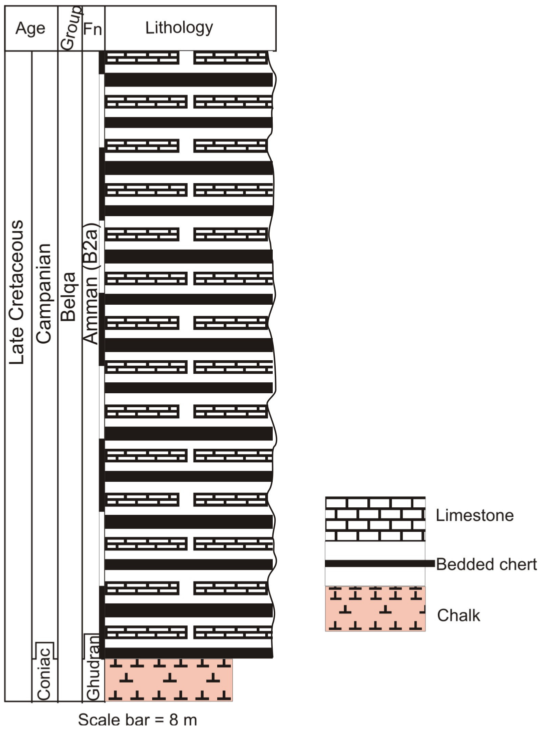

Fig. 1 Generalized columnar section showing the lithology of the Amman Formation

Aggregate containing more than 3% of chert and chalcedony are susceptible to alkali silica reaction acc0rding to (ACI 221, 1998).

The mineralogical composition of cherts varies from very fine-grained opaline silica to cryptocrystalline silica and from chalcedony to microcrystalline quartz, (Williams et al., 1982). The reactivity of an aggregate primarily depends on its geological background, internal structure and physical characteristics (Mohammad S.et al., 2013).

The following work aims at utilizing meta-kaolin and calcined brown clay in Type 1 ordinary Portland cement mortars with variable content of chert and silicified limestone as inhibitors of ASR.

2. Materials and laboratory techniques

Grayish to black chert and White creamy silicified limestone aggregates were collected from active quarries in the vicinity of Amman area. Both varieties were crushed utilizing Loss Angles machine for 10 cycles. Passing sieve # 100 (0.149 mm) was collected from each sample.

White creamy dry bulk kaolinite sample was taken from Batn Al Ghoul area, south of Jordan. Smectite rich brown clay soil was collected red soil deposits west of Amman. Both kaolin and brown clay were combusted using automatically controlled furnace at a temperature of 750C^o for three hours and allowed to cool at the lab temperature.

The produced metakaolin and calcined brown clay samples were ground and passing # No. 200 (0.073 mm) was collected and stored in tight plastic bags. The combusted samples were labeled as meta-kaolin (MK) and calcined brown clay (CBC).

Meta-kaolin and calcined smectite rich clay (brown clay) were added separately to sand cement mortars with variable chert and another mortars with variable silicified limestone content to investigate the possibility of decreasing or inhibiting the expected ASR effect in the tested mortars.

Glass sand for mortar samples was collected from Dabbet Hanout, 240 km south of Amman

Portland cement Type I according to **ASTM C150** Standard Specifications was used in all mortar samples.
Compressive strength for the various mixtures was determined using a computerized compression machine Digital Compression Machine (Type: ADR- manufactured by ELE).

X-ray fluorescence (XRF), X-ray diffraction (XRD), and scanning electron microscope techniques (SEM) were used to identify the alkali-silica and alkali pozzolanic reactions in different mortars

X-ray efflorescence (XRF) for some cured mortar samples were obtained utilizing (XRF-Pioneer F4, manufactured by Broker at the labs of Natural Resources Authority, NRA).

Scanning electron microscopy (SEM) for some 56 days cured mortars was carried out to investigate the ASR effect.

The morphology of the specimens was studied using an Inspect F50 Scanning Electron Microscope (Netherlands). The samples were pre-coated with platinum under an argon atmosphere

3 Testing procedure

3.1 Chemical analysis of raw materials

X-ray fluorescence technique was used to determine the percent of oxides content by weight for chert (CH), silicified limestone (SL), brown clay (BC), calcined brown clay (CBC), kaolin (K) and meta-kaolin (MK). The major oxide content as weight percent as SiO_2 , Al_2O_3, Fe_2O_3, Fe_2O_3, CaO, MgO, P_2O_5 and Na_2O were determined.

3.2 Mortars with Meta-kaolin

Water-cement ratio (w/c) ratio was constant in all mixtures. The fresh mixed mortars were molded in 5x5x5 cm cubes and cured at laboratory temperature until further testing at 56 days.
Meta-kaolin was added with different proportion to standard cement mortars (Rf) by weight% of cement. The purpose of these trials was to determine the effect of meta-kaolin on the compressive strength of the tested mortars. The mortars with different metakaolin content were nominated as MK5, MK10 and MK20 as shown in Table 1.

Table 1 Mortars with metakaolin

S. No.	Cement	Sand	MK %
Rf	500	1375	0
MK5	500	1375	5
MK10	500	1375	10
MK20	500	1375	20

3.3 Mortars with calcined brown clay

Calcined brown clay was added instead of Metakaolin in another set of mortars with proportions of 5%, 10% and 20%. The Mortars were nominated as CBC5, CBC10 and CBC20 as shown in Table 2. The purpose of these trials is to compare the pozzolanic activity of Meta-kaolin and calcined brown clay in sand cement mortars

Table 2 Mortars with calcined brown clay

S. No.	Cement	Sand (g)	Calcined clay%
Rf	500	1375	0
CBC5	500	1350	25
CBC10	500	1325	50
CBC20	500	1275	100

3.4 Mortars with chert (CH) and silicified limestone (SL)

The purpose of these trials is to investigate the reactivity of these silica rich fine aggregates with cement alkalis from one side and their effect on the compressive strength of the concerned mortars

A series of standard cement mortars were prepared following ASTM C109 and nominated (Rf) samples. Different proportions of chert were added to the standard mixtures by weight% of the glass sand. The samples were designated as CH1, CH2 and CH3 as shown in Table 3.

Table 3. Mortar samples with chert

S. No.	Cement	Sand (g)	Chert % by weight
Rf	500	1375	0
CH1	500	1350	1.82
CH2	500	1325	3.64
CH3	500	1275	7.27

The second set of trials, Silicified limestone was added instead of the ground chert with the same proportions of chert to another set of (Rf) mortars. The purpose of these trials was to investigate and differentiate the potential reactivity of different silica sources on ASR. The samples were nominated as SL1, SL2 and SL3. In all mortars, the total weight of glass sand and added crushed chert or silicified limestone was kept constant as 1375 g as shown in Table 4.

Table 4. Mortar samples with silicified limestone

S. No	Cement	Sand (g)	S. Limestone % by weight
Rf	500	1375	0
SL1	500	1350	1.82
SL2	500	1325	3.64
SL3	500	1275	7.27

3.5 Mortars with chert and meta-kaolin

Meta-kaolin was added as a cement substitute by weight % of cement in mortars including chert. The purpose of these trial mortars is to study the effect of metakaolin as an inhibitor of chert-cement reaction and its influence on the compressive strength results. The mortars were designated as CHM1, CHM2 and CHM3. Table 5 shows the proportions of chert and metakaolin in these mixtures.

Table 5 Proportions of chert and metakaolin in mortars

S. No.	Cement	Sand (g)	Chert % by weight of sand	MK % by weight of cement
Rf	500	1375	0	0
CHM1	475	1250	1.82	5
CHM2	450	1225	3.64	10
CHM3	400	1175	7.27	20

3.6 Mixtures with chert and calcined brown clay

In this set of mortars, calcined brown clay was used instead of metakaolin at the same proportions of metakaolin in chert-cement mortars to investigate the efficiency of calcined brown clay as a pozzolanic material.

The mortars were designated CHC1, CHC2 and CHC3as revealed in Table 6. Compressive strength values and scanning electron photomicrographs were obtained for the mortar samples.

Table 6. Chert-calcined brown clay mortars

S. No.	Cement	Sand (g)	Chert %	Calcined. Brown. Clay%
Rf	500	1375	0	0
CHC1	500	1250	1.82	5
CHC2	500	1225	3.64	10
CHC3	500	1175	7.27	20

3.7 Mortars with silicified limestone and meta-kaolin

Metakaolin was added by different proportions by weight of cement to mortars with various silicified limestone content. The purpose of these trial mortars is to investigate the reactivity of silicified limestone with cement in the presence of metakaolin as an additive with different proportions. The mortars were nominated SLM1, SLM1 and SLM1 as shown in Table 7.

Table 7 Silicified limestone and metakaolin mortars

S. No.	Cement	Sand (g)	Silicified. Limestone %	MK % by weight of cement
Rf	500	1375	0	0
SLM1	500	1250	1.82	5
SLM 2	500	1225	3.64	10
SLM 3	500	1175	7.27	20

3.8 Mortars with silicified limestone and calcined brown clay

Calcined brown clay was used at the proportions of metakaolin in silicified limestone-cement mortars to investigate the efficiency of calcined brown clay as a pozzolanic material to be used as an inhibitor of silicified limestone-cement reaction and its effect on compressive strength of such mortars with variable silicified limestone content. The mortars were designated SLC1, SLC2 and SLC3 as revealed in Table 8

Table 8 Silicified limestone and calcined brown clay mortars

S. No.	Cement	Sand (g)	Silicified. Limestone %	Calcined. Brown. Clay%
Rf	500	1375	0	0
SLC1	500	1250	1.82	5
SLC2	500	1225	3.64	10
SLC3	500	1175	7.27	20

4. Results and Discussion

Through calcination process, kaolin is converted from high plastic clay into non-plastic amorphous material. Color of kaolin was changed from whitish creamy to rosy color. Mixtures showed excellent consistency at a constant w/c ratio with all additives which are characterized by their very low absorption.

The Equivalent Alkali Content Na_2O_e of type 1OPC cement is less than 0.6. The cement is classified as low alkali cement. The SiO_2 weight % is 82.3% and 5.2% for chert and silicified limestone respectively. XRF analysis showed that both meta-kaolin and calcined brown clay revealed a pozzolanic composition with variable ($SiO_2+Al_2O_3+Fe_2O_3$) content. The chemical composition of the used ingredients in various mortars are given in Table 9.

Table 9 Chemical composition of the raw materials

Oxide Wt.%	Chert	Silicified limestone	Kaolinite	MK	Silica sand	Brown clay	Calcined brown clay
SiO_2	82.3	5.2	47.6	59.9	96.6	68.2	51.3
Al_2O_3	3.2	2.3	25.9	32.3	0.23	12.9	16.6
Fe_2O_3	2.7	3.7	8	2.5	0.03	6.8	7.5
CaO	9.7	76.8	0.7	1.2	.15	2.7	16.1
MgO	1.1	11	19.1	0.5	0 .1	1.1	3.4
P_2O_5	0.4	0.2	0.1	0.2	-	0.15	0.2
Na_2O	0.1	0.1	0.1	0.1	0 .11	0.12	0.1

The petrographic and XRD results have indicated that the chert samples are composed of micro to cryptocrystalline quartz. The kaolinite samples are composed of quartz and minor chlorite in addition to kaolinite. The brown clay is mainly composed of smectite, kaolinite, quartz and goethite.

The silicified limestone is composed of micritic calcite, dolomite and microcrystalline quartz. The silica sand is composed of pure quartz.

Compressive strength was determined for all tested cured mortars at 56 days. The mortars were cured in a water tank at 24 C°. The mortars including Meta-kaolin showed increasing of compressive strength values with increasing of Meta-kaolin. Maximum compressive strength (36 Mpa) was achieved at metakaolin content in the range of 5-10%.

The mortars including calcined brown clay showed an increase of the compressive strength, a maximum compressive strength value of (30 Mpa) was obtained at 8% of calcined brown clay content.

The compressive strength values of the Rf mixture and other mortars with different proportions of meta-kaolin (MK) and calcined brown clay content are shown in Figure 2**.**

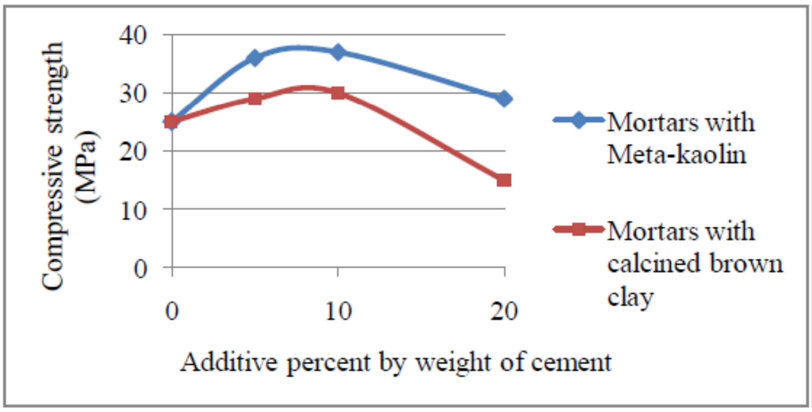

Figure 2 Compressive strength- mortars with meta-kaolin and calcined clay

According to SEM photomicrographs as shown in Figure 3, the B1 Sample showed complete reaction, fibrous Ca-silicate and Ca-Al silicate phases which are responsible for increasing the compressive strength from 25 MPa to 30 MPa.

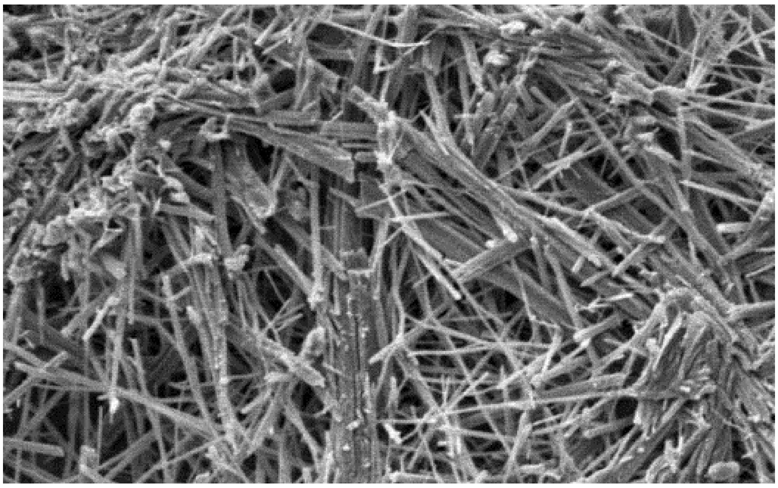

Figure 3 SEM photomicrograph for the CBC10 mortar

The chemical composition determined by Energy dispersive spectrometry (EDS) is shown in Figure 4**.**

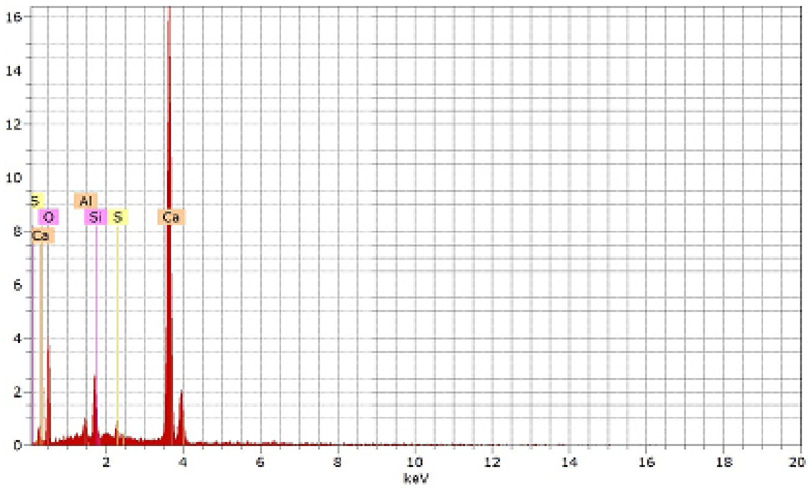

Fig 4 Energy dispersive spectrometry of the CBC10 mortar

The compressive strength values were varied according to chert and silicified limestone content in the tested mortars. The maximum compressive strength values of 32 and 31 MPa were obtained at 3.64% of chert and 3.64% of silicified limestone respectively. The results of compressive strength values for mortars with variable chert and silicified limestone content are shown in Figure 5.

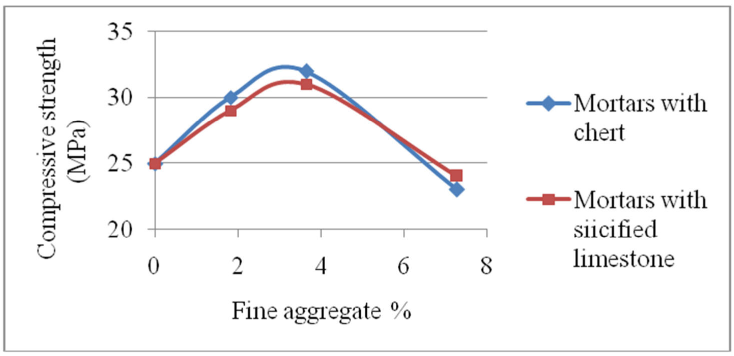

Figure 5 Compressive strength-mortars with variable chert and silicified limestone content

Meta-kaolin was added to mortars with various chert proportions to investigate the potential of Meta-kaolinite as a pozzolanic material on decreasing or inhibiting the expected ASR and the compressive strength of the tested mortars. The compressive strength values increased to 40 MPa for the sample with 10% of Metakaolin-and

3.64% of chert content. The compressive strength values decreased to 30 MPa when calcined brown clay was used instead of Meta-kaolin at the same proportion at the same chert content.
The compressive strength results for the chert-Metakaolin and chert-calcined brown clay mortars are presented in Figure 6.

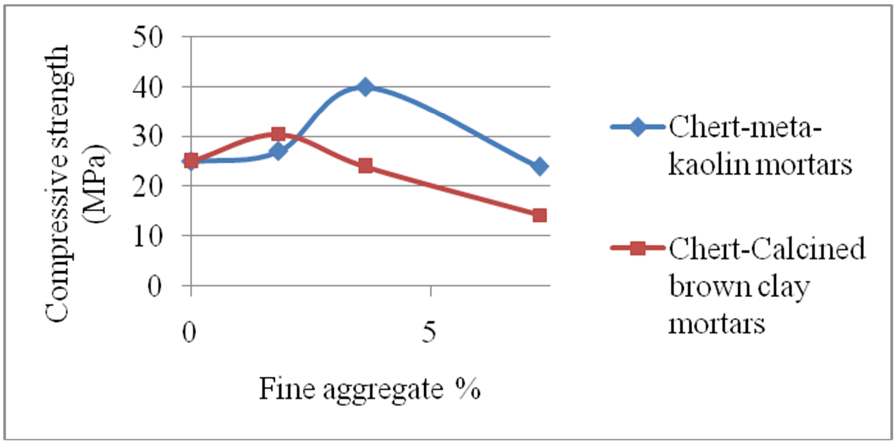

Figure 6 Compressive strength of chert-MK and CBC mortars

The SEM images of the CHC1 mixture shows fibrous Ca-silicate and Ca-Al silicate phases, un reacted calcite and quartz. The mixture shows highly porous texture as shown in Figure 7.

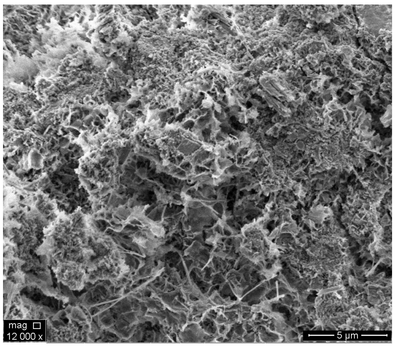

Figure 7 SEM images of Sand cement with chert and calcined brown clay mortars

The chemical composition of the CHC1mortar according to the (EDS) spectra are illustrated in Figure 8.

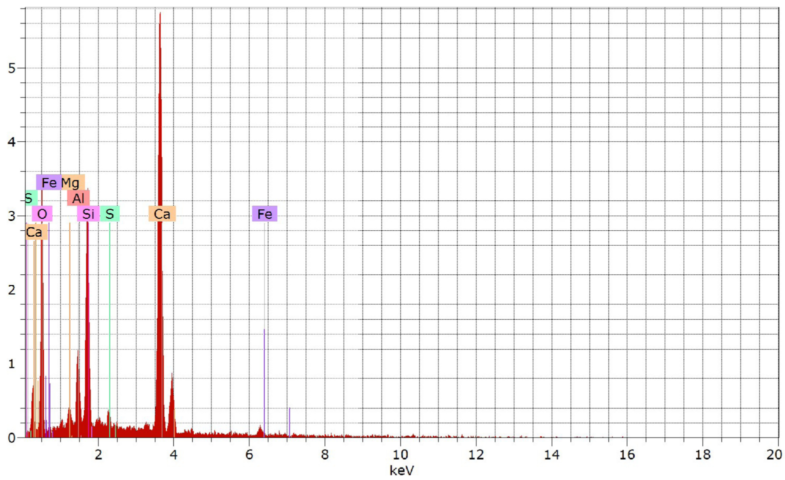

Figure 8 EDS spectra of Ca-Al silicate phase in the L1 sample

Compressive strength results for mortars with silicified limestone-Meta-kaolin and silicified limestone-calcined brown clay showed that maximum compressive strength value of 32 MPa was reached at 10% of Meta-kaolin and 5% of calcined clay at 3.65% and 1.82% of silicified limestone respectively. Variation of compressive strength for silicified limestone with Metakaolin and calcined brown clay is shown in Figure 9

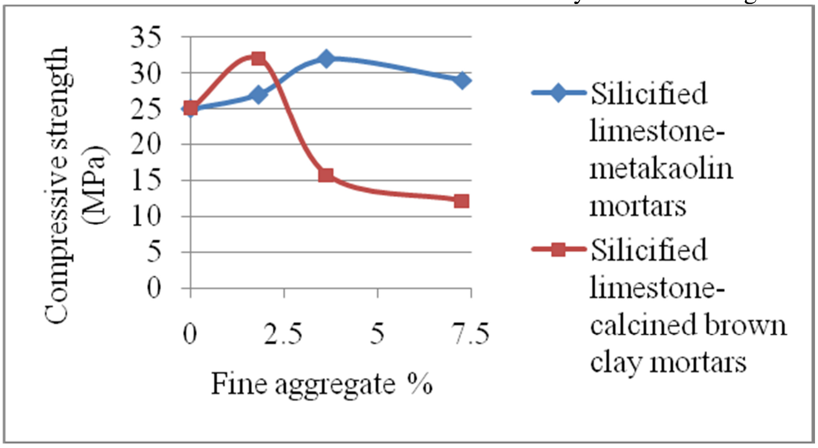

Figure 9 Compressive strength for SL with MK and CBC mortars

Scanning electron images of the CHM mixtures showed short fibrous Ca-silicate and Ca-Al silicate phases as shown in Figure 10. Unreacted kaolinite and quartz are dominant. Most of the porous texture is filled by short fibrous Ca-silicate and Ca-Al silicate phases. Reactions have consumed the produced silica gel. The XRD results have indicated that portlandite $(Ca(OH)_2)$ is also present in addition to quartz and Ca-silicate and Ca-Al silicate phases

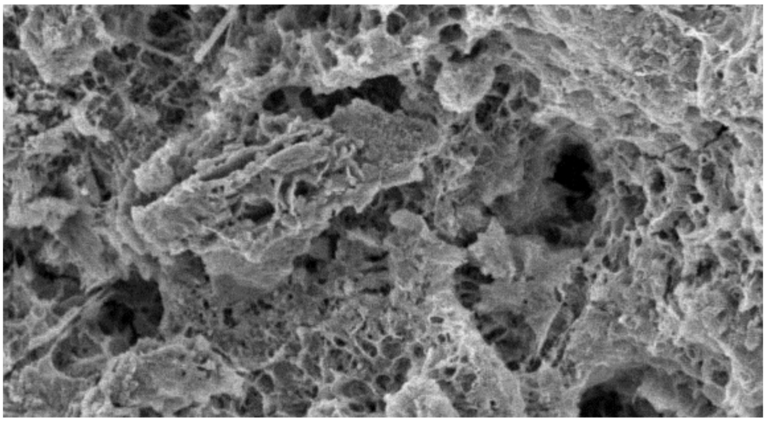

Figure 10 Sand cement with chert and metakaolin mortars

The chemical composition of the CHM mixture according to the (EDS) spectra are shown in Figure 11

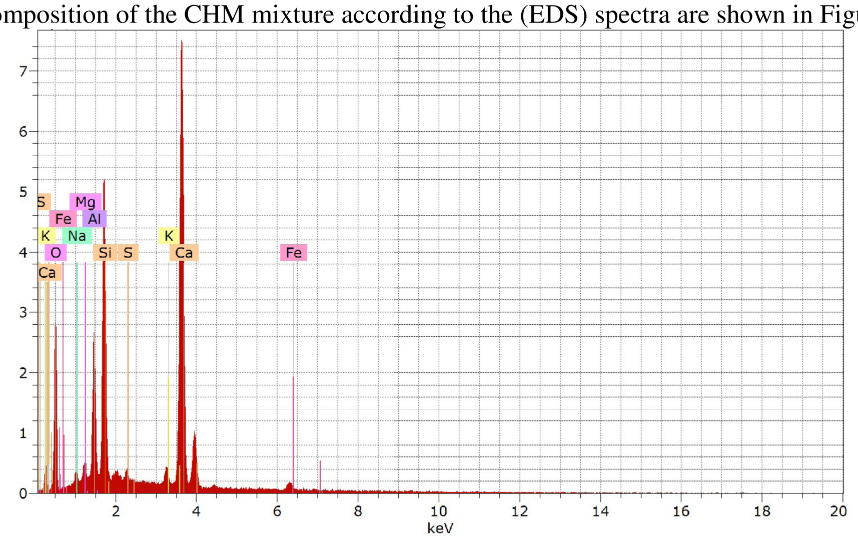

Fig 11 EDS spectra of Ca-Al silicate phase in the CHM sample

The silicified limestone and Meta-kaolin mortars(SLM) mixtures showed fibrous Ca-silicate and Ca-Al silicate phases as shown in Figure 12. Unreacted calcite and quartz. The XRD results have indicated that portlandite (Ca $(OH)_2$ is also present in addition to dolomite, calcite and Ca-silicate and Ca-Al silicate phases.

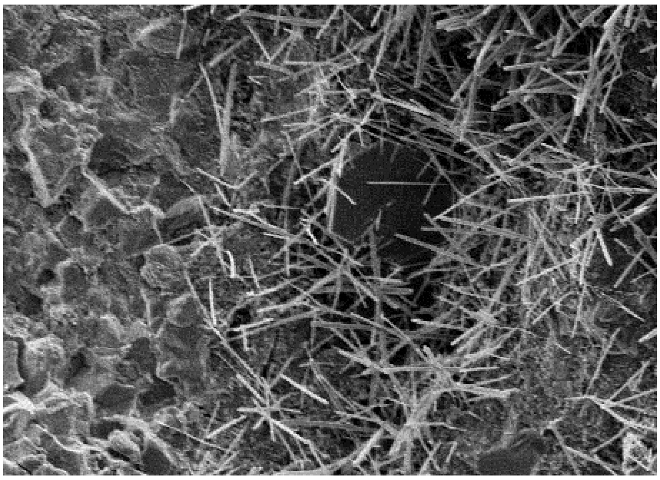

Fig 12 Sand cement with silicified limestone and Meta-kaolin mortars

5. Conclusions

Alkali silica reaction in the tested mortars with variable chert, silicified limestone, meta-kaolin and calcined brown clay utilizing SEM microphotographs showed the deleterious reaction between hydrated cement alkalis and microcrystalline silicates in aggregates has been minimized as a result of adding metakaolinite, Chert has acted as a source of alkali silica gel that reacted with metakaolinite (source of Al and Si) and portlandite (Ca(OH)2 to form fibrous Ca-silicate and Ca-Al silicate phases. The swelling characteristic of the product of the reaction was minimized. Fibrous new phases have filled the pores as indicated by the SEM images and increased the compressive strength up to 40 Mpa . The presence of the right ratios of chert in the aggregates and the addition of metakaolinite as a source of Al and Si could prevent cracking in the concrete as result of the absence of the internal tensile stresses caused by swelling pressure of the gel produced during the ASR.

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