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A Study of the Strength Activity Index of Libyan Kaolin Treated by a Thermal Method

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ABSTRACT

The strength activity index of mortar and pastes containing treated Natural Pozzolan (NP) as Libyan kaolin have been investigated. NP obtained from south Libya was treated by a thermal method via sieving, grinding by ball mill and calcined by heating at 800°C for 120 minutes in order to improve the pozzolanic reactivity of the NP.

The pozzolanic reactivity of the treated NP was evaluated by conducting strength development tests according to ASTM C311. The hydration products of hardened pastes produced raw materials; these were analysed by x-ray diffraction (XRD) and scanning electron microscopy (SEM). Particle size analysis was also done before and after in order to measure the influence of the treated NP. To measure this appropriately, NP replaced cement. Five different types of NP were obtained from different places of South Libya and tested. This study will measure the strength activity index; the starting ratio for this is 20 % NP. The result showed that after 28 days, the strength activity index of the treated NP with ordinary Portland cement exhibited very good performance and was higher than 100%. At 28 days, the strength activity index of the Sebha sample increased to 109. The strength activity index also showed an increase after 28 days for all samples of (Libyan kaolin) ; This shows that as the amount of treated NP increased ,that mean there was a reduction in the Ca (OH)2 content.

Keywords: *The strength activity index , Natural Pozzolan , kaolin*

1. INTRODUCTION

Libya is one many countries that possess large amounts of Natural pozzolan deposits; especially in the southern area. Natural pozzolan has great industrial uses which depend on the purity and quality of the raw materials.

Due to the new movement towards sustainability, the cement content of concrete mixtures has been reduced and replaced by Portland cement and the addition of supplementary cementation materials such as: fly ash, limestone powder, natural pozzolan, slag and silica fume [1]. The compounds present in ordinary Portland cement, such as C3S and C2S, are known to react with water [2]. This reaction forms approximately 70% C-S-H, 20% Ca(OH)2, 7% sulfoaluminate, and 3% secondary phases. [3]. The assessment of Natural Pozzolan is based on the strength activity index, which is outlined in ASTM C311 [4] and specified by ASTM C618 [5].

In the ASTM C311 test method [4], the 7 days and 28 days compressive strengths of a mortar was prepared with a 20 % SCM substitution for cement on a mass basis, these were compared to those of a control mortar. The control mortar is prepared with a water-to-cement ratio by mass (*w/c*) of 0.484, while the water content of the test mixture is adjusted so that there is an equivalent flow to that measured for the control. According to the ASTM C618 specification [5], the SCM mixture should have 75% strength of the control at 7 or 28 days.

In order to produce small sized particles, thermal treatment is used as this improves the pozzolanic reactivity. An important factor that must be considered is the silica content of the ash; when ash from certain agricultural by-products (such as rice husk and bagasse) is added to the cement, an additional C-S-H is formed in the hydrated cement matrix due to the silica reacting with Ca(OH)2. This additional product increases the density of the matrix and improves the pore structure [5, 7].

In the current study for the set of sample cement types (i.e. the control), and natural pozzolan samples obtained from five different areas in southern Libya, the current ASTM C311 strength activity index testing theory is contrasted to a proposed protocol where cement or any SCM).

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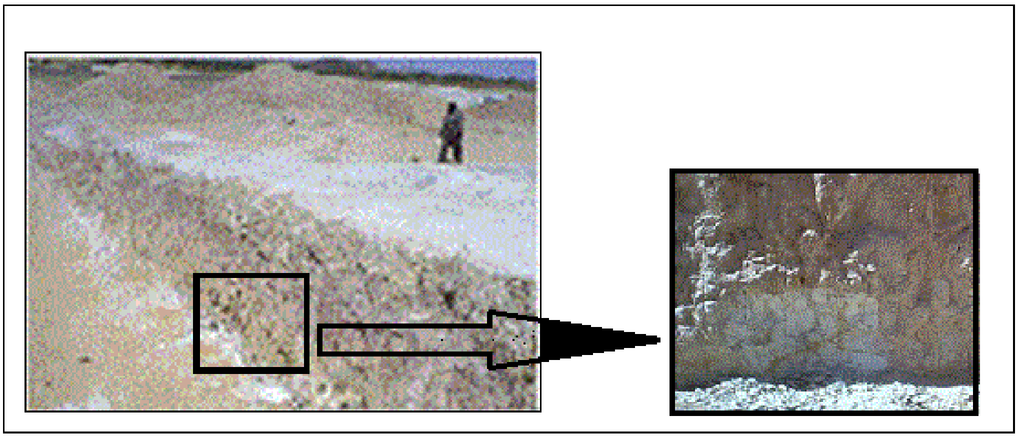

Figure1: Picture showing the location & Minerals of Sebha Libyan kaolin

The change in flow induced by the replacement of cement by SCM is measured. In addition to measuring flows and compressive strengths at various ages, a subset of the mixtures are also characterized with respect to their 7 days heat hydration. To assess the potential of quantifying pozzolanic activity through 7 days the heat released is measured. Those required for conventional compressive strength testing at 28 days. Constant volumetric proportions are maintained.

For the natural pozzolan samples, each test and reference mixture has the same volume ratio of water, sand and powder this include

Figure 2: Picture showing the Sebha Libyan kaolin (LKA) after treatment

2. MATERIALS AND METHODS

2.1 Materials

1-Ordinary Portland Cement (OPC)

OPC is used as the main binder material and is supplied by Cement Industries in England. This cement has a specific gravity of 3.15 and a Blaine surface area of 340 m2/kg. [8]

2- Natural Pozzolan (NP)

Samples were collected from a nearby Sebha city, south Libya .from five different places sebha (A, B, C, D and E) Figure 1 shows place Sebha(A) Libyan kaolin before treaded . The removal of excess carbon and other unburned organic materials present in Libyan kaolin is important as it avoids the potential negative effect on hydration.

Therefore, in order to remove these materials the NP is dried in an oven at 105ºC for 24 hours and then sieved using a set of sieves (3 mm, 600 µm, and 300 µm sieves) to remove the particles coarser than 300 µm. The average particle size of NP before milling was around 75µm. The untreated NP was then ground in a ball mill to reduce the particle size as this improves reactivity. The milling time was approximately 1 hour at 45 rpm.
To prevent glassy phase crystallization and particle agglomeration both of which could affect the pozzolanic properties, untreated NP was heated at a low temperature of 800ºC for 2 hours in an electric furnace. After the heat treatment, shown in Figure 2 the colour of the treated NP

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turned from light brown to grayish red once the unburned residue was removed. Figure 3 shows the XRD patterns of Kaolin before it underwent treatment and temperature changes .the mean compound was Quartz; Kaolinite; Muscovite

Table 1 show the chemical analysis of cement and The chemical analysis of calcined clays was carried out using XRF (X-Ray Fluorescence), the results are presented in Table (2). The table indicates that the total percentage of SiO_2, Al_2O_3 and Fe_2O_3 of calcined NP(A), calcined NP(B), calcined NP(C), calcined NP(D) and calcined NP(E) are: 95.2%, 96.7%, 92.4%, 95.1% and 93.9%, respectively. Then the main component of the treated NP (A) Sebha sample is SiO_2, the total amount of SiO_2, Al_2O_3, and Fe_2O_3 is 93.10 (shown in Table 2). All chemical compositions of calcined clays fully comply with the ASTM C618-03; this indicates good pozzolanic materials due to the silica, alumina and iron oxide total content being greater than 70%.

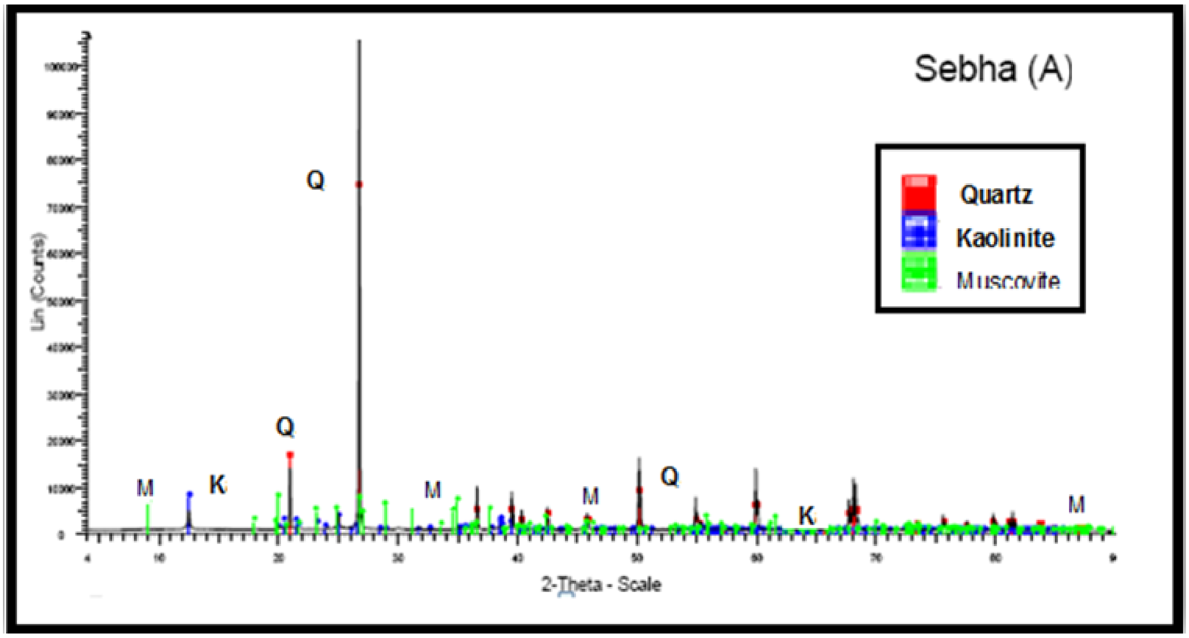

Figure 3: XRD patterns of Libyan kaolin (Sebha) before treatment (Q-Quartz; K-Kaolinite; M-muscovite)

Table 1: Chemical composition of the CEM I 52, 1 N

Oxides	Cement
SiO2	19.70
Al2O3	5.62
Fe2O3	3.08
MgO	1.21
CaO	62.44
K2O	0.89
Na2O	0.27
SO3	3.29
TiO2	0.24
P2O5	0.11
L.O.I	2.72
Specific surface area (g/cm²)	4010
Density (g/cm³)	2.93
Specific gravity (kg/dm³)	3.1

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Table 2: Chemical Constituents and Physical properties of Calcined Natural pozzolan (Libyan Metakaolin)

	A Calcined clays Sebha	B Calcined clays Temenhint	C Calcined clays Alafya	D Calcined clays Agar	E Calcined clays Tarout
Chemical composition (%)					
SiO_2	71.88	58.46	57.95	70.33	53.42
Al_2O_3	20.2	34.36	24.52	25.32	40.84
Fe_2O_3	1.84	2.266	9.973	1.o5	0.975
CaO	0.163	0.162	0.133	0.0801	0.1
P_2O_5	0.36	0.11	0.241	----	0.137
MgO	0.35	0.28	0.724	0.14	0.13
K_2O	1.06	1.44	2.919	0.366	0.16
Na_2O	0.93	0.22	0.35	0.35	0.22
TiO_2	0.966	1.37	1.12	1.51	3.019
SO_3	0.036	0.0209	0.0167	0.0063	0.034
Sum	**93.1**	**95.1**	**92.4**	**96.7**	**95.2**
LOI	**1.06**	**1.25**	**1.68**	**0.76**	**0.88**
Physical properties					
pH	7.6	9.3	7.2	6.2	8.5
Specific gravity (g/cm^3)	2.60	2.61	2.44	2.53	2.65
Particle size µm	50	60	60	70	80

The treated NP particles size was irregular in shape and in porous texture. In addition , the result shown in figures (4and5) shown sample sebha kaolin , the particle size before and after treated, there was no agglomeration of NP particles before the heat treatment. the particle size was lie between 100 to 1000 nm the with biggest diameter 400 nm and volume 12% , after treated the size lie between 0.5 to 200 nm with the biggest diameter 60 nm and volume 4.25% .
Also Figure 6 show the image by SEM the photographs of treated Sebha Libyan kaolin (a) before and (b) after treatment ,the size was more fine after treated

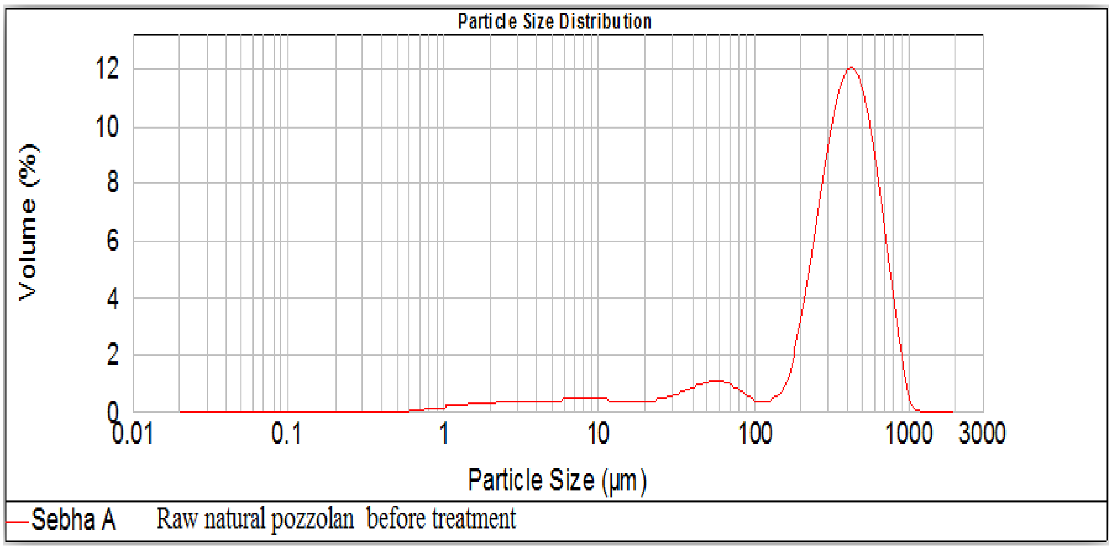

Figure 4: Cumulative particle size distribution curves of Sebha Libyan kaolin before.

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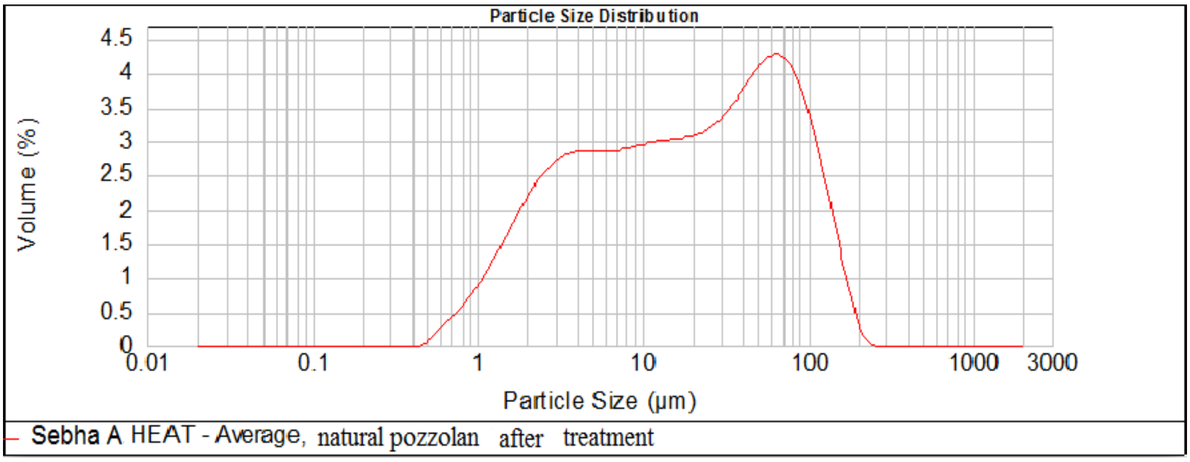
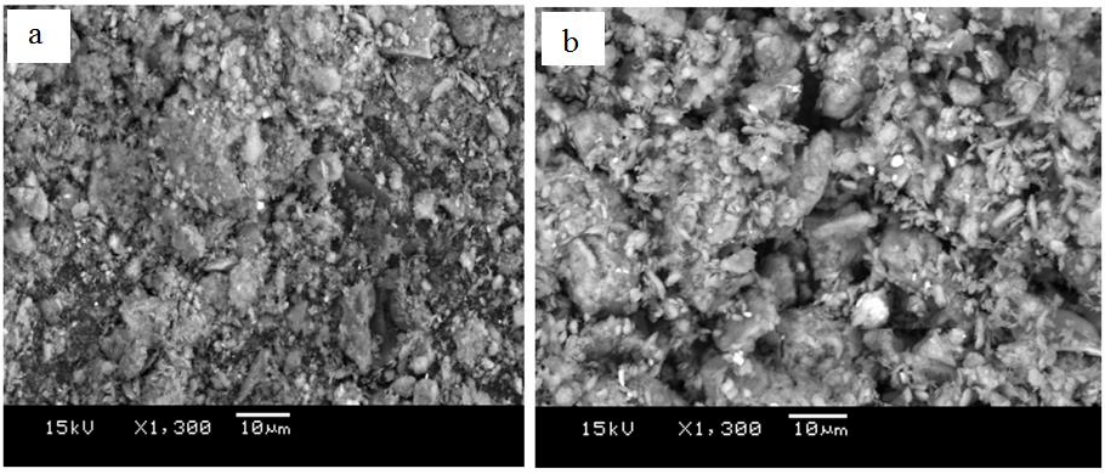


Figure 5: Cumulative particle size distribution curves of Sebha Libyan kaolin after treatment



(a) Before treatment (raw kaolin) **(b) After treatment (metakaolin)**

Figure 6: SEM photographs of treated Sebha Libyan kaolin (a) before and (b) after treatment

2.2 Pozzolanic activity method (Strength Activity Index (SAI))

The pozzolanic activity of the NP was determined based on the compressive strength set by the ASTM C311 specifications.

The OPC in the mortar was partially replaced by 20% NP and tested. The process used to replace the CEM-1 was based on BS 3892[9]. The control mortar blocks were prepared by mixing 1350g sand, 450g CEM-1 and 225ml water in a planetary orbital mixer for 5 minutes. The test mortar blocks were prepared in the same way, but 20% of CEM-1 was replaced with the test pozzolan. Table 3 shows the mix composition summary. Flow tests were done on the pastes based on EN 1015-3 [10].

The water to binder ratio was altered so that the mixture had the same flow properties as the control mortar (±5 mm). Mortar pastes were then re-mixed for 30 seconds and cast into six (50 x 50) mm cubes with the aid of a vibrating table.

Fifty millimetre cubes were cast for the present study. After moulding, the specimens and moulds were placed in the moist room and maintained 23 ± 2ºC for 24 hours. Then the cube samples were removed from the moist room, and demoulded from their respective moulds. The cubes were then placed and stored in saturated limewater. The compressive strength was determined from the average of the three specimens at the ages of 3, 7, 14 and 28 days. The cubes were tested using a universal testing machine (Model UH-F1000kNI) test system with a 1000 KN capacity loading frame. The compressive strength was measured using a constant loading rate of 21 MPa/min

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Table 3: Test mixtures used in the Pozzolanic Activity Tests

Test/material	PC (g)	Mortar sand (g)	Test pozzolan (g)	Water (ml)
SAI/control	450	1350	0	225
SAI/sand	360	1350	90	225
SAI/A	360	1350	90	260
SAI/ B	360	1350	90	260
SAI/ C	360	1350	90	260
SAI/D	360	1350	90	260
SAI/E	360	1350	90	260

All blocks were de-moulded after 24 hours and placed in a water bath at 23 °C for 6 or 27 days They were then removed from the bath, surface dried and the compressive strength was tested at7 or 28 days. The results of the total strengths are the averages of the three tests and are presented as percentage strength relative to the control mortar with the strength activity index (SAI). Therefore, the results are presented in the following format:

SAI	= (A/B) x 100 ------------- (1)

Where: **A** is the unconfined compressive strength of the test pozzolan specimen (MPa) and **B** is the unconfined compressive strength of the control mortar (MPa). According to BS 3892 [9], SAI results greater than 0.80 after 28 days indicate positive pozzolanic activity for Pozzolan. for a cement replacement of 20%. ASTM C618[11] requires a SAI greater than 0.75 after 7 and 28 days for natural pozzolan at a cement replacement of 20%.

3. RESULTS AND DISCUSSION

3.1 Strength Activity Index

Strength activity indices for all mortars are shown in Figure 6. The strength activity index is the ratio of the strength of the NP-cement mortar in relation to the strength of the reference (cement mortar) at each specific curing time.

The rate of strength development of cement mortar relies principally on its hydration rate. In contrast, the said rate relies on the cement hydration and rehydration caused by the pozzolanic reactivity of NP in NP -cement mortar. Figure 6 shows that the strength activity indices at 7 and 28 days were higher than the minimum requirement of 75% as specified in ASTM C 618-05. The NP cement for the samples presents a strength activity index of: A= 99.8%, B=95.6%, C= 93.3%, D=80% and E =101.% of the reference cement strength at 7 days respectively. However, the reference cement strength for the samples at 28 days had the following strength activity index: A= 109.1%, B=104%, C= 99%, D=80% and E =102%. At the early ages of 3 and 7 days, replacing OPC with 20% NP was found to reduce the compressive strength in comparison to the reference mortar. This could be attributed to the dilution effect and delayed onset of pozzolanic reaction of NP $Ca(OH)_2$.

The results are reported as averages of three replicates. The control mortar strength was 39.9 MPa at 7 days and 49.6 MPa at 28 days. Figure 6 shows that all test pozzolan causes a decrease in strength after 7 days in relation to the control. After 28 days, with the exception of pozzolan A, B, C samples, there is still a clear difference between the control and test sample strengths however, sample E at both 7 and 28 days still has the same results as the control. The control value is control E =101%, while the sample value E =102%. Sample D at 7 and 28 days still had a lower value than the control, the control value was 80% .

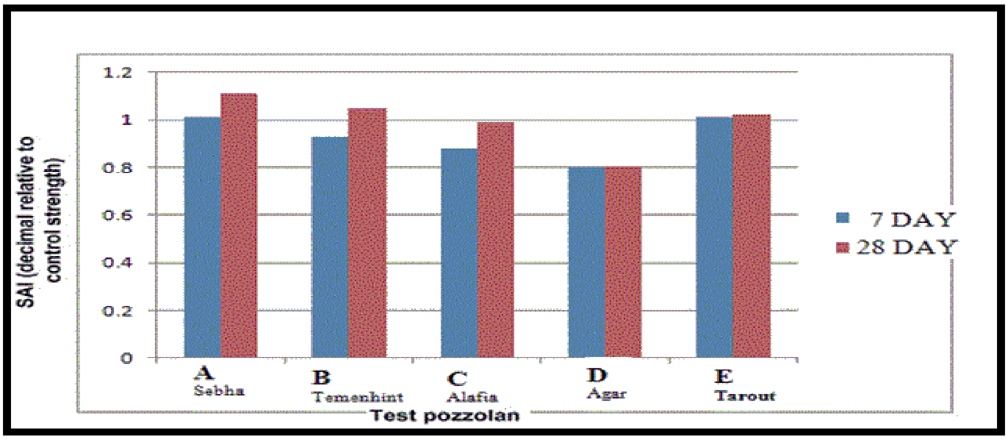

Figure 6: Strength activity index of five different test materials after 7 and 28 days

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Figure 6 shows the Strength activity index of five different test materials after 7 and 28 days. The results are the averages of triplicate determinations and expressed as % of control sample strengths, which were 39.9 MPa after 7 days and 49.6 MPa after 28 days. The SAI results for Libyan kaolin were greater than 0.80 after 28 days; this indicates a positive pozzolanic activity for Pozzolan for a cement replacement of 20%.

4. DISCUSSION

When assessing the pozzolanic activity of a material, it is important to take into account the method used. While it is common for pozzolanic activity to be investigated by more than one method, at least one of the methods is likely to be qualitative and show a trend of $Ca(OH)_2$ consumption with time. When comparing different methods a key consideration is the temperature and time of sample curing prior to testing. The SAI test is specified for 28 days at 23 °C.

5. CONCLUSIONS

Based on the experimental studies presented in this paper, the following conclusions can be drawn:

- Ground NP obtained by heating at 800ºC for 2 hours resulted in loss on ignition significantly lower than that of the untreated NP (LOI= 1.06,1.25,1.68,0.76 and 0.88 for A, B ,C ,D and E sample) . Hence, the treated NP was free from carbon and other organic matter. In addition, the treatment process yielded NP with high specific surface area, preserving the amorphous characteristic related to pozzolanic activity of NP and free from particle agglomeration.

- 2-Strength activity index of NP /cement mortar fulfilled the requirements of pozzolanic materials as per ASTM C 618-05. Compressive strength tests confirmed that after 28-days curing, the strength of treated NP was greater than that of the reference cement.

- 3- The SAI results for all samples from south Libya were greater than 0.80 after 28 days, which shows there is a positive pozzolanic activity for Pozzolan for a cement replacement of 20%.

- 4- At 28 days, the strength activity index increases to more than 100%, this may be caused by the pozzolanic reaction of NP with Ca(OH) producing C-S-H and increasing the strength. At Days, the compressive strength of the NP was higher than that of the reference cement; this could be due to the pozzolanic reaction of NP.

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