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Properties of sonochemically treated Libyan kaolin pozzolan clay

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ABSTRACT

Natural kaolin pozzolan is found in south Libya. Libyan Kaolin clay is relatively high in silica, and its physical and chemical properties could be referenced as ASTM C618 class N. Sonochemical treatment was carried out and the treated Kaolin was used as a mineral admixture in the Portland cement mortar. The Sonochemical treatment was carried by horn method with 25, 50 and 100 Watts power and 20 kHz frequencies for half an hour.
The Scanning Electron Microscope (SEM) imaging and particle size analysis were used to understand the mechanism of pozzolanic enhancement of the Libyan kaolin after treatment by Sonochemistry. This paper presents details of the physical and chemical properties of sonochemically treated Libyan Alafya kaolin. The effect of magnitude of sonic power on pozzolanic activity of treated kaolin was also investigated. The results show enhancement in particle size and pozzolanic activity of treated kaolin with increase in sonic power.

Keywords: Sonochemical treatment, Sonochemically treated pozzolan, Sonochemically treated Kaolin

INTRODUCTION

Power ultrasound influences chemical reactivity through an effect known as "cavitation". Cavitation occurs by applying high-intensity ultrasound to liquids, resulting in the superimposition of sinusoidal pressure on the steady ambient pressure. Sound is transmitted through a fluid as a wave consisting of alternating compression and rarefaction cycles. In the phenomenon called cavitation, the micro bubbles formed during the rarefaction cycle of the acoustic wave undergo violent collapse during the compression cycle of the wave. During the compression cycle, the bubble's content is estimated to be heated to 5000 K, and the implosion of the cavitation bubble also produces high-energy shock waves with pressures of several thousand atmospheres (Suslick, 1999) . The ultimate consequence of the high temperature is a chemical reaction. The high pressure leads to an increased number of molecular collisions owing to enhanced molecular mobility and decreased overall volume, leading also to high chemical reactivity (Mason, 1998). Growing interest to the application of sonochemistry in materials chemistry, electrochemistry, and environmental chemistry (Suslick, 1999, Gedanken, 2004 and Mason, 1998) demands the development of characterization methods for the sonochemical reactors. Problems related to the non-uniform acoustic energy distribution and the active zones identification in 20 kHz sonoreactors have been considered recently (Mason, 1998).

There are two types of sonication methods available, Horn and Bath methods. In this study the Horn sonochemical method is used for activation of kaolin-clay to obtain treated sonic kaolin and understand the effect of sonic waves on kaolin Pozzolanic activity.

Horn sonication is the most common way to process a sample. Energy is transmitted from the probe directly into the sample with high intensity and the sample is processed quickly.
The diameter of the probe's tip dictates the liquid volume that can be effectively processed. Smaller tip diameters (Microtip probes) deliver high intensity sonication and the energy is focused within a small, concentrated area. Larger tip diameters can process larger volumes, but offer lower intensity. Boosters and High Gain horns can be used to increase the output of large diameter probes. Probes are offered with either replaceable or solid tips and are made from titanium.

In the reactions with solids, ultrasound breaks up the solid pieces due to the energy released from the bubbles created by cavitation collapsing through them. This gives the solid reactant a larger surface area for the reaction to proceed over, increasing the observed rate of reaction. The aim of this study is to examine the effect of a partial replacement of Portland cement by chemically treated kaolin.

EXPERIMENTAL METHOD AND MATERIALS

Libyan Kaolin

The clay soils (kaolin) were collected from (Alafia site) in the south of Libya, around Alafia town regions as shown in Table 1 and in Figures 1 and 2. Most of the experimental work was carried out in laboratory of concrete technology, department of civil, architecture and building, Coventry University according to procedures as per BS and ASTM standards.

Table 1. Location and major oxides of used Libyan kaolin from Alafya

Sample	Location	Mineral in clay (%)
Lybian Kaolin (LKT) Alafya	Three km in Alafya –Brack road: 0.5m from ground surface, near the road side	Kaolinite54%, Quartz 46%

Figure 1. Libyan kaolin sample from Alafya

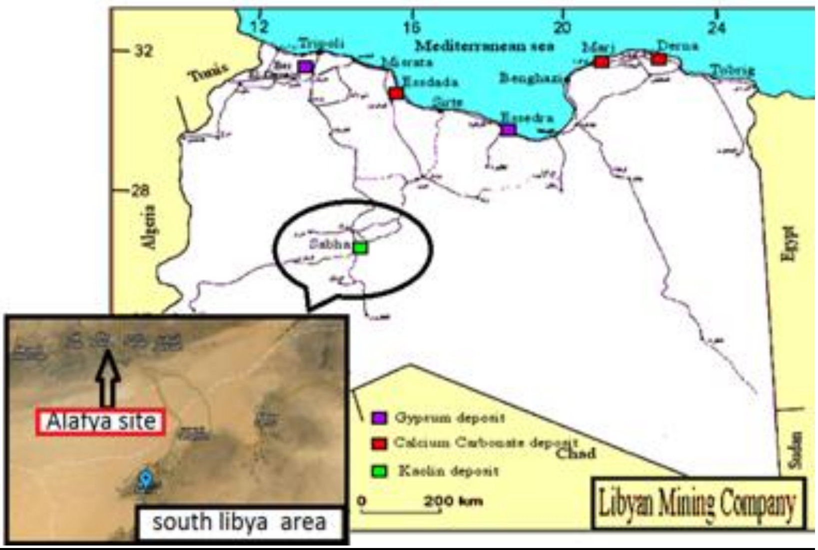

Figure 2. Map of south Libya showing the position of soil extraction (Lybian Kaolin sample from Alafya)

Portland Cement

The Portland cement CEM I was obtained from Castle cement which complies with the specifications as per the BS EN 197-1 - CEM I 52.5 R and Libyan standard 340/97.

Sand

The sand used was natural quartz sand. The grading of the sand used was within the recommended limits of ASTM C778-03. The natural sand was used to make 50mm cube mortar specimens for compressive strength test in accordance with ASTM C109-03.

Chemical Analysis

The chemical analysis of raw Kaolin from Alafya site is shown in Table 2. This was carried out by employing XRF (X-Ray Fluorescence) technique. The particle size and SEM technique were carried out at Coventry University lab before sonochemical treatment. Figure 3 shows the Scanning Electron Microscope (SEM) of Kaolin clay (alafya) before treatment

Table 2. Chemical composition of kaolin (clay) Alafya sample

Oxides	Natural clay Alafya (%)
SiO_2	56.73
Al2O3	20.73
Fe2O3	8.90
Total (SiO_2+Al_2O_3+Fe_2O_3)	**86.36**
MgO	0.55
CaO	0.01
K2O	2.76
Na2O	0.09
SO3	0.09
TiO_2	1.05
MnO	0.05
P2O5	0.16
Cr2O3	0.02
LoI	7.60

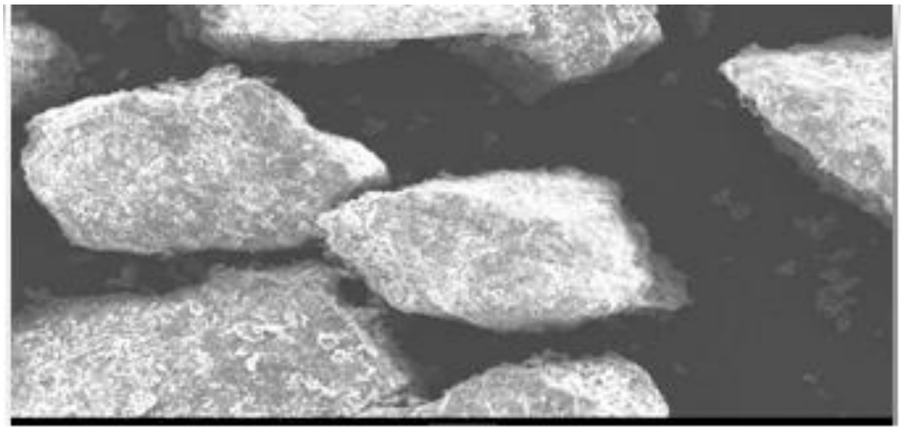

Figure 3. The Scanning Electron Microscope (SEM) of kaolin clay (Alafya) before sonochemcial treatment

Particle Size Analysis

Figure 4 shows the particle sizes of Alafya kaolin sample used in this research before treatment. It can be seen that majority of particles ranges from 700 -1000 µm and 70-100 µm. This data was obtained following a particle size analysis exercise.

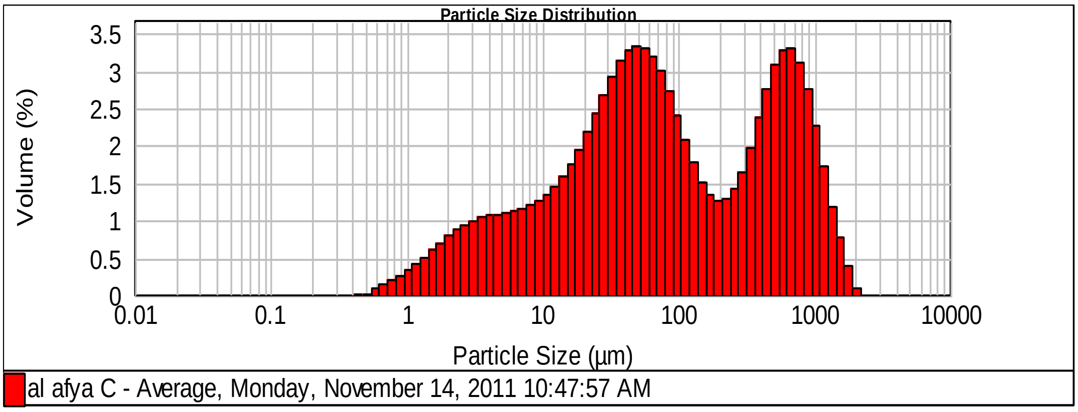

Figure 4. the particle size analysis of kaolin clay (Alafya) before sonochemical treatment.

Horn Sonication of the Kaolin in Water Solution

The application of ultrasound was carried out using a high intensity sonicator series Auto tune, model 100W with frequency of 20 kHz and titanium probe diameter 12.5 mm. The solutions (100 mL) mix with 10 grams of Libyan kaolin was sonicated by means of different powers, from 25, 50 and 100 Watts at room temperature.

In order to avoid excessive heating of the sample, the beaker was immersed in a water bath at 25°C. The temperature was maintained below 40 °C in the sample.

The sonication was carried out for 30 minutes period by sonicator. The capacity of beaker for sonication was 200 mL with dimensions 95 mm. The height of sonication horn in the liquid was 30 mm as shown in Figure 5.

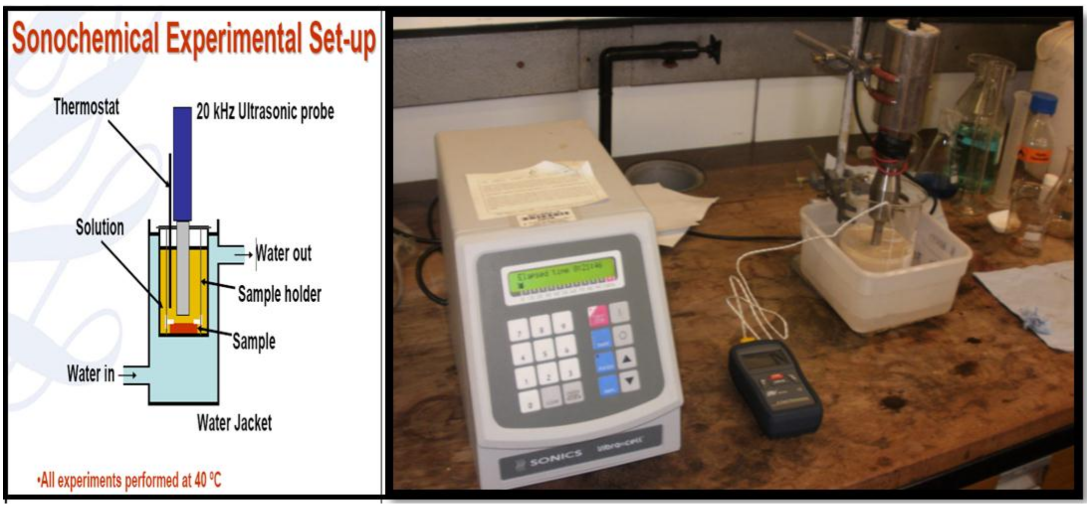

Figure 5. Horn method of treatment

Mix Proportions

Three mortar mixes containing 10% Libyan Alafya Kaolin Sonochemically treated (LKST) using 25, 50 and100 Watts power were made. The compressive strength of these mixes were compared with a control mortar mix made with 100% Portland cement.

Table 3. Mix proportions of cement and Sonochemically treated kaolin at different power.

Sample/mix	Code	Libyan kaolin treated LKST(%)	Portland Cement	Water:Cement	Sonic treated power (Watt)
OPC	Control	0	100	0.485	0
Libyan treated kaolin (LKT) from Alafya site	LKT1	10	90	0.485	25
	LKT2	10	90	0.485	50
	LKT3	10	90	0.485	100

In the mix proportions the designations LKT1, LKT2 and LKT3 represents 25, 50 and 100 watts power applications respectively.

Mortar Specimens

A constant ratio of cementitous materials (Portland cement plus Libyan kaolin) to sand was set at 1 to 2.75 by weight, and water to cementitous materials ratio was maintained at 0.485. Portland cement was replaced by Kaolin material at the rate of 10% by weight of cementitous materials. All mortars were cast in 50x50x50-mm cube moulds and removed from the moulds after casting for 24 hours and then cured at standard 21±1 °C water. Compressive strength of mortar specimens was determined at the ages of 3, 7, and 28 days. All specimens were prepared and tested in accordance with ASTM C-109 (1997).

RESULTS

Particle Size Analysis

Figures 6 and 7 represent particle size of Alafya kaolin sample after sonochemical treatment with 25 and 100 watts respectively. It can be seen that the majority of particles lie between 1 and 100 µm. It was found that treatment at 100 Watts resulted in the best particle size distribution as it produced finer particles. The analysis of the particle size for the sonic treated and non treated kaolin used are also shown in table 4. This table shows that the increase power produces finer particles as expected.

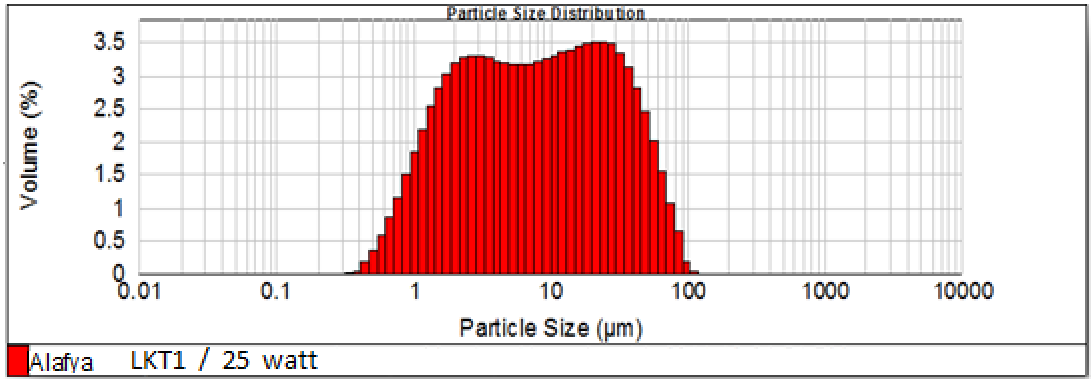

Figure 6. The Particle size after treatment by Sonochemical method (25 watts) kaolin (Alafya) sample

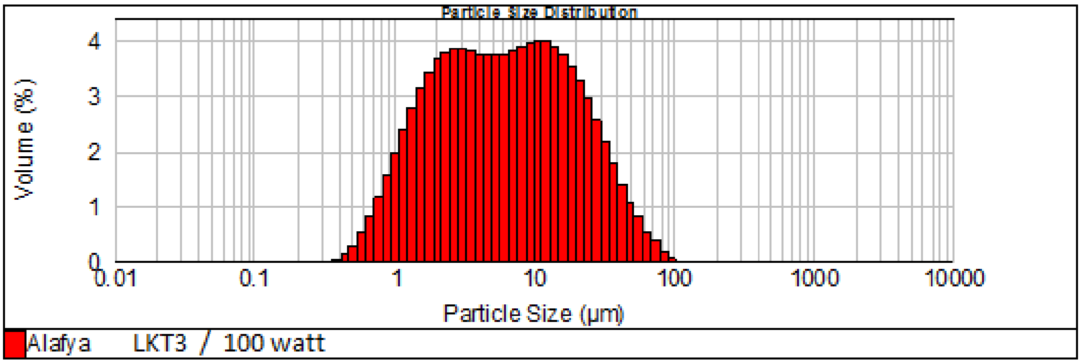

Figure 7. The Particle size after treatment by Sonochemical method (100 watts) kaolin (Alafya) sample

Table 4. Particle size diameter of Alafya kaolin before and after sonic treatment by horn method with different powers of 25, 50 and 100 watts.

sample	1SD Dia 84% Vol (µm)	2SD Dia 95% Vol (µm)	Mean Dia (µm)
Non treated kaolin	620.15	1037.79	257
LKT1	31.68	54.12	15.41
LKT2	27.07	44.17	13.19
LKT3	20.6	37.21	10.97

SEM Image and Microstructure

Figure 8 shows the SEM image of the treated Alafya kaolin. It was observed that the morphology of the kaolin particles was modified after sonochemical treatment. The SEM images confirmed that the particle size of kaolin was significantly reduced. Also the particles appeared to have more fractured surface compared to the clay before treatment.

Figure 8. The Scanning Electron Microscope (SEM) of kaolin clay (alafya) after treatment.

Compressive Strength Test

Figure 9 and Table 5 show the development of compressive strength of mortar specimens at different ages. It can be seen that different power used for sonichemical treatment of kaolin affects the strength of the mortar. The rate of strength gain increased with increase in sonic power.

Also, the sonochmical treatment of the kaolin did not have any adverse effect on normal strength development of mortar specimens with age, i.e. strength increased with time as expected.

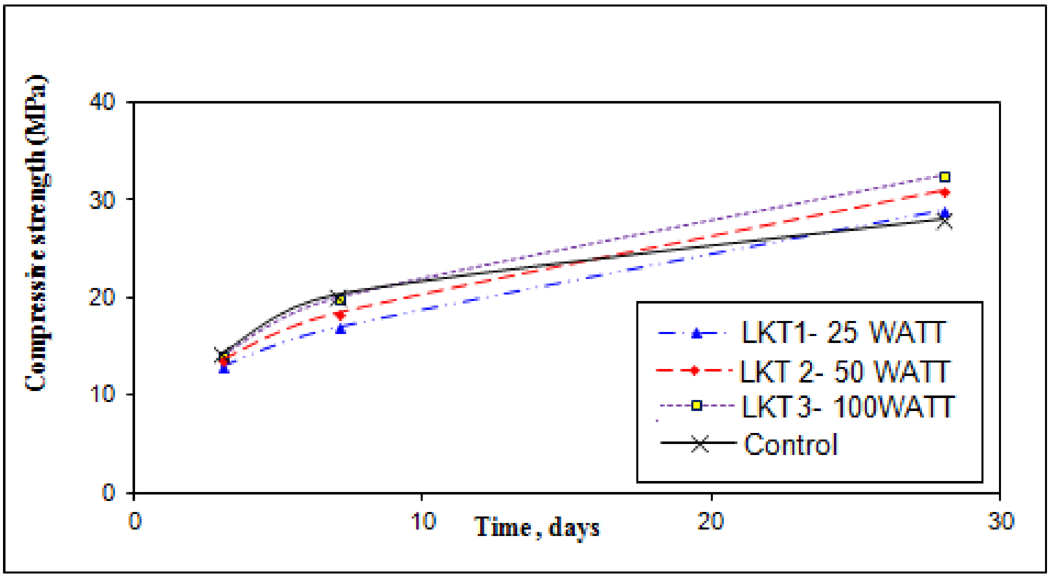

Figure 9. Compressive strength test results (MPa)

Table 5. Compressive strength test results (MPa) for kaolin treated by Horn sonic method using different power.

Age (days)	Compressive Strength (MPa)			
	Control mix	**Sonic treated at power 25 watts**	**Sonic treated at power 50 watts**	**Sonic treated at power 100 watts**
3	14.3	13	13.7	14.1
7	20.3	17	18.5	20
28	28	21	31	32.5

CONCLUSION

Lybian kaolin from Alafya site was treated using horn sonochemical technique at different sonic power and compressive strength of mortar specimens made with 10% treated kaolin as cement replacement was measured. The following conclusions can be drawn:

1) The natural clays (Libyan kaolin clays) collected from Alafya site can be used as partially replacement of OPC after treatment by sonochemical.
2) The chemical composition of the Alafya kalin before and after treatment complies with the ASTM C 618-03 and can be classified as class N.
3) Specimens made with 10% treated kaolin suing 100Watts sonic power achieved the highest compressive strength, reaching up to 32.5 MPa at 28 days.
4) The particle size and morphology of kaolin particles were modified significantly after sonichemical treatment. The higher sonic power used, the smaller particle size and higher compressive strength achieved.

5) In the laboratory, sonochemical treatment can be carried out economically. However currently, for an industrial scale, this treatment will be very expensive compared to thermal treatments.

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