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Potential of Calcined Brick Clay as a Partial Substitution in Blended Cement Mortars

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Abstract

Calcined kaolinitic clays, among other supplementary cementitious materials, have been acknowledged as having a good potential to reduce the CO2 emissions associated with cement and concrete production. However, little attention has been given to impure kaolinitic clays which are usually used in the manufacture of burnt bricks and other products. This paper has studied the potential use of less pure kaolinitic clay, which is normally used in brick production, as a pozzolan in blended cement mortar. X-ray diffraction studies revealed the presence of kaolinite, illite and quartz in the clay. The clay was calcined at 600°C and 700°C and blended with Portland cement at weight percentages of 10, 20 and 30. From the Frattini test, clay calcined at 700°C showed a better pozzolanic reactivity than that calcined at 600°C . Blended cements containing 700°C calcined clay recorded lower water demand and setting times as compared to 600°C calcined clay. There was a reduction in compressive strength at all replacement levels, both at early and later ages. Mortar containing 700°C calcined clay recorded higher compressive strength than the clay calcined at 600°C .

Keywords: Pozzolanic reactivity; Impure kaolinitic clays; Portland cement; Supplementary cementitious materials; Blended cements; Calcined clay

Abbreviations: SCMs: Supplementary Cementitious Materials; GGBS: Ground Granulated Blast Furnace; XRF: X-ray fluorescence; XRD: X-ray Diffraction; BC: Brick Clay; PC: Portland Cement

Introduction

It has become a known fact that the partial replacement of Portland cement with supplementary cementitious materials (SCMs) is the most realistic and attainable solution to reduce the release of greenhouse gases associated with cement and concrete production. Clays, heat-treated at an appropriate temperature (usually between 600-900°C) [1], have been demonstrated as suitable for use as SCM in concrete [2-6]. This is mostly due to the fact that clays are available in many regions in relatively large quantities as compared to popular SCMs such as pulverised fly ash, ground granulated blast furnace slag (GGBS) and silica fume [7,8]. Among clay types, the ones containing high kaolinite contents are mostly known to display appreciable reactivity in pozzolan-cement systems [9]. Calcined kaolinitic clays, therefore, react faster with portlandite originating from the Portland cement hydration, as compared to other types of clay. This fast reactivity is mainly due to the abundant formation of highly reactive 5-fold coordinated Al sites in kaolinite during heat treatment [8-11]. As a result, several researchers, in the past years, have studied the performance of calcined kaolinitic clays in cementitious materials systems [2,12,13].

However, in recent years, the focus of calcined clay research has shifted from pure kaolinitic clays to low-grade clays (i.e. clays containing low kaolinite mineral content) [9,11,14-20]. This is due to the scarcity and high cost of pure kaolinitic clays because of their alternate use for other industrial applications. One clay type that is known to have insufficient kaolinite content and high concentrations of minor elements is clays used for brick production, otherwise called brick clays. In England, there is a considerable availability of brick clays which is mostly utilised for the production of burnt bricks and other clay products used for housing construction. Even though the brick industry in England is colossal and booming, the valorisation of these brick clays for alternative use in the cement and concrete industry has not been fully explored. Again, there exist a threat on the availability of pulverised fly ash due to the possible complete shutdown of coal-fired power generation plants in the United Kingdom and some parts of Europe in a few years to come [11,21].

Therefore, in order to achieve a deeper comprehension of the parameters that determine the properties of calcined impure kaolinitic clays (i.e. calcination process and temperature, replacement percentages, compressive and flexural strengths, microstructural and mineralogical studies, hydration kinetics and durability), extra and advance data need to be accumulated. Hence, this work studies and characterises a type of brick clay with low kaolinite content and calcined at two different temperatures. Blended cements made from these calcined clays were tested and their effect on properties of blended cement mortar samples determined.

Materials and Methods

An impure kaolinitic clay was obtained from a local brick manufacturer in Bellingdon, England. The raw clay was initially dried in an drying oven at a temperature of 105°C for 24 hours. The clay was then crushed, pulverized using a hammer mill and sieved through a 2mm sieve. It was afterwards calcined in a muffle furnace (Nabertherm, Germany) at 600°C and 700°C for 2 hours using a heating rate of 10°C /min. The calcined clay, after cooling in the furnace, was milled into fine powder and screened through a 75 µm sieve. Samples of the raw and calcined clays have been shown in Figure 1. Portland cement (CEM I 52.5 N), manufactured by Hanson UK, was used as the main binder for the testing of blended cement pastes and mortars. Additional pastes and mortars were prepared with the same cement, without any calcined clay, and used as the reference samples. The chemical composition of the clay and cement were determined by x-ray fluorescence (XRF) using P Analytical Axios mAX WDXRF spectrometer. X-ray diffraction (XRD) analysis of the calcined clays were also carried out using a 3rd generation Malvern P Analytical Empyrean XRD Diffractometer. Particle size analysis was performed using the Laser Diffraction method with the Malvern Mastersizer 2000 analyser.

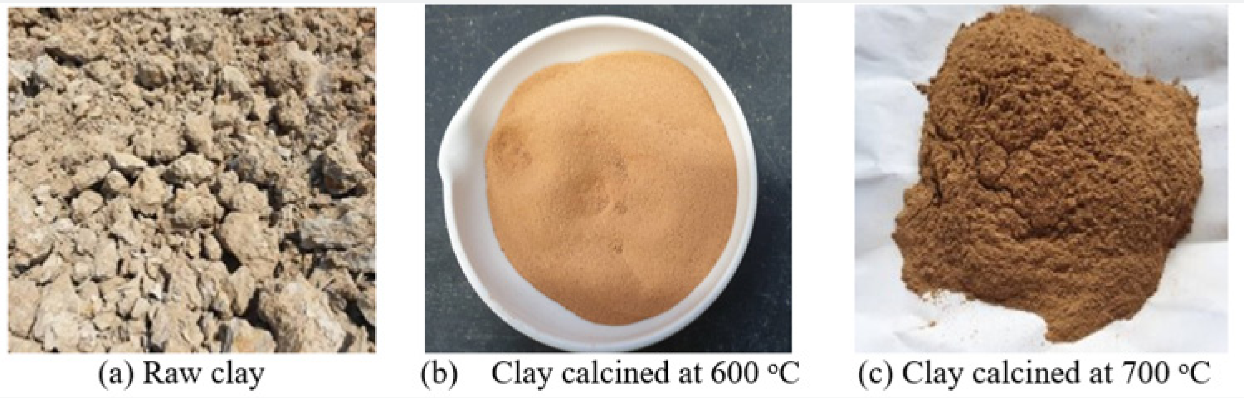
(a) Raw clay (b) Clay calcined at 600 °C (c) Clay calcined at 700 °C

Figure 1: (a) Raw clay and calcined clay at (b) 600°C and (c) 700°C .

Portions of the Portland cement was replaced with the calcined clay in percentages of 10, 20 and 30 wt.% to form Portland-calcined clay blended cements. 50 × 50 × 50mm mortar cubes were prepared according to methods specified by ASTM C109, using a cement to sand ratio of 1:3 and water/binder ratio of 0.5. Deionised water was used for all mixes and naturally occurring quartz sand with particle size ranging between 0.1-2.0mm was used as the fine aggregate. Mix proportions of the mortar are shown in Table 1. The mortar cubes were cured under water and their respective compressive strengths determined after 3, 7 and 28 days. Setting times and water demand was also determined using the Vicat apparatus as described in BS EN 196-3:2016. Frattini test was conducted with reference to ASTM C191. In order to validate the results obtained, the test was repeated three times to obtain three concentrations of CaO and OH. The average of the three was then recorded and reported.

Table 1: Mix proportions of mortar samples.

Material	Replacement, wt.%	Sand, g	Cement, g	Calcined Clay, g	Water, g
Sample 1	0	1800	600	—	300
Sample 2	10	1800	540	60	300
Sample 3	20	1800	480	120	300
Sample 4	30	1800	420	180	300

Results and Discussion

The particle size distributions of the brick clay (BC), clay calcined at 600°C and 700°C (labelled 600 CC and 700 CC respectively) and Portland cement (PC) are presented in Figure 2. Other properties of the cement, calcined clays and blended cements are also shown in Table 2. Water needed to form a workable paste increased with increase in the calcined clay content for both temperatures. The smaller particle size of the calcined clay possibly increased the surface area of blended cement and therefore required more water [22]. There was also a progressive increase in both initial and final setting times as the pozzolan content increased. It was observed that water demand and setting times decreased as calcination temperature increased from 600°C to 700°C .

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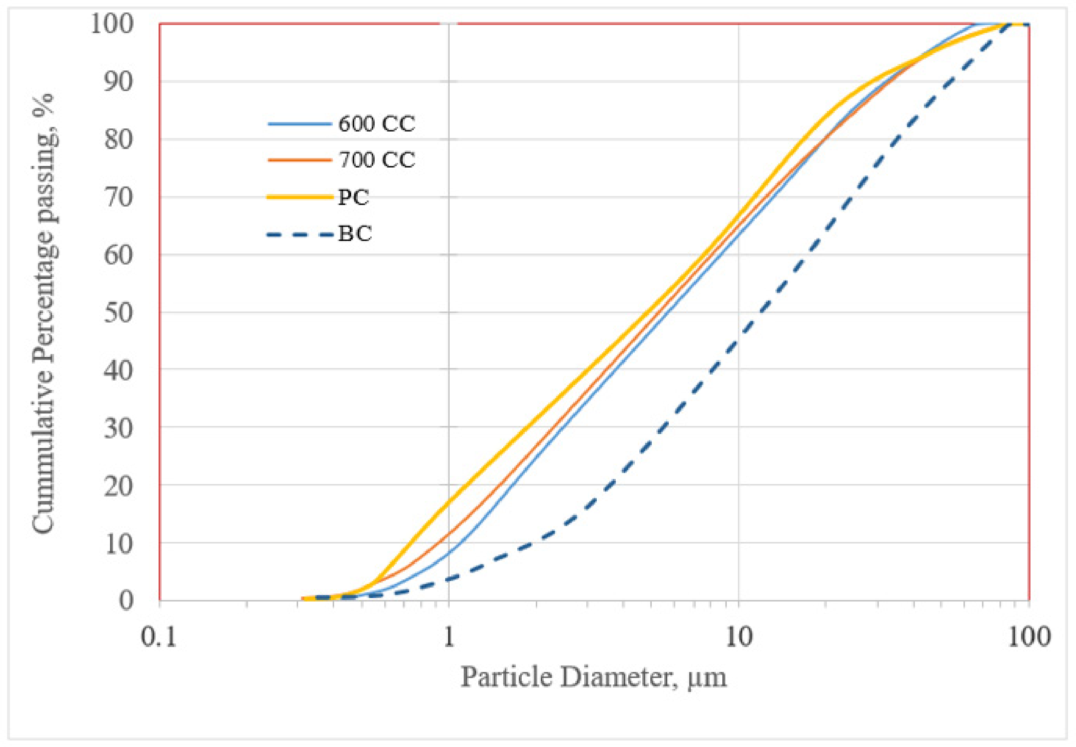

Figure 2: Particle size distribution of the starting materials.

Table 2: Properties of the cement, calcined clay (CC) and CC blended cements.

			600°C Blended Cement				700°C Blended Cement		
Property	CEM1	600°C CC	10%CC	20%CC	30%CC	700°C CC	10%CC	20%CC	30%CC
Specific gravity	3.15	28.2	-	-	-	2.78	-	-	-
water demand, %	28.2	-	32.4	33.3	39	-	31.6	32.4	35.6
Initial setting time, min	162	-	180	190	200	-	175	185	190
Final setting, min	250	-	280	295	320	-	269	275	305

Table 3: Chemical composition of the clay and cement.

Material	Chemical Composition, %									
	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	SO_3	Cl	LOI
Raw clay	59.95	18.2	6.51	1.3	0.14	1.6	1.29	0.07	0.01	10.9
CEM-1	21.0	4.4	2.7	1.6	64.7	0.6	1.99	2.27	0.01	0.73

The XRF analysis of the raw clay and the reference cement are shown in Table 3. The raw materials contained the relevant oxides needed for reaction.

XRD of the two calcined clays are seen in Figures 3 & 4 respectively. Kaolinite peaks are observed (even though with smaller intensity) after calcining at 600°C . This indicates calcining at 600°C can introduce a partial disorder to the crystal structure of kaolinite, in a process to transform kaolinite into metakaolin. There is therefore partial dehydroxylation at 600°C [17,23]. There, however, appears to be a complete dehydroxylation of kaolinite at 700°C . Illite and smectite, on the other hand, stay in their crystalline phases. There is, however, a reduction in their peaks as temperature increased from 600°C to 700°C . The transformation of illite and montmorillonite from crystalline to amorphous structure is quite different from that of kaolinite because of the limited amount of hydroxyls present in illite and montmorillonite as compared to kaolinite. This, possibly, could cause a reorganization of the crystalline structure and may not necessarily lead to complete disordering [17].

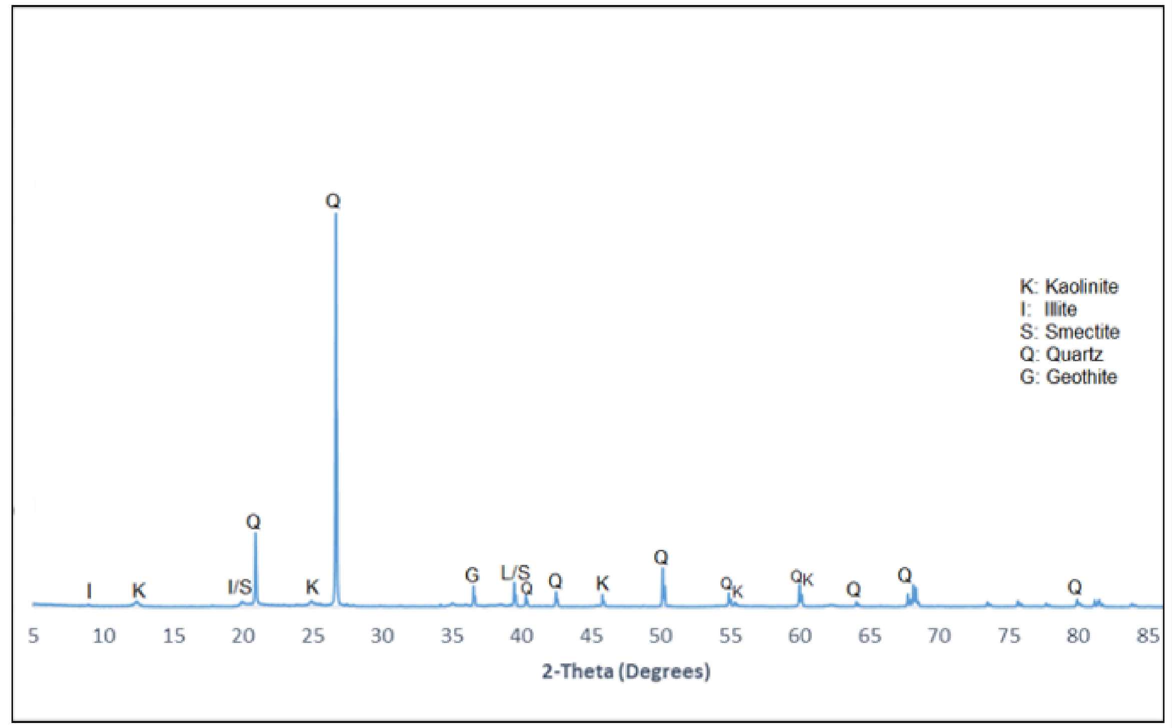

Figure 3: XRD pattern of clay calcined at 600°C .

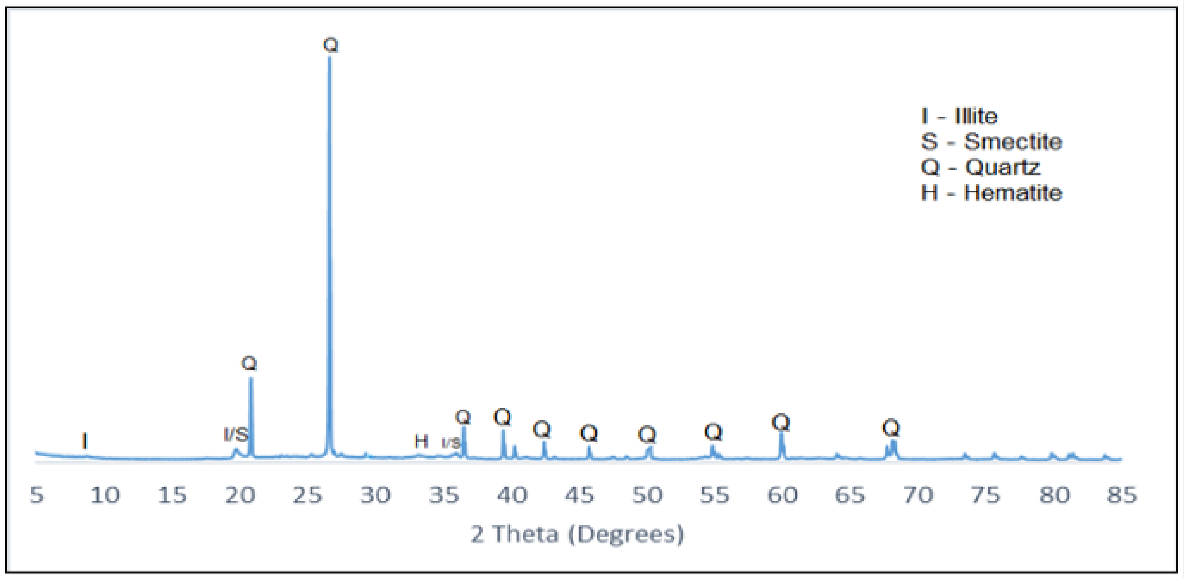

Figure 4: XRD pattern of clay calcined at 700°C .

A determination of the reactivity of the calcined clays, as demonstrated by Frattini test, is shown in Figure 5. Quartz sand, being non-reactive, is added to serve as the reference material. Blended cement containing 20% clay calcined at 600°C displays some amount of reactivity but is not reactive enough to cross the lime solubility curve into the pozzolanic region. On the other hand, blended cement containing clay calcined at 700°C is seen on the pozzolanic region of the curve, denoting pozzolanic reactivity. This suggests that calcium hydroxide from the cement system can react with the constituent of the calcined clay to produce further calcium silicate hydrates [24]. Sand, however, does not show any reactivity.

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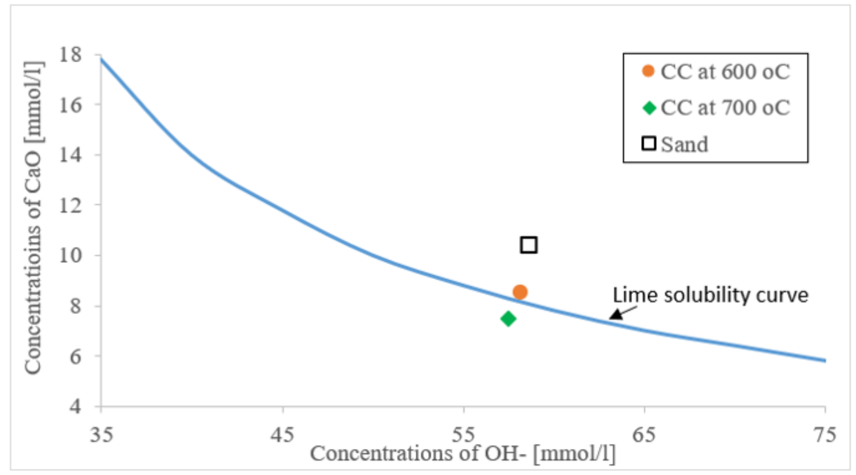

Figure 5: Reactivity of the calcined clays using Frattini test.

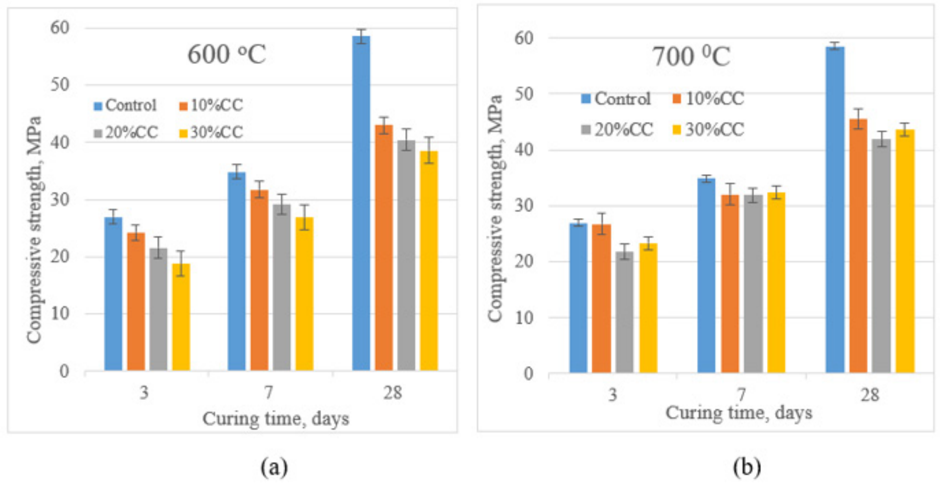

Figure 6: Compressive strength of mortars containing varying clay contents calcined at (a) 600°C and (b) 700°C .

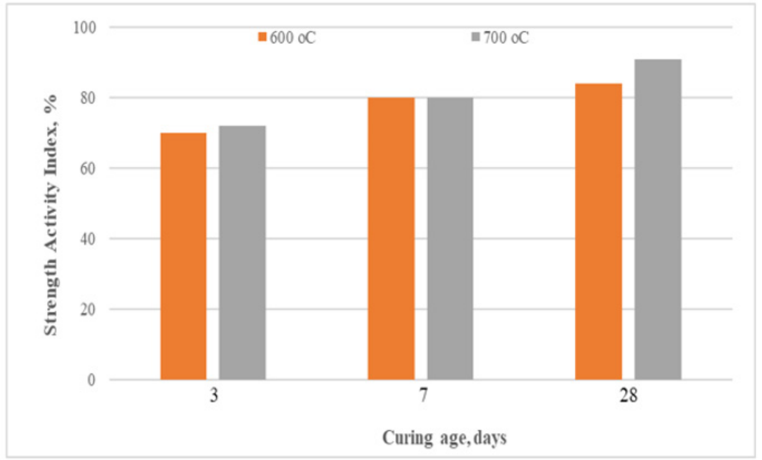

Figure 7: 28 days strength activity index results of the blended cement mortars containing 20 wt.% calcined clay at 600°C and 700°C were 84% and 91% respectively.

Figure 6 is the compressive strength results of blended cement mortars containing different percentages of calcined clay at 600°C and 700°C . The samples with non-blended cement were seen to obtain the highest compressive strength throughout the curing ages (i.e. 3, 7 and 28 days). Compressive strength generally decreased in the mixes with the blended cements, as calcined clay replacement increased. This could be attributed to dilution effect [8]. Within the first few days of curing, pozzolans in cementitious

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systems do not play a reactive role but only contributes to filler effect [17,18]. However, blended cements containing clay calcined at 700°C is seen to obtain higher compressive strength results compared 600°C calcined clay at 3, 7 and 28 days. Figure 7 shows the 28 days strength activity index results of the blended cement mortars containing 20 wt.% calcined clay at 600°C and 700°C were 84% and 91% respectively. This exceeds the 75% minimum requirement of pozzolanic activity as prescribed by ASTM C618.

Conclusions

Calcined Brick clay was found to have a good potential to be utilised as a supplementary cementitious material because of its meta kaolinite composition. Temperature at which clay is calcined is quite critical in achieving complete dihydroxylation of kaolinite. In this study, the clay was partially hydroxylated at 600°C but achieved complete hydroxylation when the calcination temperature was increased to 700°C . The setting time and water demand increased with increasing calcined clay content. However, blended cements containing 700°C calcined clay recorded lower water demand and setting times as compared to 600°C calcined clay. There was a reduction in compressive strength, both at early and later ages. Compressive strength generally decreased with the partial replacement of cement with calcined clay. Blended cements containing 700°C calcined clay recorded higher compressive strength than the clay calcined at 600°C . This could be due to the incomplete dehydroxylation of kaolinite at 600°C .

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