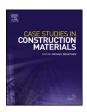
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## Case Studies in Construction Materials





# An investigation on the suitability of hydrated building lime from travertine limestone outcrop of Bogongo, South West of Cameroon



Ndigui Billong<sup>a,\*</sup>, Likiby Boubakar<sup>a</sup>, B.N. Bayiha<sup>b</sup>, S.M. Njimbouombouo<sup>a</sup>, Uphie C. Melo<sup>c</sup>, Jonathan Oti<sup>d</sup>, John Kinuthia<sup>d</sup>

- <sup>a</sup> Laboratory of Materials Analyses, Local Materials Promotion Authority (MIPROMALO), P.O. Box 2396, Yaounde, Cameroon
- <sup>b</sup> Department of Civil Engineering, Advanced Technical Teachers Training College, University of Douala, P.O. Box 1872, Douala, Cameroon
- <sup>c</sup> Department of Inorganic Chemistry, Faculty of Science, University of Yaoundé I, P.O. Box 812, Yaounde, Cameroon
- <sup>d</sup> Faculty of Computing, Engineering and Science, University of South Wales, Pontypridd, CF, 37 1DL, UK

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#### ABSTRACT

In the present study, physico-chemical investigations were carried out on hydrated lime produced from the limestone of the travertine outcrop of Bogongo in the South West Region of Cameroon. The aim was to evaluate the suitability of that hydrated lime as building lime. The raw material was characterized and then fired at 900 °C. The fired product was hydrated, dried and also characterized. Chemical and mineralogical analyses, density, BET specific surface measurements and thermal analyses were performed. Results were compared to those for an EN 459-1 CL 90-S industrial commercial hydrated lime. It has been shown that, hydrated lime production using the raw material from the Bogongo travertine could yield products with almost similar physico-chemical properties compared to imported CL 90 S hydrated lime, thus could have positive consequence in the commercial exploitation of the Bogongo travertine limestone outcrop.

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### 1. Introduction

Lime is a material that has been used as an essential binder for the production of mortars and plasters used in structures of different civilizations since antiquity [1]. Nowadays, the world consumption of lime is driven by the growing demand in chemical, industrial, metallurgical, environmental and construction applications [2]. Lime has also prominent applications in soil stabilization, coatings and lightweight insulation materials [3–5]. According to BS EN 459-1-2015 standard, lime refers to calcium oxide or hydroxide, calcium-magnesium oxide or hydroxide produced by calcination of naturally occurring calcium carbonate such as limestone, chalk and shells, or naturally occurring magnesium carbonate like dolomitic limestone and dolomite [6–8]. Although limestone deposits can be found in many parts of the world, only a small portion is pure enough for industrial lime manufacturing. Depending on the purity of the raw material, there are two families of lime: the first is air lime, which combines and hardens with atmospheric carbon dioxide, and the hydraulic lime. It is divided into two sub-families: calcium lime (CL) and dolomitic lime (DL). Quicklime is an air lime in the form of calcium oxide that reacts exothermally after contact with moisture or water. The second family is hydrated lime which is obtained by the controlled

E-mail address: nbillong@yahoo.fr (N. Billong).

<sup>\*</sup> Corresponding author.

slaking of quicklime. It can be available in powder (S), putty (S PL) or slurry milk (S ML). Based on BS EN 459-1-2015 standard, calcium and dolomitic lime types and their chemical (calculated on the basis of quicklime) and physical requirements are indicated in Table 1.

Prior to the invention of Portland cement, lime had been used in mortar, external and internal plastering and rendering, foundations, flooring, infilling of walls and casing of water conduits for the construction of several historic buildings in all the continents of the World [9,10]. With the discovery of Portland cement and the explosion of its use since World War II, the use of building lime was reduced to repair work. Since the beginning of the 21 century, lime as a building material is regaining interest in the scientific and technical community. The concern about the high amount of carbon dioxide (CO<sub>2</sub>), a major greenhouse gas responsible of global warming, released by the lime industry has been mitigated by various capture techniques that can transform the CO<sub>2</sub> to raw materials for the production of energy [11]. Numerous advantages of building lime have been documented compared to Portland cement. The less energy consumption during its production and the recovery of lime in masonry units after demolition places lime well ahead of Portland cement as a sustainable material [12,13]. From limestone to the hardened product after contact with air, the transformation process of lime is in the form of a cycle commonly referred to as « lime cycle » (Fig. 1). The lime cycle is a slow and gradual process that increases the hardening of the material and favors the self-healing of cracks, improves its durability, provides breathing property of the building and frees the indoor environment from moisture with benefit of improved humidity regulation and pollutants removal [13]. Also, the strength in lime based mortars or plaster has been improved with pozzolanic additives to produce cement-like material [14,15].

Travertine is a form of limestone deposited by mineral springs, especially hot springs. It is formed by rapid precipitation of calcium carbonate (CaCO<sub>3</sub>). Sedimentological, pretrological and geochemical studies conducted on the Bogongo travertine deposits, situated in the South-West Region of Cameroon [16,17] showed that the material consisted of dense and hard limestone concretions with high calcite (CaCO<sub>3</sub>) content (98.2 to 99.8 %), associated with detrital quartz (0.2 to 0.8 %). No existing study has been conducted to assess the suitability of the limestone from the Bogongo travertine for building lime production. Thus, the objective of the present research is to investigate the physico-chemical properties of hydrated lime obtained by calcining a sample of limestone from the Bogongo travertine deposit at 900 °C, and compare the results with those of an industrial commercial hydrated lime of BS EN 459-1 CL 90-S type. This involved chemical and mineralogical characterizations, density, BET specific surface measurements and thermal analyses of hydrated lime.

#### 2. Materials and methods

## 2.1. Materials

The materials used in the present work were a limestone sample (EKD) from the Bogongo travertine deposit in the South-West Region of Cameroon as indicated in Fig. 2, an industrial hydrated lime EN 459-1 CL 90-S type produced by SB-Mercier in France and distilled water

### 2.2. Methods

## 2.2.1. Preparation of test specimens

The limestone sample was crushed to  $500~\mu m$  using an impact mill and fired at a temperature rise rate of 10~C/minute in a laboratory Nabertherm electric oven at 900~C inside a crucible and soaking time of two hours. This production process was chosen to optimize the firing of limestone at a laboratory scale. It differs from the industrial process where vertical and rotary kilns make use of limestone pebbles with particles size between 30 to 100~mm. As soon as the cooling started, the crucible was removed from the oven and left to continue to cool in a desiccator to avoid the contact of lime with the atmospheric carbon dioxide. Excess distilled water (60% in weight of fired material) was then poured on the contents and mixed to form "lime putty" which was left to mature for 24 hours in a container having a lid to avoid contact with  $CO_2$  from air. The "lime putty" was then dried in an oven for 24 hours at 110~C and thereafter crushed for 30 minutes in a ball mill to obtain the laboratory made hydrated lime powder (CHEKD) for characterizations.

**Table 1**Chemical requirements for some building limes according to BS EN 459-1:2015.

Type of lime	Designation	Oxide content (wt.%)						
		CaO + MgO	MgO	CO <sub>2</sub>	SO <sub>3</sub>	Available lime	Soundness (mm)	
Calcium lime	CL 90	≥ 90	≤ 5	<b>≤</b> 4	≤ 2	≥ 80	Pass, according to BS EN 459-2 :2010 [7]	
	CL 80	≥ <b>80</b>	≤ 5	≤ 7	<b>≤</b> 2	≥ 65		
	CL 70	≥ <b>70</b>	≤ 5	≤ 12	≤ 2	≥ 55		
Dolomitic lime	DL 85	≥85	≥ <b>30</b>	≤ 7	<b>≤</b> 2	1		
	DL 80	$\geq 80$	≥ <b>5</b>	≤ <b>7</b>	$\leq 2$	1		

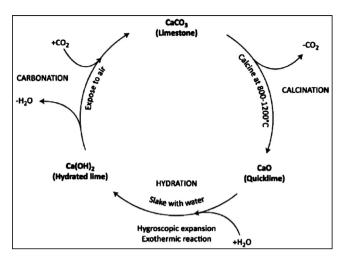


Fig. 1. Schematic representation of the « lime cycle ».

#### 2.2.2. Physico-chemical characterizations

In order to characterize the various materials used in the study, the following techniques were used:

## • Inductively Coupled Plasma (ICP) analyses

The Inductively Coupled Plasma (ICP) spectrometry method was used to chemically characterize the limestone sample (EKD), the commercial hydrated lime (CHX) and the laboratory made hydrated lime (CHEKD). The device was an Atomic Emission Spectrometer using induced argon plasma (ICP-AES). Samples were dissolved into solutions before analyses. Thus, 19 to 27 mg of dry powder sample having particle size less than 80 microns was introduced into the reactors made of Teflon tubes. Then 3 mL of hydrofluoric acid (HF) and 9 mL hydrochloric acid (HCl) were added. The reactors were then mounted in a CEM MARS 5 microwave oven equipped with pressure and temperature sensors. The reactors were heated to 180 °C at a temperature rise rate of 12 °C/minute using a soaking time of 20 minutes and pressure of 30 bars to allow the complete dissolution of the material. At the end, the liquid of the reactors was recovered in a 250 mL plastic flask. The rinsing of each reactor and its accessories made it possible to increase the volume of the solutions to 250 mL.

For analyses, a small volume of the solution was introduced into the argon plasma (temperature of about 4200 to 6000 K). The passage of the solution in the hottest region of the plasma allowed the excitation of the electrons of the peripheral layers of the elements in the solution, which then left the hot zone and returned to their ground state by emitting a photon whose wavelength characterized the original element. This wavelength was then oriented towards a sensor that made it possible to identify the elements and then to quantify them by means of calibration curves previously produced. The measurements were made at a constant temperature of 23 °C.

## • X-Ray Diffraction (XRD) test

The two starting materials (EKD and CHX) were subjected to the XRD test that allowed determining the crystalline phases present in the tested specimens. The equipment used was a Debye-Scherrer assembly equipped with a localized curved detector (INEL CPS 120 - Curved Position Detector) at the center of which the sample was placed. The X-ray source operated at 40 kV and 30 mA while the monochromatic radiation used had a wavelength of 1.540598 Å (copper  $K\alpha_1$ ). The sample holder of the apparatus had two rotational movements and a translational movement. One of these rotations was to adjust the angle of incidence  $\alpha$  of the beam while the other, controlled by a motor, was to rotate the sample during data acquisition around a perpendicular axis to its surface, thus ensuring a random distribution of the orientation of the crystallites. The translational movement helped to position the sample so that its surface intercepts the incident X-Ray beam on the axis of rotation of the assembly. The photons were diffracted in an angular range  $2\Theta$  of about  $120^{\circ}$  but during the exploitation of the curves  $2\Theta = 65^{\circ}$  was considered.

## • Absolute density measurements

The absolute density is the ratio of the dry mass of sample to its actual volume of solid matter in a porous body. The measurements of the absolute density were carried out according to ISO 5018: 1983 [18] standard. The equipment used was an Accupyc1330 helium pycnometer. The determination of the volume of the sample  $(V_{ech})$  makes it possible to calculate its density. By applying the Mariotte's law (equation 1), the principle of the technique is based on the measurement of the pressure  $P_1$  which prevails in a calibrated chamber and the pressure  $P_2$  of the cell which contains the sample. The cell volume

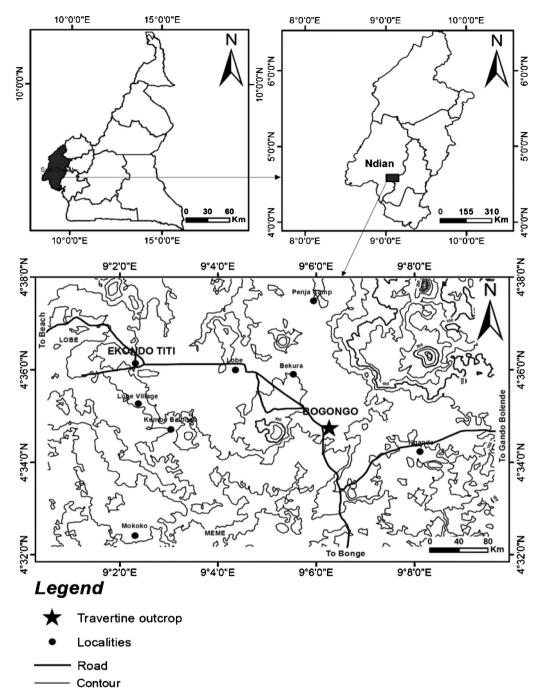


Fig. 2. Localization of the Bogongo travertine outcrop.

 $(V_{cell})$  and the expansion volume  $(V_{exp})$  are constants given by the manufacturer. After calibrating the pycnometer with a steel ball of known mass and volume, a small amount of sample (dried at 110 °C for 24 hours) was placed in a cell and then introduced into the apparatus. The helium pressure was set at 1.8 bars and the device automatically performed five volume measurements of particles and calculated the average volume (in cm<sup>3</sup>). Using the dry mass (in grams) of the sample initially recorded in the apparatus, the average density was established and displayed in the device in  $g/cm^3$ .

$$V_{ech} = V_{cell} - \frac{V_{exp}}{\frac{P_1}{P_2} - 1} \tag{1}$$

## • BET specific surface measurement

The specific surface is one of the properties that govern the reactivity of powders. The measurements were performed on CHX and CHEKD samples. The experimental determination of the BET specific surface is based on the principle of nitrogen adsorption at low temperature in materials. From a quantity of absorbate, the size of the adsorbed molecules and their possibilities of arrangement, it was possible to evaluate the surface on which the adsorbate molecules were fixed using the so-called Brunauer, Emmett and Teller (BET) calculation model. The BET method required a pre-treatment of the samples (degassing and drying between 150 and 300 °C in order to evacuate the gases previously adsorbed). The apparatus that was used included a Desorb degassing device 2300 with three stations under nitrogen sweep and a Flowsorb II 2300 measuring device under a nitrogen/helium mixture also having a degassing station. Grains of powders previously dried for 24 hours in an oven at 110 °C were introduced into cells and fixed at the degassing stations of the apparatus. The degassing was then carried out for 2 hours at 200 °C. Once the degassing was completed, the cells were passed one after another to the previously calibrated measuring station to measure the amount of gas absorbed and to determine the surface of the powder. During the measurement, the cell was immersed in liquid nitrogen and the gas was introduced into the cell. The gas was adsorbed progressively until the saturation of grains occurred. When the equipment indicated a stable surface for grains, the sample was removed from the liquid nitrogen and the gas gradually desorbed from the grains and a new equilibrium established. The equipment indicated another surface for grains which was retained as S in m<sup>2</sup>. At the end of the process, the dry mass (m) in grams of the sample was determined and the ratio between S and m gave The BET specific of the grains in m<sup>2</sup>/g.

## • Thermal analysis

The thermogravimetric analysis (TGA) revealed and quantified the loss of material weight as a function of temperature. This technique was applied to EKD, CHX and CHEKD samples. The device used was a Linseis model. About 140 mg of sample was introduced into an alumina crucible and placed in the heating block of the apparatus. Once the device was turned on, the weight loss as a function of temperature of up to 1000 °C was recorded and stored in the computer data base system. A "white test" was performed under the same conditions to establish the baseline. Differential thermal analysis (DTA) was also performed to sample EKD using DTA-TG Setsys 2400 equipment because peaks of energy displayed during thermal treatment of the raw material can give an indication on the optimal firing temperature of the raw material.

#### 3. Results and discussion

## 3.1. Densities and BET specific surfaces

The results of density and BET specific surface measurements are shown in Table 2. The high density  $(2.813 \, \text{g/cm}^3)$  for EKD compared to ordinary limestone  $(1.55 \text{ to } 2.75 \, \text{g/cm}^3)$  could be an indication of the high compactness of the Bogongo travertine. The density of CHEKD is slightly greater than that of CHX. This difference could be explained by the difference in their chemical composition with CHEKD having higher  $SiO_2$ ,  $Al_2O_3$  and  $TiO_2$  than CHX. Nevertheless the densities of the two hydrated limes are in the range of densities for common hydrated limes which are typically between 2.2 and 2.4 g/cm<sup>3</sup>. The grinding time applied to CHEKD permitted to produce fine particles with specific surface of 21.18 m<sup>2</sup>/g higher than that for the industrial hydrated lime (CHX) which was  $19.12 \, \text{m}^2/\text{g}$ . The specific surface of hydrated lime is mostly affected by the type of limestone used and its calcination and slaking processes [19]. In industry, slurry detention, paste, ball mil and batch slackers produce hydrated lime without the need of the grinding process [19,20]. The typical specific surface of hydrated lime ranges from 8 to  $58 \, \text{m}^2/\text{g}$ .

## 3.2. Chemical and mineralogical analysis

The results of chemical analysis (Table 3) indicated that EKD, CHX and CHEKD mainly consisted of CaO as major oxide with minor oxides made of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub> and MgO. Sample CHEKD contained slightly more SiO<sub>2</sub> than CHX, but CHX had more MgO than CHEKD. Small quantity of TiO<sub>2</sub> was detected in CHEKD. The presence of that oxide could be responsible for the yellowish color presented by CHEKD compared to the whitish color of CHX. Both samples CHX and CHEKD presented roughly lower loss on ignition compared to EKD. This could be mainly explained by the decomposition Ca(OH)<sub>2</sub> to form CaO and H<sub>2</sub>O during the thermal treatment, while CaCO<sub>3</sub> in EKD is transformed to CaO and CO<sub>2</sub> during heating. The heating process at

 Table 2

 Absolute densities and BET specific surface of samples

Sample	Absolute density (g/cm <sup>3</sup> )	BET specific surface (m <sup>2</sup> /g)
EKD	2.813	1
EKD CHX	2.262	19.12
CHEKD	2.322	21.18

**Table 3**Major oxides in the studied materials (nd = not detected, L.O.I.= Loss on ignition).

Oxides	EKD	CHX	CHEKD
SiO <sub>2</sub>	1.68	1.05	1.49
$Al_2O_3$	0.76	0.85	0.90
$Fe_2O_3$	0.61	0.62	0.62
CaO	53.00	65.30	64.12
MnO	0.05	nd	nd
MgO	0.69	1.91	0.78
Na <sub>2</sub> O	0.04	nd	nd
K <sub>2</sub> O	0.02	nd	nd
TiO <sub>2</sub>	0.20	nd	0.18
L.O.I. at 1000°C (%)	42.5	29.34	28.11
Total	99.55	99.07	96.20

 $1000\,^{\circ}\text{C}$  is responsible of about  $44\,\%$  weight loss in pure  $\text{CaCO}_3$  (equation 2). In the case of samples CHX and CHEKD, the major contribution to weight loss on ignition is from the release water molecules during the heating of  $\text{Ca}(\text{OH})_2$  in samples (equation 3). The Loss on ignition of CHX (29.34%) is slightly greater than 29 % expected if the hydrated lime was pure. The difference could be explained by the presence of small proportion of calcium carbonate ( $\text{CaCO}_3$ ) produced by the reaction of a small part of  $\text{Ca}(\text{OH})_2$  with the carbon dioxide of air or from unburned limestone particles. The CaO, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub> and MgO content in lime is essential for their reactivity and their classification as building lime. They can be considered as hydraulic or non-hydraulic depending on their Hydraulic Index (HI) which is calculated using equation 4.

$$CaCO_3 \rightarrow CaO + CO_2 \tag{2}$$

$$Ca(OH)_2 \rightarrow CaO + H_2O$$
 (3)

$$HI = \frac{\%Al_2O_3 + \%Fe_2O_3 + \%SiO_2}{\%CaO + \%MgO}$$
(4)

The HI calculation applied on CHX and CHEKD gave 0.037 and 0.046 respectively and suggested the two hydrated lime samples to be non-hydraulic, since their HI were less than 0.1 [21]. The sum of CaO, MgO and loss on ignition was 96.59 and 93.01 for CHX and CHEKD respectively. These values were greater than 90, further confirming the two hydrated limes as CL 90 S types according to the standard BS EN 459-1 [6].

The results of the XRD test for CHX and EKD are indicated in Figs. 3 and 4 respectively. In the case of CHX, calcium hydroxide and quartz were identified as the minerals present. Sample EKD was essentially made of calcite ( $CaCO_3$ ) and quartz ( $SiO_2$ ). After the thermal treatment at 900 °C, it can be assumed that the product will be mainly consisted of CaO, explaining the higher loss on ignition of EKD. The other minor minerals phases including those containing  $Al_2O_3$ ,  $Al_$ 

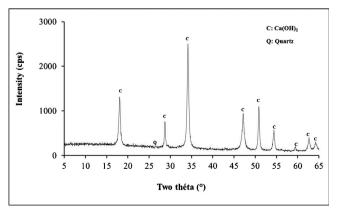


Fig. 3. XRD patterns of CHX.

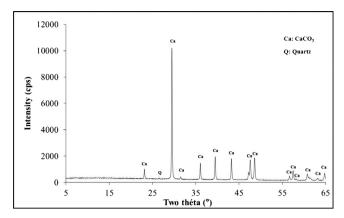


Fig. 4. XRD patterns of EKD.

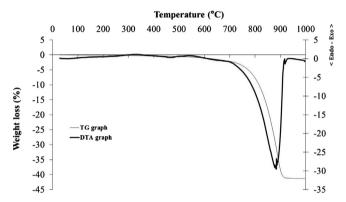


Fig. 5. DTA-TG thermograms of EKD.

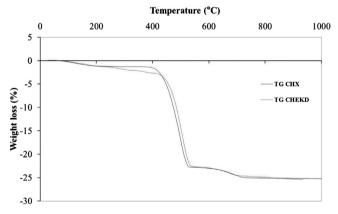


Fig. 6. TG thermograms of CHX and CHEKD.

## 3.3. Thermal properties

The thermal profiles for samples EKD, CHX and CHEKD are shown in Figs. 5 and 6. Fig. 5 did not only illustrated the thermal decomposition of phases that are present in the limestone, but also allowed the estimation of the temperature range at which the material must be calcined to convert its calcium carbonate (CaCO<sub>3</sub>) to quicklime (CaO) as indicated by the endothermic reaction that started at about 700 °C and finished at about 910 °C with optimum decomposition of the material being at 883.6 °C. The maximum weight loss was 41.3%, mainly due to the decomposition of CaCO<sub>3</sub> [22–24]. The result

confirmed that sample EKD essentially consisted of CaCO<sub>3</sub>. Consequently, by comparing the weight loss of pure CaCO<sub>3</sub>, calculated according to reaction (2), the percentage of CaCO<sub>3</sub> in EKD can be estimated at 94% if it is assumed that the contribution to weight loss was essentially due to the decomposition of CaCO<sub>3</sub>. This percentage is greater than that indicated by Bayiha et al. [25], but lower than that estimated by Tchouatcha et al. [16] or Bisse et al. [17]. The proportion of impurities such as quartz (SiO<sub>2</sub>) in the limestone sample can the estimated at 6%.

The TG curves of CHX and CHEKD samples are shown in Fig. 6. The weight loss that started at about 300 °C and ended at about 500 °C with about 22% weight loss was due to the de-hydroxylation of the hydrated lime through the reaction illustrated by equation 3. The second weight loss between 500 °C and 700 °C) with about 25% weight loss can be due to the decomposition of impurities in the form of magnesium hydroxide, residual un-burnt calcium carbonate from EKD or carbonate formed in the calcined material after its contact with carbon dioxide of air [26]. Thermogravimetric results confirmed the similarity between the industrial hydrated lime and that synthesized from EKD.It is then clear that hydrated lime production using raw material from the Bogongo travertine could yield products with almost similar physico-chemical properties compared to imported CL 90 S hydrated lime. This outcome would have beneficial social-economic dynamics in the Bogongo region and neighboring communities. For the industrial production of the lime, additional properties like available lime and soundness according to BS EN 459-2:2010 standard should be tested for conformity to BS EN 459-1 standard.

#### 4. Conclusion

From investigations carried out on the physico-chemical suitability of hydrated lime from the Bogongo travertine as building lime, the following conclusions can be made:

- The limestone of the travertine contained about 94% CaCO<sub>3</sub> and 6% impurities mainly consisting of quartz;
- The production of hydrated lime using the raw material from the Bogongo travertine could yield products with almost similar physico-chemical properties compared to imported CL 90 S hydrated lime;
- The density measurement of the travertine sample suggested a very dense and compact material compared to ordinary limestone:
- The Bogongo limestone should be fired between 550 °C to 910 °C with optimum decomposition of the material being at about 880 °C.
- The TiO<sub>2</sub> content of the Bogongo limestone could contribute to the yellowish color of the hydrated lime produced from the limestone:
- There is potential for social-economic development of the Bogongo region, for commercial activities occurring for the production of building lime from Bogongo travertine outcrop.

## **Declaration of Competing Interest**

There is no conflict of interest.

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