



Production of quicklime from Ashaka limestone through calcination process

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Abstract

This study is on the production of quicklime from Ashaka limestone through calcination process. Effects of temperature, particle size and time on quicklime yield were determined. The experiment was carried out at temperatures of 800, 900, 1000, 1100 and 1200 °C, particle sizes of 80µm, 90µm, 100µm, 300µm and 425µm and times of 0.5hr, 1hr, 2hrs, 3hrs and 4hrs. Analyses of the results showed that quicklime was successfully produced from Ashaka limestone through the calcination process. Quadratic model adequately described the relationship between quicklime yield and calcination factors of temperature, particle size and time. Recorded model F-value of 134.35 implies that the model is significant. The predicted R² of 0.9597 is in reasonable agreement with the adjusted R² of 0.9844; the difference is less than the critical value of 0.2. Optimum yield of 73.48% was obtained at optima operating conditions; temperature of 1000 °C, particle size of 90 µm and time of 3 hrs.

Keywords: Calcination; Ashaka limestone; Quicklime yield; Optimization

1. Introduction

Ashaka limestone deposit is located in Funakaye Local Government Area of Gombe State, Nigeria. Its usage has not been sufficiently diversified. Effective application of the limestone will depend on the level of its modification (treatment/processing). For instance, limestone (mainly made up of CaCO₃) will not be reactive enough to remove a desirable amount of sulfur dioxide from flue gases (in a desulphurization process). Processed limestone produces value added product(s) with wide applications in construction, agro, chemical and allied industries [1, 2, 3, 4]. Hence, there is need to calcined Ashaka limestone to quicklime (CaO). Calcination is an endothermic reaction, which consists of five process steps; heat transfer from ambient to the solid surface, heat conduction from surface to the reaction front, chemical reaction at the front, diffusion of CO₂ through the porous oxide layer to the surface, and the mass transfer into the surroundings [5]. The internal structure of a rock having open and closed pores in its texture affects the heat transfer. Lime is produced by calcining of limestone at temperatures over 800°C in a %100 atmosphere at 760 mmHg pressure. In the calcinations process, limestone decomposes by expelling carbon gas (CO₂) and converts to quicklime. In the process, there are three main requirements which are heating the stone to its dissociation temperature, providing for minimum temperature for certain duration and expelling the CO₂ gas from the stone. When a pure limestone (calcite) is heated to its dissociation temperature, it loses 44% of molecular weight by expulsing of CO₂.

In processing of limestone, typical approach involving transport phenomena, calcination and reactor models are considered [6]. It can be processed into quicklime and subsequently into slaked lime. Lime production is a process that entails complex material and energy transport and chemical reaction. Calcination is carried out in lime kilns. In ancient times, charcoal and wood were burnt as sources of energy for the calcination of limestone. There were two basic types of lime kilns, which were flare kilns and continuous kilns. In flare kilns, limestones are burnt with the heat and flames without any contact with fuel. But, for the running kilns, continuous burning was carried out in such a way that limestones and fuel took place alternately in the kilns; calcined one then the other successively. Quicklime produced in

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flare kilns were purer when compared to quicklime produced in running kilns. As such, flare kilns increased the quality of the produced quicklime.

The general procedures of processing limestone begin with initial cutting, followed by application of a finish, and then a second cutting / shaping step. Proper management of the activities is needed for the safety of the environment [7, 8]. Processing commences with transportation of the limestone from the quarry to the processing facility. Limestone is often produced with a natural surface, but finishes can be applied. In such cases, often a polished or honed finishing is given to limestone products, but a variety of other finishes are also common. Polishing and honing are manually and/or mechanically accomplished through the use of polishing pads or bricks. A secondary shaping step may be necessary if the product includes any features or custom size or shape.

In most cases, limestone application requires its calcination in shaft or rotary kilns, where carbonate is thermally decomposed to produce quicklime and CO₂. There are numerous critical variables that exert profound effect on lime burning operations [9]. The factors and variables that affect the burning of limestones must be considered in the selection, design and optimization of the calcining equipment. Limestone burning in a vertical kiln often presents complex problems which can be solved from the conception by consideration of the prevailing factors that determine the progress of calcinations reactions. The calcination reaction is endothermic [9]:



The forward reaction is favoured by higher temperatures. The reaction will proceed only if the partial pressure of CO₂ in the gas above the solid surface is less than the decomposition pressure of the CaCO₃. According to Yang and Yongping (2015), the calcination reaction of limestone is always accompanied by sintering of the calcined product. The accelerated sintering rates and a reduced specific surface area are observed in the presence of steam and carbon dioxide.

Factors that affect Calcination rate are particle size, temperature, retention time, CO₂ concentration in kiln, type of fuel used, pre-heating and cooling, size of limestone/types of kilns. Calcination rate increased with decrease in particle size. The chemical reaction and internal mass transfer of the limestone in a kiln depends on particle size. The best particle size of limestone in rotary kilns is the size that will allow for quick heating, short residence time and minimum amount of cores. Smaller size limestone is most suitable for calcination in rotary kilns and will allow optimum residence time. Temperature also affects the rate of calcination. For complete calcination, lowest temperature with the shortest possible residence time is required. Higher calcination temperature will cause increased shrinkage and reduction in volume. It will also cause recarbonation of the surface of CaO pebbles, with the presence of CO₂ released from the limestone as well as by product of combustion, which makes the lime non-porous, and thus not suitable for slaking. The temperature rise must be gradual and even so that the limestone can remain porous during the process. Retention time depends on the particle size of the limestone as well as temperature of calcination. The size of the limestone is the most critical factor in calcination.

2. Material and methods

Quicklime (CaO) was produced from limestone by calcination process. Effects of temperature, particle size and time on quicklime yield were determined. 10g of the limestone sample (80µm particle size) was weighed into pre-weighed empty crucible plates. The pre-weighed crucible plates with the limestone were set into laboratory furnace and heated at various temperatures 800 °C - 1200 °C. The first sample was removed after 30 minutes of holding time, thereafter other samples removed at the time of 1, 2, 3 and 4 hours. After the heating, it was allowed to cool for 15 minutes. The calcined samples were transferred to desiccators to maintain their integrity. The weight of the quicklime produced was measured. The experiment was carried out at temperatures of 800, 900, 1000, 1100 and 1200 °C and particle sizes of 80µm, 90µm, 100µm, 300µm and 425µm. Percentage yield of the quicklime was determined using the expression:

$$Y = \frac{W_2}{W_1} \times \frac{100}{1} \quad (2)$$

Where W₁ = Weight of limestone before calcination, W₂ = Weight of limestone after calcination, Y = percentage yield.

The calcination process was carried out using one-factor at-a-time and response surface methodology. As used by researchers [11], central composite design (CCD) tool of Design Expert Software 11 was employed in the design of the experiment. Temperature, particle size and time were the considered factors of the calcination process, while percentage yield was considered as the response.

2.1. Effects of the Process Variables on the Quicklime Yield

Effects of temperature on Ashaka quicklime are presented in Figures 1 – 5. For any particle size, the Figures showed a relationship between the yield and temperature at various times of the calcination. The quicklime yield decreased with increase in temperature, particle size and time. This observation is in agreement with previous findings [12]. The Figures showed the results of one factor at-a-time. Further analysis is required to determine effects of interactions of the calcination variables on the quicklime yields. Such analysis is shown in the response surface methodology result.

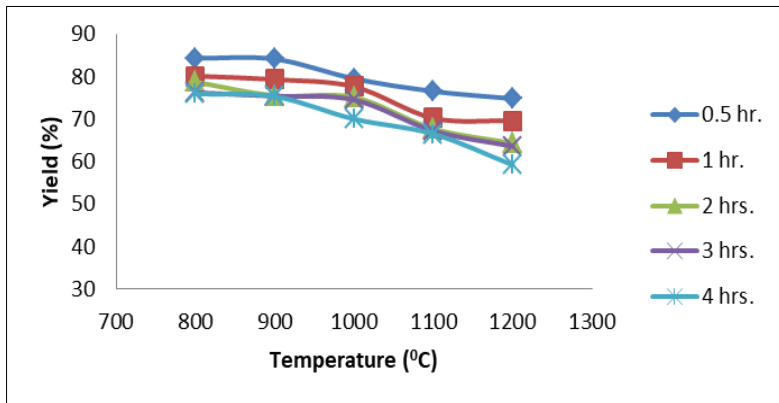


Figure 1 Effects of Temperature on the Calcination of 80 µm of Ashaka Limestone

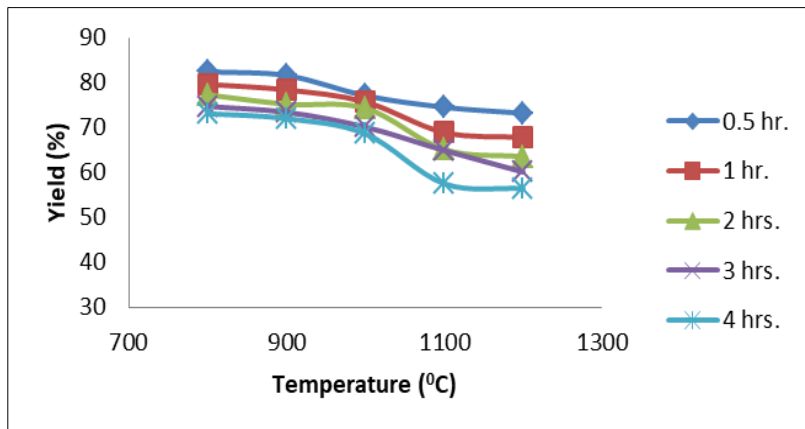


Figure 2 Effects of Temperature on the Calcination of 90 µm of Ashaka Limestone

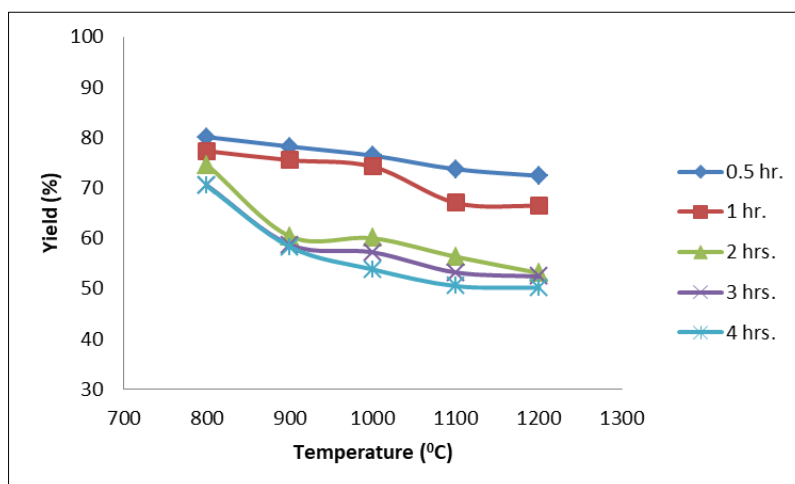


Figure 3 Effects of Temperature on the Calcination of 100 µm of Ashaka Limestone

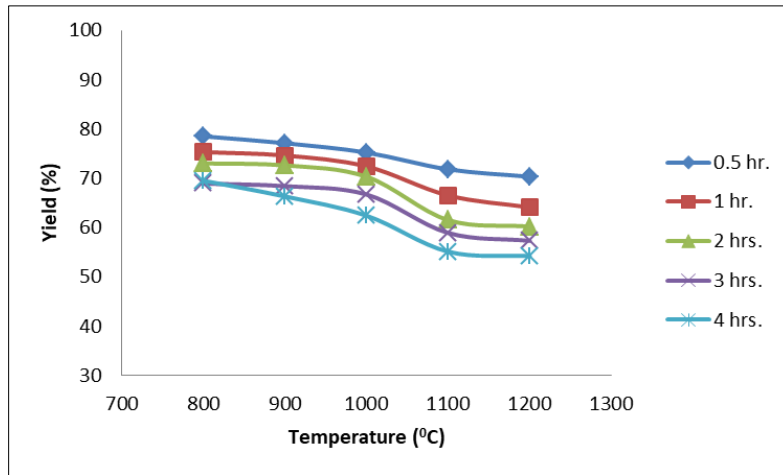


Figure 4 Effects of Temperature on the Calcination of 300 µm of Ashaka Limestone

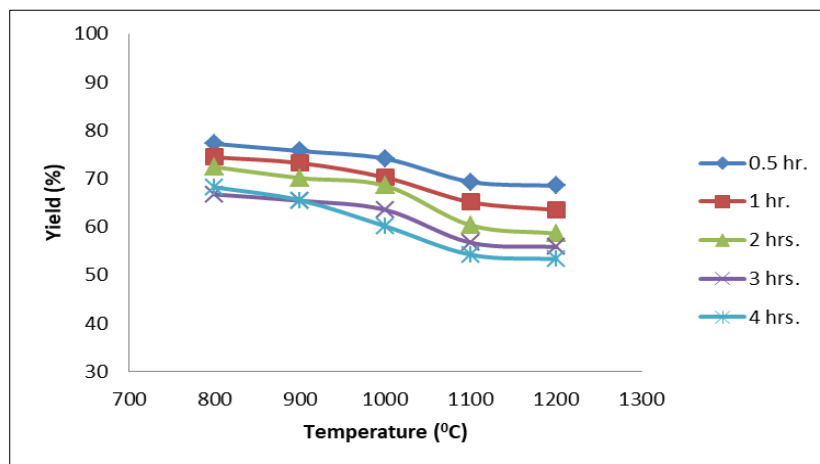


Figure 5 Effects of Temperature on the Calcination of 425 µm of Ashaka Limestone

2.2. RSM Results of the Calcination Process

The RSM result of the calcination of Ashaka limestone is shown in Table 1. It showed effects of the interactions among the factors of temperature, particle size and time on the percentage yield of the quicklime. Low yields of 50.5% and 56.3% were recorded at extreme points of temperature, particle size and time. This is an indication that at extreme calcination variables, the quality of Ashaka quicklime is low. The pattern of the data revealed that the peak of the quicklime yield is around the mid-points of the calcination variables (temperature of 1000 °C, particle size of 90 µm and time of 3 hrs). It revealed that quicklime yield is temperature, particle size and time dependent. The variations in the percentage yield may be attributed to the compositions of the limestone sample [13, 14]. The nature of variation of the quicklime yield with the factors of the calcination suggests that there are peaks (maximum points) in the relationship between the factors and response of the calcination process. Such peaks are made visible through graphical analysis. More so, further analyses of these data are required to obtain the nature of interactions (synergistic and antagonistic effects) among the considered factors of the calcination process. Such analyses include; ANOVA, modeling and optimization.

Table 1 RSM Results of calcination of Ashaka Limestone

Std	Run	Factor 1 A: Temperature (°C)	Factor 2 B: Particle Size (µm)	Factor 3 C: Time (hr)	Response Yield (%)
4	1	1100	100	2	56.3
3	2	900	100	2	60.4
1	3	900	80	2	75.4
11	4	1000	80	3	74.5
18	5	1000	90	3	74.1
12	6	1000	100	3	58.3
14	7	1000	90	4	70.6
16	8	1000	90	3	74.1
10	9	1100	90	3	64.9
19	10	1000	90	3	74.1
2	11	1100	80	2	67.7
13	12	1000	90	2	73.3
17	13	1000	90	3	74.1
5	14	900	80	4	75.3
6	15	1100	80	4	66.5
8	16	1100	100	4	50.5
9	17	900	90	3	73.4
20	18	1000	90	3	74.1
7	19	900	100	4	56.3
15	20	1000	90	3	74.1

2.3. Analysis of variance (ANOVA) of the quicklime yield

The ANOVA of the Ashaka quicklime yields is presented in Table 2. The statistical data revealed the criteria for determining the significance of the models, with regards to the considered factors of the calcination process. The model F-value of 134.35 implies that the model is significant. P-values less than 0.0500 indicate that the model terms are significant. In this case A, B, C, AB, BC, A², B² are significant model terms. It showed that Ashaka quicklime yield depends on temperature, particle size and time. Also, there were two forms of significant interactions; temperature and particle size, and particle size and time relationships. The Predicted R² of 0.9597 is in reasonable agreement with the Adjusted R² of 0.9844; the difference is less than 0.2. Adequate precision measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 36.469 indicates an adequate signal. Thus, the model of the Ashaka quicklime yield can be used to navigate the design space.

Table 2 ANOVA of Ashaka Quicklime Yield

Source	Sum of Squares	Df	Mean Square	F-value	P-value	
Model	1168.54	9	129.84	134.35	< 0.0001	Significant
A-Temperature	121.80	1	121.80	126.04	< 0.0001	
B-Particle Size	602.18	1	602.18	623.12	< 0.0001	
C-Time	19.32	1	19.32	19.99	0.0012	
AB	5.44	1	5.44	5.63	0.0390	
AC	0.9800	1	0.9800	1.01	0.3377	
BC	9.24	1	9.24	9.57	0.0114	
A ²	31.88	1	31.88	32.98	0.0002	

B ²	104.17	1	104.17	107.79	< 0.0001	
C ²	1.01	1	1.01	1.04	0.3319	
Residual	9.66	10	0.9664			
Lack of Fit	9.66	5	1.93			
Pure Error	0.0000	5	0.0000			
Cor Total	1178.20	19				
Std. Dev.	0.9830			R ²		0.9918
Mean	68.40			Adjusted R ²		0.9844
C.V. %	1.44			Predicted R ²		0.9597
				Adeq Precision		36.4686

2.4. Mathematical Model of the Quicklime Yield

The mathematical model of the quicklime yield is presented in Equation 2. The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients. As revealed by the analysis of variance, the model adequately described the relationship between the quicklime yield and the factors of temperature, particle size and time. Thus, the yield is a function of temperature, particle size and time. The positive signs in the model signified synergistic effect, while the negative signs signified antagonistic effect.

$$\text{Yield} = + 73.48 - 3.49A - 7.76B - 1.39C + 0.8250AB - 1.07BC - 3.40A^2 - 6.15B^2 \quad (3)$$

2.5. Graphical Analysis of the Quicklime Yield

Graphical representations of the quicklime yields are presented in Figures (6) – (9). Plot of predicted versus actual yield was used to test the performance of the model. It gave a linear graph. The graphs (3-D surface plots) showed the relationship between the factors and response of the designed experiment. The 3-D plots revealed the optimum yield as 73.48% with the corresponding optimal factors of temperature (1000 °C), particle size (90 μm) and time (3hrs).

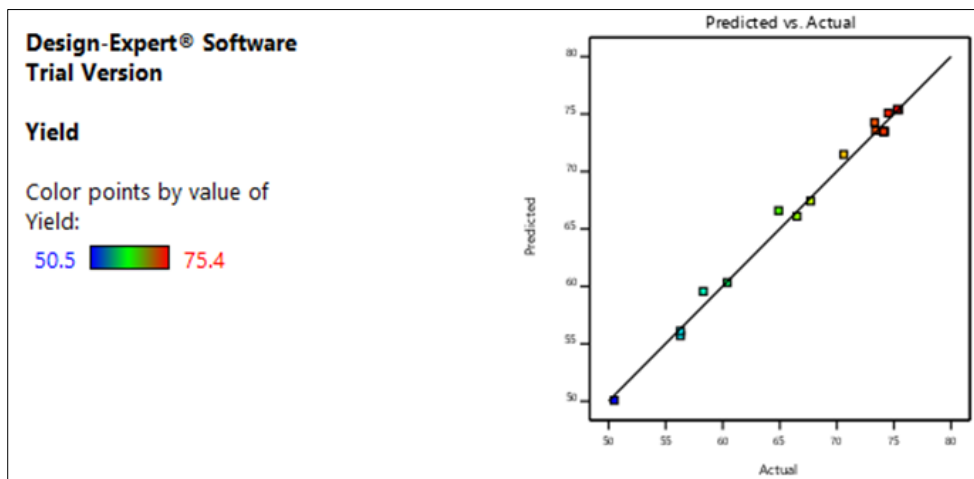


Figure 6 Predicted versus Actual Yield of Ashaka Quicklime

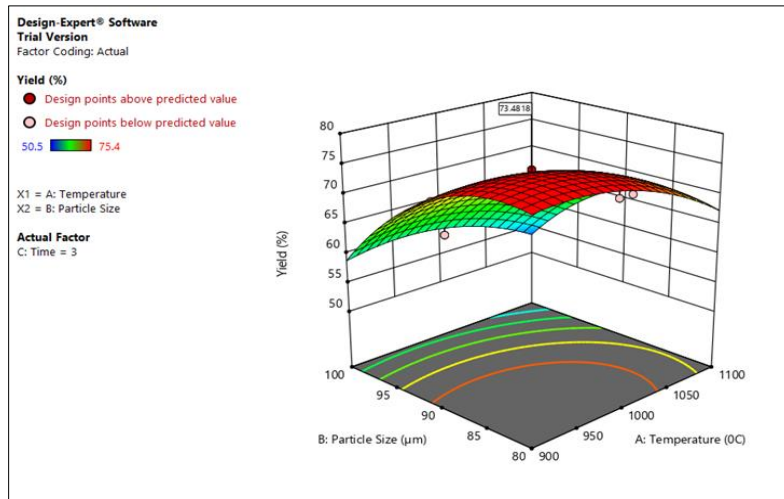


Figure 7 Effects of Temperature and Particle Size on Ashaka Quicklime Yield

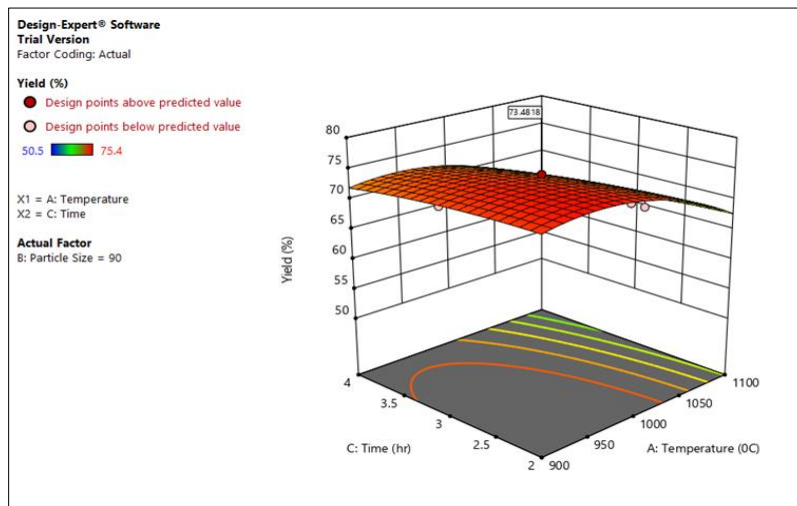


Figure 8 Effects of Temperature and Time on Ashaka Quicklime Yield

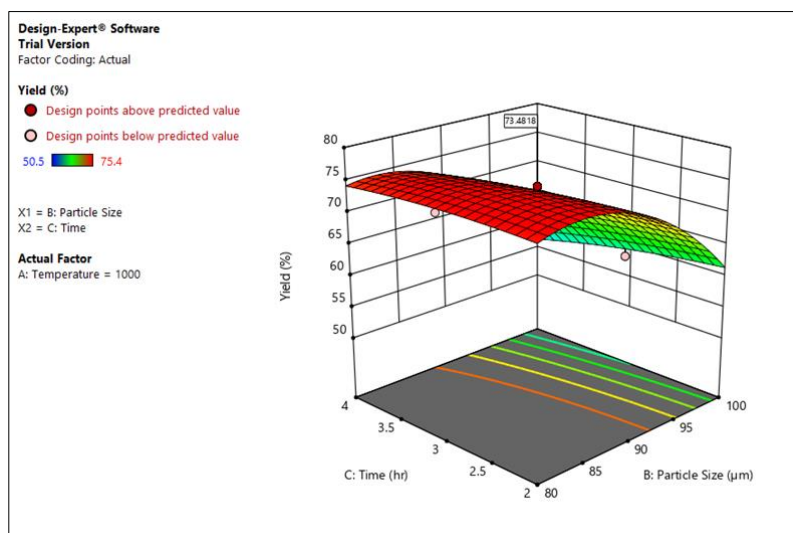


Figure 9 Effects of Particle Size and Time on Ashaka Quicklime Yield

3. Conclusion

Quicklime was successfully produced through the calcination of the Ashaka limestone. Quadratic model adequately described the relationship between quicklime yield and calcination factors of temperature, particle size and time. Optimum yield of 73.48% was obtained at optimal operating conditions; temperature of 1000 °C, particle size of 90 µm and time of 3 hrs.

Compliance with ethical standards

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Disclosure of conflict of interest

There is no conflict of interest of any kind.

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