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Effects of calcination variables on quicklime yield of Nkalagu limestone

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Abstract

Calcination of Nkalagu limestone for the production of agricultural quicklime is presented. It entails improving the quality of limestone through calcination process. Appropriate scientific instruments/techniques (x-ray diffractometer and scanning electron microscopy) were used for the characterization of the uncalcined and calcined limestone samples. Effects of calcination variables on the quicklime yield were examined. Central composite design of design expert software was used to optimize the calcination process. Analyses of the results revealed that calcite was the major limestone's mineralogical composition. Quadratic model adequately described the relationship between quicklime yield and calcination factors of temperature, particle size and time. Quadratic model adequately described the relationship between quicklime yield and calcination factors of temperature, particle size and time. The optimum yield of 74.00% was obtained at optima operating conditions; temperature of 937.41 ^{0}C, particle size of 85.99μm and time of 3.7 hrs. Characteristics of the quicklime showed that the calcination improved the quality of the sample in terms of mineralogical properties. It is recommended that the generated model should be used to develop chemical plant/equipment for limestone calcination process.

Keywords: Quicklime; Calcination; Calcite; Limestone; Quadratic model

1. Introduction

Limestone is a sedimentary rock composed primarily of Calcium Carbonate ($CaCO_3$) [1]. It is a valuable industrial raw material. The industries that use quicklime include building, agriculture, water treatment, sugar refining, tannery, paper and glass. The bulk demand for quicklime in Nigeria is for water treatment, soft drink bottling, tannery, breweries, soil amelioration and food processing. Limestone has been reported as the principal raw material for cement manufacture [2]. Nigeria is blessed with abundance of limestone deposits, spread throughout the States of the country. According to previous report [3], limestones and their varieties represent the most frequently used rocks in industry and are included among the thirty most important raw materials. Nigeria is in high need of quicklime due to its wide range of uses. Application of limestone can be enhanced through adequate processing techniques. Unfortunately, Nigerian limestone deposits are yet untapped, especially for agricultural purposes [4, 5]. Calcination is one of the techniques used for processing limestone. Such technique encourages diversification of limestone usage.

Calcination is useful for the modification of limestone [6]. Quicklime is produced by calcination of limestone (calcium carbonate or calcite, $CaCO_3$) in a kiln/furnace at a high temperature. There are numerous critical variables that exert profound effect on lime burning operations [7]. The factors and variables that affect the burning of limestones must be considered in the selection, design and optimization of the calcination 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 [7, 8]:

$$CaCO_3 \rightarrow CaO + CO_2 \ \Delta H = 182.1 \text{ kJ mol}^{-1} \quad (1)$$

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The forward reaction is favoured by higher temperatures. The reaction will proceed only if the partial pressure of CO_2 in the gas above the solid surface is less than the decomposition pressure of the $CaCO_3$. The chemical reactivity of limestone depends on the crystalline structure and the nature of impurities present. Suitability of a limestone deposit for the production of cement is largely dependent on chemical characteristics [9]. And there are several factors that affect the quality of quicklime. These factors include chemical composition of limestone, residence time and temperature, residence time, extent of CO_2 in the kiln, pressure acquired in kiln, rate of calcination, and fuel quality [7, 10]. Although Nigeria is blessed with huge deposits of limestone, there is no serious effort to process it for agricultural purposes. Thus, the aim of this study is to determine the effects of calcination variables on the quicklime yield of Nkalagu limestone.

2. Material and methods

2.1. Limestone Preparation and Classification

The limestone sample was collected from Nkalagu, Ebonyi State. Method used by previous author [11] was employed in the sample preparation and classification. It was washed to remove impurities associated with the limestone crystals. It was gradually sun dried at ambient atmospheric condition. 20000g of the sample was crushed in a hard surface. Crushed sample was classified and re-classified with the aid of the automatic vibrating sieves of 80µm, 90µm, 100µm, 300µm and 425µm arranged vertically such in descending order of magnitude and system set in vibration to the tune of amplitude 50m and for 10 minutes.

2.2. Determination of Mineralogical Composition

Mineralogical composition of finely ground limestone sample was determined by X-ray Diffractometer (XRD). The X-ray diffraction patterns were taken using Empyrean Pan Analytical. The sample was analyzed using reflection transmission spinner stage using the theta-theta (X-ray beams at certain angles of incidence) settings. Two-theta (2Θ) starting position was 4 degrees and ends at 75 degrees with a two–theta step of 0.026261 at 8.67 seconds per step. Tube current was 40 mA and the tension was 45VA. A programmable divergent ship was used with width mask. These procedures were also used in the determination of mineral content of the quicklime.

2.3. Surface Morphology of the Samples

Scanning electron microscope, (Phenom pro x-ray, phenomworld Emdhoven Netherlands) was used to study the surface morphology of the samples. The electron beam was scanned in a raster scan pattern, and the beam position was combined with the detected signal to produce an image.

2.4. Samples Calcination

Standard procedures for calcination of limestone were adopted in this experiment [7]. 10g of the limestone sample (80µm particle size) was weighed into pre-weighed empty crucibles plates. The pre-weighed crucible plate with the limestone was set to laboratory furnace and heated at various temperatures 800 ^{0}C-1000 ^{0}C. The first sample was removed after 1 hr of holding time, thereafter other samples at the time of 2 , 3, 4 and 5 hours. After heating the calcined sample, it was allowed to cool for 5-15minutes. The calcined sample was transferred to desiccators. The weight of the quicklime produced was measured. The procedure was carried out at temperatures of 800, 850, 900, 9500 and 1000 ^{0}C and particle sizes of 90µm, 100µm, 300µm and 425µm.

Quicklime yield was determined after the calcinations process. This was calculated to find the percentage ratio of the limestone weight before and after calcination:

$$\boldsymbol{Y} = \frac{\boldsymbol{W_2}}{\boldsymbol{W_1}} \text{X} \frac{100}{1} \qquad (2)$$

Where W_1 = Weight of limestone before calcination, W_2 = Weight of limestone after calcination, Y = percentage yield.

The Calcination process was carried out using one-factor at-a-time and response surface methodology. Central composite design (CCD) tool of Design Expert Software 11 was used to design the experiment. Temperature, particle size and time were the considered factors of the calcination process, while percentage yield was considered as the response.

3. Results and discussion

3.1. XRF results of the uncalcined and calcined limestone samples

The chemical compositions of the limestone sample were obtained through the XRF analysis. The analyte concentrations of the uncalcined and calcined limestones are shown in Tables 1 and 2 respectively. Predominant constituents of the uncalcined limestone include CaO, SiO_2, Al_2SO_3 and SrO. There were variations in the oxide compositions when compared with the calcined limestone. The variation of the chemical compositions of calciend and uncalcined samples is an affirmation that calcination alters the composition of limestone [7, 10].

Table 1 Chemical compositions of the uncalcined limestone

Oxides of the limestone	Compositions (%)
Fe_2O_3	0.9803
SiO_2	24.917
Al_2O_3	4.435
MgO	1.03
P_2O_5	0.2727
SO_3	1.4316
TiO_2	0.0930
MnO	0.04838
CaO	50.008
K_2O	1.2850
CuO	0.000057
ZnO	0.00447
Cr_2O_3	0.00157
V_2O_5	0.00169
PbO	0.00286
Rb_2O	0.00262
Ga_2O_3	0.000585
Cl	0.202
ZrO_2	0.0100
BaO	0.0600
Ta_2O_5	0.0017
WO_3	0.0200
SrO	2.263
CeO_2	0.00320
ThO_2	0.00058
Y_2O_3	0.002068
Nb_2O_5	0.001913

Table 2 Chemical compositions of the quicklime (calcined limestone)

Oxide of the limestone	Compositions (%)
Al_2O_3	10.7382
SiO_2	34.4248
K_2O	0.6107
CaO	51.9364
TiO_2	0.0360
MnO	0.0032
Fe_2O_3	0.4614
ZnO	0.0190
SrO	0.1534

3.2. XRD results of the uncalcined and calcined limestone

The mineralogical compositions of the uncalcined and calcined limestones, as determined by XRD, are shown in Figures 1 and 2 respectively. Nkalagu limestone is predominantly made of calcite. The compositions of the limestone can affect the quicklime yield [9]. The observed calcite-type of limestone is an indication that Nkalagu limestone can be used for agricultural, building and pharmaceutical purposes [10, 12, 13]. The calcination process enhanced the quality of the limestone. Such quality improvement can ensure versatile applications of the quicklime, which include capacity to ameliorate soil acidity [10, 12, 13].

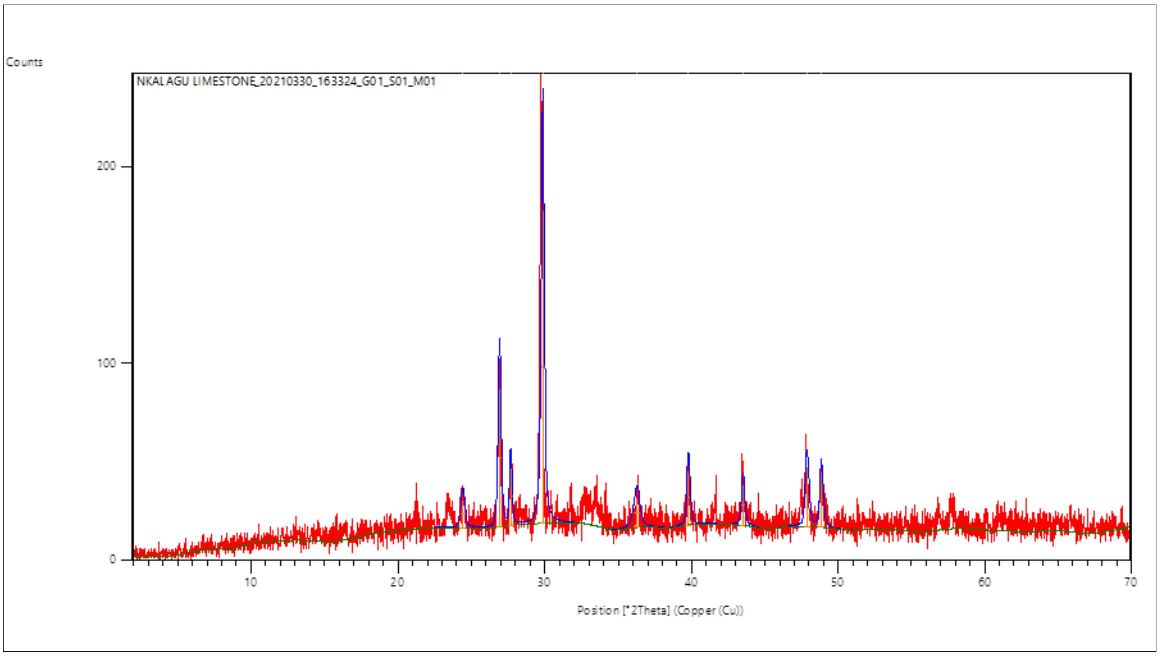

Figure 1 Spectrum of the uncalcined limestone

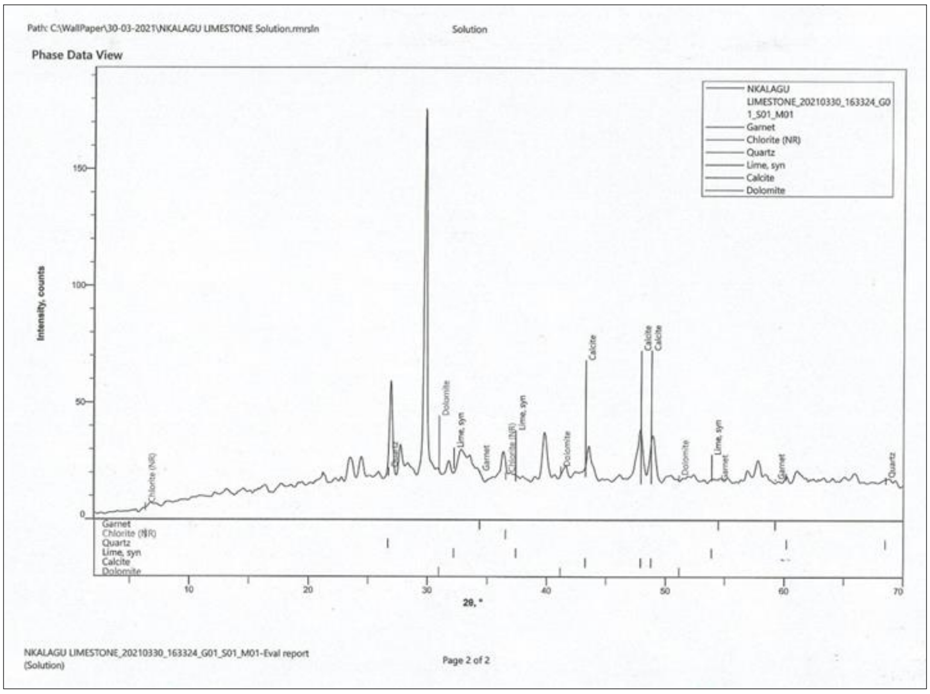

Figure 2 Spectrum of the calcined limestone

3.3. SEM results of the uncalcined and calcined limestone

The surface morphologies of the uncalcined and calcined limestones as revealed by scanning electron microscopic analyses are presented in Figure 3 and 4 respectively. In Figure 3, micrograph showed that the particles are packed together in powdered form with visible pores that will allow passage of fluids. The presence of such pores is an indication that there will be effective removal of CO_2 during the calcination process, and subsequent improvement of the CaO. In Figure 4, the surface morphology of the uncalcined sample showed that calcination altered the structural properties of the quicklime. The micrograph revealed that the particles are packed together in powdered form with visible pores. Relatively, the pores of the sample were enhanced by the calcination process [14, 15]. Also, the surface morphology (showing visible pores), indicate that Nkalagu quicklime has good hydration properties.

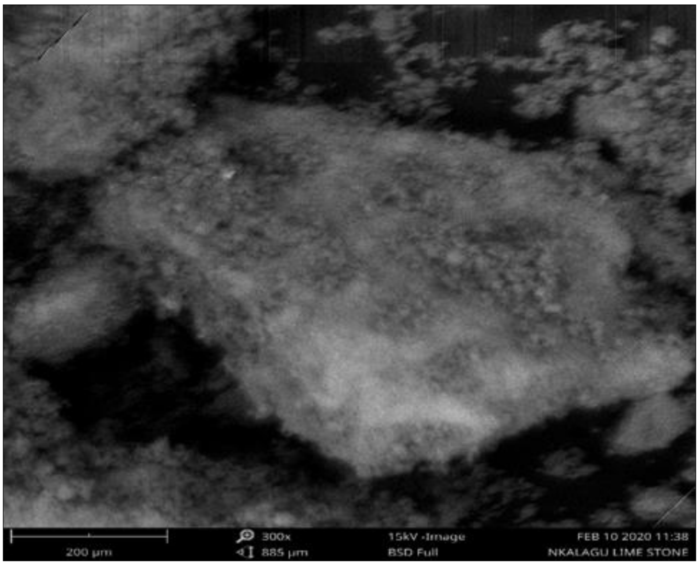

Figure 3 SEM result of the uncalcined limestone (quicklime)

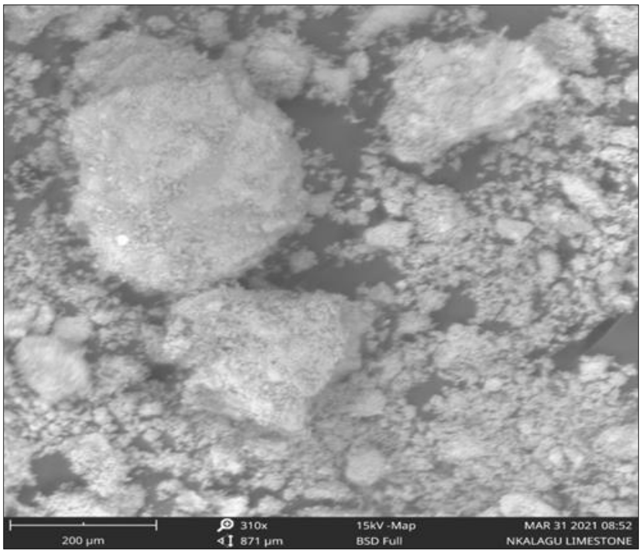

Figure 4 SEM result of the calcined limestone (quicklime)

3.4. Effects of Process Variables on the Quicklime Yield

Effects of temperature on quicklime yield are displayed in Figures 5 – 9. For a given particle size, the graphs showed the 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 report [11]. Additional analysis is necessary to determine effects of interactions of the calcination variables on the quicklime yields. Such analysis is presented in the response surface methodology result.

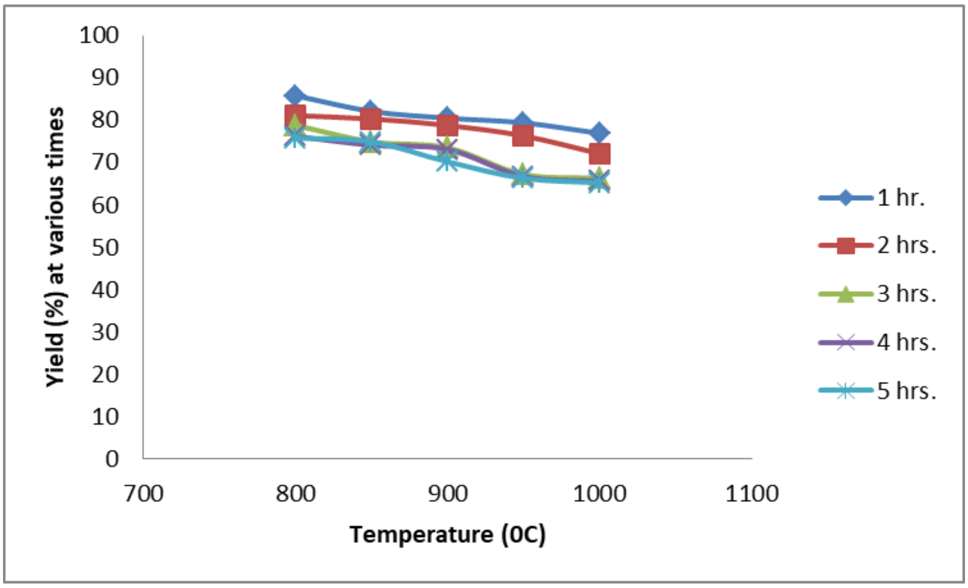

Figure 5 Effects of Temperature on the Calcination of 80 µm Limestone

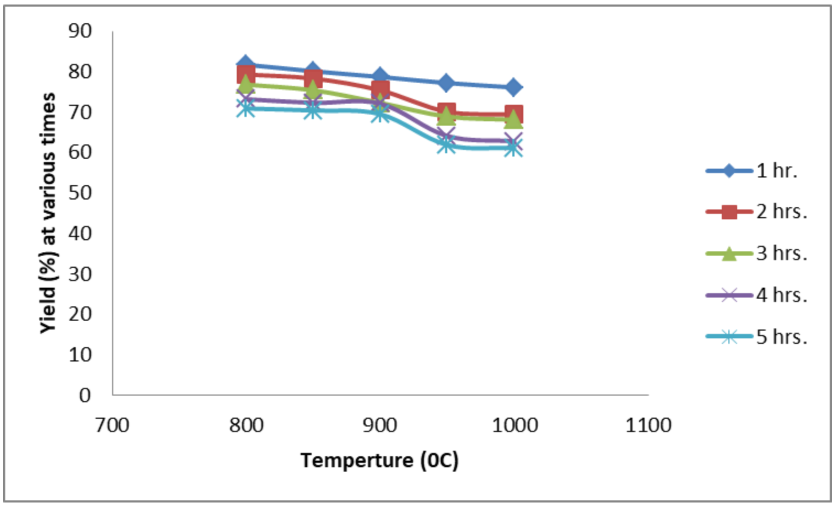

Figure 6 Effects of Temperature on the Calcination of 90 μm Limestone

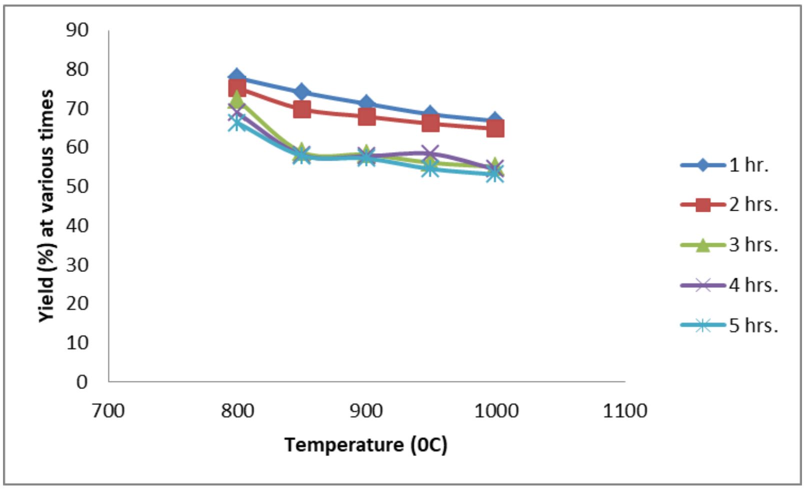

Figure 7 Effects of Temperature on the Calcination of 100 μm Limestone

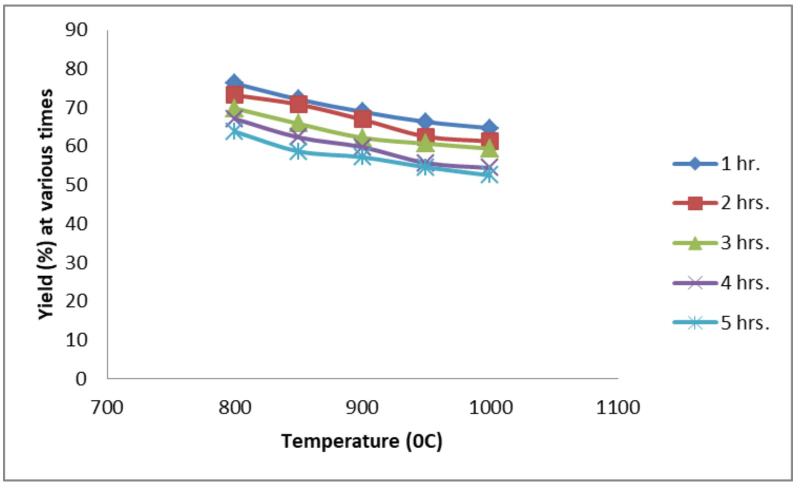

Figure 8 Effects of Temperature on the Calcination of 300 μm Limestone

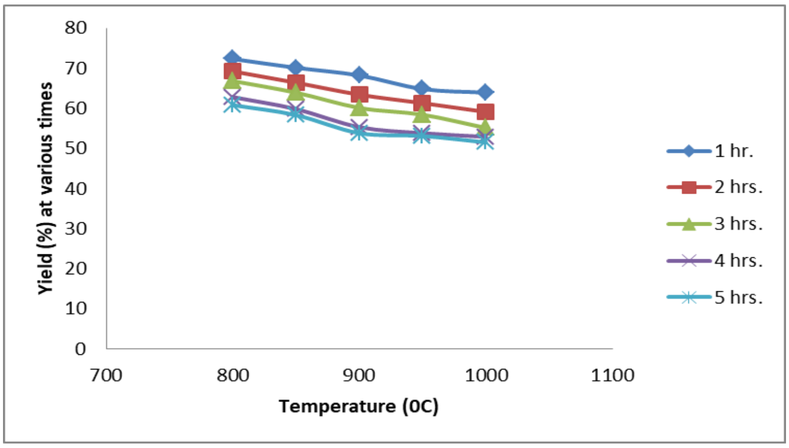

Figure 9 Effects of Temperature on the Calcination of 425 μm Limestone

3.5. RSM Result of the Calcination Process

Experimental data of the calcination process based on response surface methodology are presented in Table 3. The result showed the effects of the interactions among the factors of temperature, particle size and time on the percentage yield of the quicklime. The pattern of the data revealed that the peak of the quicklime yield is around the mid-points of the calcination variables. This is an indication that the relationship between yield and the considered factors is parabolic in nature.

Table 3 RSM result of the Calcination Process

Std	Run	Factor 1 A: Temperature (^{0}C)	Factor 2 B: Particle Size (μm)	Factor 3 C: Time (hr)	Response Yield (%)
9	1	900	90	4	71.3
13	2	950	90	3	71.8
4	3	1000	100	3	55.6
8	4	1000	100	5	54.3
12	5	950	100	4	57.3
18	6	950	90	4	71.5
14	7	950	90	5	68.9
20	8	950	90	4	71.5
3	9	900	100	3	58.3
11	10	950	80	4	72.9
15	11	950	90	4	71.5
10	12	1000	90	4	62.6
19	13	950	90	4	71.5
5	14	900	80	5	74.4
1	15	900	80	3	74.4
16	16	950	90	4	71.5
7	17	900	100	5	55.4
17	18	950	90	4	71.5
6	19	1000	80	5	65.5
2	20	1000	80	3	66.8

3.5.1. Analysis of Variance of the RSM results

The ANOVA of the experimental data is presented in Table 4. The model F-value of 73.77 implies the model is significant. There is only a 0.01% chance that an F-value as large as 73.77 could occur due to noise. P-values less than 0.0500 indicate model terms are significant. In this case A, B, C, AB, A^2, B^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model. The Predicted R^2 of 0.8901 is in reasonable agreement with the adjusted R^2 of 0.9718; 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 26.633 indicates an adequate signal. This model can be used to navigate the design space.

Table 4 ANOVA for the quicklime yield

Source	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	914.33	9	101.59	73.77	< 0.0001	significant
A-Temperature	84.10	1	84.10	61.07	< 0.0001	
B-Particle size	534.36	1	534.36	388.02	< 0.0001	
C-Time	7.06	1	7.06	5.12	0.0471	
AB	20.16	1	20.16	14.64	0.0033	
AC	0.0113	1	0.0113	0.0082	0.9298	
BC	1.05	1	1.05	0.7634	0.4028	
A^2	28.32	1	28.32	20.56	0.0011	
B^2	70.38	1	70.38	51.11	< 0.0001	
C^2	0.1002	1	0.1002	0.0728	0.7928	
Residual	13.77	10	1.38			
Lack of Fit	13.77	5	2.75			
Pure Error	0.0000	5	0.0000			
Cor Total	928.10	19				
Std. Dev.	1.17		R^2		0.9852	
Mean	66.92		Adjusted R^2		0.9718	
C.V. %	1.75		Predicted R^2		0.8901	
			Adequate precision		26.6328	

3.5.2. Mathematical model

Mathematical model in terms of coded factors of the significant and non-significant terms is expressed in Equation 3. Considering only the significant terms, the model was reduced to the expression of Equation 4. It is a quadratic model because the highest power of the variables is 2. The model 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.

$$\text{Yield} = +\,70.96 - 2.90A - 7.31B - 0.8400C + 1.59AB + 0.0375AC - 0.3625BC - 3.21A^2 - 5.06B^2 + 0.1909C^2 \quad (3)$$

$$\text{Yield} = +\,70.96 - 2.90A - 7.31B - 0.8400C + 1.59AB - 3.21A^2 - 5.06B^2 \quad (4)$$

3.5.3. Graphical analysis of the experimental results

Graphical representations of the quicklime yield are presented in Figures (10 – 13). Plot of predicted versus actual yield was used to test the performance of the model. Predicted versus actual plot gave linear graphs (Figure 10).

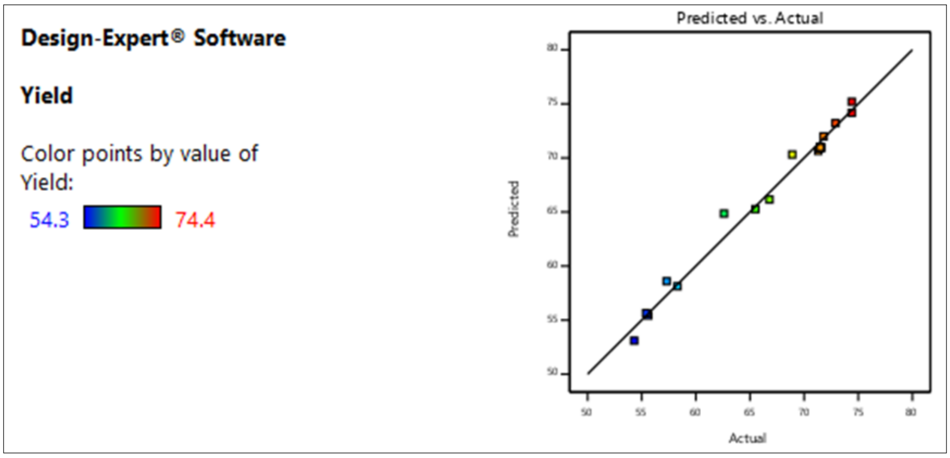

Figure 10 Predicted versus Actual Yield of Nkalagu Quicklime

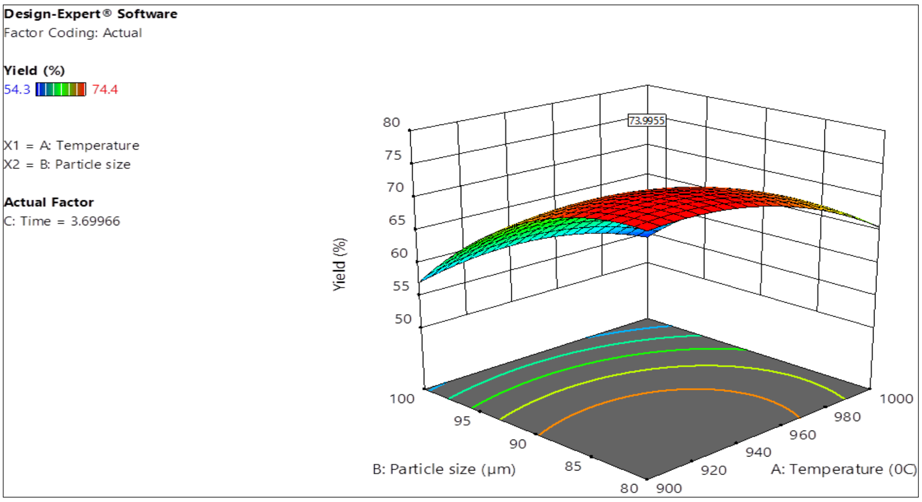

Figure 11 Effects of Temperature and Particle Size on Nkalagu Quicklime Yield

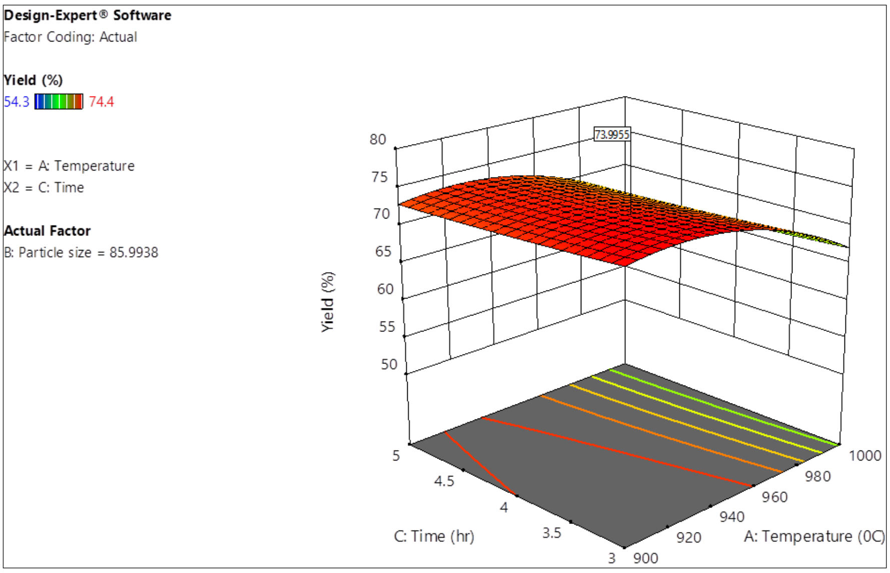

Figure 12 Effects of Temperature and Time on Nkalagu Quicklime Yield

The points clustered along the line of best fit, which showed that the model can be used to adequately predict the experimental data. 3-D surface plots of Figures (11 - 13) showed the relationship between the factors and response of the designed experiment. Optimum quicklime yield and corresponding optimal values of temperature, particle size and time were revealed. Optimum yield of 74.00% was obtained at optima operating conditions; temperature of 937.41 ^{0}C, particle size of 85.99µm and time of 3.7 hrs.

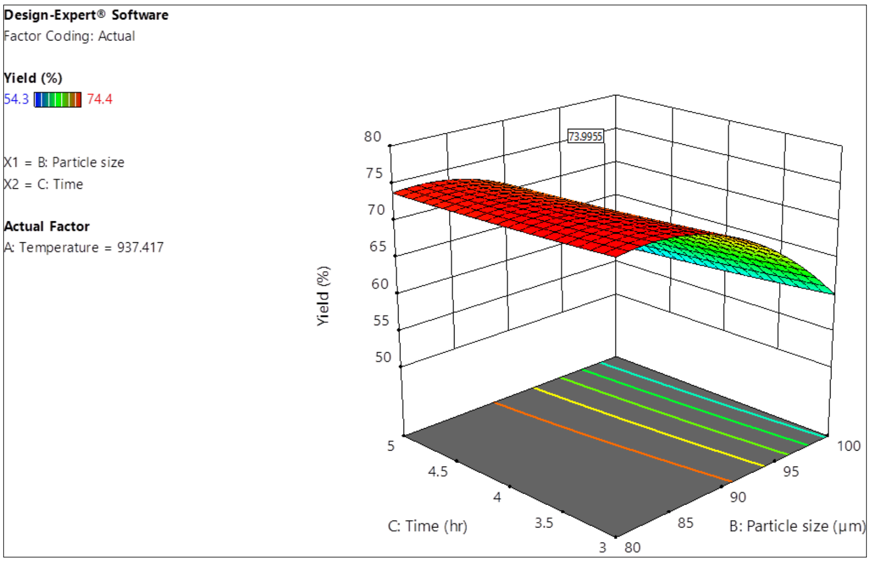

Figure 13 Effects of Particle Size and Time on Nkalagu Quicklime Yield

3.5.4. Validation of the results of the calcination process

Data for the validation of the result are presented in Table 5. The experimental result was validated by the determination of percentage deviation of experimental yield from the predicted yield. Percentage deviation is less than 5%, indicating that the model is adequate for the description of the calcination process.

Table 5 Validation of the Result of the Calcination Process

Limestone Sample	Temperature (°C)	Particle Size (µm)	Time (min.)	Experimental Yield (%)	Predicted Yield (%)	Percentage Deviation (%)
Nkalagu	937.41	85.99	3.7	72.92	74.00	1.48

4. Conclusion

From the analyses of the experimental results, the following conclusions can be drawn:

The XRD and SEM analyses revealed the mineralogical and morphological characteristics of the Nkalagu limestone. The XRD analysis revealed calcite as the major mineral of Nkalagu limestone.

Nkalagu quicklime was successfully produced through the calcination. The quicklime yield is temperature, particle size and time dependent.

Quadratic model adequately described the relationship between quicklime yield and calcination factors of temperature, particle size and time. Optimum yield of 74.00% was obtained at optima operating conditions; temperature of 937.41 ^{0}C, particle size of 85.99µm and time of 3.7 hrs.

Characteristics of the quicklime showed that the calcination enhanced the quality of the sample in terms of mineralogical properties.

It is recommended that the generated quadratic model should be used to develop chemical plant/equipment for limestone calcination process.

Compliance with ethical standards

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Disclosure of conflict of interest

There is no conflict of interest.

References

[1] Ofulume AB, Ibe KK, Ibeneme SI, Dioha EC, Chinemelu ES, Eluwa JC, Onyeise UO. The Petrography, Geochemistry and Potential Applications of Ndi-Uduma Ukwu/Ohafia-Ifigh Limestone, Ohafia, S.E. Nigeria. Journal of Geosciences and Geomatics. 2018; 6(1): 21-26.

[2] Onwe MR, Omonona VO, Ema MA. Exploring and Reserve Estimation for Industrial Mineral Potential in Parts of Calabar Area (Ewen/Iwuru/Agbangana Axis) Southern Nigeria. Journal of Geology & Geophysics. 2017; 6(6): 1-10.

[3] Leontakianakos G, Baziotis I, Ekonomou G, Delagrammatikas G, Galbenis CT, Tsimas S. A Case Study of different Limestones during Quick Lime and Slaked-Lime Production. Bulletin of the Geological Society of Greece. 2010; 43: 2485-2491.

[4] Ujoh F, Alhassan MM, Ujoh FT. Multi-temporal change detection at a limestone mining and cement production facility in Central Nigeria, American Journal of Environmental Protection. 2014; 3(3): 113-121.

[5] Osazuwa KO, Bamidele JF, Oyanoghafo OO. Vegetation Assessment of Okigwe Limestone Quarry Site at Okpilla in Etsako East Local Government Area, Edo State. Jordan Journal of Biological Sciences. 2016; 9(3): 195 – 203.

[6] Jiangyin L, Zhen Z, Chunming X, Aijun D, Pu Z. Effects of Calcination Temperature on the Acidity and Catalytic Performances of HZSM-5 Zeolite Catalysts for the Catalytic Cracking of n-Butane. Journal of Natural Gas Chemistry. 2005; 14: 213–220.

[7] Okonkwo PC, Adefila SS. Investigation of some factors that affect Jakura limestone burning African Journal of Pure and Applied Chemistry. 2013; 7(8): 280-290.

[8] Kilic O. Application of Ultrasonic Velocity Measurement and Thermal Analysis for Determination of Limestone Quality. Physicochem. Probl. Miner. Process. 2014; 50(2): 525–533.

[9] Bolarinwa AT, Idakwo SO. Evaluation of Albian Limestone Exposed at Dangote Cement Quarry, Tse-kucha Near Yandev, North Central Nigeria: A Geochemical Approach. International Journal of Science and Technology. 2013; 2(12): 847-856.

[10] Penuel BL, Maitera ON, Khan ME, Kefas WS. Quality assessment of limestone deposit found in Demsa local government area of Adamawa state, Nigeria. Direct Research Journal of Chemistry and Material Science (DRCMS). 2015; 3(2): 11-16.

[11] Suleiman IE, Abubakar A, Otuoze HS, Suleiman MA, Momoh RO and Aliyu SN. Effects of Particle Size Distribution on the Burn Ability of Limestone, Leonardo Electronic Journal of Practices and Technologies. 2013; 23: 115-130.

[12] Guemmadi Z, Resheidat M, Chabil H, Toumi B. Modeling the Influence of Limestone Filler on Concrete: A Novel Approach for Strength and Cost, Jordan Journal of Civil Engineering. 2009; 3(2): 158-171.

[13] Akinniyi AA, Ola SA. Investigation of Certain Engineering Properties of Some Nigerian Limestone Deposits for Cement Production, Electronic Journal of Geotechnical Engineering. 2016; 21:10471-10482.

[14] Maina P. Improvement of Lime Reactivity towards Desulfurization by Hydration Agents. Chem Sci Trans. 2013; 2(1): 147-159.

[15] Nurfatirah N, Zainab H, Othman H, Farizul HK, Rozaini A. Effect of Temperature in Calcination Process of Seashells. Malaysian Journal of Analytical Sciences. 2015; 19(1): 65–70.