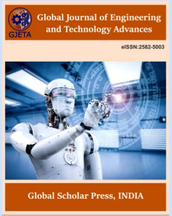
Global Journal of Engineering and Technology Advances
eISSN: 2582-5003
Cross Ref DOI: 10.30574/gjeta
Journal homepage: https://gjeta.com/



(RESEARCH ARTICLE)

Effects of process variables on the reactivity of slaked lime produced from Shuk quicklime

Benson Chinweuba Udeh *

Department of Chemical Engineering, Enugu State University of Science and Technology, PMB 01660, Enugu, Nigeria.

Global Journal of Engineering and Technology Advances, 2021, 09(03), 068–076

Publication history: Received on 08 November 2021; revised on 18 December 2021; accepted on 20 December 2021

Article DOI: https://doi.org/10.30574/gjeta.2021.9.3.0161

Abstract

This is a research report of the effects of process variables on the reactivity of slaked lime produced from Shuk quicklime. It involved the calcination (at temperature of 1000 ^{0}C, particle size of 90 µm and time of 3 hrs) of Shuk limestone and subsequent slaking of its quicklime. The quicklime was characterized by x-ray diffractometer (XRD) and scanning electron microscopy (SEM) respectively to determine its mineral content and surface morphology respectively. Effects of process variables (quicklime/water ratio, particle size and time) on the reactivity of the slaked lime were determined. The reactivity was optimized using response surface methodology (RSM). The XRD analysis revealed calcite as the type mineral of the Shuk quicklime. The surface morphology of the quicklime sample showed that the particles are packed together in powdered form with visible pores that will allow passage of water. Reactivity of the lime was influenced by the quicklime/water ratio, particle size and time. Quadratic model appropriately explained the relationship between reactivity and considered slaking factors of quicklime/water ratio, particle size and time. The optimum reactivity value of the slaked lime was obtained as 59.3 ^{o}C at quicklime/water ratio of 0.24 g/ml, particle size of 88.2 µm and time of 15.1 minutes.

Keywords: Reactivity; Slaked lime; Shuk quicklime; Slaking

1. Introduction

Shuk limestone deposit is at Sokoto State, North-West geopolitical zone of Nigeria. Application of the limestone has not been fully diversified. For preliminary study, information on the quantity and quality of limestone is required for the establishment of limestone factory. This precondition will inform other secondary complimentary geological surveillance for earth resource based industries [1]. Activities that attract establishment of limestone factories include extraction and processing of the limestone. Extraction mostly referred to as quarrying consists of removing blocks or pieces of stone from an identified and unearthed deposit. Differences in the particular quarrying techniques used often stems from variations in the physical properties (such as density, fracturing/bedding planes, financial factors, and the site owner's disposition) of the deposit itself. Mining and quarrying activities of limestone are detrimental to the environment [2, 3]. As such, proper management of mining and quarrying activities is needed for the safety of the environment. The general procedures of processing limestone begin with initial cutting, followed by application of a finish, and then a second cutting / shaping step. Generally, limestone extraction process involves the following stages; locating the stone, removing the stone using heavy machinery, securing the stone on a vehicle for transport, and moving the material to storage.

According to previous researchers [4], a typical approach to obtaining the complete process model involves the considerations of the transport phenomena, calcination and reactor models. The individual models constitute layer models which are usually combined to give the complete model. It can then be simulated over ranges of conditions

---

* Corresponding author: Benson Chinweuba Udeh
Department of Chemical Engineering Enugu State University of Science and Technology, PMB 01660, Enugu, Nigeria.

obtainable in typical operations. In addition, the layer models are simulated in order to define the utility and their effect on the overall model. Effective extraction of the limestone requires the application of appropriate scientific principles. According to previous report [5], penetration rate of drilling limestone depends on the physical and mechanical properties of the limestone. Similarly, limestone processing techniques (such as calcination and slaking) depend of the physic-chemical properties of the limestone. Hydration rate of calcium oxide for a suspended single pellet into an atmosphere of air with a controlled humidity was examined by previous researchers [6]. It was observed that the hydration reaction takes place on a sharp, well defined interface between the product layer and the unreacted core.

Lime is a material produced from the heating of limestone and its subsequent slaking with water. It can be combined with aggregate and water to produce a mortar or plaster, or diluted with water and used for lime washing. Lime was commonly used as the binding agent in the historic mortars of traditionally constructed buildings and structures until the beginning of the 20th century, when its use was largely superseded by Portland cement. It is produced by burning limestone (calcium carbonate or calcite, $CaCO_3$) in a kiln at temperatures in excess of 850°C. This drives off the carbon dioxide held within the lime to produce calcium oxide (CaO). The CaO is a highly reactive solid known as quicklime or lump lime. Slaking the calcium oxide with water is a highly exothermic reaction. It produces calcium hydroxide (slaked lime, hydrated lime or Portlanclite). Daud et al (2015) reported the rate of reactivity of quicklime for the production of optimum quality of hydrated lime. In the report, hydrated lime (calcium hydroxide, $Ca(OH)_2$) was prepared by digesting calcium oxide (CaO) in distilled water. Though Nigeria is blessed with vast limestone deposits (including Shuk limestone), there appear to be lack of technical know-how on the limestone processing techniques. For the limestone deposits to be developed, adequate and relevant data on the characteristics, extraction and processing of the limestone are needed. Thus, this study is aimed at determining the effects of process variables on the reactivity of slaked lime produced from shuk quicklime. Calcination of the limestone and subsequent slaking of the quicklime will engender diversification of the uses of shuk quicklime.

2. Material and methods

The raw materials used for this study are Shuk limestone, and distilled water. Equipment/instruments used for this study include; furnace, crucibles, tongs, automatic vibrating sieve, thermometer, weighing balance, grinder, beakers, stop watch, scanning electron microscopy (SEM) and x- ray diffractometer (XRD).

2.1. Limestone Preparation

Shuk limestone sample was collected from Sokoto State, Nigeria. Method used by previous study [7] was adopted in the sample preparation. The sample was washed to remove impurities. It was gradually sun dried at ambient atmospheric condition. 3000g of the sample was crushed, and ground into powdered form. Crushed sample was classified with the aid of the automatic vibrating sieves, and 90μm particle size was obtained for the experiment.

2.2. Production of Slaked Lime from Shuk Quicklime

The quicklime (calcium oxide) was obtained from Shuk limestone by calcination at temperature of 1000 ^{0}C, particle size of 90 μm and time of 3 hrs. Then, the slaked lime ($Ca(OH)_2$) was produced by hydration/slaking of the quicklime. Method used by previous authors [8] was adopted in the slaking process (digesting calcium oxide (CaO) in distilled water). During the hydration process, temperature changes were measured and recorded, as the reactivity of the slaked lime.

2.3. Determination of Mineralogical Composition

Mineralogical composition of each limestone sample was determined by X-ray Diffractometer (XRD). This study adopted a method used by previous works [9, 10], with slight modification. The XRD analysis was performed on finely ground sample of the limestone. The X-ray diffraction pattern was taken using Empyrean Pan Analytical. The powdered samples of each limestone were prepared by preparation block and compressed in the flat sample holder to create a flat smooth surface that was later mounted on the sample stage in the XRD cabinet. The sample was analyzed using reflection transmission spinner stage using the theta-theta (X-ray beams at certain angles of incidence) settings. Two-theta (2Ɵ) 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) to determine the mineral content of the quicklime.

2.4. Determination of Surface Morphology of the Sample

Scanning electron microscope, (Phenom Pro X-ray, phenom world Emdhoven Netherlands) was used to study the surface morphology of the sample, in line with method used by previous researchers [10]. It is a type of electron microscope that produces images of a sample by scanning the surface with focused beam of electrons. The electrons interact with atoms in the sample producing various signals that contain information about the samples surface topography and composition. The electron beam was scanned in a raster scan pattern, and the beam position was combined with the detected signal to produce an image.

3. Results and discussion

3.1. Mineralogical Composition of the Quicklime

The mineralogical composition of Shuk quicklime as determined by XRD is shown in Figure 1. It is made up of pure calcite. The observed quality of the quicklime will ensure its versatile applications [12, 13, 14].

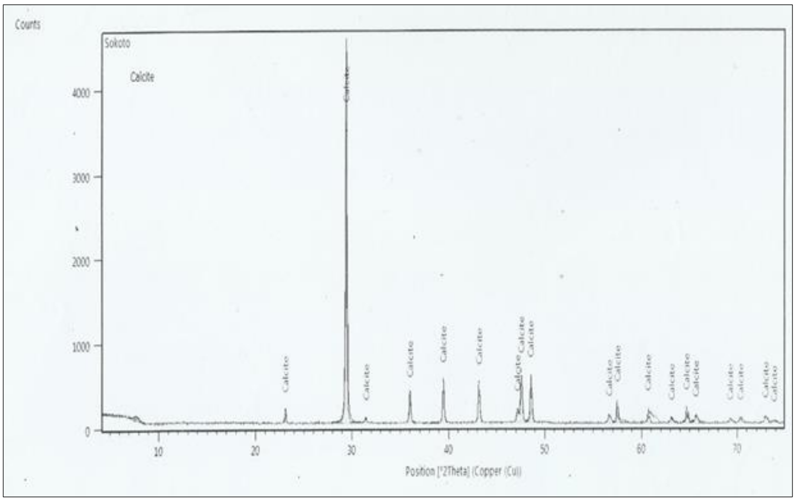


Figure 1 Mineralogical Compositions of Shuk Quicklime

3.2. SEM Analysis of the Quicklime

The scanning electron microscopic analysis of Shuk quicklime is shown in Figure 2. The scanning electron microscope (SEM) produced the image of the quicklime sample by scanning the surface with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that contain information about the surface topography and composition of the sample. The micrograph showed that the particles are packed together in powdered form with visible pores. The surface morphology (showing visible pores), indicates that the quicklime has good hydration properties [10, 11].

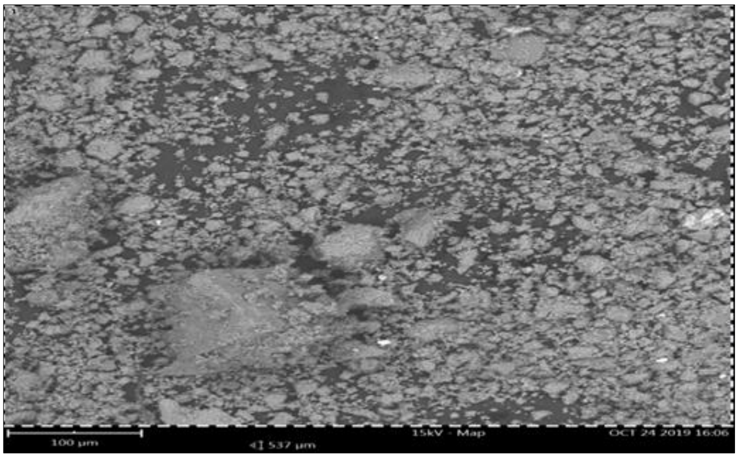


Figure 2 SEM Analysis of the Shuk Quicklime

3.3. Effects of the Process Variables on the Slaked Lime Yield of Shuk Quicklime

The effects of quicklime/water ratio, particle size and time on the reactivity of the Shuk slaked lime are presented in Figures 3, 4 and 5 respectively. At various temperatures, the graphs show the relationships between: the reactivity and quicklime/water ratio; reactivity and particle size; and relativity and time of the slaking. The reactivity increased with increase in quicklime/water ratio till it got to the peak from where decline in the reactivity was noticed. Similar trend was observed in the reactivity versus time graph. On the contrary, reactivity decreased with increase in particle size. These findings corroborate with previous reports [7, 8].

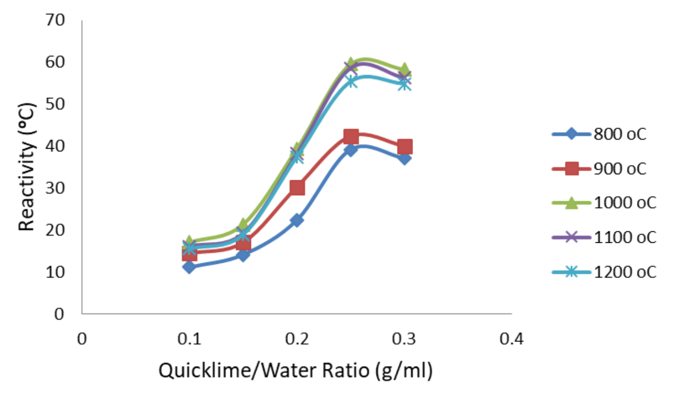


Figure 3 Reactivity of Shuk lime against Quicklime/Water ratio at various temperatures

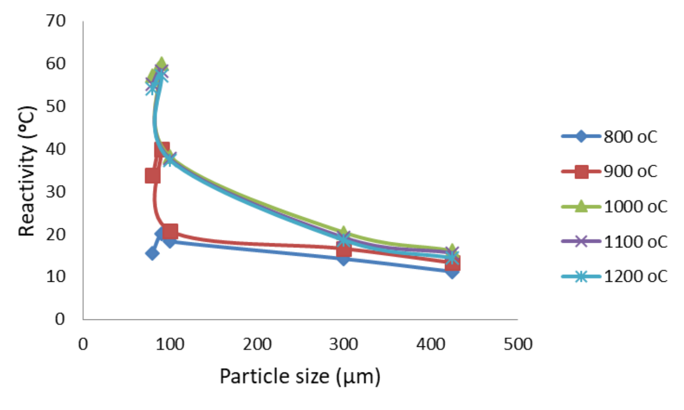


Figure 4 Reactivity of Shuk lime against Particle Size at various temperatures

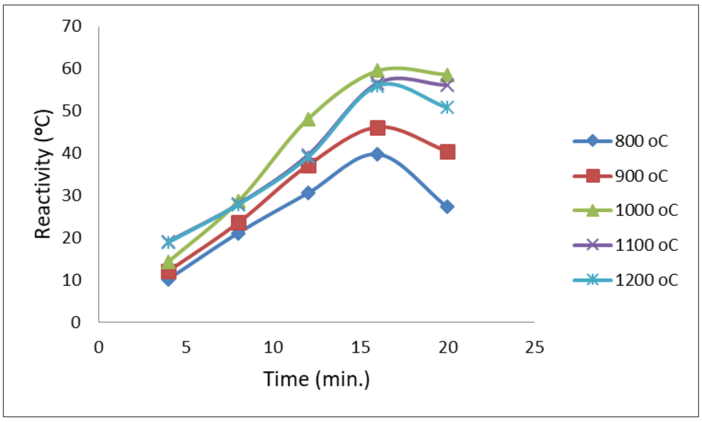


Figure 5 Reactivity of Shuk lime against time at various temperatures

3.4. RSM Results of the Slaking Process

The RSM result of the reactivity of Shuk slaked lime is presented in Tables 1. It showed the effects of the interactions among the factors of quicklime/water ratio, particle size and time on the reactivity of the quicklime. It is made up of reactivity data as function of the considered factors in a 20-run experiment. The maximum reactivity value was observed around the mid-points of the considered factors of quicklime/water ratio, particle size and time of the slaking process. This is an indication that graphical analysis of the data will be in parabolic form – quadratic model [15].

More so, it was revealed that minimum reactivity values were recorded at the extreme points of the considered factors. It showed that reactivity increases with increase in surface area (decrease in particle size). Lowest reactivity value was obtained as 14.4%. Minimum reactivity of the quicklime was obtained at lowest quicklime/water ratio, highest particle size and lowest time of slaking. This observation is in agreement with the previous findings [7, 8, 16].

Table 1 RSM Result of the Slaking of Shuk Quicklime

Std	Run	Factor 1 A: Quicklime/Water Ratio g/ml	Factor 2 B: Particle Size µm	Factor 3 C: Time min.	Response 1 Reactivity °C
14	1	0.25	90	20	55.7
16	2	0.25	90	16	59.5
13	3	0.25	90	12	52.0
1	4	0.2	80	12	26.1
12	5	0.25	100	16	57.1
4	6	0.3	100	12	28.6
3	7	0.2	100	12	14.4
2	8	0.3	80	12	29.5
15	9	0.25	90	16	59.5
20	10	0.25	90	16	59.5
8	11	0.3	100	20	31.3
10	12	0.3	90	16	48.7
9	13	0.2	90	16	40.2
18	14	0.25	90	16	59.5
5	15	0.2	80	20	31.3
6	16	0.3	80	20	33.2
7	17	0.2	100	20	17.6
17	18	0.25	90	16	59.5
19	19	0.25	90	16	59.5
11	20	0.25	80	16	55.2

3.5. Analysis of Variance (ANOVA) of Reactivity of the Slaked Lime

In Table 2, the model F-value of 77.60 implies the model is significant. There is only a 0.01% chance that an F-value this large 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, C^2 are significant model terms. The predicted R^2 of 0.9164 is in reasonable agreement with the Adjusted R^2 of 0.9732; the difference is less than 0.2. Adequate precision measures the signal to noise ratio [17]. A ratio greater than 4 is desirable. The ratio of 24.651 indicates an adequate signal. This model can be used to navigate the design space.

Table 2 ANOVA of Reactivity the Shuk Slaked Lime

Source	**Sum of Squares**	**df**	**Mean Square**	**F-value**	**p-value**	
Model	4688.77	9	520.97	77.60	< 0.0001	Significant
A-Quicklime/Water Ratio	173.89	1	173.89	25.90	0.0005	
B-Particle Size	69.17	1	69.17	10.30	0.0093	
C-Time	34.23	1	34.23	5.10	0.0475	
AB	63.85	1	63.85	9.51	0.0116	
AC	0.5000	1	0.5000	0.0745	0.7905	
BC	1.13	1	1.13	0.1676	0.6909	
A^2	921.41	1	921.41	137.24	< 0.0001	
B^2	119.96	1	119.96	17.87	0.0018	
C^2	218.05	1	218.05	32.48	0.0002	
Residual	67.14	10	6.71			
Lack of Fit	67.14	5	13.43			
Pure Error	0.0000	5	0.0000			
Cor Total	4755.91	19				
Std. Dev.	2.59		R^2			0.9859
Mean	43.90		Adjusted R^2			0.9732
C.V. %	5.90		Predicted R^2			0.9164
			Adeq Precision			24.6506

3.6. Mathematical Model of the Reactivity of the Slaked Lime

The mathematical model of the reactivity of Shuk slaked lime (in terms of significant terms) is expressed in Equation (1). The model can make adequate predictions about the response for given levels of each factor, and they are useful for identifying the relative impact of the factors by comparing the factor coefficients. As revealed by the analysis of variance, each model adequately described the relationship between the reactivity and the factors of quicklime/water ratio, particle size and time. Thus, the reactivity is a function of quicklime/water ratio, particle size and time. The positive signs in the model signified synergistic effect, while the negative signs signified antagonistic effect [17]. As such, there is a synergistic effect on the interaction of quicklime/water ratio and particle size. The model's highest power of at least one of the variables is two, which showed that the mathematical model is a quadratic equation.

$$\text{Reactivity} = +60.80 + 4.17A - 2.63B + 1.85C + 2.82AB - 18.30A^2 - 6.60B^2 - 8.90C^2 \quad (1)$$

3.7. Graphical Analysis of the Reactivity of the Slaked Lime

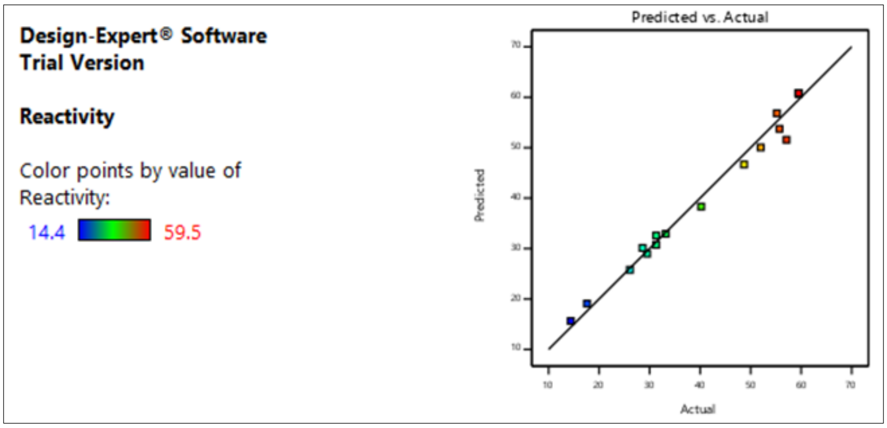


Figure 6 Predicted versus Actual Reactivity of Shuk Slaked Lime

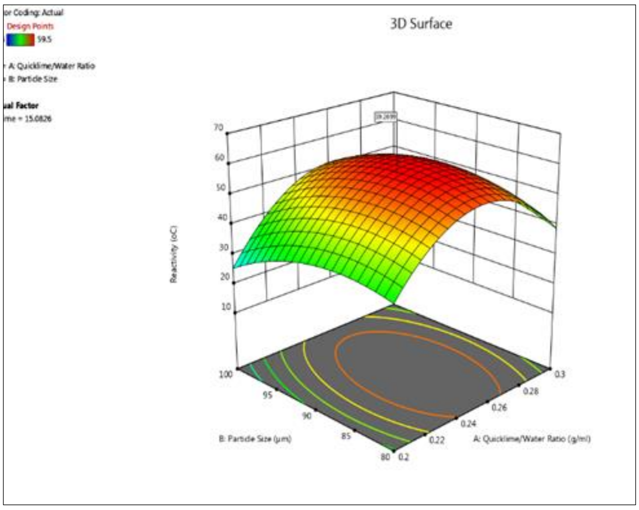


Figure 7 Reactivity versus Quicklime/Water Ratio and Particle Size for the Shuk Quicklime

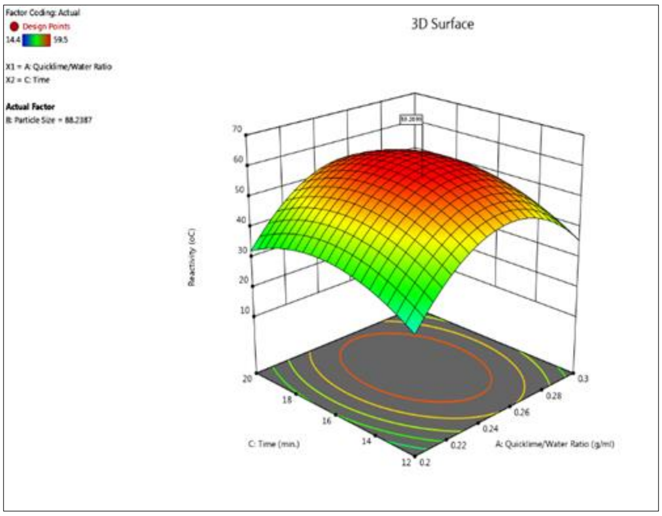


Figure 8 Reactivity versus Quicklime/Water Ratio and Time for the Shuk Quicklime

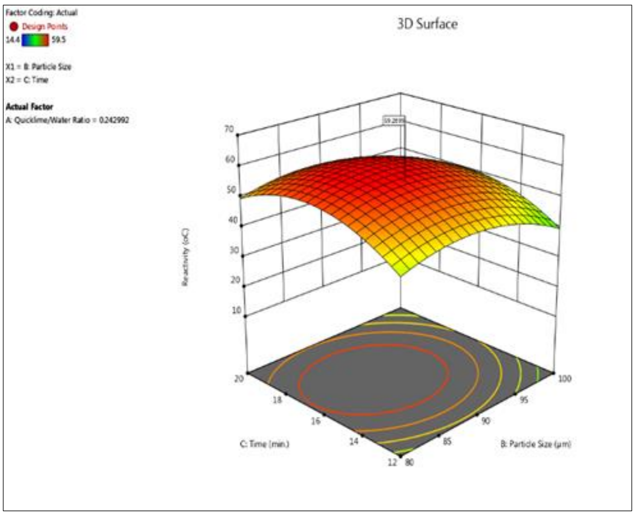


Figure 9 Reactivity versus Particle Size and Time for the Shuk Quicklime

Graphical representations of the reactivity of the slaked lime are presented in Figures (6 – 9). Plot of predicted versus actual yield was used to test the performance of the model. It revealed a linear graph, indicating that the model performed very well. 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 reactivity of 59.3 ^{0}C with the corresponding optimal factors of quicklime/water ratio, particle size and time.

3.8. Validation of the Result

Validation of the result is presented in Table 3. The result was validated by the determination of percentage deviation of experimental reactivity from the predicted reactivity. The percentage deviation (1.5%) is less than critical value of 5%. This is an affirmation that the generated model adequately described the slaking process [15].

Table 3 Validation of the Result

Quicklime/ Water Ratio (g/ml)	Particle Size (µm)	Time (min.)	Experimental Reactivity (^{o}C)	Predicted Reactivity (^{o}C)	Percentage Deviation (%)
0.24	88.2	15.1	60.2	59.3	1.5

4. Conclusion

The XRD analysis revealed calcite as the major mineral of the Shuk quicklime. The surface morphology of the quicklime sample showed that the particles are packed together in powdered form with visible pores that will allow passage of water.

Reactivity of the lime is quicklime/water ratio, particle size and time dependent. Quadratic model explained the relationship between reactivity and considered slaking factors of quicklime/water ratio, particle size and time. The optimum reactivity value of the Shuk, slaked lime was obtained as 59.3 ^{o}C at quicklime/water ratio of 0.24 g/ml, particle size of 88.2 µm and time of 15.1 minutes.

Compliance with ethical standards

Acknowledgments

I hereby acknowledged Enugu State University of Science and Technology, Nigeria, for providing suitable environment for this study.

Disclosure of conflict of interest

There is no conflict of interest.

References

[1] 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.

[2] 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.

[3] 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.

[4] Okonkwo PC, Adefila SS, Ahmed AS. Development of Process Simulation Model for Lime Production. International Journal of Engineering Research and Applications (IJERA). 2012; 2(2): 616-628.

[5] Okewale IA, Olaleye BM. Characterization of Some Selected Limestone Deposits in ogun State, Nigeria for Prediction of Penetration Rate of Drilling, IOSR Journal of Engineering (IOSRJEN). 2013; 3(7): 25-30.

[6] Maciel-Camacho A, Hernandez HR, Hills AWD, Morales RD. Hydration Kinetics of Lime, ISIJ International. 1997; 37 (5): 468-476.

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

[8] Daud MO, Noorliyana HA, Azmi R, Kamarudin H. Reactions of Limestone on Slaking Process under Different Conditions of Parameters. Material Science Forum. 2015; 819: 393-398.

[9] Ikhane PR, Folorunso AF, Nton ME. Oluwalaanu Evaluations of Turonian Limestone Formation Exposed at NIGERCEM-Quarry, Nkalagu, and Southeastern Nigeria: A Geochemical Approach. The Pacific Journal of Science. and Technology, 2009; 10 (2): 761-771.

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

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

[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] 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.

[14] 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.

[15] Onukwuli OD, Omotioma M. Optimization of the Inhibition Efficiency of Mango Extract as Corrosion Inhibitor of Mild Steel in 1.0M H_2SO_4 using Response Surface Methodology, *Journal of Chemical Technology and Metallurgy*. 2016; 51(3): 302 – 314.

[16] Mohammad R, Priscilla T, Elham D, Behdad M. Determination of carbonation/calcination reaction kinetics of a limestone sorbent in low CO2 partial pressures using TGA experiments. Energy Procedia. 2017; 114: 259 – 270. 13th International Conference on Greenhouse Gas Control Technologies, GHGT-13, Lausanne, Switzerland. November 2016; 14-18.

[17] Omotioma M, Onukwuli OD. Evaluation of Pawpaw Leaves Extract as Anti-Corrosion Agent for Aluminium in Hydrochloric Acid Medium, *Nigerian Journal of Technology (NIJOTECH)*. 2017; 36(2): 496-504.