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Assessment of the Combined Effect of Temperature and Time on Calcium and Magnesium Release in Carbonate Rocks, Central Luconia Province, Offshore Sarawak, Malaysia

Yassir Ali[1*], Padmanabhan E[2] and Andariamihaja S[2]

[1]Faculty of Earth Science, Red Sea University, Sudan
[2]Department of Geosciences, Faculty of Petroleum Engineering and Geosciences, University Petronas Technology, Malaysia

Abstract

Dissolution of carbonate rocks indicated by the release of calcium and magnesium ions was evaluated for a number of six samples of carbonate rocks representing three microfacies. Dissolution was measured progressive periods of time under different temperature ranges. The main objective of this paper was to evaluate the individual and collective effect of time and temperature on the dissolution trends for any facies. The method applied was the surface response method, in which, temperature, and time represented the factors, while amounts of calcium and magnesium released (in ppm) represented the responses. Dissolution was carried out experimentally under a constant pH of 0.1 m Hcl, continuous stirring. The resulting data in terms of concentration (ppm) of the Ca and Mg was used as input data for the model. Response surface method provided models for the dissolution of carbonates, optimization of dissolution, and individual-and-combined effect of each factor (Time and temp).

Keywords: Response surface method; Carbonate dissolution; Central Luconia province; Offshore sarawak

Introduction

Unlike siliciclastic rocks, carbonate rocks are much more susceptible to dissolution. Dissolution of carbonate rocks is significant in many aspects of geosciences, including the quality of reservoirs, carbon dioxide sequestration, reefs and platforms stability. Many factors including mineralogy, temperature and time influence the rate of carbonate dissolution. Response surface method (RSM) is a useful statistical technique which has been applied in research in complex variable [1,2].

The method is suitable for application when the output is influenced by several input factors [3]. The area is one of the most important gas provinces in the area [4]. The formation of these carbonate buildups started in the Miocene [5] and is following structural highs. Epting [4] described the architecture of the carbonate platforms in Central Luconia (Figure 1) as to be controlled by four major processes, namely: (i) the rate of skeletal carbonate production, (ii) subsidence, (iii) relative sea-level fluctuation and (iv) the supply of clastic material from the Borneo delta [6]. The sedimentation of these carbonates is suggested by [7] to be caused by turbidity currents sourcing from different platforms.

Most of the studies in the area were directed towards the genesis of these carbonate buildups, their genesis, architecture, and potentiality. Studies addressing the disintegration of these buildups mainly scoped to the physical weathering due to turbidity currents. There is a remarkable lackness in the studies regarding dissolution phenomenon in that area, and the response of different microfacies. Therefore, the aim of this paper is to predict short term response of three carbonate microfacies in those buildups as bases to understand long term impact of dissolution. The proposed method here, the response surface method is used to examine the relationship between one or more response variables and a set of quantitative experimental variables or factors [8].

Materials and Methods

Samples of limestone from different locations were selected for this study. The carbonate rocks are taken from the Central Luconia area

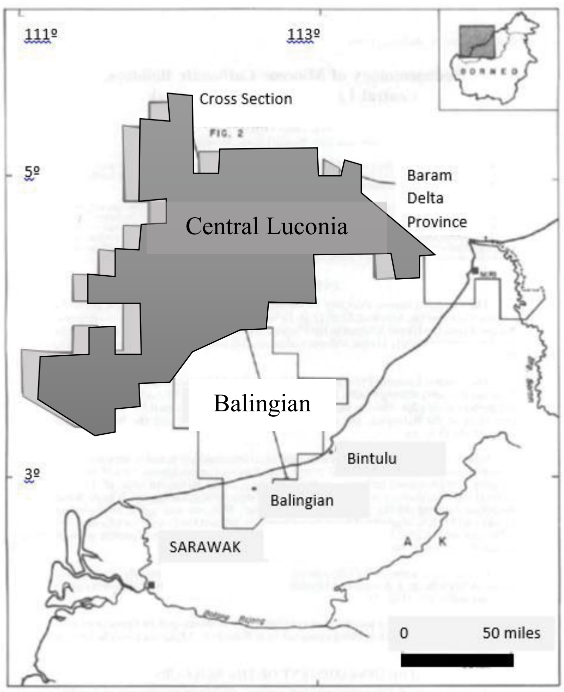

Figure 1: Location map of Central Luconia Province.

***Corresponding author:** Yassir Ali, Faculty of Earth Science, Red Sea University, Sudan, Tel: 00249000000; E-mail: yasirovic2009@gmail.com

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offshore Sarawak. A number of three facies identified based on their composition, texture and structure; these included: Moldic Wackestone, Mudstone, and, [9] Crystalline-Sparistone. Initially, dissolution was run for the different samples under three different temperatures, namely: 25, 50, and 75 C to evaluate the response of dissolution of different microfacies under different temperatures. The solvent used was diluted Hydrochloride acid (0.1), with pH level maintained constant. Dissolution process was carried out for a time period of 100 minutes. Dissolution rates for different samples were measured in terms of the release of calcium and magnesium iron (Ca^{+2} and Mg^{+2}), and is expressed in part per million (ppm). Concentrations were measured continuously at an interval of 10 minutes using inductive couple plasma (ICP) device.

In order to study the effect of time and temperature, data were transferred into Sigma Six, Minitab package for analysis. A response surface method (RSM) was selected under design of experiment (DOE) option. The method was best described, is an empirical modeling approach for determining the relationship between various operating variables and response variables. It provides a sequential experimentation strategy for building and optimizing an empirical model. Thus, RSM is a collection of mathematical and statistical procedures that are useful for the modeling and analysis of problems in which the response is affected by operating variables.

After parameters identified, model type was selected based on the items to be fit. The simplest one is that includes the effect of individual parameter, A B C D. Square model studies the combined effect for the factor A*B, A*C, B*C, etc. (Figure 2).

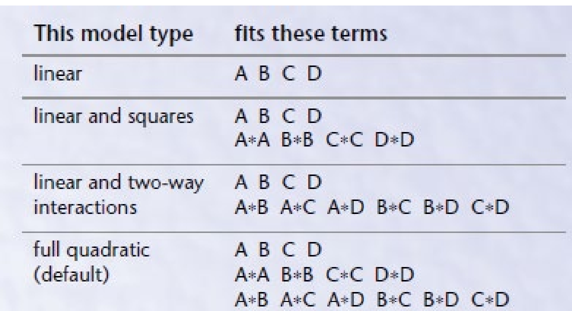
By setting a quadratic model, different relationships can be obtained, including single parameter (A) effect, two factors effect (A*B), and squared effect of the same factor (A*A). Different setting parameters of the response surface are shown in Table 1. The setting of the response surface method included with the selecting of tow input factors. According to that selection, the number of runs will be identified.

The design of experiment for two factors, the setting design of the experiments to be carried out obtained is shown in Table 1. Parameters of the model were set and nominated for calcium and magnesium individually, temperature as A, and time as (B) all through the experiment. Other settings such as the number of runs, display units were set to the default (Table 2).

Results and Discussion

Data layout

After data were imported to the software, response surface



This model type	fits these terms
linear	A B C D
linear and squares	A B C D A*A B*B C*C D*D
linear and two-way interactions	A B C D A*B A*C A*D B*C B*D C*D
full quadratic (default)	A B C D A*A B*B C*C D*D A*B A*C A*D B*C B*D C*D

Figure 2: Screen shot showing model type selected based on the factors/parameters.

Std. Order	Run Order	Pt Type	Blocks	Time (min)	Temp (C)
1	1	1	1	10.0	25.0
2	2	1	1	100.0	25.0
3	3	1	1	10.0	75.0
4	4	1	1	100.0	75.0
5	5	-1	1	-8.6	50.0
6	6	-1	1	118.6	50.0
7	7	-1	1	55.0	14.6
8	8	-1	1	55.0	85.3
9	9	0	1	55.0	50.0
10	10	0	1	55.0	50.0
11	11	0	1	55.0	50.0
12	12	0	1	55.0	50.0
13	13	0	1	55.0	50.0

Table 1: Design of the experiment.

Factors	2
Blocks	none
Runs	30
Display Order:	Run Order
Display Units:	Uncoded
Factors and their Uncoded Levels	
Factor Name A Temp B Time	Low High 25 75 10 100
Response and model	
Response: Ca Terms	A B AA BB AB
Response: Mg Terms:	A B AA BB AB

Table 2: Setting model parameters for calcium.

method module was selected. After the design was set, a model for the dissolution was selected from the "Fittings" column. The fittings gave the modeled value of dissolution while the Residuals (Res) represented the difference between the actual values of dissolution and predicted one (Figure 3).

Qualitative description of dissolution

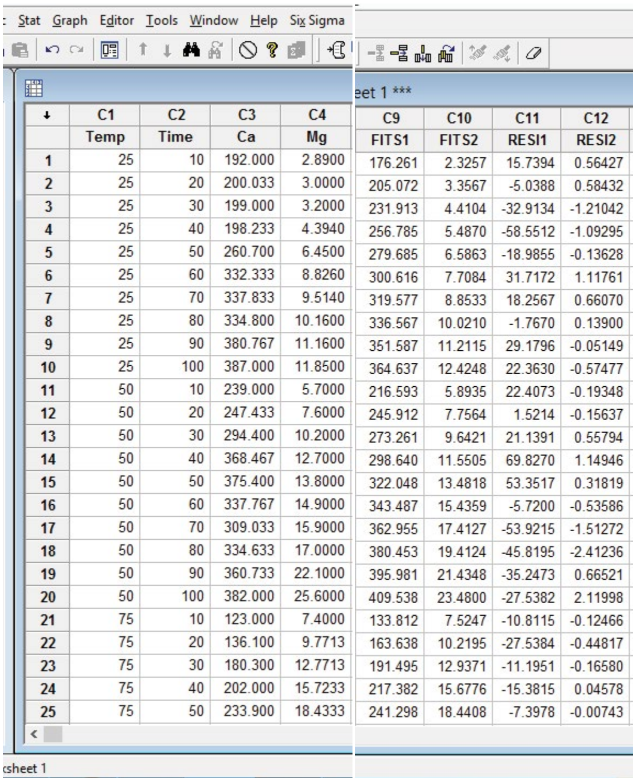
From the 3D surface plot, a qualitative description can be obtained that can show the trend of dissolution with time and temperature. Dissolution data obtained experimentally are shown for different samples. Patterns of dissolution for Mg^{+2}, the curves show less fluctuation compared to that of Ca^{+2}. In general, dissolution of Ca^{+2} is much more compared to that of Mg^{+2} which shows low dissolution, where it ranges from 10 to 340 ppm and from 2-30 ppm respectively (Figure 4).

Calcium release model

Data on Estimated Regression Coefficients can be helpful in the analysis of calcium release. Statistical data were shown in Table 3

From which, dissolution model can be constructed:

Analysis of variance: The p value (to the right side of the results shows that individual factors (i.e. Time, and temperature), and the effect of temperature* temperature are significant as they reflect values lower than 0.05 (the significance limit). The effect of time*time and time*temperature are less significant as they show values of 0.8 and 0.09 respectively. This also indicates that Time*Temp effect is more significant as compared to the effect Time*Time.



↓	C1 Temp	C2 Time	C3 Ca	C4 Mg	C9 FITS1	C10 FITS2	C11 RESI1	C12 RESI2
1	25	10	192.000	2.8900	176.261	2.3257	15.7394	0.56427
2	25	20	200.033	3.0000	205.072	3.3567	-5.0388	0.58432
3	25	30	199.000	3.2000	231.913	4.4104	-32.9134	-1.21042
4	25	40	198.233	4.3940	256.785	5.4870	-58.5512	-1.09295
5	25	50	260.700	6.4500	279.685	6.5863	-18.9855	-0.13628
6	25	60	332.333	8.8260	300.616	7.7084	31.7172	1.11761
7	25	70	337.833	9.5140	319.577	8.8533	18.2567	0.66070
8	25	80	334.800	10.1600	336.567	10.0210	-1.7670	0.13900
9	25	90	380.767	11.1600	351.587	11.2115	29.1796	-0.05149
10	25	100	387.000	11.8500	364.637	12.4248	22.3630	-0.57477
11	50	10	239.000	5.7000	216.593	5.8935	22.4073	-0.19348
12	50	20	247.433	7.6000	245.912	7.7564	1.5214	-0.15637
13	50	30	294.400	10.2000	273.261	9.6421	21.1391	0.55794
14	50	40	368.467	12.7000	298.640	11.5505	69.8270	1.14946
15	50	50	375.400	13.8000	322.048	13.4818	53.3517	0.31819
16	50	60	337.767	14.9000	343.487	15.4359	-5.7200	-0.53586
17	50	70	309.033	15.9000	362.955	17.4127	-53.9215	-1.51272
18	50	80	334.633	17.0000	380.453	19.4124	-45.8195	-2.41236
19	50	90	360.733	22.1000	395.981	21.4348	-35.2473	0.66521
20	50	100	382.000	25.6000	409.538	23.4800	-27.5382	2.11998
21	75	10	123.000	7.4000	133.812	7.5247	-10.8115	-0.12466
22	75	20	136.100	9.7713	163.638	10.2195	-27.5384	-0.44817
23	75	30	180.300	12.7713	191.495	12.9371	-11.1951	-0.16580
24	75	40	202.000	15.7233	217.382	15.6776	-15.3815	0.04578
25	75	50	233.900	18.4333	241.298	18.4408	-7.3978	-0.00743

Figure 3: Screen shot of the actual values, predicted ones, and residuals.

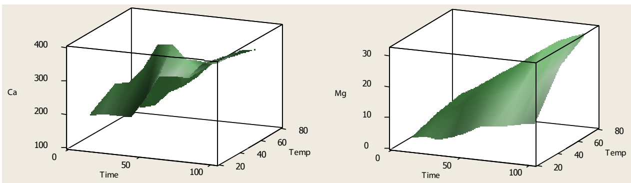


Figure 4: Impact of time-temperature (C) on Ca and Mg release, sample 1.

Term	Coef	SE	T	P
Temp	339.37	12.70	26.71	0.000
Time	40.80	9.61	4.246	0.000
Temp*Temp	76.12	13.01	5.85	0.000
Time*Time	-2.48	17.09	-0.15	0.886
Time*Temp	20.80	11.77	1.77	0.090

S=33.60 R-Sq=80.6% R-Sq (adj)=76.6%.

Table 3: Statistical data of calcium release model.

Magnesium model

Statistical data and model for magnesium is shown in Table 4. For Mg, the model fit is stronger as compared to calcium. The variance analysis for the model shows that the impact of all factors (Temp. Temp*Temp, and Temp*Time) is quite significant, with p value equivalent to zero; except for Time and Time*Time (p value =0.25 and 0.26).

Effect analysis

Normal Probability Plot utilizes residuals remaining from prediction and compares it to a hypothetical straight line of normal data. Normal probability plot provides a measure of outliers also which can be used in determining the points of maximum effect. An example taken here is sample 2 (Figure 4.1) to show the fluctuation of residuals around the hypothetical straight line. The data plotted in the probability plot shows some curvature instead of straight line; the thing that indicates alternative sources other than normal distribution. Effect analysis graphs were obtained directly from ANNOVA test. A

Statistical data and model for magnesium	Coef.	SE Coef.	T	P
Term				
Constant	`	`	4.449	0
Temp	-0.376	0.103	-3.653	0.001
Time	0.059	0.0517	1.154	0.26
Temp*Temp	0.0038	0.0009	3.886	0.001
Time*Time	-0.0005	0.0004	-1.177	0.251
Temp*Time	0.002	0.00049	4.079	0

S=1.585 R-Sq=90.0% R-Sq (adj)=87.9%.

Table 4: Statistical data and model for magnesium (mg).

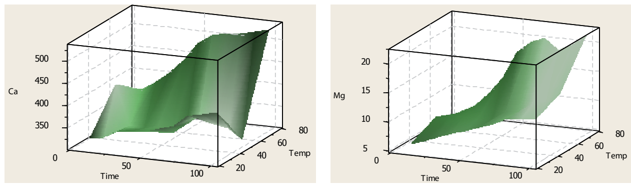


Figure 4.1: Impact of time-temperature on Ca and Mg release, sample 2.

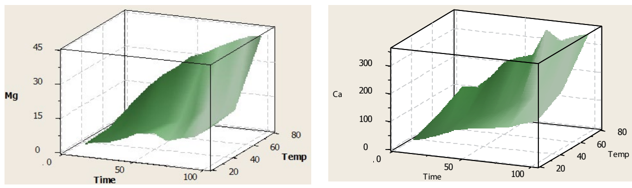


Figure 4.2: Impact of time-temperature on Ca and Mg release, sample 3.

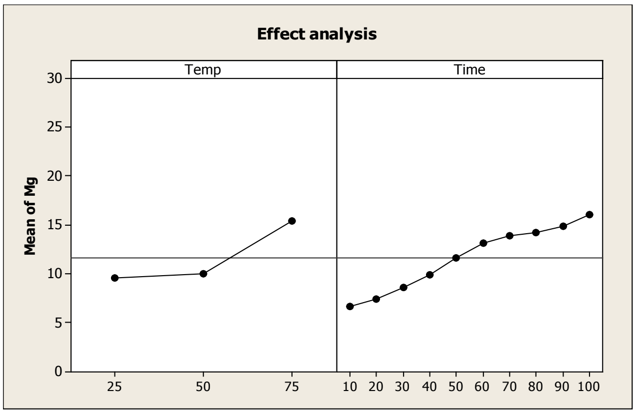


Figure 5: Effect analysis of both Time and Temp for Mg, Sample No 2.

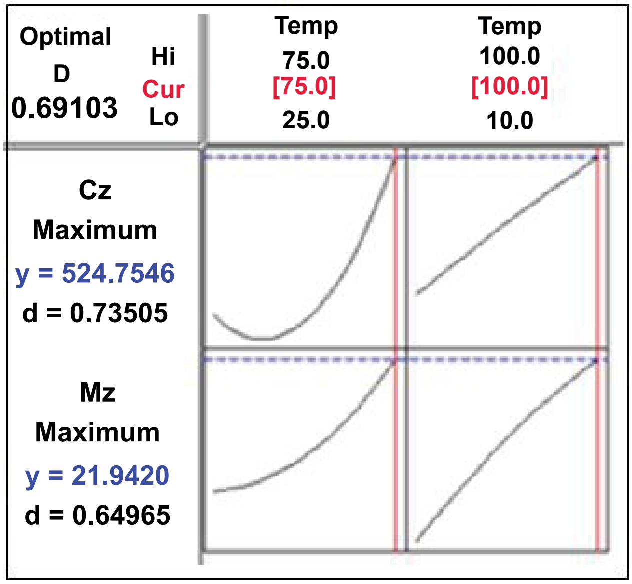


Figure 6: Optimization plot showing the individual desirability of Ca and Mg.

comparative plot the effect of both time and temperature on Mg release (Figure 4.2).

Dissolution optimization

The response optimization helps identify the combination of input parameters setting that results in a maximum single or a group of outputs (responses). In order to achieve that, Response Optimizer module was applied for both calcium and magnesium released. Individual desirability (d) was obtained for each individual response. Minimum and maximum values are set for the output before the response was maximized. The'd' value for both calcium and magnesium was found to be 0.73 and 0.64 respectively. The Results obtained (Figures 5 and 6).

Conclusion

Response surface method was applied to investigate the combined effect of temperature and time factors on the dissolution of calcium and magnesium that principally compose carbonate rocks in the area. Dissolution of carbonate rocks indicated by the release of calcium and magnesium ions was evaluated for a number of six samples of carbonate rocks representing three microfacies. The main objective of this paper was to evaluate the individual and collective effect of time and temperature on the dissolution trends for any facies. Dissolution was measured progressive periods of time under different temperature ranges, and time represented the factors, while amounts of calcium and magnesium released (in ppm) represented the responses. Dissolution was carried out experimentally under a constant pH of 0.1 m Hcl, continuous stirring. The resulting data in terms of concentration (ppm) of the Ca and Mg was used as input data for the model. Response surface method provided models for the dissolution of carbonates, optimization of dissolution, and individual-and-combined effect of each factor (Time and temp).

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