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Design of Experiment Method – An Application in Improving Quality of Magnesium Hydroxide Produced from Natural Seawater Bittern

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ABSTRACT

Seawater bittern is an important resource for production of magnesium hydroxide. It is particularly meaningful for countries of which long beaches and high temperatures are available as natural advantages. This production process bases on the main reaction between solutions containing magnesium and various alkaline agents. Among of precipitants, lime slurry is frequently used as an inexpensive reactant. However, the contamination of calcium in the final magnesium hydroxide is a major problem which limits applicability of this product in many fields required extremely high quality of magnesium hydroxide such as pharmaceuticals, and catalyst, etc. In this work, magnesium hydroxide with more than 99% purity will be recovered from natural seawater bitterns by using the design of experiment (DOE) method. Selected from nine potential factors, a full factorial design considering four main effects was applied to construct a second-order regression model which allows predicting favorable conditions to pursuit high purity of magnesium hydroxide from multi-component seawater bittern and lime milk. On the one hand, the results for a 1.0 L batch investigation proved that factors such as concentration of $\text{Ca}(\text{OH})_2$ in lime suspension and residence time have strong negative influences on the product purity, besides positive effects of reaction temperature. On the other hand, magnesium concentration in the bittern was absent in the regression model. At the optimal conditions, the chemical purity of the solid product was obtained at a value of 99.35%. Thus, this product, and therefore its corresponding production conditions, can be recommended for applications required high quality of magnesium hydroxide.

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1. Introduction

Seawater contains plenty of chemical components, such as salts of K^+ , Na^+ , Mg^{2+} , Cl^- , SO_4^{2-} , etc. Due to its reserves, this resource has been exploited for centuries to produce many valuable mineral salts in large quantities, for instance magnesium compounds, bromide salt, potassium salt, etc. [1]. Among of the seawater-extracted mineral salts, table salt ($NaCl$) is the earliest and the leading produced sea-salt. According to Statista [2], worldwide production of salt in 2020 amounted to 270 million tons. According to salt market forecast for the period from 2020 to 2025, the global salt market is expected to reach a value of more than 32 billion U.S. dollars by 2026 [3]. For areas with advantages of long coastlines and tropical climate, using solar energy is frequently considered as the most economic method to evaporate seawater and produce table salt. Fig. (1) schematically presents a common salt production in Vinh-Hao province (Vietnam). In this process, first, seawater with moderate concentrations of about 4–5 °Bé (Baumé) is continuously evaporated in evaporation bonds and lined earthen basins to increase its total concentrations above 15 °Bé. After that, table salt is crystallized during further evaporation. The obtained mother liquor after salt production is known as seawater bittern, which can reach to very high concentrations (e.g. 30–35 °Bé). At the end of this process, even though the bittern contains extremely rich magnesium, it is often treated as a waste or just partly circulated in table salt production because of potential contamination from high concentrations of multiple components. Therefore, study on recovering magnesium hydroxide with high purity from this resource plays an important role not only for environmental protection but also for further production of valuable compounds from seawater-based resources.

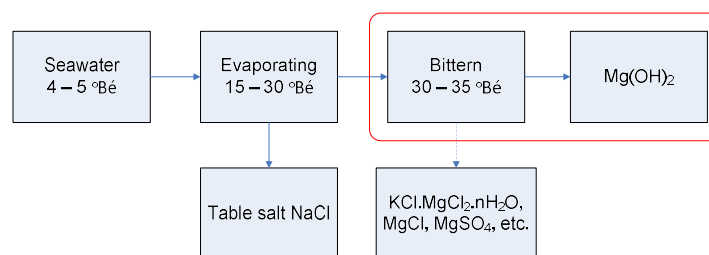


Figure 1: Recovering magnesium hydroxide from seawater bittern as a by-product from production of table salt.

High quality of magnesium hydroxide is frequently required for many applications including pharmaceuticals, advanced materials, catalyst, flame retardant, refractory coating, etc. [4]. The need of magnesium in the world is very large and it is annually increasing in various industries. For instance, annual production of magnesium over the world is 910,000 tons in 2016 [5]. In USA, magnesium hydroxide production increased from 394,000 tons in 2015 to over 410,000 tons in 2019 of which 70% was produced from seawater and natural brines [6]. Regarding to magnesium hydroxide produced from seawater-based resources using lime suspension, the major problem of this process is the contamination of calcium. This can occur due to either calcium adsorption on magnesium hydroxide or forming inclusion of mother liquor inside magnesium hydroxide. These events can rapidly trap calcium into magnesium hydroxide and, therefore, contaminate the final product. Besides diffusion and adsorption, the reaction mechanism of this process can be shortly described as the following two main Equations (1) and (2).



In this work, we focused on separation and purification of magnesium hydroxide from natural seawater bittern from Vinh-Hao province using industrial lime milk. The design of experiment (DOE) will be applied to find out the optimal conditions to obtain the highest purity of magnesium hydroxide in consideration of multiple variables.

2. Design of Experiment (DOE)

Thank to the trend of software development during last decades, DOE has been widely applied in scientific researches and industries as well. Number of publications relating to DOE has increased from 40,000 to 80,000

just in the period 2002–2015 [7]. In comparison to all other fields, about 50% of publications using DOE tools were related to medicine, engineering, biochemistry, physics and computer science. Among of them, physics and computer science, medicine, engineering and biochemistry participated about 13%, 18% and 20%, respectively [8]. The significant increase of DOE application is mainly relied on the development of computer science and software engineering. Nowadays, besides commercial DOE software such as Statistica, SPSS, SAS, Design-Expert, there are also freeware available, for example R software. Additionally, users can also develop their own programs using statistical tools. As a multipurpose tool, DOE can be used in various situations, such as for comparison, variable screening, transfer function identification, system optimization, etc. In principle, DOE identify relationship between response variables as the output functions and important input variables. Basically, DOE is a regression analysis that can be briefly described as follows for cases of full factorial designs [9]. In general, a second-order polynomial can be used with sufficient accuracy, which yields the model as Equation (3). In fact, this regression for k parameters comprises contributions of individual factors and interactions between these effects:

$$\hat{y} = b_0 + \sum_{i=1}^k b_i x_i + \sum_{i,j=1}^k b_{ij} x_i x_j + \sum_{i=1}^k b_i'^2 x_i^2 + err. \quad (3)$$

In this equation, variables x_i are input variables and y is response function. The method of least square is used to estimate parameters in Equation (3) in which the sum of the squares of the errors (err) between experiment (y_i) and model (\hat{y}_i) results will be minimized. Equation (4) is assigned for the least squares function.

$$L = \sum_{i=1}^n err_i^2 = \sum_{i=1}^n (y_i - \hat{y}_i)^2 \quad (4)$$

Procedures for parameter estimation was clearly described in literature [8]. Here, Equation (5) is used to evaluate the effects of each input variable. The significance of each coefficient and the full model will be examined by the F-test (95% confident interval).

$$b_j = \frac{\sum_{i=1}^n x_{ij} \cdot y_j}{n} \quad (5)$$

3. Experimental setup

In Figure (2), 400 mL of a lime milk suspension (1) and a calculated-volume of seawater bittern (2) with various concentrations were agitated by propellers (3) and (4), respectively. While homogeneity of lime suspension is quite easy to remain, its stability with presence of CO_2 in the atmosphere is an issue that restricts feeding time of lime suspension. Feeding rate of lime suspension was controlled. Reaction temperature was controlled by a digital Memmert water bath WNB 10 (5) in which tap water was used as a heat transfer medium.

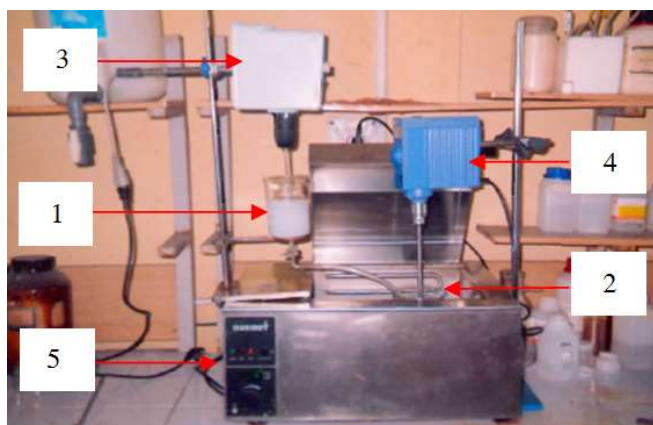


Figure 2: Experimental setup.

To achieve highest purity of magnesium hydroxide, the process must be well controlled under optimal conditions in order to promote crystal growth while nucleation rate should be diminished [10-12]. Therefore, increasing rates of diffusion and ripening as well as decreasing local super-saturation and adsorption of calcium can be considered as major tasks in this research. Herein, nine parameters ($k=9$) were studied including particle size of lime suspension, temperature, feeding time, reactant ratio, lime suspension concentration, stirring rate, seed, magnesium concentration and residence time. However, this approach will lead to a huge number of experiments (e.g. $2^k=512$ runs for a full factorial design). Therefore, in the first experimental part, some parameters will be screened and fixed to reduce the number of experiments if their contributions to variation of the response variable are not significant. Indeed, the experiments were carried out with the similar procedures as follows. The interested parameter will be gradually changed, while the other parameters are remained constants at "fixed" values denoted as "common levels". Table 1 summarized the common levels of these parameters. To increase readability, these parameters will be organized in two groups A and B in the first experimental part corresponding to fixed- and DOE-involved parameters, respectively. In the second part, just the parameters in group B will be further investigated using a full DOE approach to obtain the second-order multiple variable regression. Based on this model, an optimization will be performed in the last part. As a consequence, a comparison between experimental and the model results will be discussed.

Table 1: Parameters and their common levels in screening experiments.

Group	Parameter	Unit	Value
A	Particle size of lime	[μm]	45<
	Feeding time (400 mL slurry)	[min]	10
	Reactant ratio $\text{Mg}^{2+}/\text{Ca}(\text{OH})_2$	[-]	1.2
	Stirring speed	[rpm]	150
	Seeds of $\text{Mg}(\text{OH})_2$	[$\text{m}_{\text{seed}}/\text{m}_{\text{solution}}$]	0%
B	Lime suspension conc.	[$\text{gCa}(\text{OH})_2/\text{L}$]	40
	Magnesium conc.	[mol/L]	0.5
	Reaction temperature	[$^{\circ}\text{C}$]	50
	Residence time	[min]	30

4. Materials

Seawater bittern 30 °Bé from Vinh-Hao province (Vietnam) was used in this study with composition as seen in Table 2 (ICP-OES determination). This is a multiple-component solution with very high concentrations. This solution was pretreated by a reaction with CaCl_2 solution to remove all sulfate as gypsum $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$. The reaction was performed at the following conditions: Concentration of Ca^{2+} was 5.2 M, reactant ratio of $\text{Ca}^{2+}/\text{SO}_4^{2-}$ was 1.99 (mol/mol); reaction temperature and reaction time were 40 °C and 40 mins under well-stirred conditions.

Table 2: Chemical composition of seawater bittern 30 °Bé from Vinh-Hao province (Vietnam).

Composition, [g/L]					
Cl ⁻	SO_4^{2-}	Ca^{2+}	Mg^{2+}	Na^+	K^+
139	105	0.20	24.5	6.2	7.9

For preparation of precipitant, limestone was calcinated at 950 °C for 5 hours to convert all CaCO_3 into CaO before reacting with fresh water. Then, lime milk was sieved to have different fractions which were quickly prevented in closed containers to avoid contamination from reaction with CO_2 in the atmosphere. In the experimental part, concentrations of calcium and magnesium were determined via titration analyses using

standard EDTA 0.1 M solutions (Sigma-Aldrich). Herein, murexide (Sigma-Aldrich) and erio T black (Merck) were used as indicators. Distilled water (Millipore) was used for analytical experiments.

5. Results and discussion

5.1. Effects of Parameters in Group A

a. Effect of Particle Size of Lime Milk

As seen in Fig. (3), a study on three different fractions shows that particle size of lime in the initial suspension has strong influence on purity of $\text{Mg}(\text{OH})_2$. Indeed, using fine lime particles provides higher purity of $\text{Mg}(\text{OH})_2$ than that of coarse particles. Obviously, a comparison on lime particle size effects show that particles smaller than 45 μm results in 84.94% purity of magnesium hydroxide while the products of two other fractions 45-63 and 63-125 μm are found with purity at values of 79.15% and 73.88%, respectively. This is explained due to competition between diffusion rate of Ca^{2+} and OH^- from solid phase of $\text{Ca}(\text{OH})_2$ particles into solution (Reaction (1)) and precipitation rate of $\text{Mg}(\text{OH})_2$ (Reaction (2)). Dissolving $\text{Ca}(\text{OH})_2$ process and its constituent component diffusion can be interfered by other events occurring consecutively and/or simultaneously. For instance, new born $\text{Mg}(\text{OH})_2$ can quickly cover the surface of residual lime particles. Besides, it can also form liquor inclusion containing calcium as a contaminant. In other words, small particles dissolve faster than large particles due to their specific surface areas that leads to less residual $\text{Ca}(\text{OH})_2$ particles existing in the solution. Therefore, using fine lime particles is more favored. However, it is not applicable to have many fine fractions since just limited sieves are available. In the next part, we will fix input stream of lime suspension with particles smaller than 45 μm .

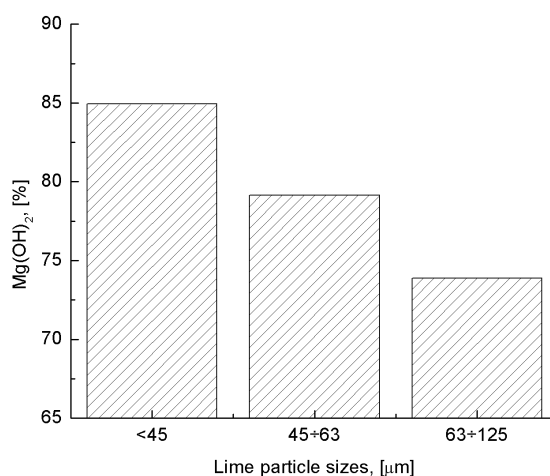


Figure 3: Effect of particle sizes of lime suspension.

b. Effect of Feeding Rate of Lime Milk

Besides effect of particle sizes as discussed above, the purity of magnesium hydroxide also strongly depends on the feeding rate of lime milk. Results in Fig. (4) show that the slower the lime milk is added, the higher the product purity is obtained. High feeding speed of lime milk will lead to increase local super-saturation and result in small particles which are easily contaminated by Ca^{2+} adsorption. Furthermore, unexpected change in super-saturation can also form mother liquor inclusion and encouraging the process of coating $\text{Ca}(\text{OH})_2$ particles. Thus, the slow feeding rate of lime milk will create favorable conditions for the diffusion processes, therefore, promoting purity of magnesium hydroxide. Nonetheless, after 20 minutes, further extended feeding time does not significantly change the product quality but it will reduce economic efficiency of the overall process. Because of the equipment conditions and instability of lime milk under presence of CO_2 in atmosphere, we fixed the feeding time of 400 mL of lime milk at 20 minutes for next experiments.

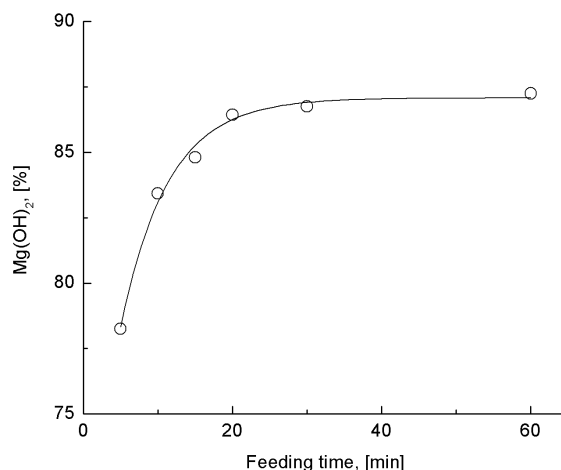


Figure 4: Feeding rate effect (counted for 400 mL of lime suspension).

c. Effect of Adding Seed of Mg(OH)₂

Due to high reaction rate between Mg²⁺ and OH⁻ as depicted in the Reaction (2), the precipitation rapidly occurs and forms tiny particles that not only promotes adsorption of calcium but also reduces filterability of the final product. Adding seeds into the system supports development of larger Mg(OH)₂ particles. In other words, the system does not need to create nuclei and the super-saturation will be mainly used for crystal growth [10-12]. Besides, promoting crystal growth based on the added seeds will also reduce covering ability of Mg(OH)₂ on the surface of residual Ca(OH)₂ particles. Since the crystal growth process is selectively developed on homogeneous nuclei than that of heterogeneous surfaces. In Fig. (5), experiments show that the use of seeds helps to increase the purity of the obtained products, but this effect is only significant up to about 5% of seeds, adding more seeds do not improve the product purity. This can be explained by the fact that the system achieves the critical required number of active sites for crystallization so that the excessive use does not show a significant change. In the following experiments, we fixed the amount of seeds at 5% of the solution.

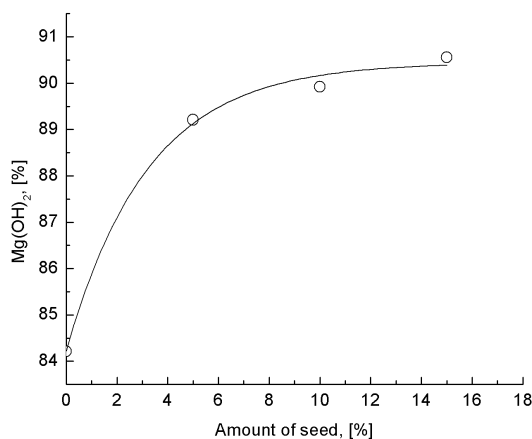


Figure 5: Effect of adding amounts of Mg(OH)₂ as seeds.

d. Effects of Mole Ratio of Reactants Mg²⁺/Ca(OH)₂ and Stirring Speed

Besides the influences of the above factors, two other parameters were also taken into account including mole ratio of reactants and stirring speed. First, amounts of Mg²⁺ were excessively used in comparison to that of Ca(OH)₂ in order to promote completion of the Reaction (2). Theoretically, the presence of various ions in solution can change ionic strength and, therefore, affect the rate of precipitation. However, experimentally, the excesses of Mg²⁺ do not significantly improve product quality as seen in Fig. (6a). Indeed, increasing magnesium excesses from 20 to 80% results in an increase of purity just about 1%. In the next part, ratio of reactants will be fixed at a value of 1.2 for further investigation.

In Fig. (6b), fluid dynamics has influence on diffusion terms of this system that can vary the purity grade of the final product. As stronger agitation is applied, as higher purity will be obtained. Nevertheless, the value of 100 rpm is selected as a suitable value since higher stirring speeds have not shown significant effects on improving product quality but consuming more energy.

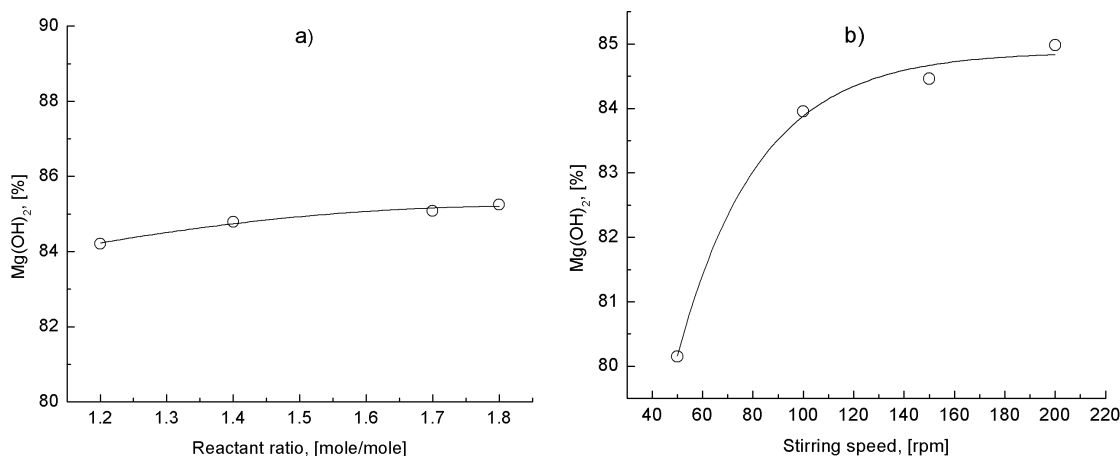


Figure 6: Effects of reactant ratio and stirring rate on the product purity.

5.2. Effects of Parameters in Group B

a. Effect of Concentration of Ca(OH)₂ in Lime Suspension

The concentration of lime milk strongly affects the purity of the obtained product. As seen in Fig. (7), the more dilute the lime milk, the higher the purity of Mg(OH)₂ is obtained. This can be explained because dilution of lime suspension will promote Reaction (1) proceeding forward right to produce more OH⁻ and reduce presence of Ca(OH)₂ as residual solid particles. Moreover, dilution also leads to reduce viscosity that is more favorable for diffusion processes. Therefore, lime milk concentration is chosen as one of important factors in the DOE investigation and the considered range is chosen from 40 to 70 gCa(OH)₂/L.

b. Effect of Concentration of Mg²⁺ in Seawater Bittern

Fig. (8) shows that the product quality is inversely proportional to the concentration of Mg²⁺ in the seawater bitterns. This is reasonable since salt effects are very significant; all components have difficulty in diffusion due to high viscosity when high magnesium concentration solutions are used. This is also related to rapid increase of local super-saturation that forms fine particles and easy to be contaminated. However, more dilution will require larger reactor volumes and reduce process performance. Thus, concentration of Mg²⁺ is also selected as a studied factor in the DOE section with a studied range from 0.1 to 0.5 M.

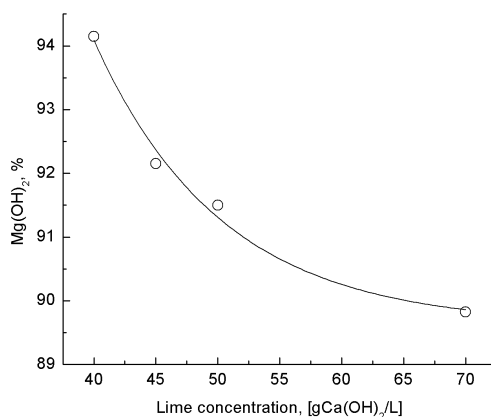


Figure 7: Effect of lime suspension concentration.

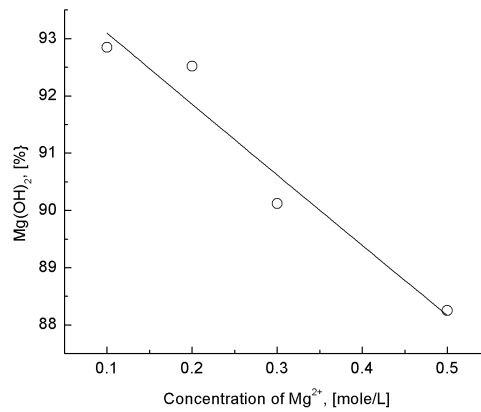


Figure 8: Effect of magnesium concentration in seawater bitterns.

c. Effect of Temperature

Reaction temperature strongly affects the purity of Mg(OH)₂ as seen in Fig. (9). In the studied temperature range, product purity increases with increasing reaction temperature. Since the reaction between Mg²⁺ and Ca(OH)₂ quickly reaches equilibrium, temperature affects the purity of Mg(OH)₂ mainly during the finishing stage of the precipitation. The increased temperature promotes the desorption of calcium ions on the surface of Mg(OH)₂. In addition, the high temperature also helps to reduce product contamination because heat will push out impurities and break up the liquid inclusion. Besides, the increasing temperature also improves filterability of the product thank to Ostwald ripening. With these characteristics, we chose temperature as a studied factor in the DOE to determine the optimal temperature value. Herein, a temperature range from 50 to 90 °C is selected for the next section.

d. Effect of Reactant Residence Time

Reactant residence time is defined as a sum of the reaction time and ripening period. Indeed, ripening periods were expected supporting completion of magnesium hydroxide product. In the period up to 110 mins, the purity of Mg(OH)₂ increases with increasing the residence time. In this stage, Ostwald ripening seems to be predominating, so that an increase in the purity of product was observed. However, after this period, the purity decreases as seen in Fig. (10). Thus, in the later periods, impurity adsorption seems to be increased. Therefore, residence time is also considered as another studied factor in the DOE with a studied interval from 60 to 180 mins.

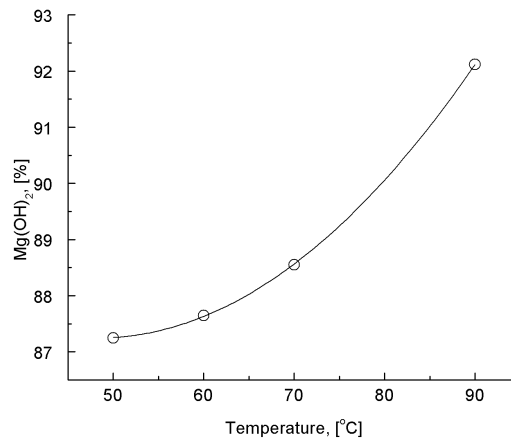


Figure 9: Effect of reaction temperature.

5.3. The 2⁴ Full Factorial Design

From the analyses as seen in part 5.2, four factors are herein selected to evaluate their contribution as well as their potential interaction in a 2⁴ full DOE model. The considered effects are: (x₁) - concentration of Ca(OH)₂ in lime

suspension, (x_2) - concentration of Mg^{2+} in bittern solution, (x_3) - reaction temperature, and (x_4) - residence time. The other parameters such as stirring speed, seeds, feeding rate, reactant ratio and size of lime particles are fixed. Meanwhile, low and high levels for four DOE-involved factors are summarized in Table 3.

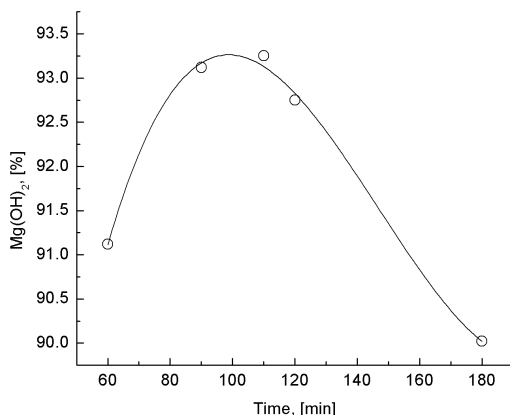


Figure 10: Effect of residence time.

Table 3: Factors of DOE investigation and their bound levels.

Factors			Low (-1)	High (+1)
x_1	concentration of $Ca(OH)_2$	[g $Ca(OH)_2$ /L]	40	70
x_2	concentration of Mg^{2+}	[mol/L]	0.1	0.5
x_3	reaction temperature	[°C]	50	90
x_4	residence time	[min]	60	180

The matrix for 2^4 full factorial design is shown in Table 4. Hereby, experiments at center point (corresponding to level 0), and axial points $\pm\alpha$ are also performed. Indeed, the α parameter is determined at a value of 1.546.

From the data in Table 4, the coefficients of the regression model were statistically analyzed. Indeed, these effects were calculated and the obtained parameters were shown in Table 5. Compatibility of these parameters were tested, then Equation (6) was obtained which includes significant contributions in real coordinates. After that, Fisher's criteria were then applied for testing the obtained equation. As a conclusion, good compatibility of this equation in comparison to experiments was achieved.

$$Y = 106.957 - 0.135 Z_1 + 0.412 Z_3 - 0.043 Z_4 + 3.10^{-4} Z_3 Z_4 + 0.0036 Z_3^2 - 2.9 \times 10^{-4} Z_4^2 \quad (6)$$

According to Equation (6), it is observed that two main effects of lime concentration (Z_1 in real coordinate or x_1 in the DOE matrix) and residence time (Z_4/x_4) are negative. Therefore, the purity improvement supposed to be performed on low levels to minimize contamination. Besides, effect of temperature (Z_3/x_3) is positive at first and second orders that means the adsorption is diminished and ripening is supported at elevate temperatures resulting high purity of the final product. Alongside main effects, there is also an interaction between temperature and residence time ($Z_3 Z_4/x_3 x_4$). Concentration of magnesium (Z_2/x_2) has so less influence that it is absent in the model. Therefore, the best setup for getting better product purity will be obtained when factors Z_1/x_1 and Z_4/x_4 are at the low level while Z_3/x_3 is at the high level.

An simple optimization was performed using derivatives of the response function according to input variables that resulted in a maximal purity of $Mg(OH)_2$ at the optimal point with the conditions listed in Table 6. Performing cross-check experiments at these conditions, we have obtained a product purity of 99.35% while the model value calculated from the regression equation is found at 98.35%. Thus, the obtained values from the DOE model is compatible with the experiment. Based on the above analysis it is possible to predict purity of magnesium hydroxide using the obtained regression model using Equation (6).

Table 4: The design matrix for 2⁴ full factorial design, experiment (y_i) vs. model (\hat{y}_i) purity (%).

No.	x0	x1	x2	x3	x4	y_i	\hat{y}_i
1	+	+	+	+	+	89.67	88.82
2	+	-	+	+	+	96.50	94.04
3	+	+	-	+	+	87.02	88.82
4	+	-	-	+	+	95.78	94.04
5	+	+	+	-	+	84.10	85.65
6	+	-	+	-	+	90.05	90.87
7	+	+	-	-	+	86.32	85.65
8	+	-	-	-	+	89.48	90.87
9	+	+	+	+	-	90.56	89.26
10	+	-	+	+	-	95.85	94.48
11	+	+	-	+	-	90.46	89.26
12	+	-	-	+	-	93.22	94.48
13	+	+	+	-	-	88.11	87.21
14	+	-	+	-	-	93.71	92.43
15	+	+	-	-	-	86.38	87.21
16	+	-	-	-	-	93.49	92.43
17	+	0	0	0	0	90.52	90.62
18	+	0	0	0	0	90.89	90.62
19	+	0	0	0	0	91.12	90.62
20	+	+ α	0	0	0	87.24	86.59
21	+	- α	0	0	0	94.56	94.66
22	+	0	+ α	0	0	90.12	90.62
23	+	0	- α	0	0	91.98	90.62
24	+	0	0	+ α	0	94.76	94.56
25	+	0	0	- α	0	91.74	90.53
26	+	0	0	0	+ α	88.55	87.27
27	+	0	0	0	- α	88.02	88.82

Table 5: Estimation of factor effects.

Model Term	Effects	Model Term	Effects	Model Term	Effects
b_0	90.75	b_{12}	-0.11	b_{34}	0.66
b_1	-2.73	b_{13}	0.11	b_1'	-0.44
b_2	0.16	b_{14}	-0.24	b_2'	0.01
b_3	1.54	b_{23}	0.36	b_3'	0.93
b_4	-0.57	b_{24}	-0.18	b_4'	-1.13

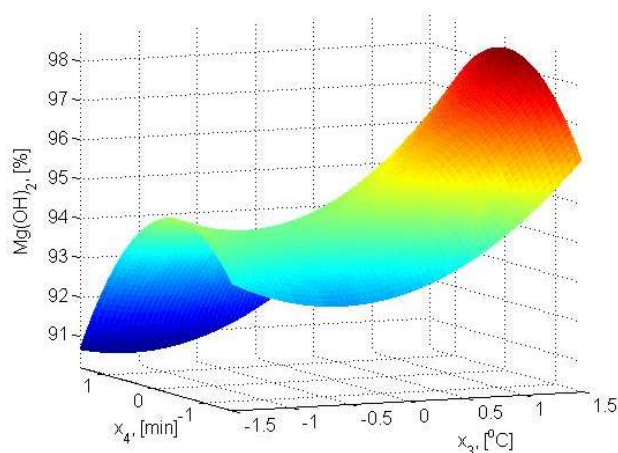


Figure 11: Graphical presentation of the surface response (plotted for a case of x_1 at the optimal point).

Table 6: The optimal solutions to obtain more than 99% purity of magnesium hydroxide.

Parameter	Lime Concentration	Temperature	Residence Time
x_i (model variable)	$x_1 = -1.546$	$x_3 = 1.495$	$x_4 = -0.162$
Z_i (real parameter)	$Z_1 = 28 \text{ gCa(OH)}_2/\text{L}$	$Z_3 = 88 \text{ }^\circ\text{C}$	$Z_4 = 110 \text{ mins}$

Quality of the obtained magnesium hydroxide under optimal conditions was also analyzed. Atomic emission spectrum shows that magnesium hydroxide is obtained with very high chemical purity, the major contaminant is calcium with the amount of 0.1%, other impurities are considered insignificant (see Table. 7). Furthermore, XRD pattern (not shown due to limited space) proved that this product contain only one phase of Mg(OH)_2 . Thus, besides chemical composition, solid phase analysis was also approved the high quality of the final product.

Table 7: Contamination of the magnesium hydroxide product (% , [m/m]).

Al	Si	Fe	Ti	Cu	Ca
0.003	0.01	0.01	0.001	0.001	0.1

6. Conclusion

The most challenging of using lime milk in magnesium hydroxide production from seawater-based resources is the contamination of calcium which leads to limited application of this compound in many fields. In this work, we focused on improving magnesium hydroxide purity produced from a real seawater bittern taken from Vinh-Hao province (Vietnam), in which extremely high concentration of many salts are available. First, the bittern was pretreated to remove sulfate as gypsum $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ by using CaCl_2 solution. Afterwards, this solution was used as a feed to produce magnesium hydroxide via a reaction with lime suspension. Regarding to this method, on the one hand, it shows advantages in terms of ability to directly handle high concentration solutions without dilution, providing good performance with very high process yields, etc. On the other hand, disadvantages of this method can also be counted for contamination of calcium as well as filterability relating to fine particle sizes of the final product. The contamination source could be resulted either from calcium adsorption or mother liquor inclusion formation. To increase purity of magnesium hydroxide, nine factors were investigated including particle size of lime suspension, temperature, feed time, reactant ratio, suspension concentration, stirring rate, amounts of seed, magnesium concentration and residence time. However, five of them can be neglected since the variation of these factors and their influences on the product's purity seem to be not significant. Besides, the other factors were further investigated in a full 2^4 DOE model to find out a regression model which allows optimizing the purity of Mg(OH)_2 . As a consequence, experiments proven that purity of magnesium hydroxide strongly depend on

concentration of $\text{Ca}(\text{OH})_2$ in the suspension. In fact, low concentration of $\text{Ca}(\text{OH})_2$ helps to improve the purity of the product but bigger volume of the reactor and/or dilution steps are required. The optimized concentration of lime suspension was found at a value of 28 g $\text{Ca}(\text{OH})_2/\text{L}$. Besides, temperature is also an important impact factor which affect to diffusion, ripening, reaction rate. A temperature at 88 °C was proven as the optimal value. Lastly, residence time is also proven as a key parameter affecting the purity and a period of 110 mins resulted in the highest purity of magnesium hydroxide. At the optimal conditions, the product purity was achieved at a value of 99.35% which is highly suggested for advanced applications. Comparing to experiments, the statistical testing showed that the estimated coefficients were reliable and the model compatibility is proven. In conclusion, the obtained 2⁴ DOE multi-variable regression can be used in predicting and controlling purity of magnesium hydroxide produced from seawater bittern and lime suspension.

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