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Research Article

Calcium Carbonate Synthesis, Optimization and Characterization from Egg Shell

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Abstract

In this study, to determine the calcium carbonate availability in eggshells waste and factors those affect its extraction. The parameters like temperature, the size of the eggshell powder and the amount of solvent have highly affected the yield of calcium carbonate obtained from eggshells. The calcium carbonate was synthesized with the temperatures of 65, 100, 150, 200 and 234°C. The eggshell powder size was 29, 50, 80, 110, and 130 μm. The volume of water to mass of eggshell ratio was (ml/g) 26, 30, 35, 40 and 43. The boiling process for 30 min of eggshell kills any microbial growth on the surface. The grinding, drying and separating process was carried out for 25, 30 and 10 min respectively. The obtained results indicated that the addition of temperature led to slightly increase calcium carbonate yield when the diameter is 29 μm, the calcium carbonate yield increases highly until the temperature reaches to 150 °C for 130 μm diameter then declines and finally the calcium carbonate yield decreases as the temperature increases for the diameter of above 125 μm. The maximum yield is 54.0%, obtained at the temperature of 200°C, the diameter of 110 μm and a ratio of 40 ml/g.

Keywords: Calcium carbonate; Eggshell; Environment; Optimization; Synthesis; Yield.

Introduction

An eggshell is the outer covering of a hard-shelled egg and of some forms of eggs with soft outer coats. On the other hand, an eggshell is a solid waste generated from homes, hotels, restaurants, food manufacturing industries and others, which possess a serious environmental problem. The eggshell is an important structure for two reasons. Firstly, it forms an embryonic chamber for the developing chick, providing mechanical protection and a controlled gas exchange medium. Secondly, it is a container for the market egg, providing the protection of the contents and a unique package for valuable food [1].

The strength of an egg can be determined by determining the calcium carbonate content of the eggshell. The percentage of calcium carbonate indicates the strength of an eggshell and the strength of the eggshell show how serious the pesticide damaged to the eggshell [2]. Eggshell makes up from 9-12% of the total egg weight; it consists largely of calcium carbonate (94%) with some magnesium carbonate and calcium phosphate deposited on the organic matrix [3]. From the chemical point of view, it consists of water (2%) and dry matter (98%). Eggshell calcium looks like a good source of calcium carbonate. It's generally low in heavy metals, reasonably absorbable, and inexpensive [4]. Calcium carbonate occurs naturally in several mineral forms including the pure calcite, aragonite and vaterite minerals as well as the impure minerals like limestone, chalk, marble, and travertine[5]. It is also the main chemical constituent of eggshells, seashells, oyster shells, snail shells, corals, etc [6].

Eggshell calcium carbonate is mainly used as a diluent in solid dosage forms in a pharmaceutical excipient [7]. It is also used as a base for medicinal and dental preparations, buffering and dissolution aid in dispersible tablets, as well as a food additive and calcium supplement [8]. Nowadays, there is a great interest in finding new pure calcium carbonate sources in order to meet the demand of calcium carbonate [9].

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In many countries around the world, egg products companies and some food manufacturers that use egg in their products generate tons of waste eggshell, this possesses loss of calcium carbonate within it, which is the best natural source of calcium carbonate[10]. At the same time, a serious environmental problem occurs from waste eggshells. Challenges associated with the disposal of eggshells include cost, availability of disposal sites, odor, flies, and abrasiveness [11]. One of the challenges for the supplement of calcium carbonate in Ethiopia is due to inappropriate use of the calcium carbonate sources. Eggshell is one of the main sources for the production of calcium carbonate [12]. So, keeping in mind the high calcium carbonate content, disposal costs, and increasing environmental concerns, it is necessary to find an alternative method that would transform the waste eggshells into a valuable item, giving financial benefits to the competitive egg processing industry [13].

Material and methods

Materials and chemicals used

The only raw material used for the synthesis of calcium carbonate from eggshell is hen eggshell. Hen eggshell: - High-quality eggshells contain 27 essential microelements but they are mostly composed of calcium carbonate, a form, and structure that is very similar to our bones and teeth. In animal and human tests, eggshell calcium shows increased bone density, less arthritic pain, and even stimulates cartilage growth.

Chemicals used

Hydrochloric acid (HCl) (32% by mass) with a molarity of10.17mol/L. Acetic Acid (CH3COOH) (36% by mass) with a molarity of 6.27mol/L. Distilled water to wash the raw materials to remove dirty material. Blue litmus paper to determine the property or acidity of final product.

Raw material pre-preparation and storage

An eggshell that was collected from different sources dries to reduce the moisture content and stored in a closed storage tank. If an eggshell was not dried it sticks and forms a bad smell during the storage period. This process takes place only when the raw material was wanted to stay in the storage tank. After drying it should be stored at room temperature in the storage tank. But if it was desired to uses the fresh eggshell it can be stored in an open storage tank. It can stay for only one day without smelling.

Raw material preparation

Once an eggshell that was collected from different sources, dried and stored in the storage tank, it was conveyed to the washing tank to wash an eggshell. This was because there were some dirties that cannot be released in the water heater unless they washed. After washing it was placed in an oven to dry the sample to dry well and it is easily crushable. Then it is crushed by using jaw crusher based on the required size of the sample. The synthesis and optimization process of calcium carbonate from eggshell was accomplished through the following procedures. Raw material collection, Washing of an eggshell, Boiling of the eggshell in hot water, Dry the eggshell, Smashing the eggshell, Grinding the eggshells to reduce its size, Separating the fine powder that contains calcium carbonate and Storage.

Raw material collection

An egg shell is solid waste that generate from homes, hotels, restaurants, food manufactures industries and others. The raw material collected was stored in storage tank until it was needed for the next process. Raw material was collected from the local community, house, hotel restaurants and etc. The laboratory work was done two times per/day or 2batch/day. The amount of raw egg shell used for one batch operation was100g for 3000ml of water. That means the ratio of water to raw egg shell used for one batch is 30:1. It was also done by increasing the amount of water used to 4000ml for the same amount of water i.e ratio of water to raw egg shell used 40:1.

Washing of an eggshell

Once an eggshell that was collected from different sources, dried and stored in the storage tank, it was conveyed to the washing tank to wash an eggshell. This was because there were some dirties that cannot be released in the water heater unless they washed.

Boiling of eggshell

The washed and cleaned eggshell was passed to a water heater that contains water at $218^{o}c$ and rinsed in it for 30 minutes to remove any wastes but without removing more membranes as these have extra nutrients. This process takes place after measuring the volume of water and the mass of the eggshell. This also separates most of the membrane which can remove if anyone concerned about long terms storage.it was easiest to do this while the water was boiling by scooping out floating solids and the foamy ring that form. This kills any bacteria and removes harmful pathogens in the eggshell.

Drying

After an eggshell was cleaned using water in the water heater the mass was measured and it was spread on glass or stainless steel baking sheet and dried in the oven for 30 minutes at a temperature of $90^{o}c$ to remove the moisture in it to make easy for grinding process. The mass of an eggshell was measured before and after drying .This is needed for calculating the moisture content of an eggshell.

Grinding

Grinding is just the process of reducing the size of an eggshell into the size needed for sieving. Once it is dried the mass was measured and it was smashed and puts in the grinder to grind it into finest. This process is one of the most important steps in calcium carbonate synthesis from the eggshell.

Sieving and measuring

Sieving is the physical separation of particle depending upon particle size. The mass of the eggshell powder was measured and passed to the sieve to separate the finest one from the coarser product. This process takes place for 10 minutes. The process was separated using 63µm,125µm, and the mass of each product was measured.

Synthesis calcium carbonate from the dry egg shell

The eggshell powder can be utilized as a food additive, anti-microbial agent and fertilizer purpose traditionally. Now in this study convert this eggshell to valuable products like calcium carbonate. The organic matter in the eggshells, the membrane and egg whites were heated and remove to maximize the yield. From the literature reviewed there are different methods listed for synthesis of calcium carbonate from the dry eggshell was selected for our project due to the following reasons. Synthesizing eggshell calcium carbonate from the dry eggshell yields the maximum amount of calcium carbonate. Synthesis eggshell calcium carbonate by soaking an eggshell in the lemon juice consumer's time to take out calcium carbonate, unlike the stone. Since the eggshell is high in minerals they will not mix into the lemon juice. It is difficult to take the waste eggshell from the lemon juice. The calcium carbonate synthesized using lemon juice is used only for consumption. Taking eggshell calcium carbonate by soaking the eggshell in lemon juice requires another extra raw material i.e. the lemon juice, which is difficult on the industrial scale. The effect of the process variables like temperature, size, and amount of water was not specifically described. But these process variables have a great effect on the calcium carbonate yield. In this research, the effects of these process variables were addressed. The parameter ranges for optimization of temperature, size (diameter) and water content shown below in a table.

Selection of process variable

Calcium carbonate synthesis was affected by several process variables, such as temperature, ratio, particle diameter, moisture content, equipment used, and residence time. The three parameters like temperature, ratio and particle diameter were the most important factors which affect the synthesis process. When compared to the three listed the others parameters have the least effect on calcium carbonate yield. So, decided to optimize those three parameters have the most important on the yield. For example, the moisture content of an eggshell before putting in the oven and after it is removed from the oven is almost the same so it has the least effect. Then table 1 shows the range of parameters that the experiments were carried out.

Table 1. Parameter ranges and level (specification) for optimization

Parameters	range	Level
Temperature, °C	65 -234	65, 100, 150, 200, 234
Size, µm	29-130	29, 50, 80, 110, 130
Ratio	30-43	26:1, 30:1,35:1, 40:1, 43:1

Process flow diagram of synthesizing calcium carbonate from hens' eggshell

Process flow diagram of synthesizing calcium carbonate from hens' eggshell is shown in figure 1.

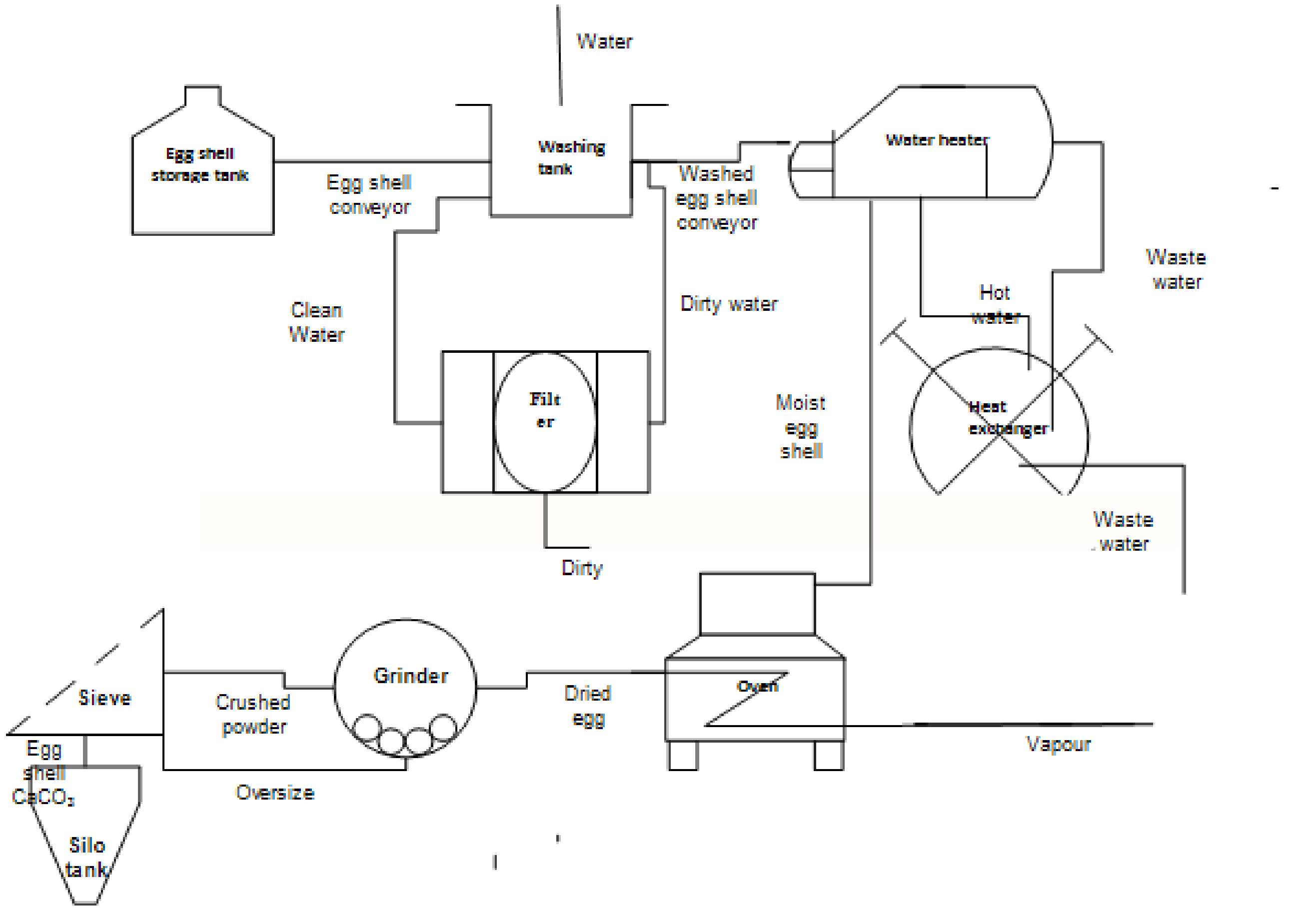

Figure 1. Process flow diagram for synthesizing calcium carbonate from egg shell

Result and discussion

The results were obtained by combing three different process variables. There are 20 experiments were carried out and the results was expressed in yield. The final synthesized product was in powder form hence it measured by using balance and expressed in a gram. The yield obtained was calculated by using equation 1. The yield obtained in a laboratory experiment by using three process variables like temperature, ratio, and diameter was shown on a table 2.

$$yield(\%) = \frac{measured\ calcium\ carbonate\ obtained}{mass\ of\ eggshell\ after\ drying} \times 100 ....1$$

Determination of moisture content of an egg shell

100g of the fresh egg shell sample was weighed and dried in an oven at 90°C for 30 minutes. Then, the weight of the egg shell was measured. Four samples were analyzed and the mass and the percentage of moisture content were measured for each sample. The mass of moisture content was measured by substructing mass of eggshell after dry from of eggshell before dry. The mass dry eggshell is calculated by using equation 2. The moisture content was calculated by using equation 3. Table 3 was used for the determination of mass of eggshell, moisture content, mass of egg shell after and befor dry.

$$mass\ of\ eggshell(\%) = \frac{mass\ of\ eggshell\ after\ drying}{mass\ of\ eggshell\ before\ drying} \times 100 ....2$$

$$moisture\ content = 100 - mass\ of\ egg\ shell(\%) ....3$$

From table 3 the average moisture content obtained from experiment was 5.20 this shows the moisture content for egg shell is small and the solid content of the eggshell was high and its results was 95.8%.

The average value for sieve analysis for the egg shell sample were determined in a table 4. The results was analysed by using equation 4,5, and 6. The average particle diameter (Dpi), mass fraction of calcium carbonate (Fi), cumulative residue fraction (FCR), and Cumulative fall fraction (FCF) were analysed by using equation 6, 7 and 8 respectively.

$$Dpi = \left(\frac{D_n + D_{n-1}}{2}\right) ....4$$

$$Fi = \frac{M_i}{M_T} \times 100\% ....5$$

$$FCF = 100 - FCF ....6$$

Table 2. Percentage yield of $CaCO_3$at different temperature, diameter and solve

Trial	T (°C)	Ratio (ml/g)	D (µm)	Yield (%)
1	100	30	50	11.5
2	200	30	50	34.6
3	100	40	50	21.5
4	200	40	50	43.8
5	100	30	110	21.5
6	200	30	110	44.8
7	100	40	110	32.02
8	200	40	110	54
9	65	35	80	14.35
10	234	35	80	52.2
11	150	26	80	24.1
12	150	43	80	41.2
13	150	35	29	25.2
14	150	35	130	42.3
15	150	35	80	33.2
16	150	35	80	34.5
17	150	35	80	33.8
18	150	35	80	33.8
19	150	35	80	34.5
20	150	35	80	32.97

Table 3.Determination of moisture content of Egg shell

Sample	Mass of egg shell before dry	Mass of egg shell after dry	Moisture content(g)	Mass of egg shell (%)	Moisture content (%)
1	100	95.10	4.9	95.10	4.9
2	100	94.68	5.32	94.68	5.32
3	100	94.55	5.45	94.55	5.45
4	100	94.89	5.11	94.89	5.11
Average moisture content of the four sample			5.20	94.805	5.20

Table 4. Sieve analysis of the product

Screen Opening (µm)	Mass of $CaCO_3$(g)	Dpi, Aver (µm)	fi (%)	FCR (%)	F CF (%)
250					
125	3.70	187.5	20.06	20.06	79.94
110	11.12	94.00	60.30	80.36	19.64
75	3.62	31.50	19.63	99.99	0.01

The effects of each parameter on a yield of calcium carbonate synthesized

The effects of temperature on a yield of calcium carbonate

From table 5 the effects of all parameter were obtained to analyze the effects on the yield obtained. Temperature had positive effects on the yield of calcium carbonate when the ratio and diameter at 35 ml/g and 110 µm. the temperature increase the amount of calcium carbonate synthesized was increased. Synthesis of calcium carbonate was carried out an endothermic reaction that means an increment in temperature favors the synthesis process. When the temperature was at 200°C the maximum yield obtained was 54. From figure 2, a temperature had significant effects than both ratios and diameter of the particle to synthesize calcium carbonate.

Table 5. The effects of each parameter on a yield of calcium carbonate

Source	Sum of Squares	df	Mean Square	F-value	p-value	Significance
Model	2439.86	9	271.10	1567.85	< 0.0001	Significant
A-Temperature	1743.02	1	1743.02	10080.54	< 0.0001	
B-Ratio	327.03	1	327.03	1891.31	< 0.0001	
C-size(D)	355.02	1	355.02	2053.24	< 0.0001	
AB	0.5618	1	0.5618	3.25	0.1016	
AC	0.0018	1	0.0018	0.0104	0.9207	
BC	0.0338	1	0.0338	0.1955	0.6678	
A^2	1.34	1	1.34	7.73	0.0194	
B^2	2.24	1	2.24	12.98	0.0048	
C^2	0.3164	1	0.3164	1.83	0.2060	
Residual	1.73	10	0.1729			
Lack of Fit	0.8403	5	0.1681	0.9455	0.5238	Not significant

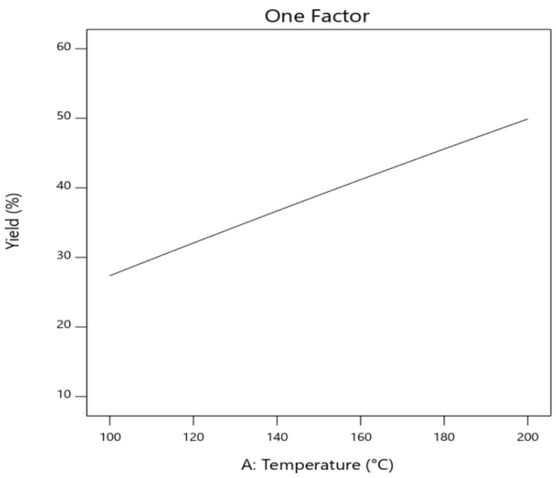

Figure 2. The effects of temperature on the yield calcium carbonate

The effects of ratio on a yield of calcium carbonate

Similarly, from the table 5 the ratios also positively affect the yield of calcium carbonate. The ratio is not extremely affects the yield of synthesized calcium carbonate like temperature and diameter of particles. But ratios affect the yields of synthesized calcium carbonate slightly compared with other factor that experimentally observed and analyzed by design expert. At the highest ratios the yield of calcium carbonate was 40 ml/g. The effect of ratio was determined at figure 3.

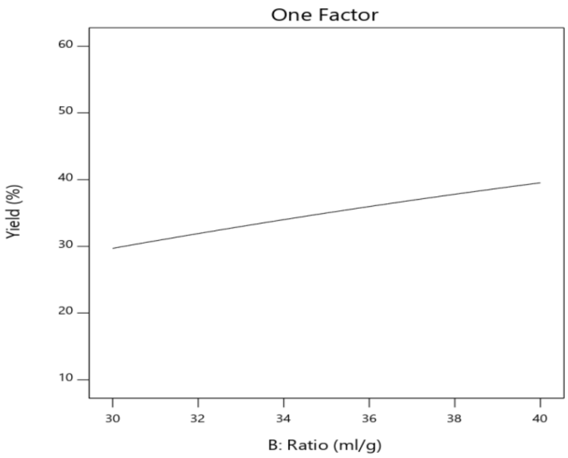

Figure 3. The effects of ratio on the yield of calcium carbonate

The effects of diameter on a yield of calcium carbonate

Size of a particle had positive effects on the yield of calcium carbonate when the ratio and temperature was at 40 ml/g and 200°C. When the size of particle was increased the amount of calcium cabinet synthesized was increased. Synthesis of calcium carbonate was increase when the size of particle is small. This was due to small sized particles have a high surface area to collision that enhance the formation of product. But these facts had its own limit on this experiment in this case the maximum results was obtained at 110 µm. There were minimum results were obtained at small sizes. Therefore, for synthesis of calcium carbonate the yield was low if the size was reduced below 150 µm. The particle size was highly affect the yield of calcium carbonate next to temperature and the effects particle size on the yield of calcium carbonate is clearly shown on the figure 4.

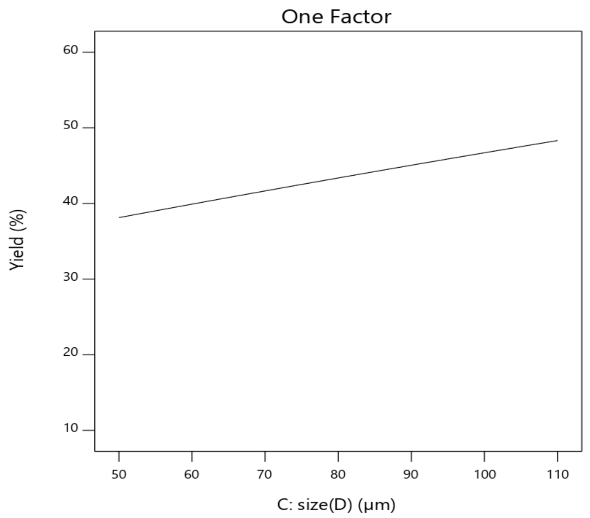

Figure 4. Effects of diameter on the yield of calcium carbonate

The interaction effects of parameter

Not only single factors affect the yield of calcium carbonate the interaction effects also. In calcium carbonate synthesis the interaction effect were significantly affect the yield. From table 6 the interaction effect of each factor was determined. The interaction effects analyzed based on the table 6 the interaction effects between temperature and size of particle negatively affects the yields. Similarly, the interaction effects between temperature and ratios affects the yields of calcium carbonate synthesized was negatively. But the interaction effects between the size of particle and ratios positively affect the yield of calcium carbonate.

Determine the effect of each parameter

In this section to determine which factors were significantly affect the yields of calcium carbonate. The factor which has high coefficients was significantly affects the yield of calcium carbonates and the factor which has small coefficients was slightly affect the yield of calcium carbonate. Positive coefficients positively affect the yield and vice versa.

Each factors have its own effects on the yield of calcium carbonate, this was determined by the coefficients of each factors. Temperature had significant effects on a yield of calcium carbonate, then the size or diameter of the particle had second significant effects on the yield of calcium carbonate, next to diameter of the particle the ratios had third significant effects on the yield of calcium carbonate. Based on these coefficients the governing equation was developed by using each factor and its interaction effects. Then equation 7 represents model equation to determine the yield of calcium carbonate by using different parameters and interaction effects.

$$Y(\%) = 33.99 + 11.28A + 4.90B + 5.10C - 0.2650AB - 0.0150AC + 0.0650BC - 0.3024A^2 - 0.3863B^2 - 0.1480C^2 \quad .... 7$$

The positive coefficient indicates that factors had a positive relation with the yield and negative coefficients had a negative relation with the yield of calcium carbonate synthesized. The slop of this equation was 33.99 this indicates that the slop value was obtained at center points. To get the good results the experiment were conducted above the center points the results value was above slop.

Table 6. Coefficient of each factor

Factor	Coefficient Estimate
Intercept	33.99
A-Temperature	11.28
B-Ratio	4.90
C-size(D)	5.10
AB	-0.2650
AC	-0.0150
BC	0.0650
A^2	-0.3024
B^2	-0.3863
C^2	-0.1480

The three dimenssional (3D) representation of optimum results

As shown in figure 5 the three dimensional representation for the yield of calcium carbon synthesized in condition the size of particle is kept constant and two variables were varied those were temperature and ratio. These two factors affect the yield of calcium carbonate positively. The synthesis of calcium carbonate is favor endothermic reaction thus the temperature increased the yield was also increased. During the reaction the temperature was increased the kinetic energy of the particle was increased it initiates the particles collision. From three dimensional representation the both temperature and ratio were increased the yield of calcium carbonate also increased.

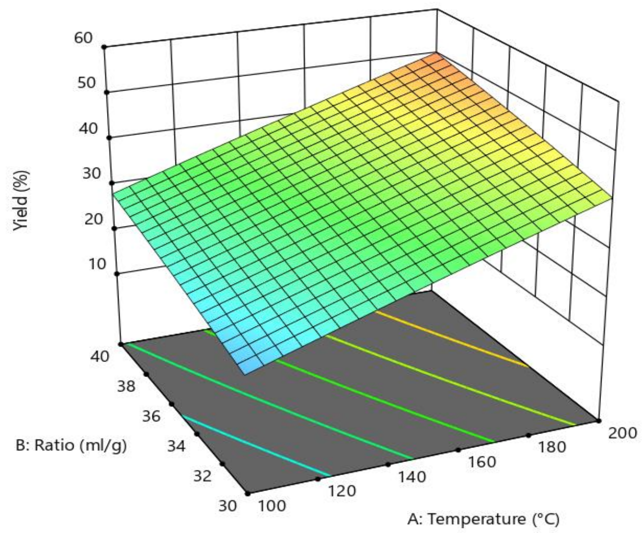

Figure 5. The yield of calcium carbonate at a constant size.

From figure 6 the yield of calcium carbonate increased if both temperature and diameter of the particle were increased. Both the temperature and diameter of the particle significantly affect the yield of calcium carbonate at a constant ratio. From the previous section discusses the reaction process was the endothermic reaction it favors the product formation when the temperature was increased. Also, the diameter of the particle was significantly affects the yield of calcium carbonate. The size of the sample was highly reduced the reaction rate was faster than coarse material. The reason was the size of the sample reduced more and more the surface area for the reaction was increased the collision between particles was increased.

Characterization of egg shell calcium carbonate

The pH of Calcium Carbonate was determined. The calcium carbonate was dissolved in distilled water. The PH of the product was determined by using PH meter by immersing it in the solution. According to the observation the PH meter reads 8.623. from the PH results the product was basic media. The specific gravity calcium carbonate was determined by using 50 ml of water was measured to dissolve calcium carbonate that produced in a laboratory. Then 25.60 gram calcium carbonate was dissolved in above 50ml distilled water. Then, total volume of the dissolved solution was reached to around 60 ml. after this it is easy to measure the specific gravity of calcium carbonate by determiningg the density and other parameter related with the specific gravity. Equation 8 and 9 is used for determine the density and specific gravity of calcium carbonates. Table 7 and 8 to determine and identify the chemical and physical properties of calcium carbonate in a laboratory scale.

$$Density\ of\ = \frac{mass\ of\ \mathrm{CaCO_3}}{volume\ of\ \mathrm{CaCO_3}} \ldots\ldots\ldots\ldots\ldots 8$$

$$= \frac{25.6\mathrm{g}}{(60-50)ml}$$

$$= 2.56\mathrm{g/ml}$$

$$\text{Specific gravity of } \mathrm{CaCO_3} = \frac{density\ of\ \mathrm{CaCO_3}}{density\ of\ water} \ldots\ldots\ldots 9$$

$$= \frac{2.56\ \mathrm{g/ml}}{1g/ml}$$

$$= 2.56$$

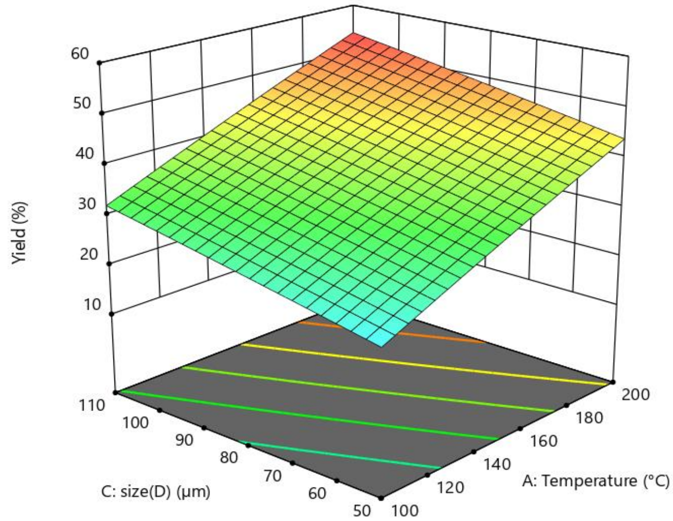

Figure 6. The yield of calcium carbonate at a constant ratio

Table 7. Table showing physical property characterization of calcium carbonate

Sl. No.	Physical properties	Egg shell calcium carbonate
1	Color	White powder
2	Odor	Odorless
3	Taste	-
4	Appearance	Solid powder
5	pH	>8.3
6	Specific gravity	2.56

Table 8. Experimental results of chemical properties of egg shell calcium carbonate

Sl. No.	Chemical properties	Egg shell calcium carbonate
1	Reactivity in water	Non-reactive
2	Reactivity in weak acid (CH_3COOH)	Partially reacted
3	Reactivity in strong acid (HCl)	Completely reacted

Conclusions

This study was intended to study the influence of different factors like temperature, diameter and amount of water on the quantity and quality of eggshell calcium carbonate. Variability of these operating conditions is the predominant factor for the quantity and quality of the product yield. There are two different methods of eggshell calcium carbonate synthesizing. In this research, to synthesize calcium carbonate the dry eggshell was used. Based on the analysis of the experimental result obtained, the optimal yield of calcium carbonate synthesized from eggshell was found to be 54% at 200°C, 110 μm and 40 ml/g ratio. This optimization of the appropriate temperature, diameter and amount of water were determined using design of expert which needs to have a consideration to get the maximum amount of product. Based on experimental data analysed the three factors had significantly affect the yield of calcium carbonate and also positive relation.

Conflict of interest

Authors declared no conflict of interests.

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