2nd International Conference Eco-Innovation in Science, Engineering, and Technology
Volume 2021
http://dx.doi.org/10.11594/nstp.2021.1401

NST PROCEEDINGS

OPEN ACCESS

Conference Paper

Synthesis of Magnesium Carbonate with Bittern Raw Material with Carbon Dioxide Gas Injection in Packing Column

Kindriari Nurma Wahyusi*, Agnes Serlina Ferdian, Martin Anderson Panangian, Ika Nawang Puspitawati

Department of Chemical Engineering, Universitas Pembangunan Nasional "Veteran" Jawa Timur, Surabaya 60294, Indonesia

*Corresponding author:
E-mail: kindrinurma@gmail.com

ABSTRACT

Magnesium carbonate is known as magnesite, which is naturally a carbonate of magnesium compounds, the main raw material for magnesium oxide (magnesia). Magnesium carbonate has many uses, one of which is an additive in the pharmaceutical field used as an additive for laxatives. In this research, The making of magnesium carbonate is the formation of magnesium hydroxide by mixing bittern and sodium hydroxide 1,875 N then reacting magnesium hydroxide and carbon dioxide gas in a packed coloum reactor. This study aims to determine the best flow rate and contact time ratio, the factors that influence, and the amount of magnesium carbonate formed. The variables used are carbon dioxide gas flow rate and contact time. The best results from this study were obtained with a flow rate of 2 liters/minute with a contact time of 30 minutes obtained 72% magnesium carbonate levels.

Keywords: Magnesium carbonate, bittern, carbon dioxide gas, packed column reactor

Introduction

Magnesium carbonate is known as magnesite, which is naturally a carbonate of magnesium compounds, the main raw material for magnesium oxide (magnesia). Magnesite can be divided into two based on its crystal form, namely macrocrystalline and cryptocrystalline. Cryptocrystalline is a crystal with higher purity than macrocrystalline, but cryptocrystalline is found less in nature than magnesite in the form of macrocrystalline crystals.

The process of magnesium carbonate, the main ingredients used are magnesium oxide obtained from bittern and sodium hydroxide. Bittern is a concentrated liquid obtained from the remaining crystallization of the salt-making process. Bittern contains various minerals, both macro minerals, and micro minerals. This mineral occurs because it does not crystallize during the manufacture of salt. Furthermore, to convert it into magnesium carbonate, namely by flowing it with carbon dioxide gas which is flowed using a packed column.

The high content of Mg deserves to be considered as a raw material for the manufacture of MgCO3. In this study, magnesium was extracted from bittern in magnesium hydroxide by adding NaOH to the bittern. After obtaining Mg (OH)2 then injected with carbon dioxide gas to form MgCO3.

Magnesium carbonate is a magnesium salt with the formula MgCO3. The hydrated forms, mainly di-, tri-, and tetrahydrate, occur as minerals. It has a role as an antacid and fertilizer. These are magnesium salts, carbonate salts, and one-carbon compounds. When heated to decomposition, it emits acrid fumes and irritating carbon dioxide fumes. (Pubchem, 2019).

Bittern is a concentrated liquid obtained from the remaining crystallization of the salt-making process. This mineral occurs because it does not crystallize during the manufacture of salt. Bittern

How to cite:
Wahyusi, K. N., Ferdian, A. S., Panangian, M. A., & Puspitawati, I. N. (2021). Synthesis of magnesium carbonate with bittern raw material with carbon dioxide gas injection in packing column. *2nd International Conference Eco-Innovation in Science, Engineering, and Technology*. NST Proceedings. pages 1-6. doi: 10.11594/ nstp.2021.1401

contains various minerals, both macro minerals, and micro minerals. Of the many minerals contained in bittern, several minerals have high concentrations, namely: Magnesium (Mg), Sodium (Na), Potassium (K), and Calcium (Ca) (Hapsari, 2008).

Table 1. Physical and chemical properties of magnesium carbonate

Physical and Chemical Properties of Magnesium Carbonate	
Molecular Formula	$MgCO_3$
Molecular Weight	84.313 g/mol
Density	2.96 g/cm^3
Solubility in Water	0.1g/L
Boiling Point	Decomposed
Melting Point	662° F
Vapor Pressure	0 mm Hg
Flavor	No Flavor
Colour	White
Form	Powder

In addition, bittern can also reduce heavy metals in liquid waste, especially Nickel, which can be reduced to 18.5%. Another benefit of bittern is to treat domestic wastewater and alkaline industrial wastewater (Sutiyono, 2006).

Factors that affect absorption performance in packed columns include:

a. Physical properties of the solvent: Viscosity does not affect mass transfer, and surface tension can affect the reaction rate in certain cases. Lower surface tension results in increased dispersion and tends to maximize the effective area of the packing. It indicates that packing with a higher surface area is expected to experience a greater increase in effective mass transfer than packing with a lower surface area under conditions of low surface tension.
b. CO_2 partial pressure or total system pressure: According to Bailey and Feron, a higher CO_2 content in the gas stream results in a higher driving force for absorption
c. Gas flow rate CO_2: The higher the gas velocity in the packing column, the longer it takes for the solvent to make direct contact between the liquid and gas phases, resulting in a decrease in the liquid flow rate.
d. Loading of Solvent CO2: If the amount of CO2 carried is high, the mass transfer force from gas to a solution will be small.
e. Packing: Packing plays a role in creating gas-liquid contact during the CO2 absorption process. Generally, the volumetric mass transfer coefficient increases with an increase in surface area. However, the surface area of the packing should not be a criterion for higher mass transfer.

A packed column is applied to improve the contact of the gas and liquid phase. Packing selection is done by considering the following: a) having a large wetted surface area per unit volume, b) Having a large enough free space so that the pressure loss is small, c) Good wetting characteristics, d) Density is so that the overall column weight is small., e) Corrosion-resistant and economical. Several types of packing that are often used include ranching rings, intalox saddles, and pall rings (Winasis, 2018).

The manufacturing process of MgCO3 differs based on different raw material routes. The process can be divided into:

a. Basic brine carbonization method: Dolomite or limestone being calcined, then it is flushed and reacted with brine to obtain precipitated magnesium hydroxide. This method requires brine, seawater, and other magnesium chloride solutions, magnesium sulfate solution as material, calcined with dolomite or limestone to produce CO2, dolomite lime or lime, and then digest carbonization, pyrolysis to obtain light grade magnesium carbonate. Magnesium Carbonate is obtained through carbonization, pyrolysis, washing, dehydration, dry and crushing processes. The chemical reaction occurs as follows

Carbonization:

$Mg(OH)_2 + 2\ CO_2 \rightarrow Mg(HCO_3)_2$

Pyrolysis:

$5Mg\ (HCO_3)_2 \rightarrow 4MgCO_3 \cdot Mg(OH)2 \cdot 4H2O + 6\ CO_2 \uparrow$

b. Classify magnesium carbonate through the magnesite: Carbonization method Put magnesite (particle size 100-300mm) and anthracite (calorific value 20000-25000 KJ/KG) into calcination kiln proportionally (7-10): 1. Temperature control at 200-250 degrees during pre-heating, 700- 800 degrees during calcination, and 100-150oC during refrigeration. Remove kiln gas from kiln crown; CO2 concentration is more than 28%. Get the burning magnesia from the kiln bottom. The broken magnesium reacts with water to get Mg(OH)2, then the carbonation reaction with CO2 gets magnesium bicarbonate. Then through precipitation, separation, pyrolysis, filter to obtain light magnesium carbonate. The chemical equation is as follows:

Calcine $MgCO_3 \rightarrow MgO + CO_2 \uparrow$

Digestion $MgO + H_2O \rightarrow Mg(OH)_2$

Carbonization $Mg(OH)_2 + 2CO_2 \rightarrow Mg\ (HCO_3)_2$

Pyrolysis $5Mg\ (HCO_3)_2 \rightarrow 4MgCO_3 \cdot Mg(OH)_2 \cdot 4\ H_2O + 6\ CO_2 \uparrow$

c. Asbestos tailings carbonization method: The chemical components in asbestos tailings are similar to dolomite. Different proportions of calcium and magnesium in different tailings. In some tailings, MgO ≦ 20%, CaO ≦ 29% but SiO2≧ 20%, LOI ≦ 30%. The energy consumption of the unit product is higher than that of the dolomite carbonization method. So, It is best to clean the solution in HCl when taking this method to produce magnesium carbonate.

The chemical equation is as follows:

C $MgCO_3 \cdot CaCO_3 \rightarrow MgO \cdot CaO + 2CO_2 \uparrow$

Digestion $MgO \cdot CaO + 2\ H2O \rightarrow Mg(OH)2 + Ca\ (OH)\ 2$

Carbonization $Mg(OH)_2 + Ca(OH)_2 + 3CO2 \rightarrow Mg(HCO_3)2 + CaCO_3 \downarrow + H_2O$

Pyrolysis $Mg\ (HCO_3)\ 2 \rightarrow 4\ MgCO_3 \cdot Mg(OH)_2 \cdot 4H_2O + 6\ CO_2 \uparrow$

Application

According to different applications of magnesium carbonate, it can be divided into industrial-grade MgCO3, food-grade MgCO3, medical-grade MgCO3, transparent MgCO3, electrical MgCO3 grade, pumped MgCO3, and aciculiform MgCO3 (Liangjiang. 2019). The global (surface) concentration of carbon dioxide (CO2) in the atmosphere has increased since the industrial revolution due to the rapid growth of human activities. Currently, there is sufficient scientific evidence to show that the increasing concentration of CO2 in the atmosphere is the main cause of global change and climate change. Carbon dioxide is a chemical compound consisting of two oxygen atoms covalently bonded to a carbon atom. CO2 is a gas at standard temperature and pressure and is present in the atmosphere. The average concentration of carbon dioxide in the atmosphere is approximately 387 ppm by volume, although this can vary depending on location and time of day. (Astuti, 2017).

Sodium hydroxide is the most widely used strong base in the chemical industry. The form of sodium hydroxide is a white, odorless, crystalline solid at room temperature. Sodium hydroxide is a very corrosive and toxic substance and is called caustic soda or lye. Sodium hydroxide is used as an alkali in producing various products, including detergents, paper, synthetic fabrics, cosmetics, and pharmaceuticals (Sese, 2018).

Atomic Absorption Spectroscopy is a tool used in analytical methods to determine metallic elements and metalloids whose measurements are based on light absorption with a certain wavelength by metal atoms in a free state. This method is very appropriate for the analysis of substances at low concentrations. This technique has several advantages over conventional emission spectroscopy methods. That method is based on the absorption of light by atoms; the atoms will absorb the light at certain wavelengths depending on the nature of the elements. The Lead (Pb) absorbs light at a wavelength of 217 nm Light at this wavelength has the electronic level of an atom. The electronic transition is specific by absorption of energy, meaning to gain a lot of energy. Suppose light with a certain wavelength is passed through a cell containing free atoms. In that case, some light will be absorbed, and the absorption intensity will be directly proportional to the number of free metal atoms in the cell. This Atomic Absorption Spectroscopy (AAS) method is based on the absorption of light by atoms. The atoms absorb the light at certain wavelengths, depending on the properties of the elements. Light at a certain wavelength has enough energy to change the electron level of an atom. The absorption of energy means that an atom in the ground state is increased in the excited state.

Each wavelength produces sharp spectral lines with maximum intensity, usually called resonance lines. The atomic spectrum for each element consists of resonance lines. Other lines that are not resonance lines can be spectra associated with the energy level of the molecule, usually broad bands.

This method is very appropriate for analyzing substances at low concentrations and has several advantages, such as having high sensitivity. In addition, elements with low excitation energies can be analyzed by flame photometry but are not suitable for high excitation energies. Flame photometry has an optimum size range at a wavelength of 400–800 nm. While Atomic Absorption Spectroscopy (AAS) has an optimum size range at a 200-300 nm wavelength (Kumalawati, 2016).

Material and Methods

This research was conducted at the Research Laboratory of the Chemical Engineering Department, University of UPN Veteran Jawa Timur, Surabaya. The tools used in the study included carbon dioxide gas cylinders, magnetic stirrer, analytical balance reactor packed column. The materials used in this research are bittern, carbon dioxide gas, and sodium hydroxide.

The formation of magnesium carbonate begins with the stage of making magnesium hydroxide by mixing 150 ml of bittern with sodium hydroxide 1.875 N using a magnetic stirrer at a speed of 300 rpm for 30 minutes at a temperature of 25-30 C until a precipitate forms. The magnesium hydroxide solution obtained is allowed to stand for 24 hours. Later, it will be filtered and washed 5 times to reduce the Na content in the solution. Then the residue obtained is dried using an oven at 100ºC. For the magnesium carbonate manufacturing stage, the magnesium hydroxide precipitate obtained was dissolved in 700 ml of distilled water using a magnetic stirrer at a speed of 100 rpm at a temperature of 100 ºC for 1 hour, then prepared a series of packed column reactors and connected to a carbon dioxide gas tube, the packed column reactor will fill with magnesium hydroxide solution that has been dissolved then the packed column reactor is flowed with carbon dioxide gas with a predetermined flow path for a predetermined time as well. Then the solution that has been injected with carbon dioxide gas is filtered with filter paper, the filtrate will be discarded, and the residue will be dried using an oven with a temperature of 110 ºC. This study uses a variable flow rate of carbon dioxide gas and time. The flow rate of carbon dioxide used is 1000 ml/minute, 1500 ml/minute, 2000 ml/minute, 2500 ml/minute, 3000 ml/minute, and the contact time used is 10 minutes, 15 minutes, 20 minutes, 25 minutes, 30 minutes. The magnesium carbonate content was determined using Atomic Absorption Spectroscopy (AAS).

Results and Discussion

From the analysis result, the relation between the Magnesium carbonate content and the carbon dioxide flowrate at the various contact time can be seen at figure 1

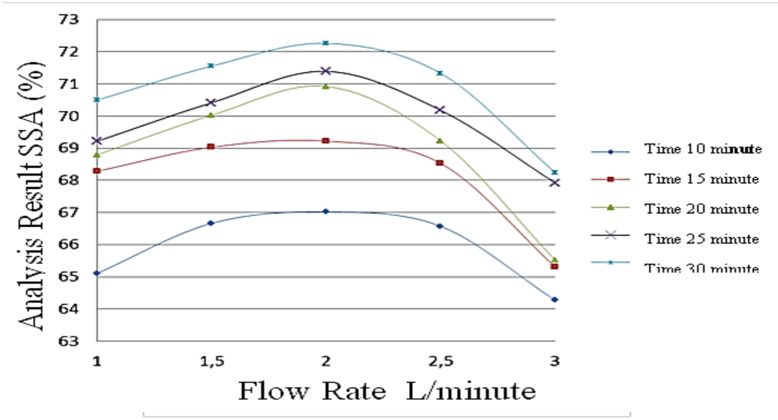

Figure 1 Effect of flow rate on the results of AAS analysis

Figure 1 shows that that the highest $MgCO_3$ analysis result is at 30 minutes with a flow rate of 2 L/minute with an AAS analysis result of 72.26 percent. From the results obtained, it can be said that this flow rate is the optimum condition. The carbon dioxide gas has been saturated when it reacts with a solution of magnesium hydroxide resulting in the formation of a supersaturation atmosphere. This situation will accelerate the deposition of magnesium carbonate so that after optimum conditions will eliminate the supersaturation atmosphere. The yield slowly decreases in the flow rate of 2.5 liters/minute and 3 liters/minute. However, with each addition of the flow time, the higher the analysis results obtained.

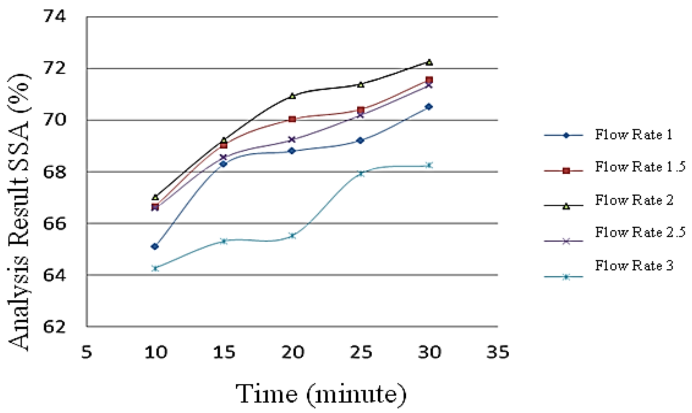

Figure 2Effect of time on the results of AAS analysis

In figure 2, it is found that the highest $MgCO_3$ analysis result is at 30 minutes with a flow rate of 2 L/minute with an SSA analysis result of 72.26 percent. From the results obtained, it can be said that this flow rate is the optimum condition. So that the carbon dioxide gas has been saturated when it reacts with the magnesium hydroxide solution resulting in the formation of a supersaturation atmosphere will accelerate the deposition process of magnesium carbonate. In addition, the column filling (Rasching) will expand the contact surface and prolong the contact time so that a precipitate occurs. But the longer the time. However, every time the flow is added, the analysis results get higher

Conclusion

The manufacture of magnesium carbonate from bittern by injection of carbon dioxide gas can be obtained from raw materials containing magnesium such as bittern. Based on the tests that have been carried out, bittern contains MgCl2 32.63% and MgSO4 10.43%. Based on flow rate can be obatained the best result is at 30 minutes with a flow rate of 2 L/minute with an SSA analysis result of 72.26 percent.

Acknowledgment

The manufacture of magnesium carbonate from bittern by injection of carbon dioxide gas can be obtained from raw materials containing magnesium such as bittern. Based on the tests that have been carried out, bittern contains MgCl2 32.63% and MgSO4 10.43%. Based on the flow rate that can be obtained the best result is at 30 minutes with a flow rate of 2 L/minute with an SSA analysis result of 72.26 percent.

References

Astuti. (2017). *Analisis kandungan CO_2 dengan Sensor dan berbasis logger pro di daerah Yogyakarta*. Jakarta Timur: Indraprasta PGRI

Hapsari. (2008). *Pengambilan mineral elektrolit dari limbah garam (bittern) untuk suplemen mineral ionic pada air minum.* Surabaya: UPN Veteran Jawa Timur

Kumalawati, O. R. (2016). *Analisis kadar logam timbal (Pb) pada bedak tabor dengan variasi zat pengoksidasi dan metode destruksi basah menggunakan Spektroskopi Serapan Atom (SSA)*. Undergraduate thesis, Universitas Islam Negeri Maulana Malik Ibrahim.

Liangjiang. (2019). *MgCO3 CAS No 2090-64-4 Magnesium carbonate untuk berbagai bidang* m.indonesian.lj-group.com/ -industrial-grade-mgco3-cas-no-2090-64-4-magnesium-carbonate-formanyfields.html. 26 Desember 2020.

Pubchem. (2019). Chemical and physical properties. https://pubchem.ncbi.nlm.nih.gov/compound/magnesiumcarbonate#.(27 Desember 2020)

Sese,N. M. (2018). *Pengaruh konsentrasi aktivator NaOH pada proses aktivasi kimia terhadap kualitas karbon aktif dari kulit singkong*. Samarinda: Politeknik Negeri Samarinda

Sutiyono. (2006). *Kinetika reaksi pembentukan magnesium sulfat dari bittern dan asam sulfat*. Surabaya: UPN Veteran Jawa Timur

Winasis, Y. S. (2018). *Produksi amorphous precipitated silica berbahan baku abu bagasse dan gas CO_2 pada Kolom Berpacking*. Surabaya: UPN Veteran Jawa Timur