WHITENING OF CALCIUM CARBONATE TAILINGS USING ATTRITION PROCESS: A CASE STUDY OF KHOKTOOM DEPOSIT, LOPBURI PROVINCE, THAILAND

Mr. Bancha Wongkaewphothong

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| Thesis Title | WHITENING OF CALCIUM CARBONATE TAILINGS USING ATTRITION PROCESS: A CASE STUDY OF KHOKTOOM DEPOSIT, LOPBURI PROVINCE, THAILAND |
| By | Mr. Bancha Wongkaewphothong |
| Field of Study | Georesources and Petroleum Engineering |
| Thesis Advisor | THEERAYUT PHENGSAART, Ph.D. |

Accepted by the FACULTY OF ENGINEERING, Chulalongkorn University in Partial Fulfillment of the Requirement for the Master of Engineering

-------------------------------------------- Dean of the FACULTY OF ENGINEERING

(Professor SUPOT TEACHAVORASINSKUN, D.Eng.)

THESIS COMMITTEE

-------------------------------------------- Chairman

(Assistant Professor Apisit Numprasanthai, Ph.D.)

-------------------------------------------- Thesis Advisor

(THEERAYUT PHENGSAART, Ph.D.)

-------------------------------------------- Examiner

(Associate Professor Kreangkrai Maneeintr, Ph.D.)

-------------------------------------------- External Examiner

(Associate Professor SOMSAK SAISINCHAI)

บัญชา วงศ์แก้วโพธิ์ทอง : การเพิ่มความขาวของหางแร่แคลเซียมคาร์บอเนตด้วยกระบวนการขัดสี: กรณีศึกษาแหล่งโคกตูม จังหวัดลพบุรี ประเทศไทย . ( WHITENING OF CALCIUM CARBONATE TAILINGS USING ATTRITION PROCESS: A CASE STUDY OF KHOKTOOM DEPOSIT, LOPBURI PROVINCE, THAILAND) อ.ที่ปรึกษาหลัก : อ. ดร.ธีระยุทธ เพ็งสะอาด

แคลเซียมคาร์บอเนต ($CaCO_3$) คือหนึ่งในแร่อุตสาหกรรมที่ถูกประยุกต์ใช้อย่างหลากหลายใน อุตสาหกรรมต่าง ๆ (เช่น พลาสติก, ยาง, สี, กระดาษ) อย่างไรก็ตามผลิตภัณฑ์แคลเซียมคาร์บอเนตที่ขายได้ จำเป็นต้องมีคุณสมบัติที่ตอบสนองความต้องการของแต่ละการใช้ประโยชน์ ความขาวคือหนึ่งในคุณสมบัติที่ สำคัญโดยเฉพาะอย่างยิ่งการใช้งานที่ให้ความสำคัญเรื่องสี เช่น อุตสาหกรรมสีและกระดาษ ในการศึกษานี้ เพื่อ เก็บกลับคืนและใช้ประโยชน์แร่ที่มีคุณสมบัติไม่ตรงกับข้อกำหนด ผลกระทบของกระบวนการขัดสี (เช่น เวลาใน การขัดสี และของแข็งในของผสม) ต่อความขาวของหางแร่แคลเซียมคาร์บอเนตถูกศึกษา ตัวอย่างแคลเซียม คาร์บอเนตถูกคัดขนาดออกเป็นช่วงขนาด −300, +300–600, +600–1000, +1000–2360, +2360–4000, และ +4000 ไมโครเมตร จากการวิเคราะห์วัสดุ พบว่าอนุภาคขนาดละเอียด (−300 ไมโครเมตร) แสดงค่าความขาวที่ ต่ำที่สุด (น้อยกว่าร้อยละ 80) ในขณะที่ช่วงขนาดอื่น ๆ มีค่าสูงกว่า (มากกว่าร้อยละ 80) เพื่อเพิ่มค่าความขาว เครื่องขัดสีถูกประยุกต์ใช้เพื่อกำจัดมลทินบนผิวของแคลเซียมคาร์บอเนตในที่ร้อยละของแข็ง (ร้อยละ 40, 45, 50, 55, 60, และ 70) และเวลาในการขัดสี (10, 15, 20, และ 25 นาที) ที่สภาวะแตกต่างกัน ผลการทดลองใน ระดับห้องปฏิบัติการพบว่าวิธีการขัดสีสามารถเพิ่มค่าความขาวและลดค่าเหล็กในหางแร่แคลเซียมคาร์บอเนต สภาวะที่เหมาะสมคือร้อยละของแข็งที่ 55 ซึ่งได้ถูกนำไปใช้ในการทดลองระดับนำร่อง ผลการทดลองที่ได้ สอดคล้องกับการทดลองในระดับห้องปฏิบัติการ ค่าความขาวถูกทำให้เพิ่มจนอยู่ในระดับมาตรฐานของตลาด ดังนั้นกระบวนการ ขัดสีสามารถเพิ่มคุณภาพของหางแร่แคลเซียมคาร์บอเนตได้

จุฬาลงกรณ์มหาวิทยาลัย

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| สาขาวิชา | วิศวกรรมทรัพยากรธรณีและ ปิโตรเลียม | ลายมือชื่อนิสิต ............................................. | |
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# # 6372806921 : MAJOR GEORESOURCES AND PETROLEUM ENGINEERING

KEYWORD: Attrition scrubber, Whiteness, Yellowness, Calcite, Marble, Sandwash

Bancha Wongkaewphothong : WHITENING OF CALCIUM CARBONATE TAILINGS USING ATTRITION PROCESS: A CASE STUDY OF KHOKTOOM DEPOSIT, LOPBURI PROVINCE, THAILAND. Advisor: THEERAYUT PHENGSAART, Ph.D.

Calcium carbonate ($CaCO_3$) is one of the industrial minerals that usually use in various applications in many industries (e.g., plastic, rubber, paint, paper). However, salable $CaCO_3$ products need to meet the requirement for each application. Whiteness is one of property that really important especially the application that concern about the color like paint and paper industries. In this study, to recover and utilize out of spec ores, the effects of attrition scrubbing process (i.e., retention time and solid in pulp) on the whiteness of the $CaCO_3$ tailings were investigated. The $CaCO_3$ samples were screened to obtain −300, +300–600, +600–1000, +1000–2360, +2360–4000, and +4000 µm size fractions. From the material characterization, the fine particles (−300 µm) showed the lowest whiteness index (i.e., < 80%) while other size fractions were higher (i.e., > 80%). To increase the whiteness index, attrition scrubber was applied to remove the impurities on the $CaCO_3$ surface with difference solid percentage (i.e., 40, 45, 50, 55, 60, and 70%) and retention time (i.e., 10, 15, 20, and 25 min) as condition. The result in laboratory-scale found that the attrition scrubbing method can improve whiteness property and reduce iron content in calcium carbonate tailing. The optimum condition was at 55 solid percentage which was used in the pilot-scale experiment. The result was in line with the laboratory-scale, the whiteness was improved to meet the market standard. Therefore, the attrition scrubbing process can be used to improve the quality of calcium carbonate tailing.

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| Academic Year: | 2022 | Advisor's Signature | ............................ |

# ACKNOWLEDGEMENTS

# TABLE OF CONTENTS

Page

# LIST OF TABLES

# LIST OF FIGURES

จุฬาลงกรณ์มหาวิทยาลัย

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# CHAPTER 1 – INTRODUCTION

## 1.1    Background

Lacking mineral resources causing by the high demand of raw materials and limitation of non-renewable resources is one of serious issues in our modern society. To obtain the products with acceptable properties, the quality of raw materials used need to be controlled under the market and/or industrial standards. So, mostly high-quality ores have been used while those of unaccepted quality (i.e., tailings and/or wastes) were rejected. However, to achieve the United Nations' Sustainable Development Goals (UN-SDGs), most resources should be used properly in economic, environmental, and social viewpoints. Therefore, the quality of these materials should be improved into acceptable standard requirement to optimize the resources utilization that including in UN-SDGs #Goal 12 "Responsible Consumption and Production".

Calcium carbonate is one of the numerous minerals on the earth's plate. It is being used as raw material in various industries (e.g., cement, plastic, paint, paper, and pharmaceutical industries). The chemical grade of $CaCO_3$ requires strict quality control in properties such as whiteness, yellowness, and impurities. Mostly, the calcium carbonate raw material rather specifies grade classification by its color property than chemical composition. The premium grade with high whiteness and low yellowness index could be found in a few locations. In Thailand, it can be produced from Khoktoom deposit in Lopburi province. However, by-product which is a low-quality calcium carbonate with the particle size less than 5 mm called "sandwash" is also generated.

Besides high utilizing the resource once the recovering method of the sandwash would be achieved, it also helps the earth in other points of view. For example, the carbon emission would be decrease because there is no additional new extraction in place the unusable materials or the loss of the energy consumed through the process, but it is become to waste or unsalable goods. Furthermore, the environmental impact, there will more less than the normal if the waste or tailing can be utilized more; additionally, the waste piles could be reduced management cost and pollution generated from them could be decreased too.

In this study, the attrition scrubbing process using water was applied to improve the quality of this waste to be salable. The effects of retention time and solid percentage on whiteness and yellowness indexes were investigated. In addition, the results of laboratory- and pilot-scales were compared.

## 1.2    Objectives

1.    To investigate the effect of attrition scrubbing process on the removal of stained impurities from the calcium carbonate tailings.

2.    To optimize the conditions of calcium carbonate tailings whitening process using attrition scrubber to meet the market standard requirement of filler grade

## 1.3    Scope

In this study, the attrition scrubbing process in water was applied to improve the properties of calcium carbonate by increasing the whiteness and decreasing the yellowness. The effects of retention times (i.e., 10, 15, 20, and 25 min) and percentage of solid (i.e., 40, 45, 50 55, 60 and 70%) on the whiteness and yellowness of calcium carbonate products after the attrition scrubbing experiments (i.e., laboratory scale, and pilot scale) were investigated.

## 1.4    Expected benefits

1.    The result of this study could be applied and developed to actual whitening process for industrial minerals.

2.    The wastes in mining and processing of industrial minerals could be utilized that tend to the decreasing of amount and handing cost of waste.

3.    The outcome of this research could be a part to solve the problem relating to the decreasing of mineral resources, increasing of demand, and higher waste generation that including in the UN-SDGs: Goal 12 "Responsible Consumption and Production".

## 1.5 Research plan

The plan of this study is shown in **Table 1.1**.

**Table 1.1** The plan of this study

| Activities | 2022 | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Jan | Feb | Mar | Apr | May | Jun | Jul | Aug | Sep | Oct | Nov | Dec |
| 1. Specific of an interesting topic for study | ■ | | | | | | | | | | | |
| 2. Defining the objective, expected benefit, and scope of the study | ■ | | | | | | | | | | | |
| 3. Literature reviews | | | ■ | ■ | | | | | | | | |
| 4. Experiment methodology design | | | | ■ | ■ | | | | | | | |
| 5. Sample characterization | | | | | ■ | | | | | | | |
| 6. Pre-liminary experiments | | | | | | ■ | ■ | ■ | ■ | | | |
| 7. Optimization | | | | | | | | ■ | ■ | ■ | | |
| 8. Result discussion | | | | | | | | ■ | ■ | ■ | ■ | |
| 9. Publication, writing, and defense | | | | | | | | | | | ■ | ■ |

## 1.6    Research flowchart

**Figure 1.1** shows the flowchart described the procedures of this research. The study started with topic of study selection then, objective, scope, and expected benefit were defined. Literature review as well as the sample preparation and characterization were conducted for experimental methodology design. The attrition scrubbing experiments were carried out both laboratory scale and pilot scale. The obtained results were compared and discussed then, the findings of this research were concluded.
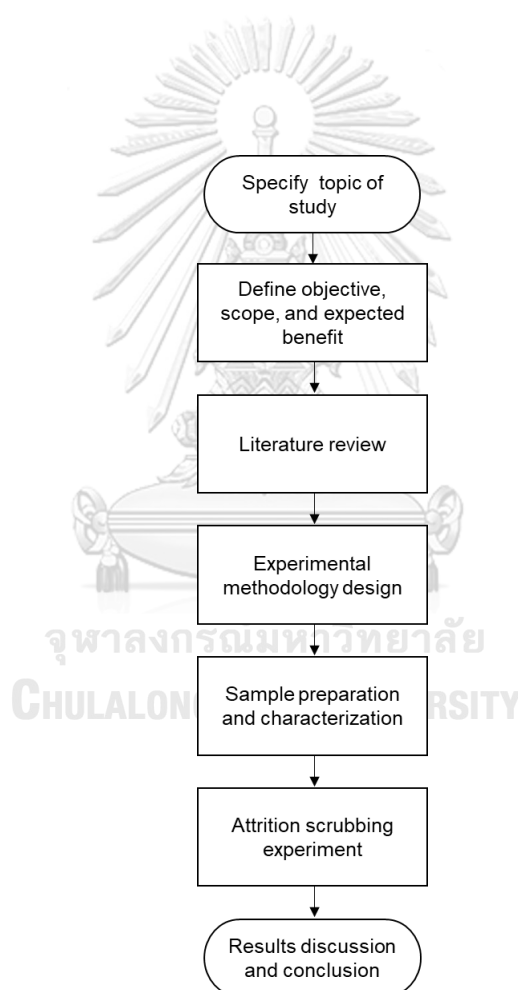


**Figure 1.1** Schematic flowchart of the present study.

## 1.7    Order of presentation

In order to present this research and make it easy to understand for reader, the author has divided this research into 5 following chapters:

**Chapter 1** Introduction: background; objective; scope; expected benefit; research plan; and research flowchart.

**Chapter 2** Literature review: mineralization of calcium carbonate; calcium carbonate deposits in Thailand; calcium carbonate deposits in Lopburi; calcium carbonate production; specification of calcium carbonate in the market; color property standard; and the beneficiation for the elimination of stained mineral surface.

**Chapter 3** Materials and methods: sample preparation and characterizations; and attrition scrubbing experiments

**Chapter 4** Results and discussions: material characterization; and attrition scrubbing experiment results

**Chapter 5** Conclusion: conclusion; and recommendation.

## CHAPTER 2 - LITERATURE REVIEW

### 2.1    Mineralization of calcium carbonate

Calcium carbonate ($CaCO_3$) is found in the earth more than 4% (Ortega-Villamagua et al., 2020). It is usually formed by the sedimentation of the shell of the small fossils (e.g., snails, shellfish, and coral) (Omari et al., 2016). It is generally found in various form of minerals and rocks together with the impurities like iron, carbonate, iron oxides and sulfides, silica, alumina, clay, carbonaceous matter, sulfur, and phosphorous (Sezer, 2013; Smith, 1915).

Calcium carbonate is found in various from of three rock types as explained below.

### (1) Sedimentary rock

In sediment conditions, calcium carbonate could be found in from of limestones and chalk. Limestone is a natural stone containing calcium carbonate in the majority, and also occurs with other impurities such as quartz, and ferromagnetic minerals, and phosphate. However, in some aspects, it also contained magnesium (Mg) that come from the replacement of dissolved ion of Mg and calcium (Ca) in sea water. This process is called as dolomitization (Farkas et al., 2012; Tegethoff et al., 2001).

Furthermore, calcium carbonate minerals in sedimentary deposits also present in other forms such as:

(i)    **Chalk**, a porous calcium carbonate mineral with fine particle and low density. Its formation is occurred by coccoliths on the seabed sedimented and gradually consolidated by pressure, that cause water removal from the structure. It could also form via the

uncompleted formation of limestone with less or non-cementation properties (Tegethoff et al., 2001).

(ii) **Marl** is a non-solidified material containing shell and sand. Sometimes, it is formed together with limestone, called as "marly limestone" (Tegethoff et al., 2001).

**(2) Igneous rock**

Calcium carbonate, called as "carbonatites", is found in volcanic rocks which is formed by hydrothermal fluid bearing with sulfide dominant ores and/or the interaction with seawater during the lava cooling (Morichita, 2012; Sklyarov et al., 2017). carbonatites can also originate from magmatism contribution in both conditions of intrusion and extrusion while carbonatite derived carbonate has been formed inside of the volcanic rock (Stoppa, 2021).

**(3) Metamorphic rock**

Marble has a similar chemical composition as sedimentary limestone or dolomite which has changed calcite crystal pattern by recrystallization in a metamorphic process. The process occurs by the high temperature and high pressure when limestone contact to the molten mantle underneath the earth's crust so, the interlocking calcite crystal marble is formed (Li et al., 2014; Tegethoff et al., 2001). Moreover, it also can be found from hydrothermal veins contact (Klein & Philpotts, 2017), or it also found in caverns (Davies & Morgan, 1991). In the market, it is also called as "calcspar" and/or "Iceland spar" (Bhavan, 2020).

### 2.2 Calcium carbonate deposits in Thailand

The information about the deposits of the most general sources of calcium carbonate like limestone and marble are described below.

### (1) Limestone

Limestone deposits are found in all regions of Thailand. All rock formation ages are varied from Precambrian until Quaternary. Most of the limestone occurrences are dominated Permian and Ordovician ages (Kuentag, 1983).

Kuentag (1983) reported the limestone deposit in Thailand can be classified based on their geological time scales as below.

- Precambrian: Pranburi district, Prachup Khiri Khan province
- Ordovician: Muang Chonburi district, Chonburi province; Ko Sichang district, Chonburi province; Thung Song distric Nakhon Sithammarat province
- Devonian-silurian: Muang Nakhon Sawan district, Nakhon Sawan province; Ban Dan Lan Hoi district, Sukhothai province
- Permo-carboniferous: Muang Saraburi district, Saraburi province; Kaeng Koi district, Saraburi province; Phra Phuttabat district, Saraburi province; Muang Loei district, Loei province; Phanat Nikhom district, Chonburi province; Muang Kanchanaburi district, Kanchanaburi province; Kaset Sombun district; Chaiyapum province; Ta Khli district, Nakorn Sawan province; Muang Ratchaburi district, Ratchaburi province; Pak Tho district, Ratchaburi province; Klaeng district, Rayong province; Sa Kaeo district, Prachinburi province; Pakphraek district, Kanchanaburi province
- Triassic: Muang Lampang district, Lampang province; Klaeng district, Rayong province
- Quaternary: Tha Muang district, Kanchanaburi province; Ban Mo district, Saraburi province

**(2) Marble**

Department of Mineral Resources (DMR, 2002) reported the marble deposits of Thailand can be summarized as main locations of deposits and minerals characteristics as below.

- Thung Salium, Sukhothai; white, yellowish-white, pale grey, and pinkish-white marble are found.
- Pran Krathai, Kampaeng Phet; white, pale grey, and pinkish-white marble that found along Khao Pra mountain, Khao Sawang Arom mountain and Khao Kong Ruen mountain.
- Mae Phrik, Lampang; yellowish white, pale grey, creamy white and pale green marble that found along Doi Phom Nam mountain.
- Thern, Lampang; pinkish white, pale grey and banded creamy white marble that exposed as mountain and underground.
- Khoktoom, Lopburi; white to creamy marble found as underground marble deposits.
- Phra Putta Bat, Saraburi; white, light grey and dark grey marble.
- Thong Saen Kan, Uttradit, white, grey, and greenish-grey marble that found as limestone interbedded marble mountain.
- Hua Hin, Prachuap Kirikan; very large grain crystal, creamy white, greenish-white, and brown-banded marble.

## 2.3 Calcium carbonate deposits in Lopburi

In Lopburi, massive lower-middle permian limestone is the general type of carbonate rock. The thickness is more than 865 m. overlying the sandstone and shale formations, respectively. These regional rocks (i.e., limestone, sandstone, and shale) contact with intruded diorite in Paleozoic age. So, the limestone closed to these

contact zones was recrystallized and metamorphosed to marble, while sandstone and shale have become quartzite, and hornfels, respectively (Shawe, 1984).

Department of Primary Industries and Mines (DPIM, 2020) reported that high-grade calcium carbonate could be produced in Lopburi province that is mostly from marble.

## 2.4    Calcium carbonate production

### 2.4.1    Calcium carbonate mining and quarrying

Most calcium carbonate is mined using surface mining method while only few underground mining also available (Newman, 2020). In Thailand, all calcium carbonate mines including limestone, calcite as well as decorative marble are mined via surface mining. Annually, about 1.44 million tons of calcium carbonate are produced in Thailand with steady amounts during 2015 to 2019. Department of Primary Industries and Mines (DPIM, 2020) report that the consumption of calcium carbonate in domestic market is only from Lopburi.

### 2.4.2    Calcium carbonate processing

After mining or quarrying, the run-of-mine is processed by crushing, grinding, screening, and classification to produce the products with required size fractions. In the market, the calcium carbonate products could be divided into two types as below.

(1) **Ground calcium carbonate (GCC)**: GCC is extracted from natural minerals and/or rocks (Renaudin et al., 2007). The pulp paper industry is the largest consumer of GCC which use more than 2.5 million tons annually (Tegethoff et al., 2001).

(2) **Precipitated calcium carbonate (PCC)**: PCC is synthetical calcium carbonate that is produced from lime (CaO) that is the product from

natural calcium carbonate via the calcination process (Eq. 1). The lime could be turned into hydrated lime ($Ca(OH)_2$) by the reaction with water as shown in Eq. 2. Then, PCC could be produced by passing carbon dioxide ($CO_2$) into a solution of hydrated lime. This process is called as the "re-carbonation process" as the chemical reaction as shown in Eq. 3 (Jimoh et al., 2016).

$$CaCO_3 \text{ (s) + Heat} \rightarrow CaO \text{ (s)} + CO_2 \text{ (g)} \qquad \text{.... (1)}$$

$$CaO \text{ (s)} + H_2O \text{ (aq)} \rightarrow Ca(OH)_2 \text{ (aq)} \qquad \text{.... (2)}$$

$$Ca(OH)_2 \text{ (aq)} + CO_2 \text{ (g)} \rightarrow CaCO_3 \text{ (s)} + H_2O \text{ (aq)} \qquad \text{.... (3)}$$
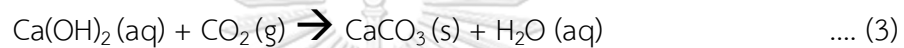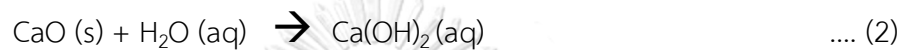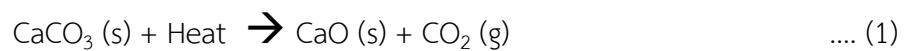
GCC is used in several proposes such as the flooring, paint, and paper due to its mechanical and optical properties (Industrial Minerals Association - North America, 2022). The quality of PCC is less varying in properties (i.e., whiteness, yellowness, and purity) but it cost is higher than GCC in the same grade.

### 2.4.3 Calcium carbonate wastes

The mining and size reduction processes generally generate calcium carbonate by-product and wastes with a variety in their qualities (i.e., whiteness, yellowness, and purity) (Marras et al., 2010). Calcium carbonate by-products with high qualities (i.e., high whiteness, low yellowness, and high purity) are saleable as chemical grade (e.g., white cement), filler grade (e.g., painting), and construction materials (e.g., aggregate) (Baghel et al., 2020; Patel, 2017; Saygili, 2015). While calcium carbonate wastes could not be sold due to it low qualities (i.e., low whiteness, high yellowness, and low purity). However, these qualities are able to be

improved to meet the market standard requirement (section 2.5 and 2.6) using mineral processing that will be explained later in section 2.7.

## 2.5    Specification of calcium carbonate in the market

The specification of calcium carbonate in Thailand is summarized in **Table 2.1**. It was found paper coating, paper filling, and paint/coating required the brightness quality while other (e.g., polyolefins, adhesives, food grade, pharmaceutical, and rubber) do not require.

**Table 2.1** Functions and specifications of calcium carbonate by main application
(Iannicelli & Murray, 1989)

| Applications | Function | Specifications | |
|---|---|---|---|
| | | PSD | Brightness |
| Paper Coating | Provide high brightness, improved runnability, higher solids capability, excellent printability, acceptable gloss, and lower binder demand | Fine PSD (90–99% < 2 μm) | High brightness (95 ISO, 96 GE) |
| Paper Filling | Provides high brightness and high strength of paper. | Fine PSD (60–80% < 2 μm) | Good brightness (>90–95 ISO) |
| Paint/Coatings | Provides non-toxic, low intrinsic color, weather resistance, low abrasiveness, low electrolyte content, and pH stabilizing effect | Fine PSD (60–80% < 2 μm) | Good brightness (>90–95 ISO) |
| Polyolefins | Diaper film ("breathable") market expanding allows diaper/nappy to absorb moisture | Fine PSD (60–80% < 2 μm) | N/A |
| Adhesives | Reduce cost and improve physical properties. Allow for high loading levels and reduce resin demand | PSD minimize coarsely | N/A |
| Food Grade | As an additive in food industry | PSD (4–16 μm) | N/A |
| Pharmaceutical | Functional filler for medical pills, reduces cost, non-toxic | PSD (4–16 μm) | N/A |
| Rubber | Reduce costs of the other ingredients with higher price | Controlled PSD | Controlled color |

Note: "PSD" means particle size distribution.

"ISO" means International Standards Development.

"GE" means General Electric.

"N/A" means not applicable.

## 2.6 Color Property Standards

There are many standards to measure the color properties of materials as shown in **Table 2.2**.

**Table 2.2** The summary of color properties measurement standards (Xrite, 2012).

| Standards | Measured Parameter | Whiteness Formula | Yellowness Formula |
|---|---|---|---|
| ASTM E313 | The lightness of blue (B) and green (G) | 4B-3G | 100(1-B/G) |
| Hunter Lab (1960) | The lightness (L), redness-greenness (a), and yellowness-blueness (b) | L-3b | N/A |
| Stensby (1967) | lightness (L), redness-greenness (a), and yellowness-blueness (b) | L-3b+3a | N/A |
| Tappi R457 | Reflecting of light of the spectrum in range 457-nm wavelength | Reflective Brightness | N/A |
| CIE Lab | Correlation 3-dimensional between lightness (L), green-red (a), and blue-yellow (b) | L*a*b. | N/A |

## 2.7 The stain removal from the mineral surfaces using attrition scrubbing process

In this study, the quality of low-grade calcium carbonate was aimed to be improved by the increasing of WI. From the visual observation, it was found that there are the yellowish stains on the surface of whitish of calcium carbonate particles. To increase the WI as well as decrease the YI, these stains need to be removed.

To remove these stains from the mineral surfaces, the attrition scrubbing process is one of the methods that might be potentially applied. There are many researchers studied about the improvement of mineral quality using attrition scrubbing process. Most studies focused on the quality improvement of quartz however, this technique is not applied with calcium carbonate minerals/rocks yet.

The attrition scrubbing process was applied to remove clay mineral that contained $Al_2O_3$ and $Fe_2O_3$ from quartz including silica sand (Aguiree-Villafana & Leal-Gonzalez, 1994; Du et al., 2019; Raghavan et al., 2006). The parameters that were studied are retention time and solid percentage. For retention time, Du et al. (2019) treated quartz by attrition scrubbing process at retention time of 25 min. The result showed that the $Al_2O_3$ and $Fe_2O_3$ was decreased from 0.040% and 0.048% to 0.033% and 0.020%, respectively. While Raghavan et al. (2006) studied about the effects of retention time on the reduction of iron content in silica sand. The retention time was varied at 5, 10, 20, and 30 min. It was found that longer retention time could cause the lower iron content in the product. At the optimum condition, the iron content could be reduced from 0.025% to 0.011%. For solid percentage, Raghavan et al. (2006) also studied about the effect of solid percentage on the the reduction of iron content in silica sand. The solid percentage was varied at 10 to 50% and the best condition was obtained at solid percentage 45%.

In addition, the attrition scrubbing process was also applied to improve the boron content of the soft mineral like Borax (Acarkan et al., 2005; Garrett, 1998). The parameter that was investigated is the retention time. Acarkan et al. (2005) reported that higher retention time could achieve higher boron content in the product. At the optimum condition the boron content could be increased from 23% to 34%.

## CHAPTER 3 – MATERIALS AND METHODS

### 3.1    Samples preparation and characterizations

The samples used in this study were provide from a calcium carbonate processing plant in Lopburi, Thailand. **Figure 3.1** shows the processing flowchart of this processing plant. The calcite is extracted from the mine using surface mining methods. After that, the run-of-mine is separated manually for quality control. After that, the qualified calcite is sent to the crushing process. The crushed products are fed into the washing process using a rotating screen. The +5 mm were sorted manually again then the qualified product will store in bunkers for sale. The –5 mm were discarded as low-quality calcite, sometime called "sandwash".

**Figure 3.2** shows the pictures of (a) salable calcium carbonate products with whiteness index (WI) higher than 90 and (b) out-spec calcium carbonate with WI less than 90 (under Stensby standard). It was found that the salable product is white while that of out spec is more yellow.

Samples were collected from three points of the tailings pile then, the blending were carried out. These blende sample were used to represent the out-spec calcium carbonate in the tailings pile. The samples were sampled by the sampling process using Jones' riffle splitter for sample characterization.

For particle size distribution (PSD) analysis, sieve analysis was carried out in both dry and wet conditions. The samples were sieved into 6 fractions (i.e., –300, +300–600, +600–1000, +1000–2360, +2360–4000, +4000 µm) as show in **Figure 3.3(a)**. After that, the obtained data were plotted in semi-log graph of particle size and cumulative percent passing.

For chemical composition, the samples with each size fractions were analyzed using x-ray fluorescent (XRF) spectrometer **(Figure 3.3(c))** and x-ray

diffractometer (XRD) **(Figure 3.3(d))** for quantitative and qualitative analysis, respectively.

Finally, for color properties, Colorimeter® (Check 3, Datacolor, USA) **(Figure 3.3(b))**—a high accuracy of color spectrophotometer embedded readiness reflectors and software—was used to analyze the whiteness index (WI) and yellowness index (YI) under Stensby standard.
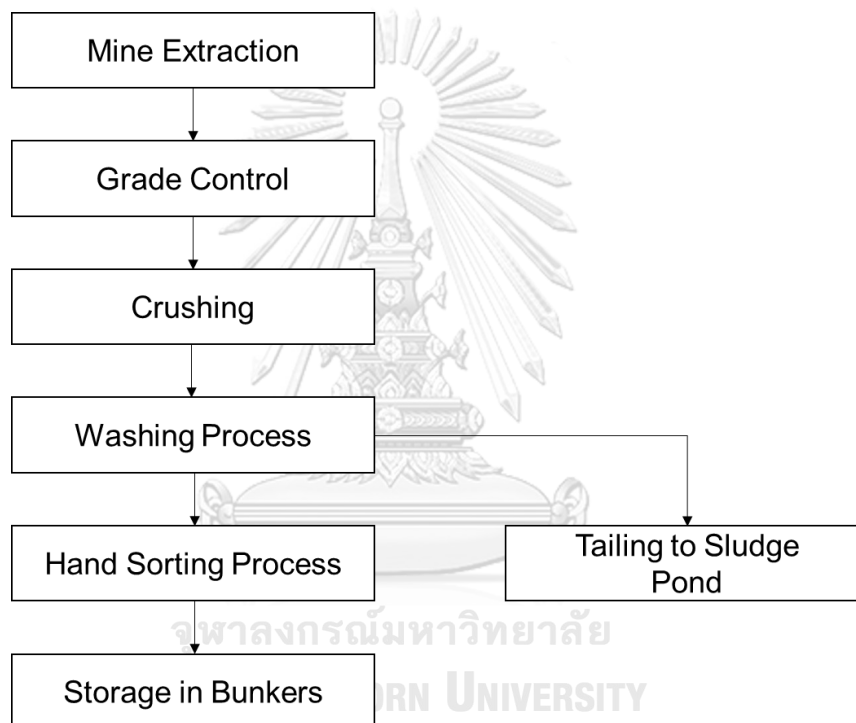


**Figure 3.1** The processing flowchart of this processing plant.
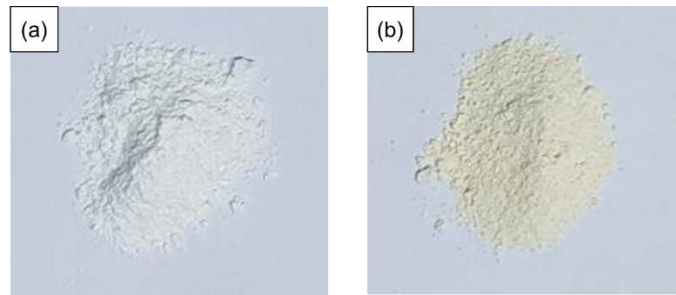
**Figure 3.2** The picture of (a) salable calcium carbonate product with whiteness index 91 and (b) calcium carbonate with whiteness index 84.
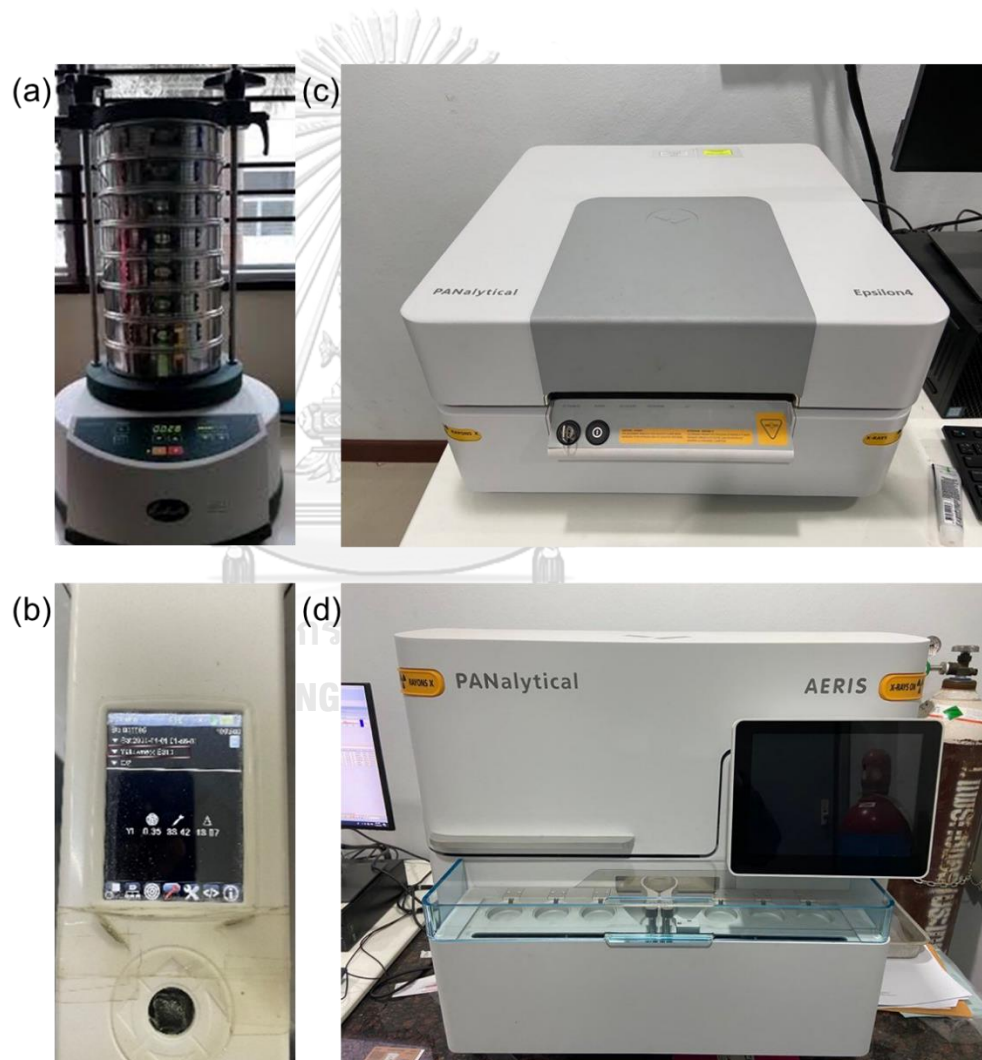


**Figure 3.3** The picture of (a) sieve, (b) Colorimeter®, (c) x-ray fluorescent spectrometer, and (d) spectrometer and x-ray diffractometer.

## 3.2    Attrition scrubbing experiments

### 3.2.1    Laboratory-scale experiments

The experiments were conducted in both laboratory- and pilot-scales. For the laboratory experiments, a laboratory-scale attrition scrubber with 30x30x26 tank (35 L) was used. The experiments were carried out with controlled agitator speed of 1425 rpm while the solid percentage (i.e., 40, 45, 50, 55, and 60%) and retention time (i.e., 10, 15, 20, and 25 min) were varied as shown in **Table 3.1**.

The samples and water were mixed at the selected condition then the mixture was fed into attrition scrubber. The attrition scrubbing process was carried out then, small amount of products were firstly collected at 10 min. After the collection, water was added to adjust solid percentage to be the same as before sample collection then, the attrition scrubbing process was continued. After that, the products were collected with similar process as explained earlier at 15, 20, and 25 min, respectively. The slurry was removed from the products manually by pouring then, rinsed once by water. After that, rinsed products were dried in oven at 105$^{\circ}$C for 24 h. Dried products were weighted and sieved under dry condition to obtain the size fraction of −300, +300–600, +600–1000, +1000–2360, +2360–4000, +4000 μm. Each size fractions were analyzed by XRF spectrometer and Colorimeter$^{®}$ to identify the chemical compositions and color properties (i.e., WI and YI), respectively.

**Table 3.1.** The conditions used in laboratory experiments.

| Batch No. | Solid percentage [%] | Sample weight [kg] | Water weight [kg] | Time [min] |
|-----------|----------------------|---------------------|--------------------|------------|
| 1 | 40 | 13.37 | 20.05 | 10 |
| 2 | 40 | 13.37 | 20.05 | 15 |
| 3 | 40 | 13.37 | 20.05 | 20 |
| 4 | 40 | 13.37 | 20.05 | 25 |
| 5 | 45 | 15.70 | 19.19 | 10 |
| 6 | 45 | 15.70 | 19.19 | 15 |
| 7 | 45 | 15.70 | 19.19 | 20 |
| 8 | 45 | 15.70 | 19.19 | 25 |
| 9 | 50 | 18.24 | 18.24 | 10 |
| 10 | 50 | 18.24 | 18.24 | 15 |
| 11 | 50 | 18.24 | 18.24 | 20 |
| 12 | 50 | 18.24 | 18.24 | 25 |
| 13 | 55 | 21.18 | 17.16 | 10 |
| 14 | 55 | 21.18 | 17.16 | 15 |
| 15 | 55 | 21.18 | 17.16 | 20 |
| 16 | 55 | 21.18 | 17.16 | 25 |
| 17 | 60 | 24.11 | 16.07 | 10 |
| 18 | 60 | 24.11 | 16.07 | 15 |
| 19 | 60 | 24.11 | 16.07 | 20 |
| 20 | 60 | 24.11 | 16.07 | 25 |

### 3.2.2 Pilot-scale experiments

After the laboratory experiments, the optimum condition was used for the pilot experiments to investigate the possibility of scale-up. **Figure 3.4** shows the equipment used in pilot experiments located at a calcium carbonate processing plant in Lopburi that is specially designed for this research.

In this pilot plant, the samples were fed from the hopper into an attrition scrubber via a screw feeder. For the attrition scrubbing process, most of parameters were controlled to be similar as laboratory scale experiments. The experiments were also carried out with controlled agitator speed of 1425 rpm while the solid

percentage and retention time were selected based on the optimum conditions of laboratory experiments.

After the attrition scrubbing process, the products were classified using spiral classifier to remove the slurry similar to the pouring process that was done in the laboratory scale. After that, clarification was carried out using an impelled clarifier to recirculate the water into the process.

In the clarification process, the clear water was separated from the from the pulp by sedimentation. In this pilot process, the clarifier with a single agitating impeller is not only used for clarification but also used to clean the solid particle from attrition scrubbing process similar to the rinsing that was carried out in the laboratory experiments. Solid percentage of this stage was controlled at about 30%. After finished this process, the products were pumped to the final stage.

The pulps were screened using a single-deck round-screen with aperture size selected from the results of laboratory-scale. The liquid part with fine particles were passed through the screen then, flowed into the sedimentation pond. While the solid part that retain on the screen were collected as the final products. After the experiments, the products were analyzed using XRF spectrometer and Colorimeter® same as the laboratory experiments.

**Figure 3.4** Equipment used for pilot experiments: (a) hopper, (b) screw feeder, (c) attrition scrubber, (d) spiral classifier, (e) impelled clarifier, and (f) rounded screen.

## CHAPTER 4 - RESULTS AND DISCUSSION

### 4.1    Sample characterization

**Table 4.1** and **Figure 4.1** show results of sieve analysis of feed sample under dry and wet conditions. Both dry and wet sieve analysis show almost the same results. The diameter of particle at cumulative passing equal to 50% ($D_{50}$) is 395 µm. These results indicated that the water usage could not disaggregate the aggregate particles and/or aggregated particles are not presence in the feed samples. It was also found that the products of each size fraction in both dry and wet sieving showed the similar color, and it seems that the stains on the surface could not be removed even water is used.

Since dry and wet sieving showed the same results so, the dry condition was used in all sieve analysis latter. **Table 4.2** shows the chemical composition of feed samples at different size fractions. It was found that there is no significant difference of chemical composition in each size fractions. The majorities contained in samples are CaO and combustible (Lost on Ignition; LOI) that represent the presence of $CaCO_3$ while impurities are $SiO_2$, $Al_2O_3$, MgO, $Fe_2O_3$ and others. Compared with other size fractions, the coarsest size fraction (i.e., +4000 µm) and the finest size fraction (i.e., – 300 µm) showed the low value of CaO. For the impurities (e.g., $SiO_2$, $Al_2O_3$, MgO, $Fe_2O_3$), high concentrations were detected in the size fraction of +4000, +2360–4000, and –300 µm.

**Figure 4.2** shows the XRD results of feed samples in each size fractions. The results showed that the major mineral contained in the feed samples are calcite that was in line with the results from XRF (**Table 4.2**). However, for the impurities only peak of quartz ($SiO_2$) was detected due to high concentration of calcite.

**Figure 4.3** shows the color properties (i.e., WI and YI) feed samples with different size fractions. The results showed that WI are between 78–88% and YI are

25–45%. It was found that WI has an inverse relationship with YI, indicating that WI will be high when YI is low. The lowest WI (i.e., highest YI) were found in −300 μm while the lowest WI (i.e., highest YI) were +300–600 μm size fraction. These may cause by the impurities like clay minerals containing in this size fraction and/or the stains on the surface showed more significant in this size fraction due to the higher surface area of fine particles compared with the coarser particles at the same amount.

**Table 4.1** Weight retained in each size fractions of the feed samples in this study obtained from sieve analysis under wet and dry conditions

| Size fraction [μm] | Weight retained [%] | |
| :---: | :---: | :---: |
| | Dry condition | Wet condition |
| +4000 | 1.36 | 1.38 |
| +2360–4000 | 2.06 | 2.05 |
| +1000–2360 | 9.09 | 9.03 |
| +600–1000 | 18.12 | 17.80 |
| +300–600 | 27.94 | 27.55 |
| −300 | 41.40 | 42.19 |
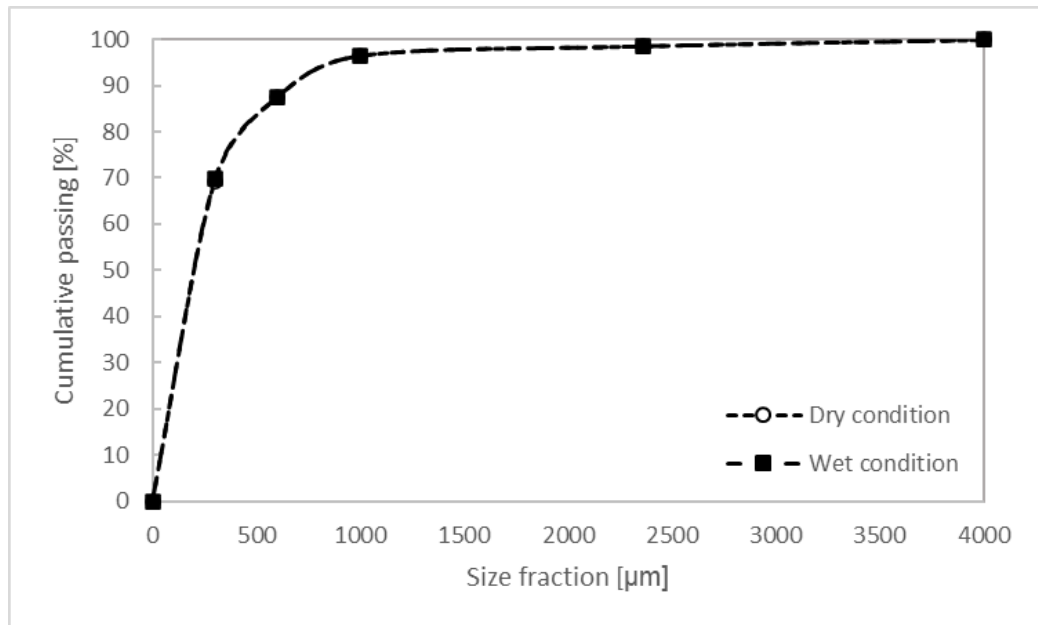
**Figure 4.1** Particle size distribution of feed samples.

**Table 4.2** The chemical composition of feed samples in each size fraction

| Size fraction | Chemical composition [%] | | | | | | |
|---|---|---|---|---|---|---|---|
| [μm] | CaO | SiO$_2$ | Al$_2$O$_3$ | MgO | Fe$_2$O$_3$ | Combustible | Others |
| +4000 | 54.86 | 1.30 | 0.48 | 0.32 | 0.30 | 42.48 | 0.26 |
| +2360–4000 | 56.09 | 1.42 | 0.64 | 0.34 | 0.47 | 40.75 | 0.29 |
| +1000–2360 | 56.02 | 0.38 | 0.17 | 0.16 | 0.10 | 43.03 | 0.14 |
| +600–1000 | 56.87 | 0.20 | 0.08 | 0.18 | 0.06 | 42.47 | 0.14 |
| +300–600 | 56.04 | 0.30 | 0.13 | 0.20 | 0.09 | 43.10 | 0.14 |
| −300 | 55.50 | 1.12 | 0.42 | 0.25 | 0.26 | 42.27 | 0.18 |

**Figure 4.2** XRD results of the particle sizes (a) +4000 μm, (b) −4,000+2,360 μm, (c) −2,360+1,000 μm, (d) −1,000+600 μm, (e) −600+300 μm, (f) −300 μm.
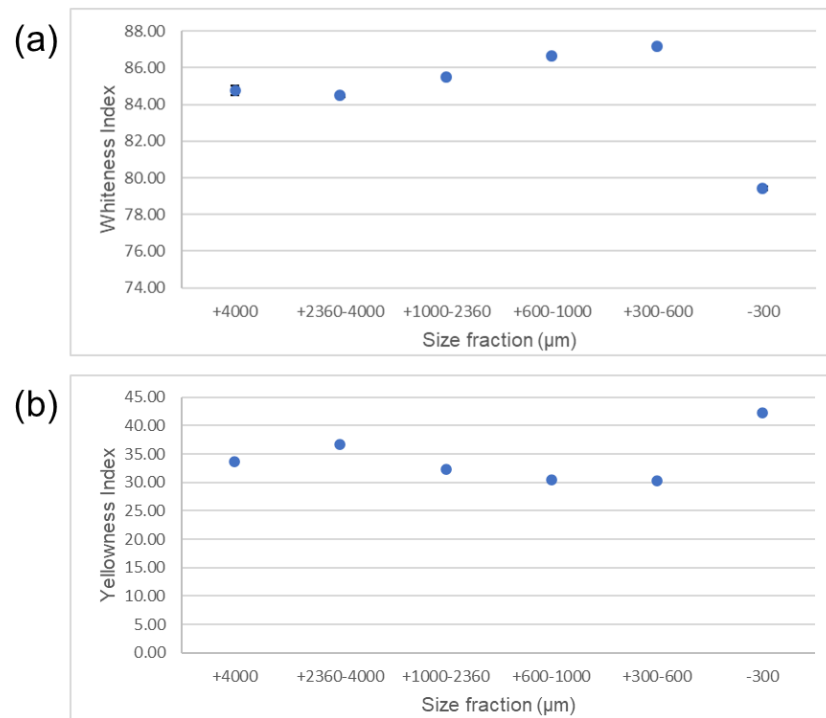
**Figure 4.3** Feed samples tested results with (a) Whiteness index (b) Yellowness
index using Colorimeter® Model Check 3.

## 4.2 Attrition scrubbing experiment results

### 4.2.1 Laboratory-scale

**Figures 4.4 and 4.5** show the particle size distribution (PSD) and $D_{50}$ of
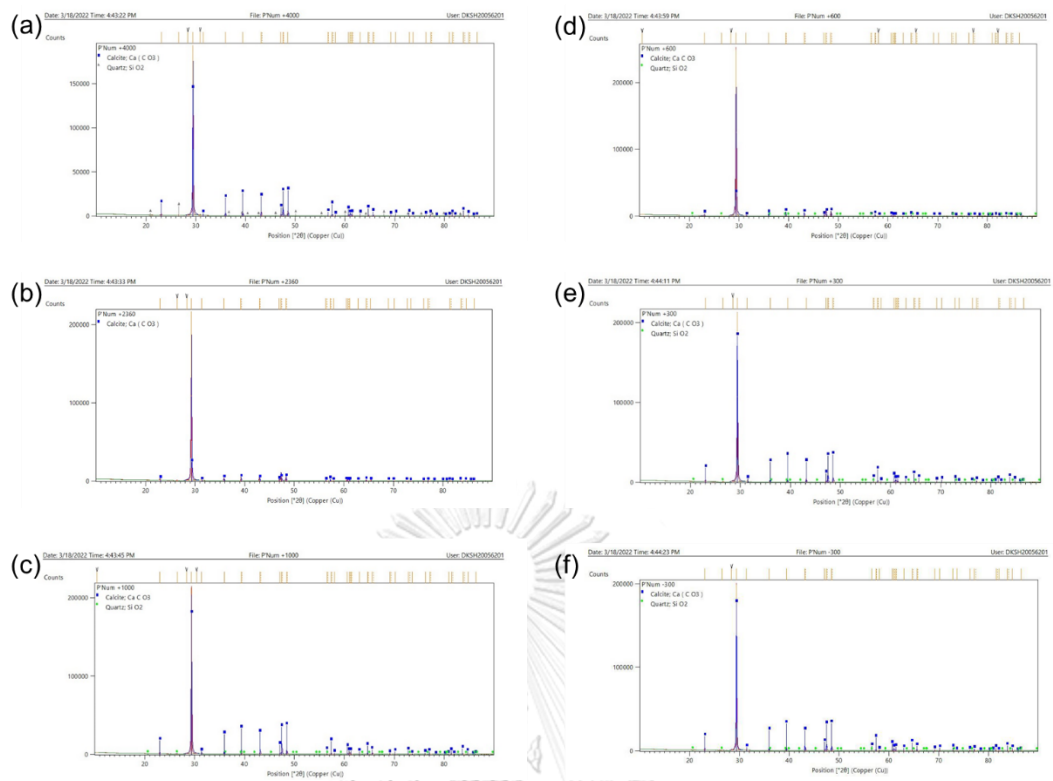products after attrition scrubbing process at difference retention time (i.e., 10, 15, 20,
and 25 min) and solid percentage (i.e., 40, 45, 50, 55, and 60%), respectively.

For 40% solid, (**Figure 4.4(a)**) the particle size distributions (PSD) as well as
$D_{50}$ were almost the same at all retention times. The $D_{50}$ in this condition was
between 210–280 μm that were finer than that of feed (390 μm). These results
indicated that the size reduction was occurred with low reduction ratio. The reason
may cause by low amount of particles in pulp that make the low probability of

collision. The low probability of collision could cause the low attrition of particle as well as size reduction.

For other solid percentage, it was found that the particles size is increasing with retention time, especially at 25 min. These results are occurred by the size reduction due to impact and attrition forces between particles. The fine particles that removed from the surface are rinsed with water so only coarse particles were remained.

It seems that higher solid percentage could enhance the probability of collision that caused the higher attrition of particle as well as particle size reduction so, more experiments were tested at 70% solid. However, from visual observation, most of particles are settle down to bottom surface when 70% solid were used. These results indicated that the attrition scrubbing process may effective when the solid percentage are about 50–60%.

**Figures 4.6 and 4.7** show calcium oxide content and iron oxide content of product after attrition scrubbing process at difference retention time (i.e., 10, 15, 20, and 25 min) and solid percentage (i.e., 40, 45, 50, 55, and 60%), respectively. It was found that there is no significant difference in each condition that was similar as explained in material characterization or feed.

**Figure 4.8 and 4.9** show WI and YI of product after attrition scrubbing process at difference retention time (i.e., 10, 15, 20, and 25 min) and solid percentage (i.e., 40, 45, 50, 55, and 60%), respectively. It was found that all products have WI higher than feed samples while for YI, the results showed that all products have YI lower than feed samples. These results indicated that the attrition scrubbing process could improve the quality of $CaCO_3$. Similar to the results of feed samples, the lowest WI were found in −300 μm size fraction while other products showed similar value with WI about 90%. These results suggest that if −300 μm size fraction was removed after

the attrition scrubbing process, the remain +300 µm size fraction might be sold. From the obtained results the optimum condition was at 55% solid. The product obtained in this condition at every retention time and size fractions except −300 µm could be sold in most industrial except paper coating industrial.
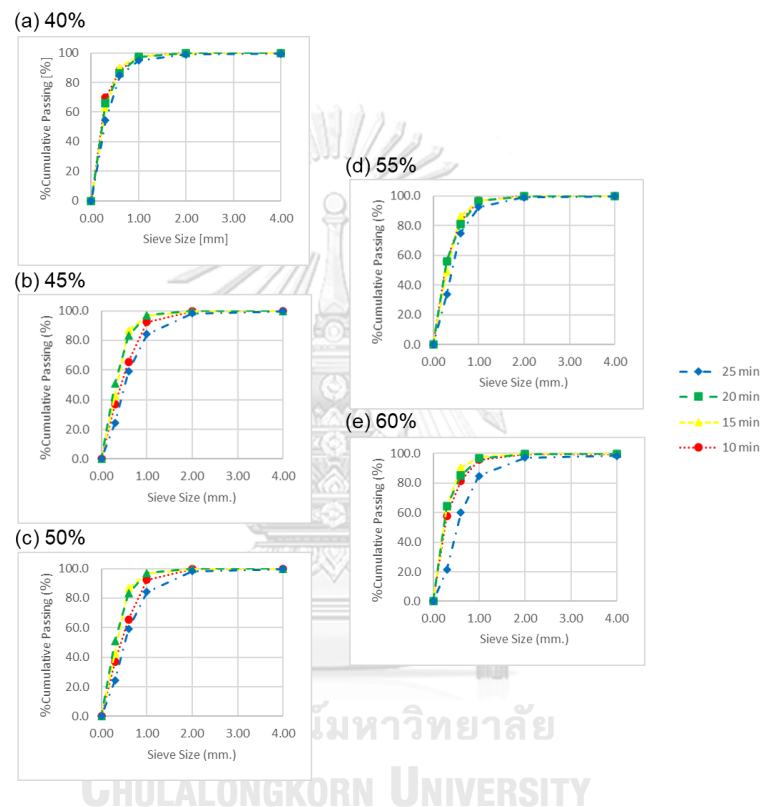


**Figure 4.4** Particle size distribution of sample after attrition scrubbing experiments in laboratory-scale at different retention time (i.e., 10, 15, 20, and 25 min) and solid percentage of (a) 40,    (b) 45, (c) 50, (d) 55, and (e) 60.
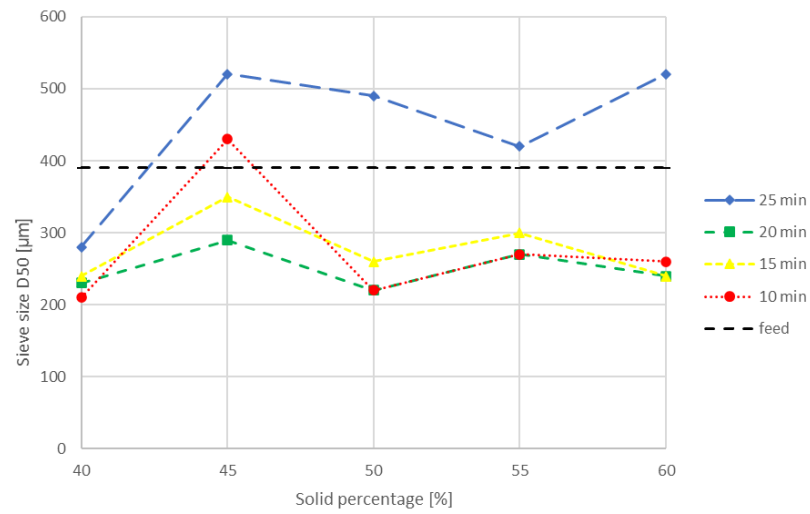
**Figure 4.5** Diameter of particle at cumulative passing equal to 50% of products after attrition scrubbing experiments in laboratory-scale at different retention time (i.e., 10, 15, 20, and 25 min) and solid percentage (i.e., 40, 45, 50, 55, and 60%).
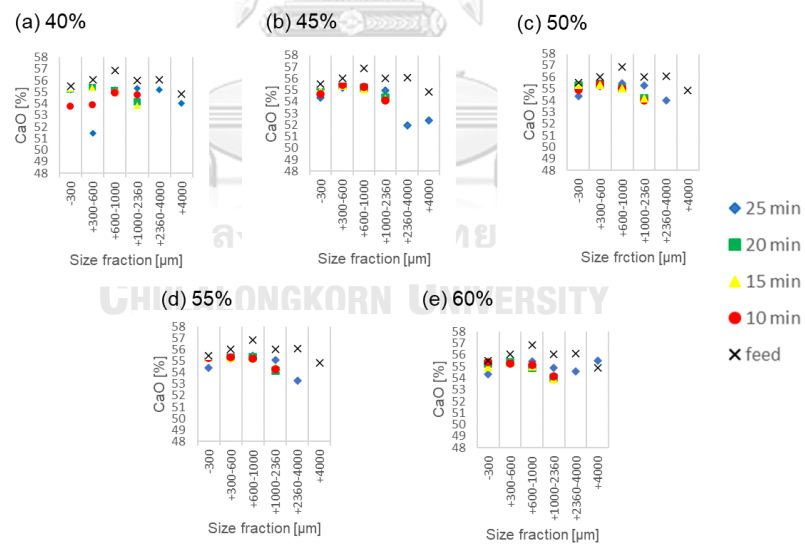


**Figure 4.6** Calcium oxide contents of products after attrition scrubbing experiment in laboratory-scale at different retention time (i.e., 10, 15, 20, and 25 min) and solid percentage of (a) 40, (b) 45, (c) 50, (d) 55, and (e) 60.
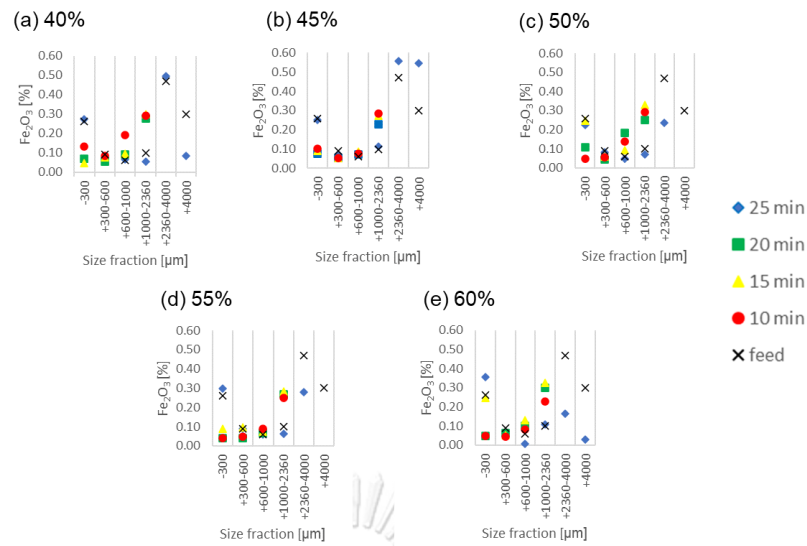
**Figure 4.7** Iron oxide contents of products after attrition scrubbing experiment in laboratory-scale at different retention time (i.e., 10, 15, 20, and 25 min) and solid percentage of (a) 40,    (b) 45, (c) 50, (d) 55, and (e) 60.
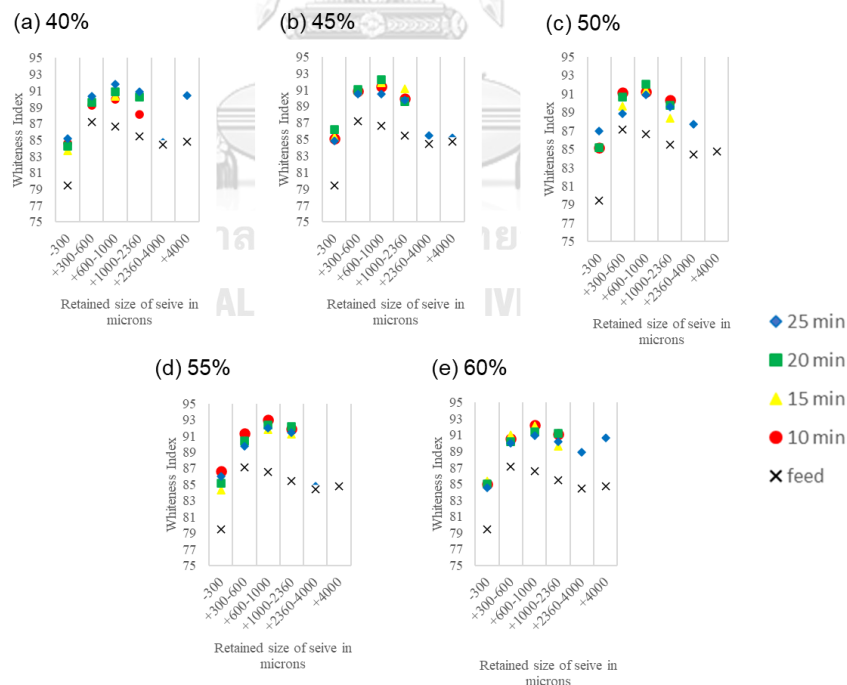


**Figure 4.8** The values of the whiteness index of products after attrition scrubbing experiment in laboratory-scale at different retention time (i.e., 10, 15, 20, and 25 min) and solid percentage of (a) 40, (b) 45, (c) 50, (d) 55, and (e) 60.
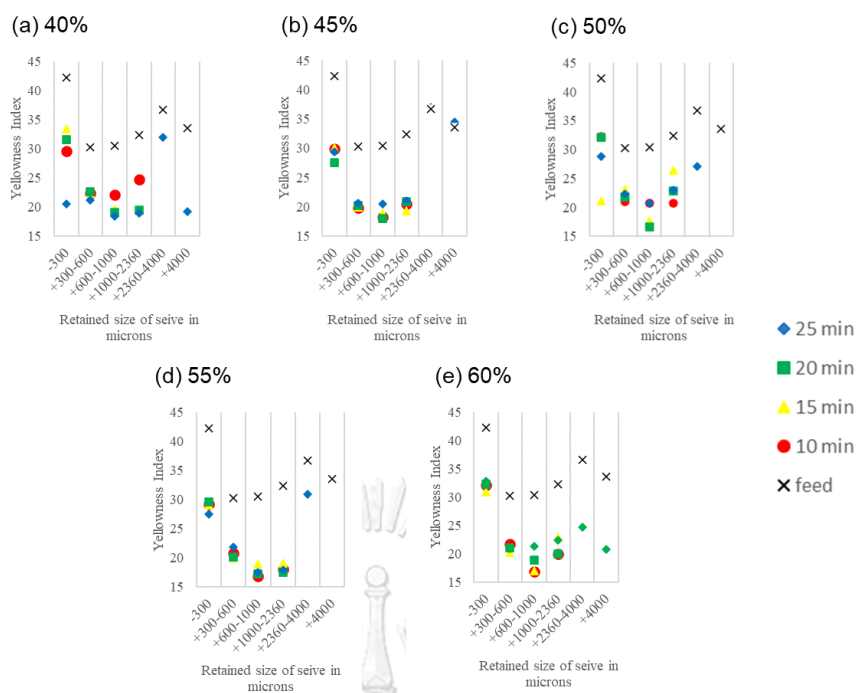
**Figure 4.9** The values of the yellowness index of products after attrition scrubbing experiment in laboratory-scale at different retention time (i.e., 10, 15, 20, and 25 min) and solid percentage of (a) 40, (b) 45, (c) 50, (d) 55, and (e) 60.

### 4.2.2 Pilot-scale

From the laboratory-scale experiments, the results showed that the optimum condition of solid percentage was 55%. To investigate the possibility of scaling up, the attrition scrubbing experiments were carried out in pilot-scale at solid percentage of 55% with retention time of 10, 15, 20, and 25 min. Since the results from laboratory-scale experiments −300 µm should be removed from other size fractions after attrition scrubbing process to improve the quality of products so, a single-desk round-screen with aperture size of 300 µm were used at the final step of pilot scale experiments.

**Figure 4.10** shows the PSD of products from pilot-scale experiments. The results show that there is no significant difference for each retention time that was in line with the laboratory-scale results. For chemical composition, since the results of

laboratory-scale do not show the significant results so, XRF analysis was not carried out while only color properties (i.e., WI, and YI) were analyzed.

For whiteness index, the results showed that the products in every size fractions have WI between 90 to 93% that was higher than feed and could be able to sell (**Figure 4.11**). For yellowness index, the results showed that all size fractions of the product have YI between 14 to 26% that was lower than feed **(Figure 4.12)**. These results indicated that the scale-up of attrition scrubbing process was possible and it could improve the quality of $CaCO_3$ to be able to sell to most industrial except paper coating industrial.



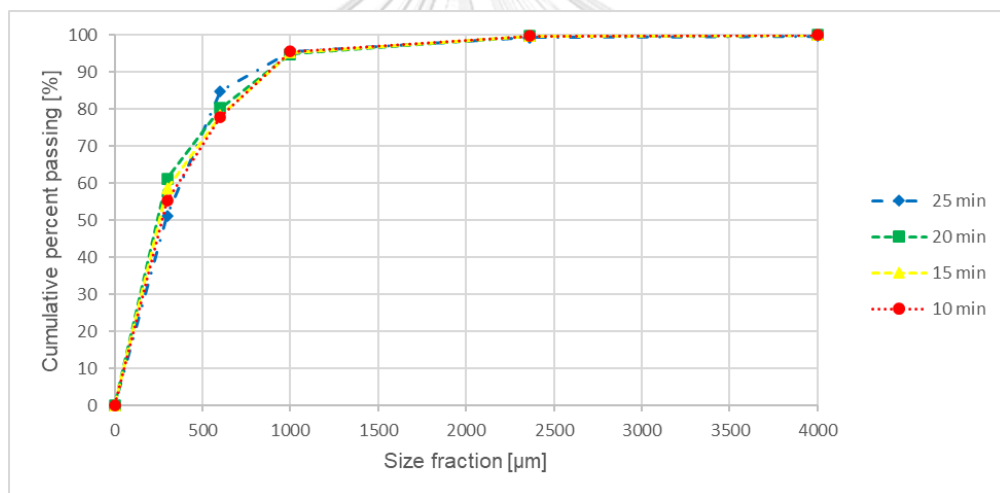**Figure 4.10** Particle size distribution of products from pilot scale experiment under 55% solids condition at different retention times (10, 15, and 20 min).
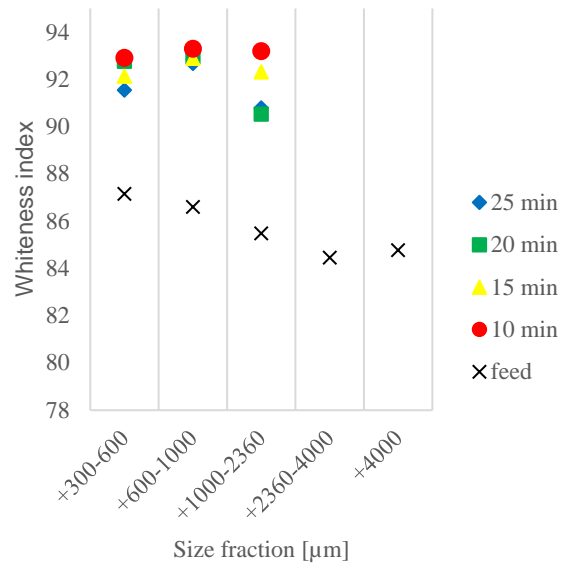
**Figure 4.11** The whiteness index values of 55% solids on pilot experiment at different retention times (10, 15, 20, and 25 min)
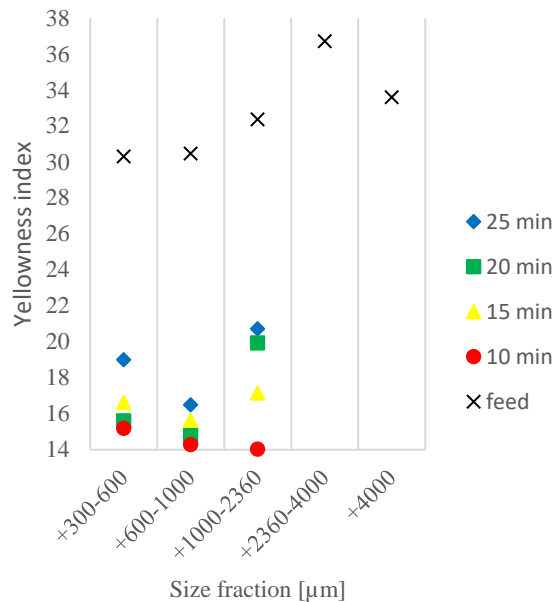


**Figure 4.12** The yellowness index values of 55% solids on pilot experiment at different retention times (10, 15, 20, and 25 min)

As shown in the results, the attrition scrubbing process could improve the qualities (i.e., WI, and YI) of calcium carbonate tailings. This method is also environmentally friendly since the chemical reagents are not used. For the economical viewpoint, the beneficiation of the waste or tailings recovering thus the main cost benefit will be taken into account from resource price. For the market price, the high-quality calcium carbonate (WI=91) is sold at 500 THB per ton while the tailing (WI<91) is sold at 200 THB per ton as a by-product for animal feeding, soil amendments, pH neutralization in water treatment, and construction material. The estimation of capacity, cost and income of pilot-plant attrition scrubbing process are shown in **Tables 4.3 to 4.6 and Figure 4.13**. A new additional equipment of the attrition scrubbing process comprises as same as the equipment list in the pilot scale (e.g., hopper, screw feeder, attrition scrubber, spiral classifier, impelled clarifier, scrubber, and rounded screen classifier). The five-year-capital cost in commercial scale can be estimated 2 million Baht with power system installed in total 50 kilo-watts that cost 4.5 baht/kilowatt-hour, and the new raw material purchasing and by-product selling is expensed more than 300 THB/ton, approximately.

Feed

| 40 t/h

**Attrition scrubber**

| 40 t/h

**Clarifier**

| 40 t/h

**Round screen** ———— 22.1 t/h → Waste

| 17.9 t/h

**Product**
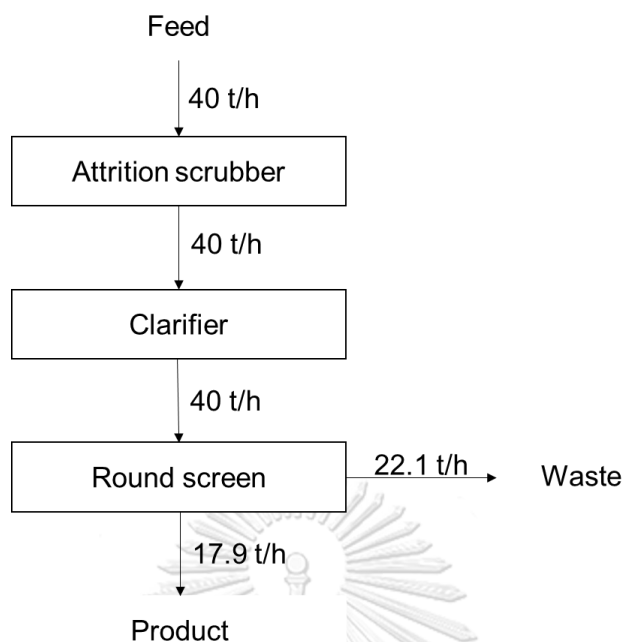
**Figure 4.13** Estimation of capacity of pilot-plant attrition scrubbing process.

**Table 4.3** Details of the equipment cost on each equipment (e.g., hopper, screw feeder, attrition scrubber, spiral classifier, impelled clarifier, scrubber, and rounded screen classifier)

| Equipment list | Dimension | Power | Unit | Estimated price | Unit |
|---|---|---|---|---|---|
| Hopper | 2.0m x 2.5m x 1.5 m | - | - | 100,000 | THB |
| Screw feeder | Dia 0.6m x 2.5m | 10 | kW | 250,000 | THB |
| Scrubber | Dia 1.5m x 1.5m | 15 | kW | 450,000 | THB |
| Clarifier | Dia 1.5m x 1.5m | 10 | kW | 200,000 | THB |
| Hydro classifier | Dia 0.6m x 3.5m | 10 | kW | 300,000 | THB |
| Round screen | Dia 1.0m x 3.5m | 5 | kW | 250,000 | THB |
| Other | - | - | - | 450,000 | THB |
| Total | - | | 50 | kW | 2,000,000 | THB |

**Table 4.4** The calculation of operating cost, maintenance cost, and by-product selling income

| Description | | Unit |
|---|---|---|
| Operating hours | 30,000 | hour/ 5 years |
| Throughputs of attrition scrubbing process | 40 | ton/hour |
| Operating cost per hour | 225 | THB/hour |
| Maintenance cost (10% of operating cost) | 22.5 | THB/hour |
| Total operating cost for 5 years (30,000 hours x 247.5 THB/hour) | 7,425,000 | THB/ 5 years |
| Product output of attrition scrubbing process | 17.9 | Ton/hour |
| By-product selling | 300 | THB/ton |
| Total by-product selling income for 5 years (30,000 hours x 17.9 tons x 300 THB/ton) | 161,100,000 | THB/ 5 years |

**Table 4.5** Saleable price and scrubbing process cost of calcium carbonate in each ore grade (i.e., SEMI-91%, and tailing)

| Ore grade | Saleable price (THB/ton) | Scrubbing process (THB/ton) | | |
|---|---|---|---|---|
| | | Capital cost | Operating cost | Total cost |
| SEMI-91% | 500 | | | |
| Tailing | 200 | 1.7 | 6.2 | 7.9 |

**Table 4.6** Estimation of cost and income of pilot-plant attrition scrubbing process

| Description | THB |
| --- | --- |
| Depreciation of the capital cost (5 years) | 2,000,000 |
| Operating and maintenance cost (5 years or 30,000 hours) | 7,425,000 |
| By-product selling income (300 THB/ton, 1,200,000 ton/ 5 years) | 161,100,000 |
| Net income | 151,675,000 |

**CHAPTER 5 – CONCLUSIONS AND RECCOMENDATION**

**5.1    Conclusions**

This study, the improvement of calcium carbonate tailings was carried out to meet the market and/or industry standard requirements. From the materials characterization, it was found that calcite is the major mineral. For the impurities quartz was detected with small amount of other minerals containing Al2O3, MgO, and Fe2O3. The WI was between 78–88 that could not be sale in most of industries. The lowest WI was found in −300 μm with WI equal to 78 while other size fractions were 84 to 88. Similarly, the YI at −300 μm size fraction was around 45 while the other size fractions were 25 to 40. These results may cause by the clay minerals containing in this size fraction.

The attrition scrubbing experiments were carried out to remove the strains on the surface of low-grade calcium carbonate. The results showed that the size reduction was occurred. It was found that the attrition scrubbing process may effective when the solid percentage are about 50–60%. For the chemical composition, no significant difference was found. While for color properties, it was found that all products have WI and YI was improved from that of feed. These results indicated that the attrition scrubbing process could improve the quality of $CaCO_3$. Similar to the results of feed samples, the lowest WI were found in −300 μm size fraction while other products showed similar value with WI about 90%. These results suggest that if −300 μm size fraction was removed after the attrition scrubbing process, the remain +300 μm size fraction might be sold.

From the laboratory-scale experiments, the results showed that the optimum condition of solid percentage was 55%. To investigate the possibility of scaling up, the attrition scrubbing experiments were carried out in pilot-scale was investigated. It

was found that the scale-up of attrition scrubbing process was possible and it could improve the quality of CaCO3 to be able to sell to most industrial except paper coating industrial.

## 5.2    Recommendation

Iron matters was one of the interested issues that correlated with whiteness of calcium carbonate. In particle size ranges smaller than 300 microns, it contaminates in particle size fraction nearly 50% of total. Though, the cut off size by classification is such a good solution for upgrading the whiteness property and also reducing the impurity like iron matter too, but the unusable material amount is still high up to 50 to 60%. Further studies could be also figure out how to remove iron contaminants from the finer particle as well in order to minimize waste regeneration and saving the new resource to supply in place the fine particle of the calcium carbonate tailing loss during the manufacturing process. Some beneficiation processes to get rid the iron matters could be considered, for example, high intensity magnetic separator (HIMS), wet high intensity magnetic separator (WHIMS), gravitational classifiers, etc. Furthermore, sizing of the iron impurities could be intensive study in order to indicate what exact particle size range is contained these impurities.

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จุฬาลงกรณ์มหาวิทยาลัย

CHULALONGKORN UNIVERSITY

# VITA

NAME                       Bancha Wongkaewphothong

DATE OF BIRTH              28 August 1979

PLACE OF BIRTH             Thailand

INSTITUTIONS ATTENDED      Chulalongkorn University

HOME ADDRESS               33/10, The connection Onnuch Village, Chalerm Phrakiat

                           Rama 9 Road, Soi 87, Prawet Subdistrict, Prawet District,

                           Bangkok 10250

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