# CHARACTERIZATION AND FORMULATION OF NATURAL FACE POWDER

**NWAY YU HNIN**

**MASTER OF SCIENCE**
**IN**
**COSMETIC SCIENCE**

**SCHOOL OF COSMETIC SCIENCE**
**MAE FAH LUANG UNIVERSITY**
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EXAMINATION COMMITTEE

……………………Nattaya Lourith.……………………CHAIRPERSON
(Assoc. Prof. Nattaya Lourith, Ph. D.)

……………………Mayuree……………………ADVISOR
(Assoc. Prof. Mayuree Kanlayavattanakul, Ph. D.)

……………………Chaisak Chansriniyom……………………EXTERNAL EXAMINER
(Asst. Prof. Chaisak Chansriniyom, Ph. D.)

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# ABSTRACT

This study was aimed to study the physicochemical properties of natural powders which were used to develop natural face powder formulations. Thanaka powder, corn flour, mung bean starch and rice bran flour were reduced sizes to less than 45 µm. These powders were fine with yellowish light brown to white. Corn flour was agglutinate, mung bean starch and rice bran flour slightly agglutinate. Those of prohibited metals, e.g. Hg, Pb, As and Cd which were crucial for cosmetic products, were detected below the limitations as regulated by ASEAN guidelines in these natural materials. They showed melting temperature at 70.25-74.92 °C by DSC, which made them applicable for solid dosage form. The percentage of crystallinity were 22.52-38.55% by XRD, which Thanaka powder had the highest value so it would be more resistant to high temperature. The flowability of Thanaka powder and mung bean starch were good, whereas corn flour and rice bran flour were good cohesiveness. They were able to absorb water (7.84-17.31%) and oil (soybean oil; 5.47-13.39%, olive oil; 5.06-12.65% and mineral oil 5.58-11.98). Under long term stability test at ambient temperature and 45 °C for 3 months without exposing to light, the color difference ($\Delta E$), of Thanaka powder, corn flour, mung bean starch and rice bran flour were less than 1.5 ($\Delta E \leq 1.43$, 0.87, 1.45 and 1.40, respectively for each natural material), thus the color changes of them could not be detected by human eyes. Thermal properties of corn flour, mung bean starch and rice bran flour had minor shifts, while Thanaka powder was more resistant for higher temperature. Water absorption, oil absorption capacities and % crystallinity of Thanaka powder, corn flour, mung bean starch and rice bran flour were insignificantly ($p \geq 0.061$) changed. Hence, certain natural raw materials were considered to be safe (in terms of total elemental analysis) and stable, and would be suitable for cosmetic solid dosage form development.

Natural loose powder and natural compact powder formulations were prepared by using Thanaka powder as pigment and absorbent, corn flour as adhesive, mung bean starch and rice bran flour as fillers and *Helianthus annus* seed oil, *Rosmarinus officinalis* leaf oil and *Foeniculum vulgare* oil as preservative, except olive oil as liquid binder was added into the later one. Natural loose powder was yellowish white powder but natural compact powder was slightly darker than the other one. The developed products were good slip, absorbency, adhesiveness and bloom but poor coverage. Both developed natural face powders showed SPF values as 1.41-1.55. Under 6 heating-cooling cycles, the color changes could not be detected by naked eyes because the color difference was ≤ 1.05. In terms of UVA and UVB protection, the values of both developed natural face powders changed less than 4.02±0.82% after 6 heating-cooling cycles.

**Keywords:** Natural Face Powder, Thanaka Powder, Corn Flour, Mung Bean Starch, Rice Bran Flour

# TABLE OF CONTENTS

# TABLE OF CONTENTS (continued)

# LIST OF TABLES

**Table** **Page**

# LIST OF FIGURES

**Figure** **Page**

# LIST OF FIGURES (continued)

**Figure** **Page**

# LIST OF FIGURES (continued)

**Figure** **Page**

# LIST OF FIGURES (continued)

**Figure** **Page**

# ABBREVIATION AND SYMBOL

| | |
|---|---|
| µm | Micrometer |
| As | Arsenic |
| Cd | Cadmium |
| Pb | Lead |
| Hg | Mercury |
| ICPMS | Inductive Coupled High Frequency Plasma Mass Spectrometry |
| ppb | Parts per billion |
| ppm | Parts per million |
| FTIR-ATR | Fourier Transform Infrared Spectroscopy-Attenuated Total Reflectance technique |
| FEG-SEM | Field Emission Gun-Scanning Electron Microscopy |
| keV | Kilovolt |
| kx | Thousand times |
| DSC | Differential Scanning Calorimetry |
| w/w | Weight by weight |
| % | Percentage |
| °C | Degree Celsius |
| hr | Hour |
| min | Minute |
| mL | Milliliter |
| WAC | Water Absorption Capacity |
| OAC | Oil Absorption Capacity |
| g | Gram |
| s | Second |

## ABBREVIATION AND SYMBOL (continued)

| | |
|---|---|
| µL | Microliter |
| rpm | Revolutions per minute |
| xg | Relative centrifugal force |
| SPF | Sun Protection Factor |
| PMMA | Polymethyl Methacrylate |
| UV | Ultraviolet |
| nm | Nanometer |
| cm | Centimeter |
| TA | Ambient Temperature |
| *p* | Probability |
| SD | Standard Deviation |

# CHAPTER 1

# INTRODUCTION

## 1.1 Background of the Study

The global color cosmetics market size was valued at USD 57.2 billion in 2018. The color cosmetics market is forecasted as Compound Annual Growth Rate (CAGR) of 6.2% from 2019 to 2025. Asia Pacific is expected to register the fastest CAGR of 9.4%. That forecast product outlook on facial products, eye products, lip products, hair products, nail products, and others. Facial products held more than 25.0% share, which is the largest share in that category. Face powder is one of the leading products in among them (Grand View Research, 2019a).

Natural cosmetics become to be more open to consumers and producers, and both of them use a variety of natural cosmetic resources and materials. Due to the awareness of environmental and health, this effect can be seen in the behavior of not only the producers but also the consumers (Amberg & Fogarassy, 2019). The size of global natural cosmetics market was estimated at USD 34.12 billion in 2018. It is projected to register a CAGR of 5.01% from 2019 to 2025 (Grand View Research, 2019b). According to the research of Amberg & Fogarassy (2019) in Hungary, from 197 responses, 70% of the participants wish to buy natural cosmetics.

One issue about face powder is that talcum, the major component of face powder (Faber, 2012; Schlossman, 2001; 2006), sometimes found asbestos in it which is known as carcinogen and can cause harm on different organ systems of the human body (Chaudry & Chrisopherson, 2020; Awam et al., 2019; American Cancer Society, 2017). Chaudry & Chrisopherson (2020) and Teeratanan (2018) mentioned that natural or modified starches e.g. corn starch and mung bean starch could be marketed as popular alternative to talcum. Furthermore, the other synthetic ingredients of face powder act as adsorbent, slipping agents, pigment or sun sunscreen, are added. According to the data from Lim et al. (2021) and Isler (2017), natural face powder brands in the market do not live up to their marketing claims and use a number of synthetic ingredients in their natural cosmetics, and that is also relevant to the report of Beerling (2014).

Since the increasing demand of natural cosmetics and main ingredient of face powder links to potential cancer, this research study would like to develop natural face powder by substituting talcum with natural starches, and combining with other natural actives e.g. Thanaka, and vegetable oils, etc. Because the research study of Thanaka is to substitute as an absorbent in the face powder since it contains tannins which could possibly have oil control property and it is not fully soluble in water (Aung, 2006; Wangthon et al. 2010). Besides, Thanaka has yellowish-white color which is suitable for Asian skin. In the previous studies, there are several thanaka researches concerning of anti-oxidant, anti-inflammatory (Wangthong et al., 2010), anti-microbial (Jantarat et al., 2018; Wangthong et al., 2010), skin lightening (Wangthong et al., 2010) and sun protection properties (Joo et al., 2004; Kanyavattanakul & Lourith., 2012a). Moreover, some vegetable oils e.g. olive oil is natural oil which could be as oil binder in compact face powder (Faber, 2012). According to Kaur & Saraf (2010) it also has sun protection factor values so that this is the extra advantage function for usage.

Furthermore, after formulating the natural face powder, the physicochemical characteristics and stability test of the developed face powder would be observed in this study.

## 1.2 Objectives of the Study

1.2.1 To test physicochemical properties and stability of each natural ingredients

1.2.2 To formulate the stable face powders containing natural ingredients and examine their stability

## 1.3 Scopes of the Study

Some natural powder ingredients e.g. Thanaka powder, corn flour, mung bean starch and rice bran flour were prepared and evaluated on their physiochemical properties. Furthermore, all of natural ingredients would be formulated in face powder, and evaluated upon heating-cooling cycling stability test.

## 1.4 Outcome of the Study

The stable face powder containing natural ingredients

# CHAPTER 2

# LITERATURE REVIEWS

## 2.1 Face Powder

Face powder is a cosmetic product which can give smoothness to overall texture, give translucency when excess is buffed, give the impression more refined skin, help to set the makeup base and add longevity, and suppresses surface oil and shine. Basically, there are numerous raw materials used in powdered cosmetics (Schlossman, 2001). In fact, in a good face powder there is a blend of constituents is employed to be able to get the following essential characteristics (Faber, 2012).

According to Faber (2012), the essential characteristics of a good face powder is as follows;

Covering: The ability to cover skin defects such as shiny skin, enlarged pores, and minor flaws.

Slip: The ability to spread the powder over the skin without dragging, and give smooth and even feeling.

Absorbency: The ability to absorb skin secretions (sweat and sebum) without showing any evidence about such absorption.

Adhesiveness: The ability to adhere on the skin.

Bloom: The ability to convey a velvety, peach-like setting to the skin.

### 2.1.1 Types of Face Powder

2.1.1.1 Loose face powder: Essentially, the raw materials of pressed powder and loose face powder are identical (Faber, 2012). Except, loose powder does not contain a binder to press the cake into a tin-plate pan. This type is less popular than the pressed face-powder products because of application is easy and less messy to apply (Schlossman, 2001; 2006).

2.1.1.2 Cake makeup: Originally known and patented as “Pancake” makeup. Powder from this compressed cake is applied with a wet sponge, and is for heavier coverage. Wet or dry face powders can be used as a foundation or face powder. The applied film contains enough oil for binding and possess water-repellent properties. Cake makeup may contain up to 25% of oil or wax component including humectants (Schlossman, 2006).

2.1.1.3 Compact face powder: Compact face powder was introduced in America during 1930 and it flowed in popularity because of it can be easily applied and can be stored conveniently. A compact powder is a dry powder which has been compressed into a cake and is usually applied with a powder puff (Faber, 2012).

The major raw materials of pressed powder are almost identical with loose face powder. The base for a compact cake must be compressed, should not crumble nor chip under normal conditions of usage and or during shipment. To accomplish such kind of result, binding agents are required. The important characteristics for pressed powder, which do not present in loose powder, is binding ability and payoff (Faber, 2012).

Variations of compact powders are available on the market as powder or powder with built-in foundation. The difference between these two types is debatable, as both contain emulsions and oils. The major difference is that the compact powder puff is packed in with the cake powder, while the sponge used for cake makeup has to be packed separately (Schlossman, 2006).

Women buy powder mainly because of its shade. Explained into the language of manufacturer, it means covering power. Hence color and covering power are the important considerations followed by slip, adhesiveness, dullness, shine and odor. There are also other terms, “light,” “medium,” “heavy,” which actually refer to covering power (Schlossman, 2006).

1. Good characteristics of compact face powder

A compact powder must also have sufficient payoff. When rubbed with a powder puff, it must come off easily onto the applicator. Too low a pressure will result in a cake disintegrate, whereas, too high a pressure will result in a hard, glazed cake which will not payoff sufficiently (Faber, 2012).

According to Schlossman (2006), the following are the most important tests recommended for the finished product.

1) A puff is used to check the release of the powder for application to the face.

2) The cake strength or hardness is checked with a penetrometer. The cake is checked for glazing.

3) Lastly, drop and shipping tests are of value.

2. Compression methods of compact face powder

The amount of pressure required for compacting a cake can be considered, according to the basic formulation, type and quantity of binder necessary, powder moisture content, and other variables (Faber, 2012). According to Faber (2012), there are three general procedures employed in the manufacture of compact powders industrially: the wet molding process, the damp compression method, and the dry compressing method. The wet molding process is now virtually obsolete in the United States, and most cosmetic firms make use of the damp or dry processes for manufacturing compressed face powder.

1) Wet molding process

The basic material (powder), color and binders are combined in the form of paste by adding water. Then the pastes are compressed into molds and the products are dried by air gradually. A possibility of producing cracks and other faults in the preparation can happen (Sharma & Gadhiya, 2018). This method requires very little binding agent. Although possibly more difficult to work out at the start, the basic procedure can be adhered to more consistently once proper conditions have been attained. It is easier to duplicate than the other two methods and more adaptable to large-scale production.

2) Damp compressing method

In the damp compression method, the base powder, color and perfume are uniformly mixed. The mixture is then moistened with a liquid binder, and blended until the desired elasticity has been achieved. The powder is later screened and passed to compression machines. The finished pressed tablets are dried at high temperatures.

3) Dry compressing method

In the dry compression method, the powder base, color and perfume are grinded and the grinded powder are moistened with binder. The mixture is then carefully combined and the powder is compressed.

### 2.1.2 Compositions of Face Powder

A face powder is a blend of detailed raw materials to be able to obtain the desired specific features. Thus, it would be stick with the basic ingredients as well as the belongings of each may convey to the finished powder formulation (Faber, 2012). Essentially, the raw materials are similar to those of the pressed powder and loose face powder. However, the main different characteristic is that pressed powder present binding ability and payoff that loose powder do not conform. Binding agents are essential for compact powder or compact cake (Faber, 2012). Primary raw materials are mentioned below.

2.1.2.1 Talcum: Talc is the major component in face or compact powder. Chemically, it is a hydrated magnesium silicate. There are many different qualities of talc and the ultimate finished product usually depends on the choice of talc. Cosmetic talcs are extracted in Italy, France, Norway, India, Spain, China, Egypt, Japan and the United States. Typically, talcs are purified by gamma irradiation. Talc is washed and ground to a particle size of 200 mesh or finer. Cosmetic talc are white in color, free from asbestos, and able to spread well or slip with minimum coverage (Schlossman, 2006; 2001). Micronized talc is lighter and fluffier but the disadvantage is that it is not so smooth on the skin. Although talc is fairly hydrophobic, treated talcs have been used to enhance its texture. In some products, talc is used up to 70% in the formulation (Schlossman, 2001). Its outstanding key point are easy spreadability (slip) and low covering ability. Dermatologically, talc is an innocuous material (Farber, 2012)

2.1.2.2 Kaolin: Kaolin, is otherwise known as China clay, which is a native hydrated aluminum silicate, and has been made free of gritty particles by elutriation. It does not have the ability of a high degree of slip. Kaolin is a good absorbent, is dense, and sometimes used to reduce the bulk density in loose-powder goods. It offers a matte surface result that can reduce slight sheen residue left by some talc products (Schlossman, 2001; 2006). It owns good covering powder and adhesion. Since it is a compound of high density, it is used in formulations to adjust fluffiness and regulate bulk. Kaolin is hygroscopic and is a dermatologically innocuous material (Faber, 2012).

2.1.2.3 Calcium carbonate: Calcium carbonate, or precipitated chalk, has brilliant absorption abilities. It affords a matte finish and has moderate covering powder. High levels should be avoided; because it might give an undesirable, dry, powdery feel on the skin (Schlossman, 2001). It is also exceptional for developing the “bloom” effect on the skin when face powder is applied. Precipitated chalk is a slightly alkaline, white, odorless microcrystalline powder. It is quite dull and has a chalky feeling (Faber, 2012).

2.1.2.4 Magnesium carbonate: Magnesium stearate is a very light, fluffy powder that can absorb well and is often prepared to absorb perfume before mixing into face powders (Schlossman, 2001). When it is use overly, it will give a drying effect on the skin (Faber, 2012).

2.1.2.5 Metallic soap: Zinc and magnesium stearate are significant supplies for conveying adhesion to face powders. Stearates enhance the ability of water repellency to recipes. Too-high levels give a blotchy outcome on the skin. Zinc stearate makes adhesion and gives a smoothing power to face powders. Aluminum stearate and lithium stearates have also been used. High levels can make pressed formulations becoming too hard to apply (Schlossman, 2001).

2.1.2.6 Starch and four: Starch is used in face powders and give a “peach-like” bloom by providing a smooth surface on the skin. One of the drawbacks is that the moistened starch tends to cake and the wet product may deliver an environment for bacterial growth (Schlossman, 2001; 2006). Several decades ago, the use of starch is one of the basic ingredients in face powder (Faber, 2012). Modified starch has been extensively used to rise the flow of powders (Schlossman, 2006). Natural face powder by flour can be a substitute from harmful ingredients (Thomas, 2013). Also, Jarupinthusophon & Anurukvorakun (2021) successfully and safely developed rice flour compact powder by replacing talcum also.

1. Mung bean starch

From Teeratanan (2018) thesis research, to develop the pressed powder form natural starches, the researcher analyzed and compared flow property, oil absorption, moisture absorption, and sensory character assessment of tapioca starch, potato starch, and mung bean starch. The researcher, at the end, confirmed that mung bean starch is suitable and can replace talcum in the formulation for the development of pressed powder.

2. Corn flour

According to Anderson et al. (2011), corn flour functions as an absorbent, binder, and bulking agent. The water absorption capacity and oil absorption capacity of yellow corn is the highest among dark purple corn, light purple red corn, and light purple corn (Akaffou et al., 2018). Corn flour absorbs oil from the skin and make it look fresh and radiant, and vitamins and minerals from corn flour help to delay the appearance of fine lines and wrinkles on the skin (Syeda, 2022).

3. Rice bran flour

Rice bran flour is a by-product from a rice milling industry which is the most important staple food for major parts of the human populations. It contains vitamins and minerals and able to serve as an important raw ingredient for nutraceutical development (Sharif et al., 2014: Sardarodiyan & Salehi, 2016). At the same time, the fiber components of rice bran flour give the properties for texture and stabilizing of the formulations (Sardarodiyan & Salehi, 2016). Rice bran extract, obtained from rice bran flour, showed effects on the sunscreen efficacy of two oil in water emulsions and one gel emulsion preparations, and increased the SPF values of them (Thongtan, 2018).

2.1.2.7 Mica: Mica is also known as potassium aluminum silicate dihydrate. Mica is available as creamy, wet ground or matte, dry ground ones. Sericite is a mineral, comparable to white mica in shape and composition. It is a very fine powder with silky shine effect. It is soft and smooth and also give a slippery feel on the skin. Sericite may be coated with silicone and other treatments for water repellency and skin adhesion characteristics (Schlossman, 2001).

2.1.2.8 Polymers: Polymers are mainly to increase the texture. Among these polymers, Nylon-12 and Nylon-6, lauroyl lysine, boron nitride, polyethylene, polypropylene, ethylene acrylates, polymethyl methacrylate (PMMA) and silica beads, polyurethane powders, silicone powders, borosilicate, microcrystalline cellulose, acrylate copolymers, Teflon® and Teflon® composite, polyvinylidene copolymers, and composite powders that are coated on inexpensive beads to reduce costs and increase effectiveness, like nylon/mica, silica/mica, lauryl lysine/mica and boron nitride/mica.

Many of these polymers are treated with silicones, titanates, lecithin, etc. for increased effectiveness (Schlossman, 2001).

2.1.2.9 Colorants: Titanium dioxide and zinc oxide gives pigment and they are ultrafine organics, inorganics, carmine, and pearlescent pigments either predispersed or treated are found in all face powders because the textures of these colorants are not actual pleasing (Schlossman, 2001). Color grinding can bring into maximum shade development. Poor grinding of color will outcome in underdevelopment of shade intensities and in tones that differ from those obtained by well-organized grinding. To bring out the maximum color in each batch of a face powder, the pigments used must be of uniform size of particle (Faber, 2012).

2.1.2.10 Perfumes: The use of perfumes is important for face powder, which needs them because most of the raw materials used in face powders are earthy sniffing and should be masked properly. Perfumes should express stability and low volatility (Schlossman, 2001). It is important that the perfume used be nonirritating. The fragrance must also be compatible with all of the other powder ingredients as problems with rancidity, heterogeneity of odor, and discoloration might happen from inappropriate scent choice. Perfume may be mixed into one of the very potential absorbent raw ingredients or it may be sprayed into the entire batch to combine properly and uniformly distributed (Faber, 2012).

2.1.2.11 Preservatives: Preservation of face powders are usually not a problematic because they are used dry, however, minor quantities of antibacterial are suggested (Schlossman, 2001; 2006). There are indeed some natural mediators having antimicrobial activity. When compared with synthetic preservatives, however, the activity is generally lower and directed to a less broad spectrum of microbes. The preservative effect of most natural ingredients is principally based on their antioxidant activity preventing oils and fats from becoming rancid. Typical natural preservatives are extracts (grapefruit seeds, rosemary), essential oils (tea tree, neem seed, thyme), and vitamins (vitamin e, vitamin c) (Bombeli, 2021). Nowadays, essential oils attract attention for cosmetic and pharmaceutical industries die to their potential as active pharmacological compounds or natural preservatives. They usually considered as safe due to their natural origin. Although most of them are regarded as safe, some of them may cause risk of irritation, sensitization, phototoxicity or allergic reactions

(Dreger & Wielgus, 2013). The global essential oils market has led by the growth in demand for organic and natural hygienic products owing to increasing awareness of health problems among consumers. 29% demand are for fragrances, cosmetics and aromatherapy (Sharmeen et al., 2021).

2.1.2.12 Binding agents: The properties of a binder provide creaminess to the face powder, helps to improve compression and adhesion, advances pigments, and improves water resistance, pick-up, and deposit. If the binder level is used too high, it may be hard to remove the powder with a puff. Hence, high levels may encounter glazing of powder surface, making it waxy looking, with very little or no pay-off (Schlossman, 2001).

According to Farber (2012), the number of binders employed in face powders is varied and extensive. There are, however, five basic types of binders used:

1. Dry binder

The dry binders such as the metallic stearates (zinc or magnesium stearate) increase pressure for firm compacting.

2. Oil binder

Oils alone, such as mineral oil, isopropyl myristate, and lanolin derivatives, can be most useful for incorporation in formulas as binding agents. They are finding widespread use in a variety of compressed cake formulas. If natural oil is used as oil binder, anti-oxidant needs to be added.

1) Olive oil

Olive oil contains fatty acids, triglycerides, tocopherols, squalene, carotenoids, sterols, polyphenols, chlorophylls, volatile and flavor compounds. The anti-inflammatory effect is employed by both unsaponifiable and polar compounds, and the free radical-scavenging effect of virgin olive oil is because of the presence of polyphenols. It is applied topically to treat skin damage, such as contact dermatitis, atopic dermatitis, xerosis, eczema, rosacea, seborrhea, psoriasis, thermal and radiation burns, other types of skin inflammation and aging. When the oil is topically applied after UVB exposure, it can effectually decrease UVB-induced skin tumors, possibly via its antioxidant effects. Adverse cutaneous reactions to topically applied olive oil is seldom reported, but olive oil is considered in general as very weakly irritant

(Aburjai & Natsheh, 2003). At the present, it is widely accepted that minor components of olive oil also exert potent anti-inflammatory activities (Lin et al., 2018).

2) Coconut oil

Coconut oil from the nuts is used as an ingredient in remedies for skin infections and it is stable at high temperatures up to 76.6 ºC (Aburjai & Natsheh, 2003). Coconut is self-possessed of many free fatty acids including lauric acid, myristic acid), palmitic acid, caprylic acid, capric acid, oleic acid, linoleic acid, and stearic acid. The expression of inflammatory profile was lower in the coconut oil-treated group after exposure to UVB radiation. Topical coconut oil protects the skin from UV radiation. Coconut oil in concentrations of 5% to 40% (w/w) revealed bactericidal action in contrast to Pseudomonas aeruginosa, Escherichia coli, Proteus vulgaris, and Bacillus subtilis Cellular studies have also shown that monolaurin exhibits antiviral and antifungal activity (Lin et al., 2018).

3) Sweet almond oil

Almond oil has sclerosant assets, which have been used to expand complexion and skin tone. In a nonrandomized study, sweet almond oil in creams are more effective than the base cream at ameliorating the itching of striae and avoiding its development. Topical almond oil prevents the structural damage caused by UV irradiation (Lin et al., 2018).

3. Water-soluble binder

If water-based binders are used, aluminum godets should be avoided to prevent corrosion (Schlossman, 2001). The water-soluble binders used in the past were generally solutions of gums such as tragacanth, karaya, and arabic. In this category, synthetics such as PVP (polyvinylpyrollidone), methyl cellulose, and carboxymethyl-cellulose have also been used in water solutions. A preservative is essential in a gum medium and is useful in all binding solutions of this type to present bacteriological growth problems.

4. Water-repellent binder

Water-repellent binders are broadly used in compact face powders. Mineral oil, fatty esters of all types, and lanolin derivatives may be used and are mixed with a considerable quantity of water to aid in the formation of a smooth, solidly pressed cake. Inclusion of a wetting agent will help to uniformly distribute the moisture throughout the powder.

5. Emulsion binder

Because of the difficulties incurred when attempting to incorporate water-repellent binders homogeneously in compact face powder, researchers have developed the emulsion-type binding agents that are now widely used. Such emulsions allow for a uniform distribution of both the oil and water phase. Since an emulsion binder will not lose moisture as readily as a water-repellent one (due to its homogeneity), its use allows for a smoother running manufacturing procedure. The use of the oils in emulsion form also tends to prevent the lumping that may occur when oils alone are incorporated as binders in face powder. Emulsion binding agents may hire soaps such as triethanolamine stearate, nonionic emulsifiers, and glyceryl monostearate in mineral oil-water mixtures. Such formulations also make use of waxes, lanolin and lanolin derivatives, fatty esters, glycols, and numerous other raw materials.

## 2.2 Thanaka

### 2.2.1 Morphology of Thanaka

**Note** Scientific Name- *Hesperethusa crenulata* (Roxb) Roem (Shinmataung Thanaka)
Family - Rutaceae (Citrus family)
Subfamily - Aurantioidea
Tribe - Citrinae
Subtrive - Citrinae
Local Name - Thanakha
English Name - Chinese Box Tree

**Figure 2.1** Shinmataung Thanaka Tree

Thanaka tree originally grow in western and southern India, Punjab State, and the regions up to 4,000 feet high in northwestern Himalaya Mountains, Assam State, Orihssa and dry plains in Dakhina highlands, Zinmay, Thailand and the arid regions from Shwebo district in central Myanmar to Pyay in south. They also thrive in the dry forest in Myanmar (Win, 2007). It can be assumed that the habit of applying Thanaka started over 2400 years ago (Win, 2007). All parts of this plant are useful as indigenous medicine, cosmetics and perfumery. Application of Thanaka enhances beauty, acts as perfume and has sun protection, astringent and antiseptic properties (Aung, 2014). Shinmataung Thanakha has its legend and is more popular than other kinds. The bark of Shinmataung is regarded to be superior to that of other kinds because it is more aromatic and compact. Shinmataung Thanakha, well known in Myanmar, belongs to the family “Rutaceae” and genus “*Hesperethusa* M.Roem” (Aung, 2006). The bark of Thanaka tree is ground in circular motion with adequate amount of water on a circular, flat stone slab. Then, the yellowish white Thanaka paste is applied on the skin in a thin layer and let it dry.

Most Myanmar women use branches, stems and roots of Thanaka plant (Aung, 2014). Application of moisten wood powder on face undergoes in Burmese culture including some part of Northern Thailand, which noticeably preserve its visual productivities to prevent acne and oil skin, and give a soft and fresh skin texture as well as some skin brightening property with sun protection prevent sunburn, which accordingly causes wrinkle, freckle and dry skin condition incorporation into skin aging (Kanlayavattanakul et al., 2009).

### 2.2.2 Benefits of Thanaka

Thanaka has been using for various reasons. Seiverling et al. (2017) did research about the reasons for applying Thanaka. The researchers had interviewed twenty-five people from different regions in Myanmar and the participants were both men and women of ages 12-55. The reasons for application of Thanaka are for photo protection, skin cooling, skin lightening, acne treatment and prevention, beautification (make up), odour prevention, pruitus (itchy skin) relief, rhytid (wrinkle) reduction and scar reduction and the total number of the participants’ responses are as the following table 2.1.

**Table 2.1** Dermatologic Uses of Thanaka

| Reasons for application of thanaka | Number of responses |
|---|---|
| Photo protection | 25 |
| Skin cooling | 14 |
| Skin lightening | 13 |
| Acne treatment and preventation | 10 |
| Beautification (make up) | 9 |
| Odour prevention | 8 |
| Pruitus (itchy skin) relief | 8 |
| Rhytid (wrinkle) reduction | 7 |
| Scar reduction | 3 |

**Source** Seiverling et al. (2017)

Sun protection was the most frequently described purpose for applying thanaka. Thanaka extracts contain marmesin. Marmesin, a fourocumarin (like psoralen) has a $\lambda_{max}$ of 335 nm resulting in absorption of ultraviolet A radiation. If comparing with titanium dioxide, marmesin has equal efficacy in UVA absorption (Joo et al., 2004; Seiverling et al., 2017). In contrast to psoralens, the result of marmesin absorption of UVA is not mutagenic. Thus, the use of thanaka is for protection from the sun and to reduce rhytids (wrinkle) is supported by the presence of marmesin (Seiverling et al., 2017).

Younger contributors, acne prevention and cure was the most normally described purpose for applying Thanaka. Thanaka bark has strong anti-inflammatory, significant antioxidant, and slight antibacterial properties (Wangthong et al., 2010; Seiverling et al., 2017). Thus, Thanaka may impact two of the important etiologic pathways in acne pathogenesis-inflammation and bacterial overgrowth (Seiverling et al., 2017).

Skin cooling, whitening and beautification were extra reported reasons for claim of Thanaka (Seiverling et al., 2017). Thanaka contains menthol and alcohol, which can serve as its cooling properties. The skin lightening effects are connected to arbutin, which inhibits melanin invention by reducing tyrosinase activity (Wangthong et al., 2010; Seiverling et al., 2017).

Some contestants informed use of Thanaka for treatment of rashes and for prevention of bad odor. Thanaka may have anti-prutitic properties since it contains menthol, as is found in pramoxine and other anti-itch products. Anti-itch properties may also be due to the anti-inflammatory components skin to steroid creams. Martini & Valle (2015) also found out after six weeks of a mere application of a natural paste from Burma and a simplest chemical reagent are more than sufficient to hold off psoriasis, almost for one whole year (Martini & Valle, 2015). For odor prevention, Thanaka alone has a fragrance similar to sandalwood, and it is sometimes mixed with sandalwood, an aromatic plant derivative used in perfumes (Martini & Valle, 2015; Seiverling et al., 2017).

Marmin and marmesin, which are contained in the bark, the main constituent of Thanaka powder, are the very precursors of psoralens. Psoralens are anyhow contained in the root at extremely high percentages (Martini & Valle, 2015).

From the Thanaka bark powder, Kanlayavattanakul & Lourith (2012b) made sunscreen liquid foundation. SPF of the bark powder was comparable to titanium dioxide. That herbal foundation with high sunscreen strength protects UVA and UVB, is meeting the consumers’ demand on natural cosmetics (Kanlayavattanakul & Lourith, 2012b). Also, the stem bark powder of Thanaka was prepared by Jantarat et al. (2018) for use of the herbal ball along with *Andrographis paniculata* and *Centella asiatica* for acne treatment, which is also known as Benchalokawichian remedy, *in vitro* antimicrobial activity of gel study (Jantarat et al., 2018).

Cytotoxicity of Thanaka extract to human skin fibroblasts showed no toxicity at the highest concentration is 100µg/ml and cell morphology did not change. UVB-irradiation upregulates MMP-1 and reduces type I pro-collagen levels in human skin. However, it was found that treating fibroblasts with thanaka extract before UVB-induced. MMP-1 expression was reduced and type I pro-collagen was upregulated (Amornnopparattanakul et al., 2012).

Natural cosmetics are of important and receiving a highly demand in the cosmetic consumer. There are several herbal cosmetics have been approached meeting the consumers’ interest (Kanlayavattanakul & Lourith, 2012a). The world has noticed the significant attractiveness of Thanaka now. Many people know the beauty benefits of it. This leads to promote the business industry making the cosmetic products by the use Thanaka and break through the world cosmetic new market.

## 2.3 Quality Control

### 2.3.1 Ingredients

2.3.1.1 Flowability of powder: The flowability of powder is as according to European Pharmacopoeia 6.0 (2008) and European Pharmacopoeia 7.0 (2010). The extensive use of powders in the pharmaceutical business has generated a variety of methods for portraying the flow of powder. It is clear single and simple method cannot sufficiently describe the flow properties of pharmaceutical powders. The four commonly reported methods for testing powder flow are:

1. Angle of repose
2. Compressibility index or Hausner ratio
3. Flow rate through an orifice
4. Shear rate

1) Compressibility index and Hausner ratio

In recent years, the compressibility index and the thoroughly related Hausner ratio and it have become the simple, fast, and popular methods of forecasting powder flow characteristics. The compressibility index and the Hausner ratio are determined by measuring both the bulk volume and tapped volume of a powder

2) Basic methods for compressibility index and Hausner ratio

The basic procedure is to measure the unsettled apparent volume, ($V_0$), and the final tapped volume, ($V_f$), of the powder after tapping compressibility index and the Hausner ratio are calculated as follows:

$$\text{Compressibility Index} = 100 \times \frac{V_0 - V_f}{V_f}$$

$$\text{Hausner Ratio} = \frac{V_0}{V_f}$$

Alternatively, the compressibility index and Hausner ratio may be calculated using measured values of bulk density ($\rho_{bulk}$) and tapped density ($\rho_{tapped}$) as follows:

$$\text{Compressibility Index} = 100 \times \frac{\rho_{tapped} - \rho_{bulk}}{\rho_{tapped}}$$

$$\text{Hausner Ratio} = \frac{\rho_{tapped}}{\rho_{bulk}}$$

For the compressibility index and the Hausner ratio, the generally accepted scale of flowability is given in table 2.2.

3) Experimental considerations for the compressibility index and Hausner ratio

Compressibility index and Hausner ratio are not intrinsic properties of the powder. They are dependent upon the methodology used. Several important considerations affecting the determination are as follows;

A. The diameter of the cylinder used

B. The number of times the powder is tapped to achieve the tapped density

C. The mass of material used in the test

D. Rotation of the sample during tapping

In a free-flowing powder, the bulk and tapped densities will be closer in value. For more-poorly flowing materials, a greater difference between the bulk and tapped densities will be observed. These differences are reflected in the compressibility index and the Hausner ratio.

**Table 2.2** Scale of Flowability

| Compressibility index | Flow character | Hausner ratio |
|---|---|---|
| 1-10 | Excellent | 1.00-1.11 |
| 11-15 | Good | 1.12-1.18 |
| 16-20 | Fair | 1.19-1.25 |
| 21-25 | Passable | 1.26-1.34 |
| 26-31 | Poor | 1.35-1.45 |
| 32-37 | Very poor | 1.46-1.59 |
| > 38 | Very, very poor | > 1.6 |

**Source** European Pharmacopoeia 6.0 (2008); European Pharmacopoeia 7.0 (2010)

#### 2.3.1.2 Water absorption capacity

Water absorption capacity (WAC) is the capacity to absorb water and swell and the consistency improved. The WAC measures the water holding ability by the sample after swelling in excess water. It depends on the availability of the hydrophilic groups which bind the water molecules and also on the gel-forming capacity of the macromolecules. WAC relates to other functional properties such as emulsification, solubility, adhesion, dispersibility, wettability, cohesion, viscosity and gelation (Twinomuhwezi et al., 2020). The sample with high water absorption may have more hydrophilic constituents. Water absorption capacity can be tested not only in ingredients, but also in finished products.

#### 2.3.1.3 Oil absorption capacity

Oil absorption capacity (OAC) is largely qualified to the entrapment of oils physically (Twinomuhwezi, 2020). OAC varies in the presence of non-polar side chain. This is potentially useful in extension of shelf life particularly products where fat absorption is desired (Chandra et al., 2015). Oil absorption capacity can be tested not only in ingredients, but also in finished products.

### 2.3.2 Finished Products

Faber (2012) mentioned time is a critical factor in modern laboratories and manufacturing industries. Nevertheless, product quality is a major necessary, and excessive urgency may result in disaster. It is to correlate speed with accuracy and a comprehensive coverage of details. And quality is also significant, since business is very largely dependent on reputation.

2.3.2.1 Packaging: In the case of compressed powder, the pans being glued into the compact must be regulated against underfilling or overfilling. Underfilling will result in compacts which are aesthetically undesirable. Overfilling will result in the breakage of tablets and the dusting of powders in the compact. It is imperative that the formula of the powder be the one which will not result in any excessive dusting in the case. The fit of the pan in the compact and the glues used are also extreme importance, since an imperfect fit may result in the pans falling out of the compacts holding them (Faber, 2012).

2.3.2.2 Dispersion of color: The pigment of a face powder must be homogeneously dispersed in the powder base. There must be no evidence of color streaks or incompatibility in the powder dispersion due to poor pulverization or bleeding of color. The uniformity of a powder can easily be checked by spreading it on a white paper and examining it with a magnifying glass. If any nonuniformity is detected, further processing to achieve maximum color development should result in homogeneity (Faber, 2012).

2.3.2.3 Pressure test: In a compressed powder, the pressure applied must be of a uniform nature, since the presence of air pockets will result in a cake which is easily broken. Uniformity and hardness of a cake are best checked by means of a penetrometer. Readings on the tablet should be taken at various points to ensure that the product is of proper hardness and that the pressure has been applied homogeneously (Faber, 2012).

2.3.2.4 Breakage test: The most successful manner of checking the tendency to a pressed powder tablet to break or chip is to drop the assembled compact on a wooden surface several times from a height of 8 to 10 in. If the cake remains unbroken, it is an indication that the compact can travel and will undergo normal handling without unsatisfactory results (Faber, 2012).

2.3.2.5 Stability test (International Federation of Societies of Cosmetic Chemists, 1992): Stability test is to control a given product in the container has a tolerable shelf-life underneath the situations of marketplace, which it is to be traded. The test conditions are as follows:

1.Temperature and humidity test: There are a few standard test conditions. 4 ºC/ambient humidity is useful for control purposes. 20 ºC or 25 ºC/ambient humidity is preferable rather than room temperature because ambient temperature and humidity can subject to considerable variation such as seasonal and geographical. Storing the samples at elevated temperatures is a more constant degree of acceleration and make accurate stability predictions. 37 ºC/ambient humidity, 45 ºC/ambient humidity, and 37 ºC/80 % relative humidity are also proposed as standard conditions to stability test samples.

For duration, 4 ºC and room temperature should be for the projected shelf-life of the product. 37 ºC is 3-6 months maximum, 45 ºC is 1-3 months maximum, and 37 ºC/80 % relative humidity is for 1 month maximum.

2. Cycling test: Cycling test is the test under conditions which are periodically changed. It reveals inadequacies faster than storage at a constant temperature. For example, cycle 24 hourly at 45 ºC and room temperature. Another suggested condition is 37 ºC/80 % relative humidity, alternating 24 hourly.

3. Freeze/Thaw test: Freeze/Thaw (-30 ºC and room temperature) test is for solutions, emulsion, creams and all other liquid or semi-solid products. It provides evidence of emulsion stability, tendency to crystallization, deposition and clouding.

4. Light or UV exposure test: Light can take about change in products. Goods that are possible to being exposed to light in the market should be tested by exposure to light. Recommended test condition is north-facing daylight. Direct sunlight exposure should be avoided because products are seldom exposed to it. Another suggested condition is constant contact in a light testing cabinet. This test is up to a maximum of 1 month.

5. Mechanical test: This is the vibration test on a suitable vibrator for some hours. Different samples should be treated with different frequencies and amplitudes of vibration. Emulsions are centrifuged to access the stability.

2.3.2.6 Preference test

According to Faber (2012), the essential characteristics of a good face powder is covering, slip, absorbency, adhesiveness and bloom. To examine the satisfaction of the compact powders, Jarupinthusophon and Anurukvorakun (2020) investigated the products on 20 volunteers using questionnaire compared to the satisfaction with the commercial product in the blinded experiment. The questions concerning compact powder efficacy were about concealment, slipperiness, skin adhesion, color, spread, and skin softening ranging from 1 (strongly dissatisfied) to 5 (strongly satisfied).

According to Faber (2012), payoff of a pressed powder should always be checked on the skin. If the pressure on the cake is too high, there will be insufficient adhesion of the material to the puff. If the pressure is too low, the cake will have a tendency to crumble and break.

## 2.4 Related Research

Kanlayavattanakul and Lourith (2012a) analyzed the stem and bark powder of Thanaka or *Naringi crenulate* cultivated from Thailand. They found out that the Carr index and Hausner ratio of Thanaka stem and bark were 47.50 ± 0.71 % and 1.91 ± 0.03, and 42.10 ± 0.42 and 1.73 ± 0.01, respectively. Although bark was better flowed than stem, according to the Carr index and Hausner ratio both of them were greater than 25 % and 1.5 respectively, so both Thanaka stem and bark were poor flowability. The SPF of stem was 1.13 ± 0.04 and for bark was 1.51 ± 0.31, but less than the standard sunscreen, titanium dioxide (1.99 ± 0.09). $L^*$ of bark (81.49 ± 0.01) was lower than stem (86.03 ± 0.01) makes Thanaka stem in lighter color. After accelerated test and 3 months storage at 3 different temperatures, $L^*$, $a^*$, and $b^*$ of both bark and stem were stable in particular ΔE was less than 1.5 as it was 0.02 – 0.43. Then, the researchers chose Thanaka stem for loose powder preparation by combining with zinc stearate, zinc oxide, kaolin, magnesium carbonate, jojoba oil, titanium dioxide, mica, and pigment. The preference of the developed products was comparatively evaluated by covering, adhesiveness, slip, and color, ranging from poor to excellent (+1 - +5), and the highest score Thanaka loose powder formula was continued for stability test.

Teeratanan (2018) did a thesis to develop pressed powder from natural starches. The researcher analyzed three starches, tapioca starch, potato starch, and mung bean starch on physical characteristic, repose angle, flow property, oil absorption, moisture absorption, and sensory assessment (spreadability, coverage, adherence, and feeling). For overall, mung bean starch was chosen to replace talcum and develop into compact powder. The formulated mung bean pressed powders were examined durability and selected the most suitable formulation on touch, adhesion and durability. After that, the selected formula was explored physical characteristics, color distribution, sensory evaluation (spreadability, coverage, adherence, feeling, pick up, flyaway), and stability. Lastly, that formula was compared texture, shift, feeling, concealment, and satisfaction with talcum compact powder on the forearm of 20 volunteers. The overall satisfaction of mung bean pressed powder was more than talcum pressed powder was found out by statistically ANOVA analysis, and concluded that mung bean starch can be used as main component instead of talcum in the future market.

Jarupinthusophon and Anurukvorakun (2021) developed jasmine rice flour compact powder by modifying the rice flour with PEG-50 shea butter and performed chemical property analysis using fourier transform infrared spectroscopy (IR). Then, physical properties, such as flowability and water resistance, of modified rice flour, rice flour and talcum are compared. And then, compact powder was formulated with talcum and rice flour replaced talcum compact powders. After that, payoff test and breakage test were performed. Finally, concealment testing, *in vitro* sun protection factor, patch testing and satisfaction testing (concealment, slipperiness, skin-adhesion, color, spread, and skin-softening) were followed by comparing with commercial product.

Shinde et al. (2021) formulated semi herbal anti acne compact face powder by A*llium sativum* and M*yristica fragrans* extract. *Myristica fragrans* (Nutmeg) and *Allium Sativum* (Garlic) were extracted (powder) and applied into the compact face powder. Physical evaluation, Organoleptic evaluation (nature, odor, color, texture), physicochemical evaluation (total ash, water soluble ash, acid insoluble ash, moisture content, and pH), powder characteristics (particle size, angle of repose, bulk density, and tapped density), antimicrobial evaluation (Escherichia coli and Pseudomonas aureginosa), evaluation of final formulation (shade test, pay-off test, pressure test, breakage test, and abrasive test), and irritancy test (irritancy, redness, swelling) were performed.

# CHAPTER 3

# METHODOLOGY

## 3.1 Materials & Chemicals and Equipments

### 3.1.1 Materials & Chemicals

| | |
|---|---|
| 3.1.1.1 Beaker | (Duran/ Duran Group GmbH, Germany) |
| 3.1.1.2 Compact powder pan | (No brand, Thailand) |
| 3.1.1.3 Cuvette | (VWR/ VWR International, Germany) |
| 3.1.1.4 Dropper | (Duran/ Duran Group GmbH, Germany) |
| 3.1.1.5 Finger cot | (Glove/ DK Group Co., Thailand) |
| 3.1.1.6 Graduated cylinder | (ISOLAB/ ISOLAB GmbH, Germany) |
| 3.1.1.7 Microcentrifuge tube | (Corning/ Eppendorf, Germany) |
| 3.1.1.8 PMMA plate | (Helioplate HD6/ Helioscreen, France) |
| 3.1.1.9 Corn flour | (PR Foodland, Thailand) |
| 3.1.1.10 Danox Preservative 8000 Organic | (All Ingredients Group Co., Ltd, Thailand) |
| 3. 1.1.11 Mung bean starch | (Pine brand, Thailand) |
| 3. 1.1.12 Nitric acid | (Merck & Co., Germany) |
| 3. 1.1.13 Rice bran flour | (Thai Edible Oil Co., Ltd, Thailand) |

| | |
|---|---|
| 3. 1.1.14 Soybean oil | (Thai Vegetable Oil Public Company Limited, Thailand) |
| 3. 1.1.15 Thanaka | (Chiang Rai, Thailand) |
| 3. 1.1.16 White petrolatum | (H&R Gruppe, Germany) |

### 3.1.2 Equipments

| | |
|---|---|
| 3.1.2.1 3-digit balance | (JB3002-L-G/ Mettler Toledo, Switzerland) |
| 3.1.2.2 4-digit balance | (SI-234 Denver/ Becthai Co., Ltd., Thailand) |
| 3.1.2.3 Autopipette | |
| 1. 20-200 µl auto pipette | (Pipeman G/ Gilson, France) |
| 2. 100-1000 µl auto pipette | (Eppendrof, Germany) |
| 3.1.2.4 Compressing machine | (Charn Intertech Co., Ltd., Thailand) |
| 3.1.2.5 Differential scanning calorimetry | (Mettler Toledo AF/ Analytical Co., St., Switzerland) |
| 3.1.2.6 Dry blender | (HR 2056/ Philips, China) |
| 3.1.2.7 FTIR-ATR spectroscopy | (Nicolet™ iS50, Thermo Fisher Scientific, USA) |
| 3.1.2.8 Hammer mill | (20/CMC, Thailand) |
| 3.1.2.9 Hot air oven | (UNE-500 Memmert/ K.K. Scientific Co., Ltd., Thailand) |
| 3.1.2.10 Mettler Toledo press | (ME-00026763/ Mettler Toledo, Switzerland) |
| 3.1.2.11 Microcentrifuge | (Spectrafuge™ 16M/ Labnet, USA) |

| | |
|---|---|
| 3.1.2.12 Microwave digestion system | (Ethos™ UP/ Milestone, Italy) |
| 3.1.2.13 Refrigerator | (SJ-W40J 13.3/ Sharp, Japan) |
| 3.1.2.14 Scanning electron microscope | (MIRA 4/ Tescan, Czech Republic) |
| 3.1.2.15 Sieve shaker | (AS 200 digit/ Retsch, Germany) |
| 3.1.2.16 Spectrophotometer | (UltraScan® Pro/ Hunter Lab, USA) |
| 3.1.2.17 SPF analyzer | (UV-2000S/ Labsphere®, USA) |
| 3.1.2.18 Trace element analyzer | (iCAP™ RQ ICP-MS/ Thermo Fisher Scientific, USA) |
| 3.1.2.19 Vortex Mixer | (Vortex Genie 2™/ Scientific Industries, USA) |
| 3.1.2.20 X-ray diffractometer | (X’Pert Pro MPD/ PANalytical, Almelo, Netherlands) |

## 3.2 Research Methods

### 3.2.1 Preparation of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour

Thanaka powder, corn flour, mung bean starch and rice bran flour were prepared as follows:

3.2.1.1 Preparation of Thanaka powder

Thanaka stem was purchased from Mae Sai District, Chiang Rai. The bark was removed from the stem. Thanaka bark was chipped down into appropriate size and sun dried. Before reducing into smaller size by hammer mill machine, the bark was placed in hot air oven at 45ºC to avoid stickiness caused by the moisture. After grinding for 3-4 times, the powder was sieved by manual or by sieve shaker. Grinding was proceeded again by using blender. The powder, which is less than 45 µm, was kept in air tight plastic zip lock bags for future use.

3.2.1.2 Preparation of corn flour

Corn flour was ground by blender to reduce the size, and sieved. The powder which is less than 45 µm, was kept in air tight plastic zip lock bags for future use.

3.2.1.3 Preparation of mung bean starch

Mung bean starch was sieved. The powder, which is less than 45 µm, was kept in air tight plastic zip lock bag for future use.

3.2.1.4 Preparation of rice bran flour

Rice bran flour was sieved. The powder, which is less than 45 µm, was kept in air tight plastic zip lock bag for future use.

### 3.2.2 Evaluation of Physicochemical Properties of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour

3.2.2.1 Total elemental analysis

Total elemental analysis was slightly modified to the method of Tanase et al. (2012). Four elements, Arsenic (As), Cadmium (Cd), Lead (Pb) and Mercury (Hg), present in Thanaka powder, corn flour, mung bean starch and rice bran flour were investigated. Inductive Coupled High Frequency Plasma Mass Spectrometry (ICP-MSMS) was analyzed in ppb unit by Aligent Technology calibration standard solution, and was performed duplicate. For element detection, 0.1 g of sample was digested with 10 mL of nitric acid (65%, Suprapur®) in digestion system microwave (Ethos™ UP, Milestone, Italy), fitted with PTFE vessels. The resulting solution was diluted into 100 with Deionized water for As, Cd, Pb, and Hg detection. The ICP-MSMS was evaluated by using the trace element analyzer (iCAP™ RQ ICP-MS, Thermo Fisher Scientific, USA).

3.2.2.2 Fourier Transform Infrared Spectroscopy (FTIR)

This method was slightly modified to the method of Jaiturong et al. (2020). Identification of Thanaka powder, corn flour, mung bean starch and rice bran flour were compared by Infrared Fourier Transform Spectroscopy with Attenuated Total

Reflectance technique, FTIR-ATR (Nicolet™ iS50, Thermo Fisher Scientific, USA). The sample were scanned between the wavenumber range of 400-4,000 $cm^{-1}$ for 32 scans. The spectra resolution was 4 $cm^{-1}$.

#### 3.2.2.3 Color

The color of Thanaka powder, corn flour, mung bean starch and rice bran flour were evaluated by both eyes and spectrophotometer (UltraScan® Pro, Hunter Lab, USA). $L*$(whiteness to blackness), $a*$ (redness to greenness) and $b*$ (yellowness to blueness) were recorded three times.

#### 3.2.2.4 Scanning Electron Microscopy (SEM)

This method is slightly modified to the method of Almeida et al. (2020). The morphology of Thanaka powder, corn flour, mung bean starch and rice bran flour were analyzed by field emission gun-scanning electron microscopy (FEG-SEM) in a MIRA 4 (Tescan, Czech Republic). The samples are prepared with gold plating to avoid charging. 5-10 keV of voltage and magnification of 2 kx will be used to obtain the images of each sample.

#### 3.2.2.5 Differential Scanning Calorimetry (DSC)

This method was slightly modified to the method of Morales-Sanchez et al. (2009). A calorimeter, DSC Mettler Toledo (Mettler Toledo AF, Analytical Co., St. Schwerzenbach, Switzerland) equipped with a thermal analysis data station was used, where Thanaka powder, corn flour, mung bean starch and rice bran flour with distilled water 3:20 (w/w), respectively, were placed in an aluminum pan with pin (ME-00026763) and then sealed with a Mettler Toledo press (ME00119410), and allowed to equilibrate for approximately 1 hr. The samples were heated from 20 to 95 ºC at a rate of 5 ºC/min with $N_2$ 30 ml/min. The gelatinization onset ($T_o$), peak ($T_p$), and end ($T_e$) temperatures were computed automatically.

#### 3.2.2.6 X-ray Diffraction (XRD)

X-Ray diffraction patterns of Thanaka powder, corn flour, mung bean starch and rice bran flour were examined by slightly modified to the method of Jaiturong et al. (2020) by using an X-ray diffractometer (X’Pert Pro MPD, PANalytical, Almelo, Netherlands). Diffractograms were obtained from 5º-90º (2θ) at a scanning speed of 6 ºC/min. The % crystallinity was calculated by X’Pert HighScore Plus Program.

% Crystallinity = $\frac{A_C}{(A_C+A_B)}$ × 100%, where

$A_C$ = area of crystalline peaks of diffraction, and

$A_B$ = area of amorphous peaks of diffraction

3.2.2.7 Bulk density

Bulk density, or otherwise, apparent density, of Thanaka powder, corn flour, mung bean starch and rice bran flour were determined by referring to European Pharmacopoeia 7.0 (2010) method. The gradated cylinder of 250 mL should be readable to 2 ml. The sample powder was passed through a sieve with apertures greater than or equal to 1.0 mm to break up the agglomerates that may have formed during storage. Typically, 100 ± 0.1 g of sample is placed into that cylinder without compacting. Read the apparent volume ($V_0$). When the powder is more than 250 mL or less than 150 mL, different amount of powder between 250-150 mL was selected. Bulk density was calculated by mass of sample by apparent volume, $m/V_0$, in g/mL unit. The determination was replicated for three times for sample powders.

Bulk Density = $\frac{m}{V_0}$

3.2.2.8 Tapped density

Tapped density of Thanaka powder, corn flour, mung bean starch and rice bran flour were determined by referring to European Pharmacopoeia 7.0 (2010) method. The gradated cylinder of 250 ml should be readable to 2 ml. The mass of that graduated cylinder should be 220 ± 44 g. The sample powder was passed through a sieve with apertures greater than or equal to 1.0 mm to break up the agglomerates that may have formed during storage. Typically, 100 ± 0.1 g of sample is placed into that cylinder without compacting. The apparent volume ($V_0$) was recorded. If the powder is more than 250 mL or less than 150 mL, different amount of powder between 250-150 mL was selected. It was tapped for 10, 500 and 1,250 times at the rate of 250 ± 15 taps from a fixed height of 3 ± 0.2 mm per minute. $V_{10}$, $V_{500}$ and $V_{1250}$ were recorded. When the difference between $V_{500}$ and $V_{1250}$ is less than 2 ml, $V_{1250}$ would be final tapped volume, $V_f$. However, when the difference exceeds 2 mL, the increments e.g., 1,250 taps is repeated until the difference between successive measurements is less than 2 ml, and it was recorded as final tapped volume ($V_f$). The tapped density was calculated by the formula $m/V_f$ in g/ml. The determination was replicated for three times for samples powders.

$$\text{Tapped Density} = \frac{m}{V_f}$$

3.2.2.9 Flowability

From European Pharmacopoeia 6.0 (2008) and European Pharmacopoeia 7.0 (2010), in order to evaluate flowability of Thanaka powder, corn flour, mung bean starch and rice bran flour, compressibility index and Hausner ratio were calculated. The results were compared with table 2.2, and the flowability of sample powders was obtained.

3.2.2.10 Water absorption capacity

For water absorption capacity of Thanaka powder, corn flour, mung bean starch and rice bran flour, it was slightly modified to Chandra et al. (2015) method. 0.1 g of sample was mixed with 1,000 µL distilled water. The mixture was vortexed at the highest speed intensity for 60 s to be fully homogenous. It was allowed standing at ambient temperature (30 ± 2°C) for 30 min, and centrifuged at 3,000 rpm or 2,000 xg for 60 min to get clear liquid above. The water was decanted carefully. Water absorption was determined as percent water bound for 0.1 g of sample powder. The determination was replicated for three times for sample powders.

3.2.2.11 Oil absorption capacity

For oil absorption capacity of Thanaka powder, corn flour, mung bean starch and rice bran flour, it was slightly modified to Chandra et al. (2015) method, and was analyze by using soybean oil as from the reference, and also with olive oil and mineral oil. 0.1g of sample was mixed with 1,000 µL soybean oil. The mixture was vortexed at the highest speed intensity for 60 s to be fully homogenous. It was allowed standing at ambient temperature (30 ± 2°C) for 30 min, and centrifuged at 3,000 rpm or 2,000 xg for 60 min to get clear oil above. The oil was decanted carefully. Oil absorption was determined as percent oil bound for 0.1g of sample powder. The determination was replicated for three times for sample powders.

### 3.2.3 Stability of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour

Long term stability test of Thanaka powder, corn flour, mung bean starch and rice bran flour were slightly modified to the method of Kanlayavattanakul & Lourith (2012a) and Kanlayavattanakul et al. (2013) by keeping at ambient temperature (TA) and 45 ºC for 3 months without light exposure. During this test, the color, SEM, DSC, XRD, water absorption and oil absorption of natural powders were recorded and compared with initial.

For color stability measurement, color differences were calculated. The difference in color could not be detected by eyes when ΔE is less than 1.5 (Lourith & Kanlavattanakul, 2012). They could be analytically be detected as trace level where ΔE = 0-0.5, slight where ΔE = 0.5-1.5, noticeable where ΔE = 1.5-3.0, appreciable where ΔE = 3.0-6.0, large where ΔE = 6.0-12.0 and obvious where ΔE > 12.0 (Moreira et al., 2015). The color difference was calculated as the following formula.

$\Delta E = \sqrt{(L_2 - L_1)^2 + (a_2 - a_1)^2 + (b_2 - b_1)^2}$, where

ΔE = the color difference

$L_1$, $a_1$, $b_1$ = the coordinates of initial color

$L_2$, $a_2$, $b_2$ = the coordinates of final color

### 3.2.4 Formulation of Natural Face Powders

3.2.4.1 Formulation of natural loose powder (Table 3.1)

Method

1. All the ingredients in part A were prepared and mixed well in motor and pestle by trituration method.

2. After mixing well, the homogeneous mixture was filled into the package.

3. The best natural loose powder was selected by sensory evaluation, dispersion test and payoff test, and the developed natural loose powder was prepared after adding with natural preservative, which is as L2/222-Int.

4. The developed natural loose powder (L2/222-Int) was ready to investigate color, SPF analysis and FTIR measurements, and stability test can be performed

**Table 3.1** Natural Loose Powder Formulations

| Part | Ingredients | Function | % w/w | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | **L1** | **L2** | **L3** | **L4** | **L5** | **L5.1-Int** |
| A | Corn flour | Adhesion | 10 | 10 | 10 | 10 | 5 | 5 |
| | Thanaka powder | Pigment & absorbent | 22 | 22 | 27 | 32 | 37 | 37 |
| | Mung bean starch | Filler | 68 | 58 | 53 | 53 | 53 | 52.5 |
| | Rice bran flour | Filler | 0 | 10 | 10 | 5 | 5 | 5 |
| B | *Helianthus annus* seed oil, *Rosmarinus officinalis* leaf oil and *Foeniculum vulgare* oil | Preservative | - | - | - | - | - | 0.5 |

3.2.4.2 Formulation of natural compact powder (Table 3.2)

Method

1. All the ingredients in part A were prepared and mixed well in motor and pestle by trituration method.

2. Liquid binder was added into part A.

3. After mixing well, the homogeneous mixture was filled into the package and pressed.

4. The best natural compact powder was selected by sensory evaluation, dispersion test, payoff test and breakage test, and the developed natural compact powder was prepared after adding with natural preservative, which is as C3/32111-Int.

5. The developed natural loose powder (C3/32111-Int) was ready to investigate color, SPF analysis and FTIR measurements, and stability test can be performed.

### 3.2.5 Evaluation of Physicochemical Properties of Natural Face Powders

3.2.5.1 Sensory evaluation

To select the best formula of natural face powders, sensory assessment was evaluated. Natural face powders were applied with puff on the back of hand. Score the covering, slip, absorbency, adhesiveness and bloom between 1-5, where 1 is very poor, 2 is poor, 3 is fair, 4 is good, and 5 is very good (Teeratanan, 2018; Faber, 2012). The sensory evaluation of natural loose powder was compared with commercial translucent powder and commercial oil control powder benchmark products and natural compact powder was compared with commercial translucent compact powder benchmark product as control.

3.2.5.2 Dispersion test

To approve the uniformity of natural face powders, dispersion test was performed. Natural face powders were spread on a white paper by using a latex glove and examined. There must not be any evidence of color streaks or incompatibility in the powder dispersion of the chosen natural face powder (Teeratanan, 2018; Faber 2012).

**Table 3.2** Natural Compact Powder Formulations

| **Part** | **Ingredients** | **Function** | **%w/w** | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | **C1** | **C2** | **C3** | **C4** | **C5** | **C6** | **C7** | **C7.1-Int** |
| A | Corn flour | Adhesion | 4 | 4 | 19 | 19 | 29 | 29 | 29 | 29 |
| | Thanaka powder | Pigment & absorbent | 36 | 36 | 21 | 21 | 21 | 21 | 36 | 36 |
| | Mung bean starch | Filler | 47 | 37 | 37 | 31 | 21 | 31.5 | 16.5 | 16 |
| | Rice bran flour | Filler | 10 | 20 | 20 | 20 | 20 | 9.5 | 9.5 | 9.5 |
| B | Olive oil | Liquid Binder | 3 | 3 | 3 | 9 | 9 | 9 | 9 | 9 |
| | *Helianthus annus* seed oil, *Rosmarinus officinalis* leaf oil and *Foeniculum vulgare* oil | Preservative | - | - | - | - | - | - | - | 0.5 |

3.2.5.3 Payoff test

To approve the adhesive property of natural face powders, payoff test was performed. Natural face powders were analyzed with the puff (Jarupinthusophon & Anurukvorakun, 2021; Shinde et al., 2021; Teeratanan, 2018).

3.2.5.4 Color

The color of developed natural face powders were evaluated by both eyes and spectrophotometer UltraScan® Pro (Hunter Lab, USA), and record *L**(whiteness to blackness), *a** (redness to greenness) and *b** (yellowness to blueness) three times. The color difference before and after of stability test was calculated and compared (Lourith & Kanlavattanakul, 2012).

3.2.5.5 Sun Protection Factor (SPF) analysis

SPF of developed natural face powders were measured by using SPF analyzer (Labsphere®, UV-2000S, USA) and roughened PMMA (Polymethyl Methacrylate) plates roughness 6 µm. This method was slightly modified to the method of Lourith et al. (2017). First, blank is prepared by applying 0.0150 g of white petrolatum on the plate and spread well with finger cot. For sample preparation, 0.0150 g of white petrolatum was poured onto PMMA plate by spatula and spread by finger cot. Then, 0.0287 ± 0.0050 g of sample was transferred and spread well again within 1 min. Incubate both blank and sample in dark area for 15 min. After that, measurement was performed by user defined SPF method that the samples were measured at 9 different scanning spots on the plates. UVB protection efficacy as SPF, UVA protection efficacy as Boot star rating, and critical wavelength were recorded.

3.2.5.6 Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR of developed natural face powders were measured by slightly modified to the method of Jaiturong et al. (2020). Identification of the selected natural loose powder and natural compact powder were identified by Infrared Fourier Transform Spectroscopy with Attenuated Total Reflectance technique, FTIR-ATR (Nicolet™ iS50, Thermo Fisher Scientific, USA). The samples were scanned between the wavenumber range of 400-4,000 $cm^{-1}$ for 32 scans, and the spectra resolution was 4 $cm^{-1}$.

3.2.5.7 Breakage test

To approve the compact powder, which can resist for normal handling by user, breakage test is performed. Natural compact powders were dropped onto a wooden surface from a height of 8 inches 10 times and examined at how many times it was broken. Natural compact powders were examined, and the ones which remain unbroken for 2-3 times were selected for further development (Jarupinthusophon &

Anurukvorakun, 2021; Teeratanan, 2018). Breakage time of natural compact powder was compared with commercial translucent compact powder benchmark product as control

When examining the sensory evaluation, dispersion test, payoff test, color, SPF analysis, FTIR and breakage test, one natural loose powder and one natural compact powder were selected to be developed and continued for the stability test.

### 3.2.6 Stability of Developed Natural Face Powders

The developed natural face powders were continued for stability test. Heating-cooling cycling stability test of certain products were conducted by 6 cycles, 24 h each of heating and cooling, 45 ºC and 4 ºC (Kanlayavattanakul & Lourith, 2012a). Sensory evaluation, dispersion test, payoff test, color, SPF analysis, FTIR and breakage test, before and after of this stability test, were recorded and compared.

### 3.2.7 Statistical Analysis

Data are presented as the mean ± SD. The parameters are compared and analyzed using with a significance level of $p$ value < 0.05 by SPSS program.

# CHAPTER 4

# RESULTS AND DISCUSSION

## 4.1 Preparation of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour

The prepared Thanaka powder, corn flour, mung bean starch and rice bran flour were less than 45 µm. The appearance of natural powders were shown in Figure 4.1 and their characteristics were as mentioned in Table 4.1.

(A) (B) (C) (D)

**Figure 4.1** Appearance of Thanaka Powder (A), Corn Flour (B), Mung Bean Starch (C) and Rice Bran Flour (D)

**Table 4.1** Characteristics of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour

| Natural Materials | Characteristics |
|---|---|
| Thanaka powder | Yellowish light brown, fine powder |
| Corn flour | Pale yellow powder, very adhere to each other |
| Mung bean starch | White, fine powder, adhere to each other |
| Rice bran flour | Pale yellow powder, very adhere to each other |

## 4.2 Evaluation of Physicochemical Properties of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Four

### 4.2.1 Total Elemental Analysis

To ensure the safety of materials for topical product application, elemental analysis of Thanaka powder, corn flour, mung bean starch and rice bran flour was performed. The detection of elements from certain natural materials can be seen in Table 4.2. According to ASEAN guidelines on limits of contaminants for cosmetics, maximum limits of Arsenic (As) is not more than 5 ppm, maximum limits of Cadmium (Cd) is not more than 5 ppm, maximum limits of Lead (Pb) is not more than 20 ppm and maximum limits of Mercury (Hg) is not more than 1 ppm (Association of South East Asian Nations (ASEAN), 2019). Therefore, all of the natural materials were detected below from the certain regulated limitations and would be safe for applying in cosmetic formulations.

**Table 4.2** Total Elemental Analysis of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour

| **Natural Materials** | **Elements** | | | |
|---|---|---|---|---|
| | **As (ppm)** | **Cd (ppm)** | **Pb (ppm)** | **Hg (ppm)** |
| Thanaka powder | < 0.00001 | < 0.00004 | 0.65401± 0.07025 | < 0.00010 |
| Corn flour | < 0.00001 | < 0.00004 | 0.98608± 0.04106 | < 0.00010 |
| Mung bean starch | < 0.00001 | 0.00293± 0.00254 | 1.70108± 0.01795 | < 0.00010 |
| Rice bran flour | < 0.00001 | 0.06486± 0.05479 | 0.6035± 0.02577 | < 0.00010 |

### 4.2.2 Fourier Transform Infrared Spectroscopy (FTIR)

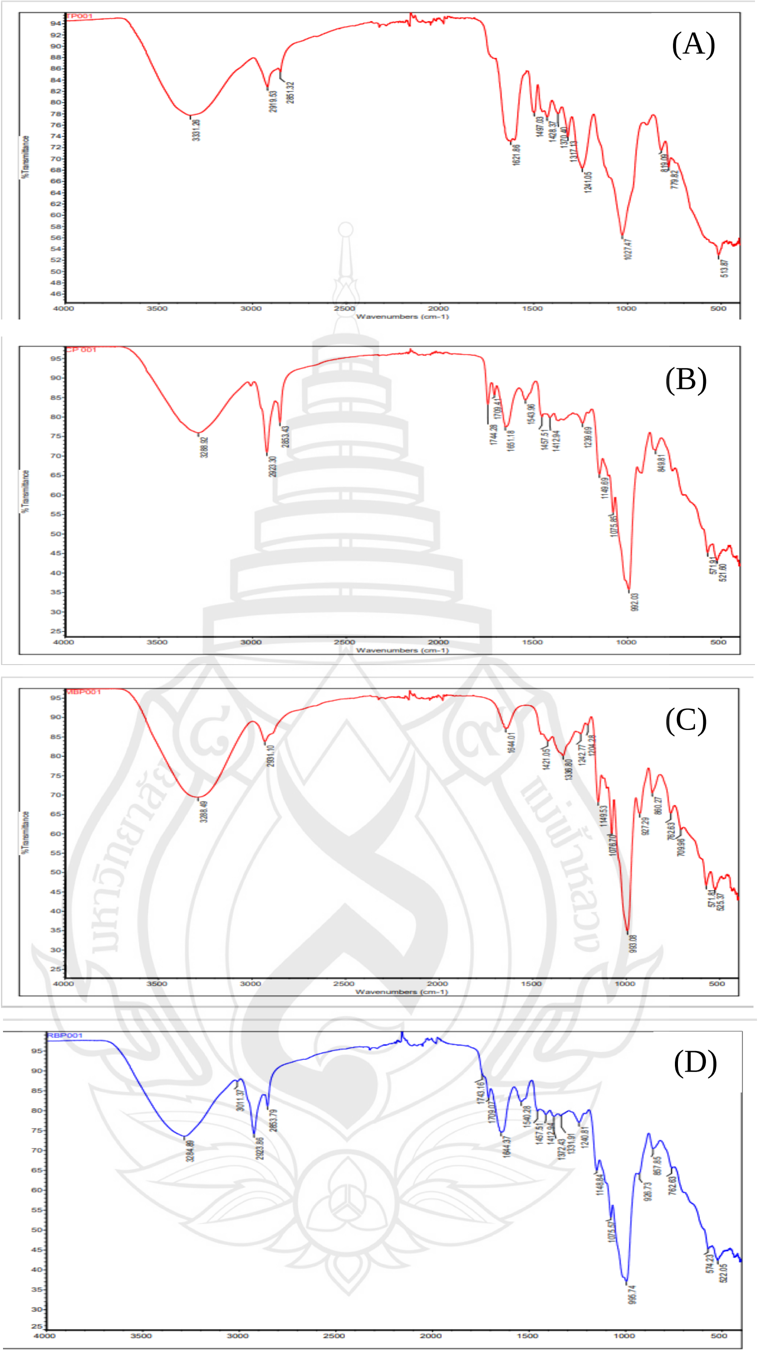


**Figure 4.2** Fourier Transform Infrared Spectroscopy (FTIR) Spectrums of Thanaka Powder (A), Corn Flour (B), Mung Bean Starch (C) and Rice Bran Flour (D)

FTIR spectrums of Thanaka powder, corn flour, mung bean starch and rice bran flour were as shown in Figure 4.2. Corn flour and rice bran flour provided IR spectrum very similarly. Mung bean starch did not have peaks in 1,670-1,780 cm$^{-1}$, which was C=O Carbonyl compounds (Sitanggang, et al., 2020). Hydroxyl group intensity in 3,700-3,000 cm$^{-1}$ could be found in all samples (Sitanggang, et al., 2020; Salim et al. 2021). Thanaka powder had peaks in the area 1,510 cm$^{-1}$, which presented lignin and lignin cellulose in the sample powder (Cho & Kobayashi, 2018).

### 4.2.3 Color

The color of natural powders was measured by spectrophotometer and their *L**, *a** and *b** values were followed in Figure 4.3 and Table A1. The color of Thanaka powder resembled the Asian yellowish light brown skin color. And this also related the combination of light shade from the positive value of *L** 75.63±0.01 with yellowness from the positive value of *b** 23.45±0.02 and a tiny amount of redness from the positive value of *a** 5.58±0.02. Corn Flour showed pale yellow color that light shade from *L** 85.76±0.01 and yellow shade from *b** 12.93±0.01 with minor green shade from *a** (-0.33±0.02). Mung bean starch was a white powder with major white shade from *L** 96.78±0.02 with a tiny amount of yellowness from *b** 1.73±0.02 and a tiny amount of greenness from *a** -0.04±0.00. Rice bran flour gave pale yellow color with light shade from *L** 85.47±0.02 and yellow shade from *b** 11.30±0.01 with a very small amount of redness from *a** 0.94±0.02.

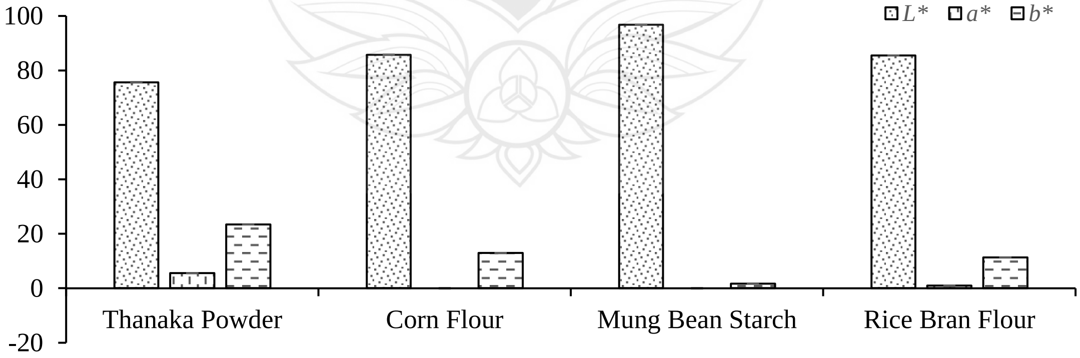


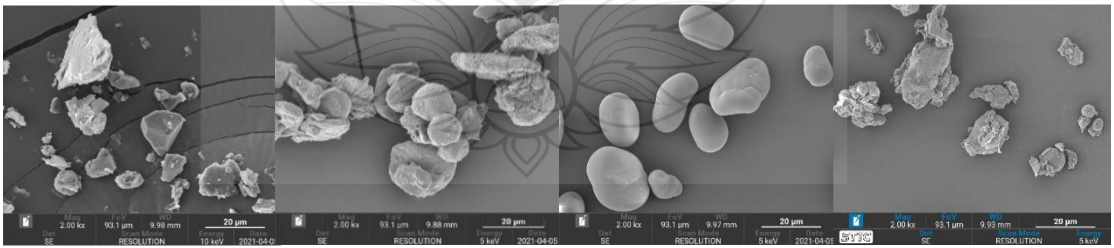
**Figure 4.3** *L**, *a** and *b** of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour

### 4.2.4 Scanning Electron Microscopy (SEM)

The morphology of Thanaka powder, corn flour, mung bean starch and rice bran flour were as shown in Figure 4.4. Thanaka powder presented plate like crystals with fractures on the surface. Corn flour was both small and large particles, round and rounded-angular shaped clusters with no smooth surface which was similar to the report of Sahi et al. (2016). Mung bean starch is pea-shaped, varying from oval to round irregular granules, smooth surface with shallow grooves without any cracks which was similar to the report of Hoover et al. (1997) and Zou et al. (2019). Rice bran flour was round and rounded-angular shaped clusters with no smooth surface and had some soft edges which was similar to the report of Coutinho et al. (2013) and Sansuwan et al. (2014).

According to Tri-County Technical College (2013), the shape of particle can influence the texture feeling when rubbed between fingers. Mung bean starch could give smooth feeling the most among them, followed by corn flour, rice bran flour and thanaka powder where the smoothness was gradually reduced and this result was conformed with the SEM images.

The shape of particle could also influence the compact of the powder. The more angular particles with a rough surface texture (for e.g., Thanaka powder) could resist compactive effect since they could interlock together, where the more rounded particles with smooth surface texture (for e.g., mung bean starch) would not be stable under load since the particles would shift against each other (Tri-County Technical College, 2013).



(A) (B) (C) (D)

**Figure 4.4** Morphology of Thanaka Powder (A), Corn Flour (B), Mung Bean Starch (C) and Rice Bran Flour (D) by Scanning Electron Microscopy (SEM)

### 4.2.5 Differential Scanning Calorimetry (DSC)

The thermal properties of Thanaka powder, corn flour, mung bean starch and rice bran flour were as shown in Figure 4.5. Thermal properties can affect the spreadability and wearability of cosmetics (Perkinelmer, 2011).

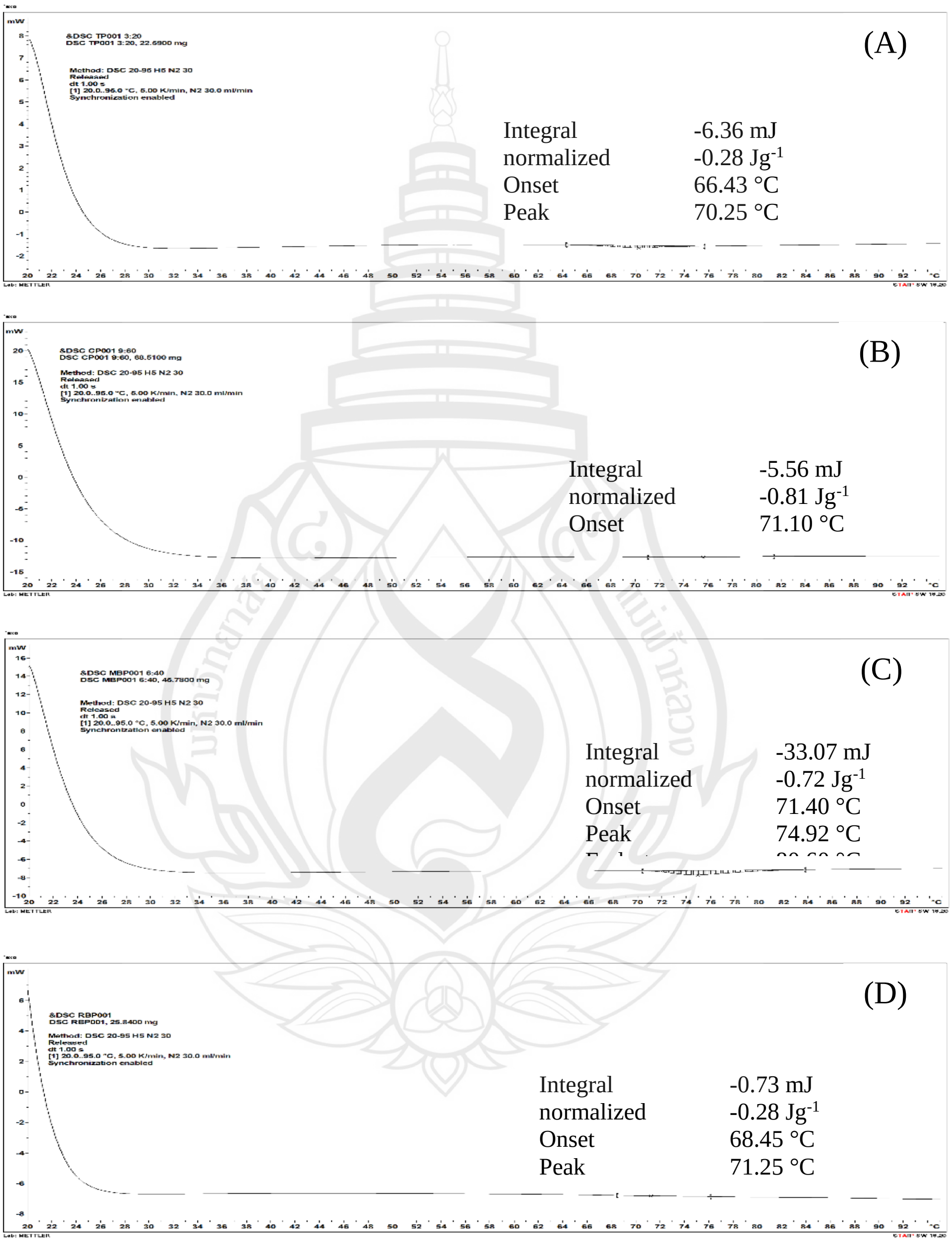


**Figure 4.5** Thermal Properties of Thanaka Powder (A), Corn Flour (B), Mung Bean Starch (C) and Rice Bran Flour (D) by Differential Scanning Calorimetry (DSC)

Thanaka powder started to melt ($T_o$) at 66.43 °C, melting peak ($T_m$) was at 70.25 °C, and melting ended ($T_e$) at 73.39 °C.

Corn flour's $T_o$ was at 71.19 °C, $T_m$ was at 75.58 and $T_e$ was at 80.85. According to the report by Budi et al. (2015), the thermal properties of corn flour were lower $T_o$ (65.42 °C), lower $T_m$ (73.21 °C) with higher $T_e$ (81.84 °C) than these results and this would be because corn flour was obtained from different country source.

Mung bean starch's $T_o$ was at 71.39 °C, $T_m$ was at 74.92 °C, and $T_e$ was at 80.60 °C. This result related to the report by Zou et al. (2019), the ranges of thermal properties of mung bean starch were $T_o$ 57.11-70.70 °C, $T_m$ 67.19-74.60 °C and $T_e$ 77.46-83.67 °C depending on different methods of starch extraction.

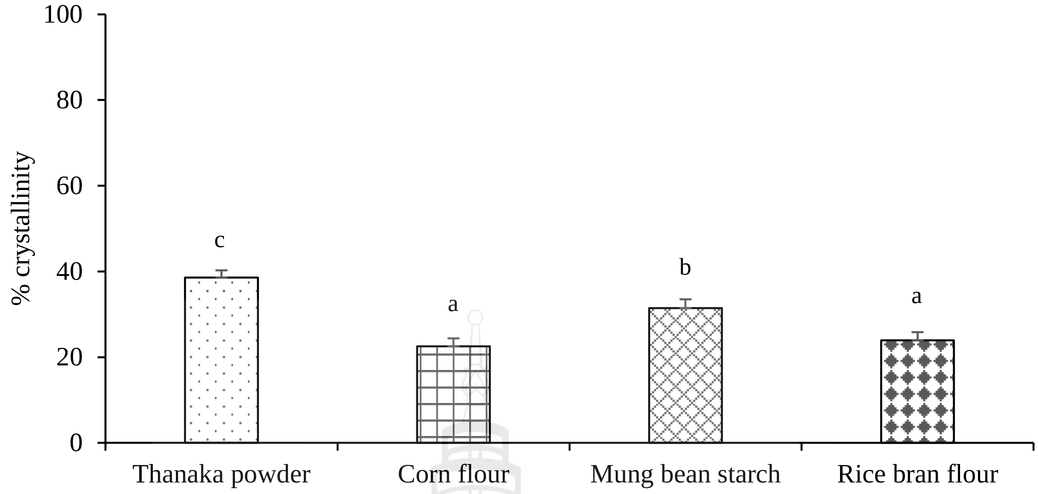
Rice bran flour's $T_o$ was at 68.45 °C, $T_m$ was at 71.25 °C and $T_e$ was at 76.21 °C. This result related to the report by Thiranusornkij et al. (2018), which the thermal properties of rice bran flour from different source and different preparation methods were $T_o$ 65.5 °C, $T_m$ 72.0 °C and $T_e$ 78.1 °C.

Since the peaks of melting temperature (slope) of Thanaka powder, corn flour, mung bean starch and rice bran flour were low and all of their melting temperatures were higher than 50 °C, they would cause stiff when applying on the skin, would stay hard on application, would be low in smoothness for topical application and would reduce the spreadability (Perkinelmer, 2011; Hnin & Kanlayavattanakul, 2022). For this reason, certain natural powders should be more applicable for formulations of solid dosage form cosmetic product rather than the semi-sold one (Hnin & Kanlayavattanakul, 2022).

### 4.2.6 X-ray Diffraction (XRD)

% crystallinity of Thanaka powder, corn flour, mung bean starch and rice bran flour were as shown in Figure 4.6 and Table A2.

Natural powders are composed of crystalline region and amorphous region. High % crystallinity would be rigid and thermally stable powder, but impact resistance is low. Low crystallinity would be soft, have lower melting point and impact resistance at high temperature (The American Ceramic Society, 2015). According to Thiranusornkiji et al. (2018) the thermal properties are influenced by milling method, size, crystalline structure, chemical components and starch composition also.



**Note** Values with different letters are significantly different ($p < 0.05$).

**Figure 4.6** % Crystallinity of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour by X-ray Diffraction (XRD)

Thanaka powder % crystallinity was 38.55±1.71%, and it was lower than Thanaka heartwood % crystallinity, which was 68.5% by the report of Cho & Kobayashi (2018).

Corn Flour % crystallinity was 22.52±1.87%, and it was lower than the report of Liu et al. (2019), which was 32.43-27.18% depending on different milling methods.

Mung Bean Starch % crystallinity was 31.4±42.05%, and it was higher than the report of Zou et al. (2019), which was 17.12-23.26% depending on different starch extraction methods.

Rice bran flour % crystallinity was 23.92±1.91%, and it is higher than the report of Qi et al. (2015), which was around 20%, but lower than the report of Sansuwan et al., (2014), which was 33.16-27.84%, since they were obtained from different sources and countries.

Among the natural materials, Thanaka powder had the highest crystalline region, and so the hardness would also high. Consequently, thanaka powder can be better stable to thermal properties than corn flour, mung bean starch and rice bran flour.

### 4.2.7 Bulk Density

The bulk densities of Thanaka powder, corn flour, mung bean starch and rice bran flour were shown in Figure 4.7 and Table A3. These values would be used to examine the flowability of powder by the calculation of compressibility index and Hausner ratio, which are important for analyzing the solid dosage form development.

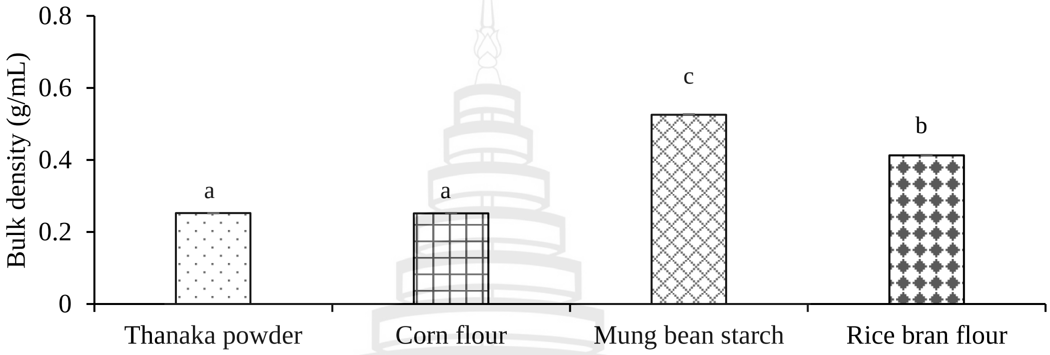


**Figure 4.7** Bulk Densities of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour

### 4.2.8 Tapped Density

The tapped densities of Thanaka powder, corn flour, mung bean starch and rice bran flour were shown in Figure 4.8 and Table A4. These values would be used to examine the flowability of powder by the calculation of compressibility index and Hausner ratio, which are important for analyzing the solid dosage form development.

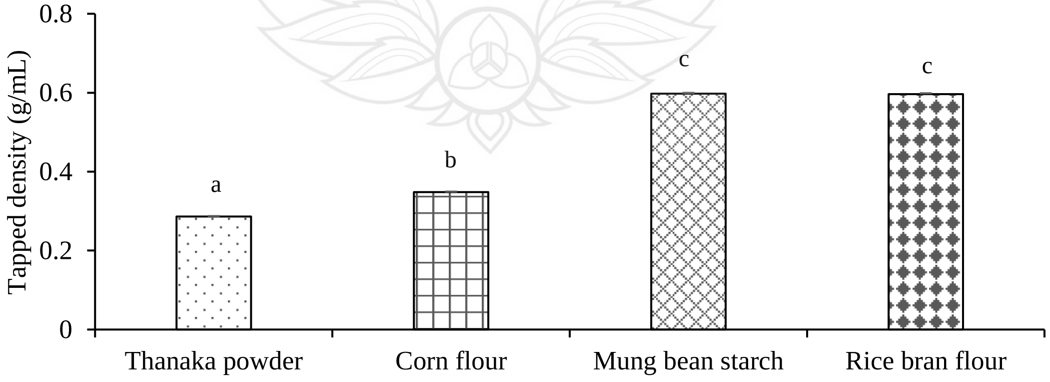


**Figure 4.8** Tapped Densities of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour

### 4.2.9 Flowability

From bulk and tapped densities, compressibility index and Hausner ratio were calculated. According to European Pharmacopoeia 6.0 (2008) and European Pharmacopoeia 7.0 (2010), compressibility index and Hausner ratio could measure the propensity of a powder to be compressed and could measure the interparticle interactions in a free-flowing powder. Poor-flowing powder has greater interparticle interactions and has higher compressibility index, whereas, free-flowing powder has small compressibility index. Compressibility is a measure of the volume change in the sample powder as a result of an applied consolidating stress (Freeman, 2014). According to European Pharmacopoeia 6.0 (2008) and European Pharmacopoeia 7.0 (2010), flowability of Thanaka powder, corn flour, mung bean starch and rice bran flour could be obtained from compressibility index and Hausner ratio calculations which was compared with table 2.2. The flowabilities of natural powders were shown in Table 4.3.

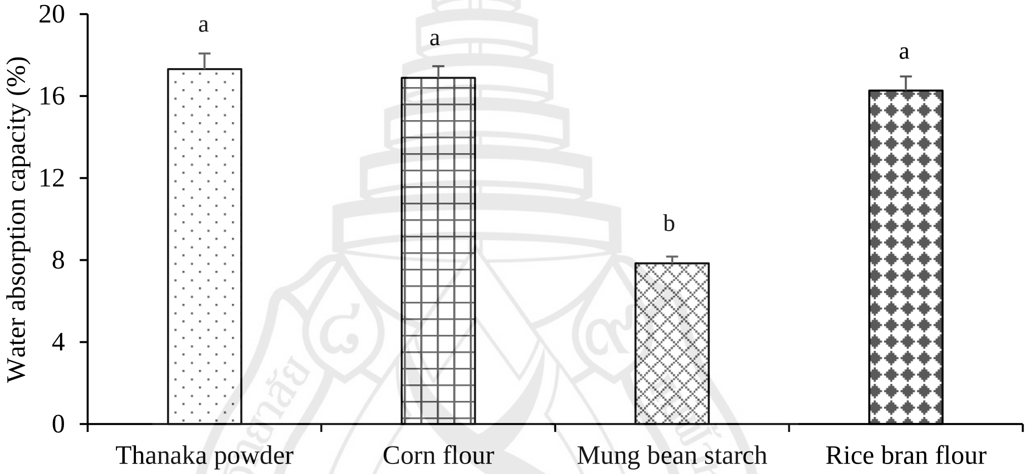
Compressibility index and Hausner ratio were inversely related to flowability and cohesiveness. Cohesiveness is a mechanism between particles which has the tendency to bond one particle to its neighbor (Freeman, 2014). According to the result, Thanaka powder and mung bean starch were not as good in cohesiveness as corn flour and rice bran flour (European Pharmacopoeia 6.0, 2008; European Pharmacopoeia 7.0, 2010). This result was also conformed with the characteristic of Thanaka powder, corn flour, mung bean starch and rice bran flour.

**Table 4.3** Flowabilities of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour

| **Natural Materials** | **Compressibility Index** | **Hausner Ratio** | **Flowability** |
|---|---|---|---|
| Thanaka powder | 12.04±0.24 | 1.14±0.00 | Good |
| Corn flour | 27.69±0.07 | 1.38±0.00 | Poor |
| Mung bean starch | 12.08±0.04 | 1.14±0.00 | Good |
| Rice bran flour | 30.81±0.40 | 1.45±0.01 | Poor |

### 4.2.10 Water Absorption Capacity

Water absorption capacities of Thanaka powder, corn flour, mung bean starch and rice bran flour were shown in Figure 4.9 and Table A5. These natural materials absorbed water and this would enhance the consistency by entrapping sweat and moisture from skin and environment, which are applicable for cosmetic solid dosage forms (Twinomuhwezi et al., 2020). Water absorption capacity of Thanaka powder, corn flour and rice bran flour were significantly higher than mung bean starch ($p < 0.001$).



**Note** Values with different letters are significantly different ($p < 0.05$).

**Figure 4.9** Water Absorption Capacities of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour

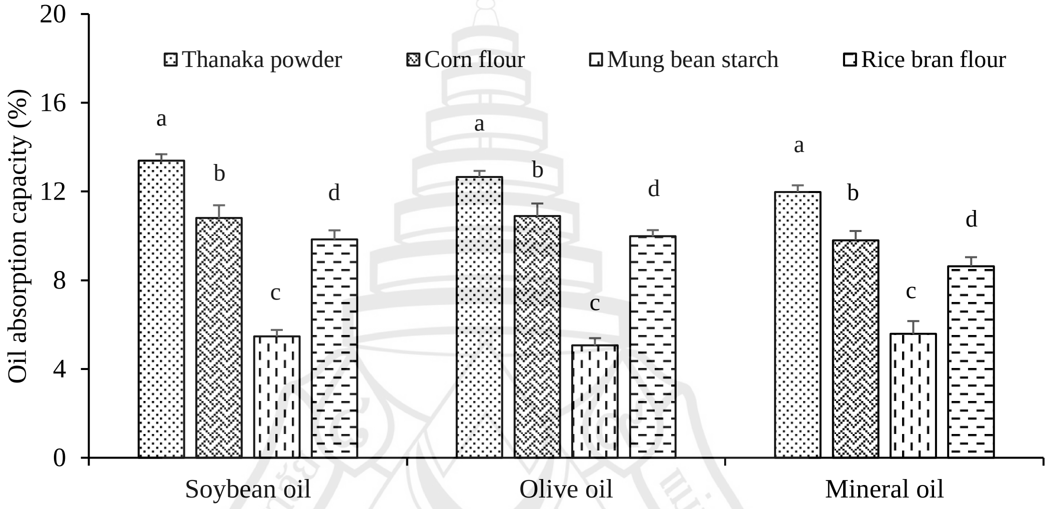
### 4.2.11 Oil Absorption Capacity

Oil absorption capacities of Thanaka powder, corn flour, mung bean starch and rice bran flour were shown in Figure 4.10 and Table A6. These natural materials absorbed oil and this would enhance the consistency by entrapping sebum from the surface of the skin, which are applicable for cosmetic solid dosage form (Twinomuhwezi et al., 2020). Among these natural materials, Thanaka powder could absorb soybean oil, olive oil and mineral oil the most.

Soybean oil absorption capacity of Thanaka powder was significantly higher than corn flour, mung bean starch and rice bran flour (*p*< 0.001).

Olive oil absorption capacity of Thanaka powder was significantly higher than corn flour, mung bean starch and rice bran flour (*p*< 0.001).

Mineral oil absorption capacity of Thanaka powder was significantly higher than corn flour, mung bean starch and rice bran flour (*p*< 0.001).



**Note** Values with different letters within each oil are significantly different ($p < 0.05$).

**Figure 4.10** Oil Absorption Capacities of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour

## 4.3 Stability of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour

Thanaka powder, corn flour, mung bean starch and rice bran flour under long term stability test, for keeping 3 months at ambient temperature (TA) and at 45 °C and compared the results with initial ones as follows.

### 4.3.1 Color

The color of Thanaka powder, corn flour, mung bean starch and rice bran flour were shown in Figure A1-A4 and the color difference values (ΔE) under stability test were mentioned in table A7. *L** of Thanaka powder, corn flour, mung bean starch and rice bran flour had minor shifts only (Figure 4.11). *a** of Thanaka powder, corn flour, mung bean starch and rice bran flour had minor shifts only (Figure 4.12). *b** of Thanaka powder, corn flour, mung bean starch and rice bran flour had minor shifts only (Figure 4.13). According to ΔE result, the color difference value of all natural materials under stability test were less than 1.5, each. For this reason, the difference in colors of natural materials under accelerated stability test could not be detected by human eyes (Lourith & Kanlayavattanakul, 2012).

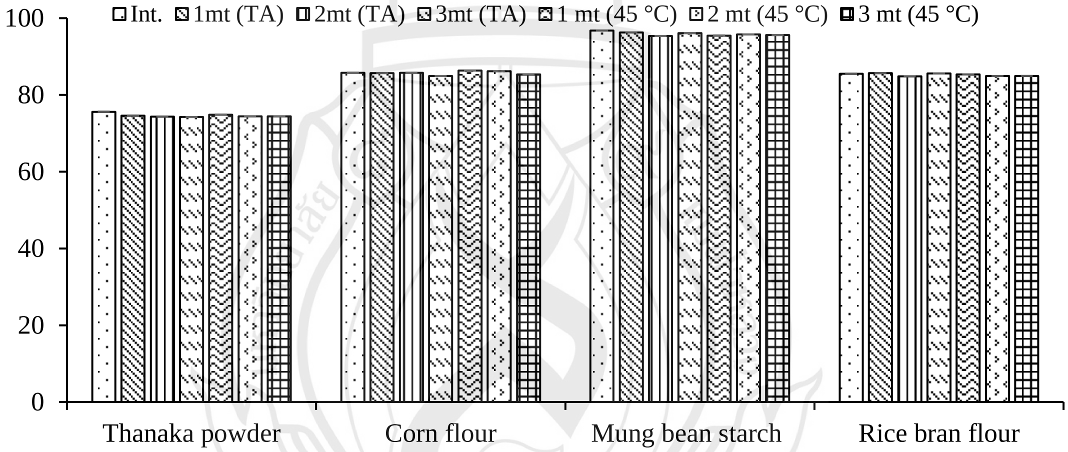


**Figure 4.11** *L** value of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour under Long Term Stability Test

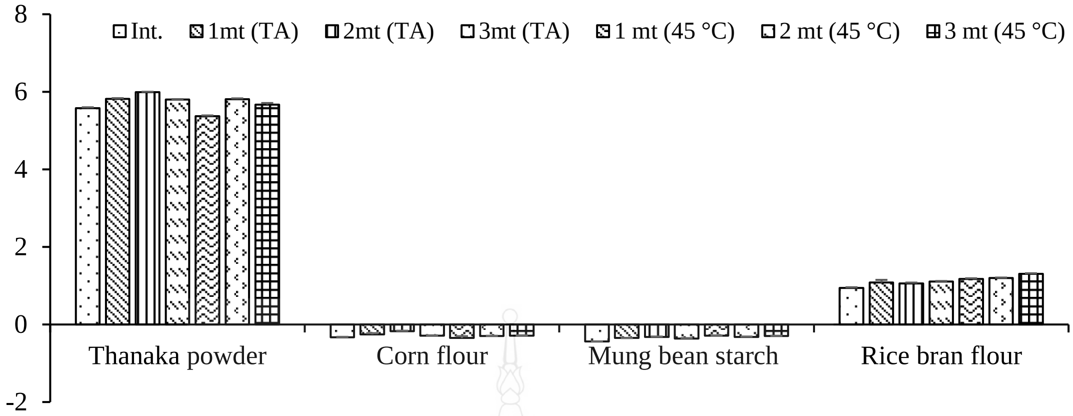


**Figure 4.12** *a** value of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour under Long Term Stability Test

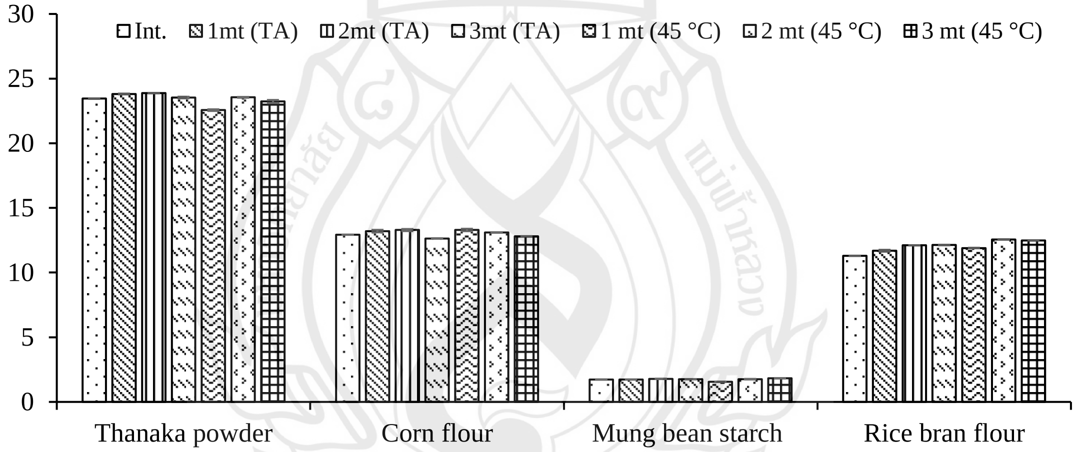


**Figure 4.13** *b** value of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour under Long Term Stability Test

### 4.3.2 Scanning Electron Microscopy (SEM)

The morphology of Thanaka powder, corn flour, mung bean starch and rice bran flour were mentioned in table 4.4 and figure A5-A8. The morphologies of natural materials had only some minor shifts under stability test and the morphology of them were as shown in figure A.5-A.8.

**Table 4.4** Morphology of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour under Long Term Stability Test

| **Natural Materials** | **Condition** | **Month** | **Morphology** |
|---|---|---|---|
| Thanaka powder | TA | 1 | plate-like-crystals with fractures on the surface |
| | | 2 | plate-like-crystals with fractures on the surface |
| | | 3 | plate-like-crystals with fractures on the surface |
| | 45°C | 1 | plate-like-crystals with fractures on the surface |
| | | 2 | plate-like-crystals with fractures on the surface |
| | | 3 | plate-like-crystals with fractures on the surface |
| Corn flour | TA | 1 | non-homogeneous clusters with bubble surface and soft edges |
| | | 2 | non-homogeneous clusters with bubble surface and soft edges |
| | | 3 | non-homogeneous clusters with bubble surface and soft edges |
| | 45°C | 1 | non-homogeneous clusters with bubble surface and soft edges |
| | | 2 | non-homogeneous clusters with bubble surface and soft edges |
| | | 3 | non-homogeneous clusters with bubble surface and soft edges |
| Mung bean starch | TA | 1 | pea-shaped non-cluster powder, even and non-drappled edges |
| | | 2 | pea-shaped non-cluster powder, even and non-drappled edges |
| | | 3 | pea-shaped non-cluster powder, even and non-drappled edges |
| | 45°C | 1 | pea-shaped non-cluster powder, even and non-drappled edges |
| | | 2 | pea-shaped non-cluster powder, even and non-drappled edges |
| | | 3 | pea-shaped non-cluster powder, even and non-drappled edges |
| Rice bran flour | TA | 1 | non-homogeneous clusters with bubble surface and have some edges |
| | | 2 | non-homogeneous clusters with bubble surface and have some edges |
| | | 3 | non-homogeneous clusters with bubble surface and have some edges |
| | 45°C | 1 | non-homogeneous clusters with bubble surface and have some edges |
| | | 2 | non-homogeneous clusters with bubble surface and have some edges |
| | | 3 | non-homogeneous clusters with bubble surface and have some edges |

### 4.3.3 Differential Scanning Calorimetry (DSC)

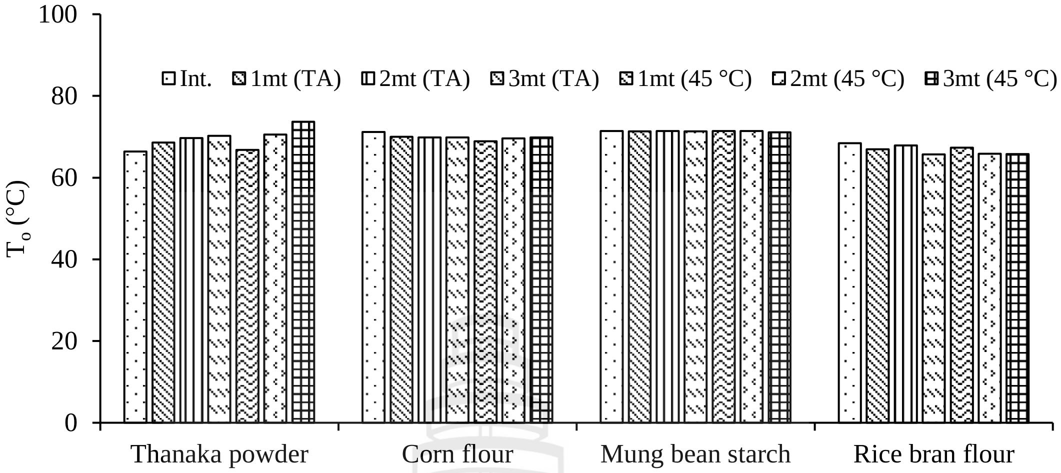


**Figure 4.14** Onset Temperature ($T_o$) of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour under Long Term Stability Test

Onset temperature ($T_o$) of Thanaka powder under stability test became gradually higher than the initial one. This can be predicted as Thanaka powder will be resistant for higher temperature after the stability test. Corn flour, mung bean starch and rice bran flour had some minor shifts only. Onset temperature of Thanaka powder, corn flour, mung bean starch and rice bran flour under stability test are mentioned in figure 4.14 and table A8.

Melting temperature ($T_m$) of Thanaka powder under stability test became gradually higher. This can be predicted as Thanaka powder will be resistant for higher temperature after the stability test. Corn flour, mung bean starch and rice bran flour had some minor shifts. Melting temperature of Thanaka powder, corn flour, mung bean starch and rice bran flour under stability test are mentioned in figure 4.15 and table A8.

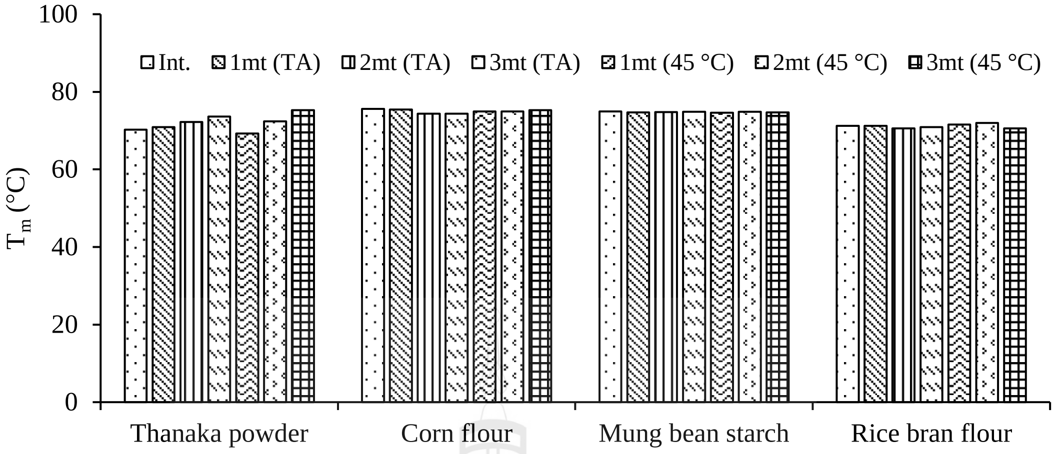


**Figure 4.15** Melting temperature ($T_m$) of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour under Long Term Stability Test

End temperature ($T_e$) of Thanaka powder under stability test became gradually higher than the initial one. This can be predicted as Thanaka powder will be resistant for higher temperature after the stability test. Thermal properties of corn flour, mung bean starch and rice bran flour had some minor shifts. End temperature of Thanaka powder, corn flour, mung bean starch and rice bran flour are mentioned in figure 4.16 and table A8.

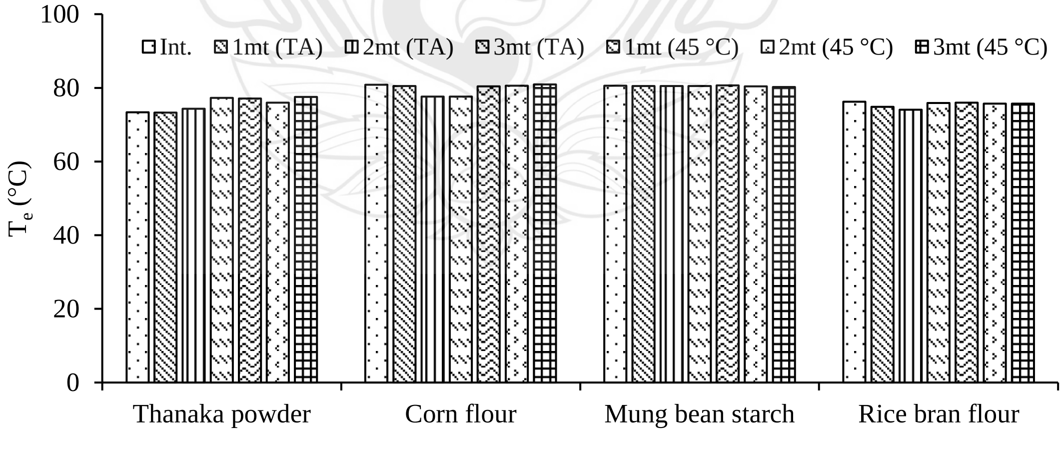


**Figure 4.16** End temperature ($T_e$) of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour under Long Term Stability Test

### 4.3.4 X-ray Diffraction (XRD)

Crystalline structure of Thanaka powder, corn flour, mung bean starch and rice bran flour under stability test were very similar, and this can be seen in Figure 4.17 and Table A9. Since % crystallinity did not significantly change (*p*≥0.459), the texture of each natural materials can be considered as stable under stability test.

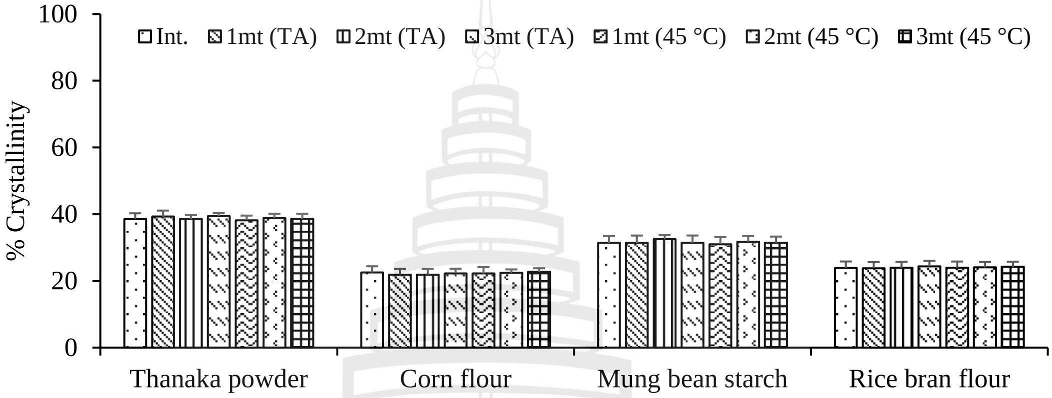


**Figure 4.17** % Crystallinity of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour under Long Term Stability Test

### 4.3.5 Water Absorption Capacity

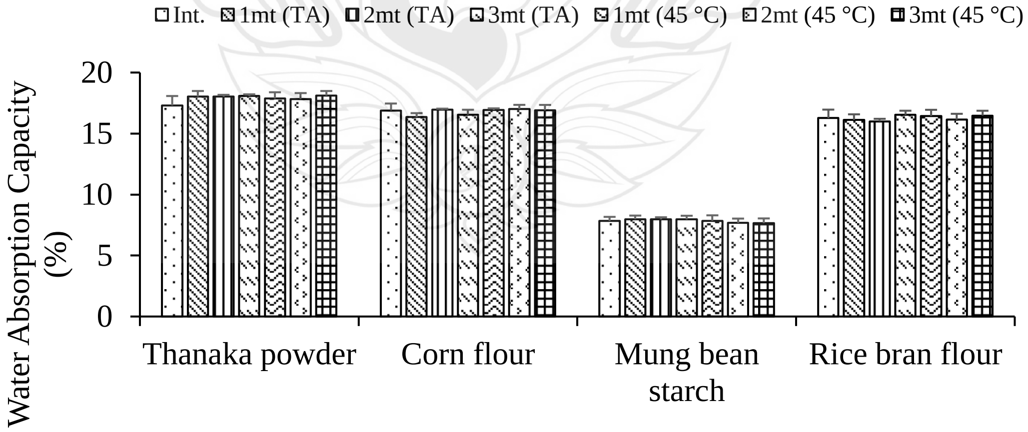


**Figure 4.18** Water Absorption Capacities of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour under Long Term Stability Test

Water absorption capacity of Thanaka powder, corn flour, mung bean starch and rice bran flour did not significantly change ($p \geq 0.061$) as *p*-values were greater than 0.05, respectively, under accelerated stability test respectively (Figure 4.19 and Table A10).

### 4.3.6 Oil Absorption Capacity

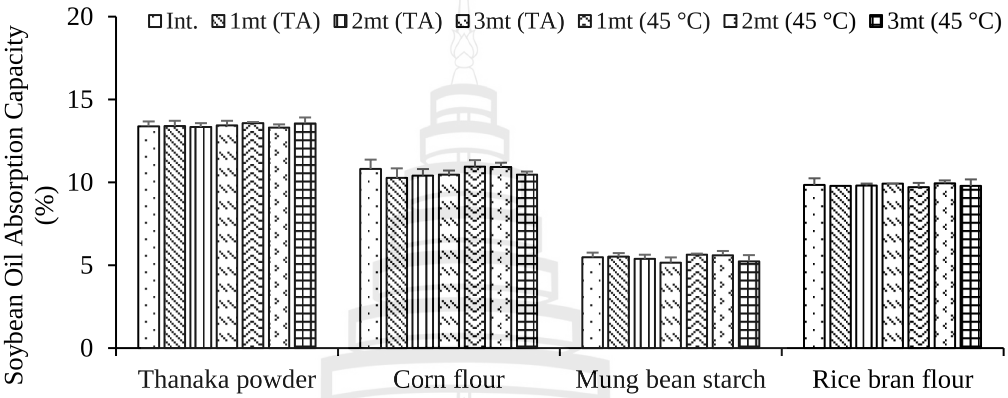


**Figure 4.19** Soybean Oil Absorption Capacities Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour under Long Term Stability Test

Soybean oil absorption capacity of Thanaka powder, corn flour, mung bean starch and rice bran flour were not significantly different ($p \geq 0.124$) as *p*-values were greater than 0.05, respectively, under accelerated stability test (Figure 4.19 and Table A11).

Olive oil absorption capacity of Thanaka powder, corn flour, mung bean starch and rice bran flour were not significantly different ($p \geq 0.083$) as *p*-values were greater than 0.05, respectively, under accelerated stability test (Figure 4.20 and Table A12).

Mineral oil absorption capacity of Thanaka powder, corn flour, mung bean starch and rice bran flour were not significantly different ($p \geq 0.189$) as *p*-values were greater than 0.05, respectively, under accelerated stability test (Figure 4.21 and Table A13).

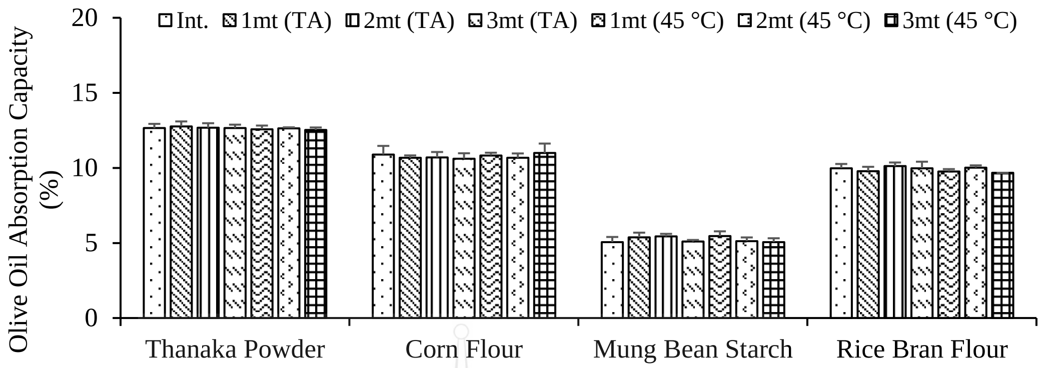


**Figure 4.20** Olive Oil Absorption Capacities of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour under Long Term Stability Test

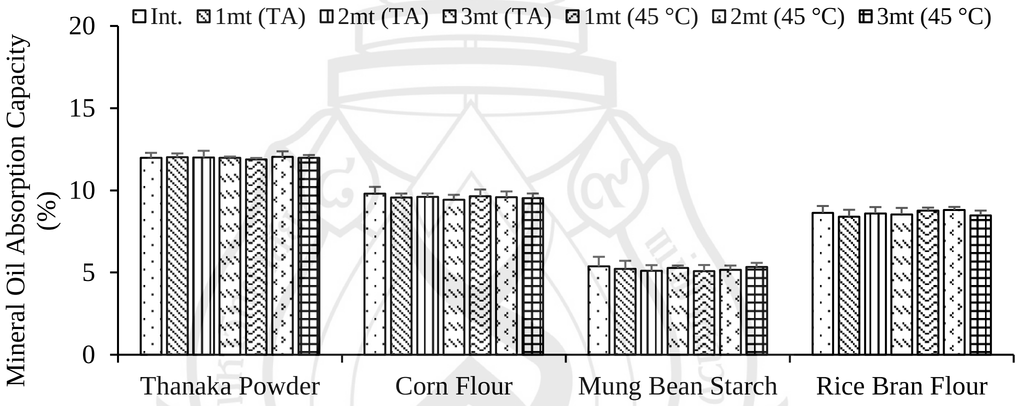


**Figure 4.21** Mineral Oil Absorption Capacities of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour Natural Materials under Long Term Stability Test

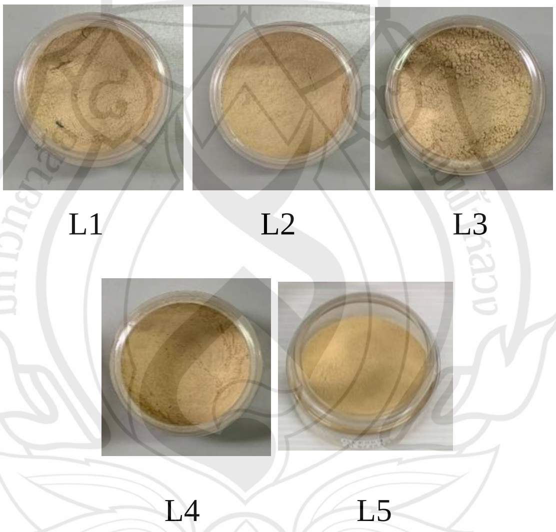
## 4.4 Formulation of Natural Face Powder

### 4.4.1 Formulation of Natural Loose Powder

Natural loose powder formulations were prepared and developed by considering the sensory evaluations, dispersion test and payoff test. The approaching method and the final selection of natural loose powder are as table 3.1. The appearance of natural loose powder formulations is followed in Table 4.5 and Figure 4.22.

**Table 4.5** Appearance of Natural Loose Powder Formulations

| **Natural loose powder** | **Appearance** |
|---|---|
| L1 | Yellowish-white powder |
| L2 | Yellowish-white powder |
| L3 | Yellowish-white powder, slightly more yellowish in color |
| L4 | Yellowish-white powder, slightly more yellowish in color |
| L5 | Yellowish-white powder, slightly more yellowish in color |



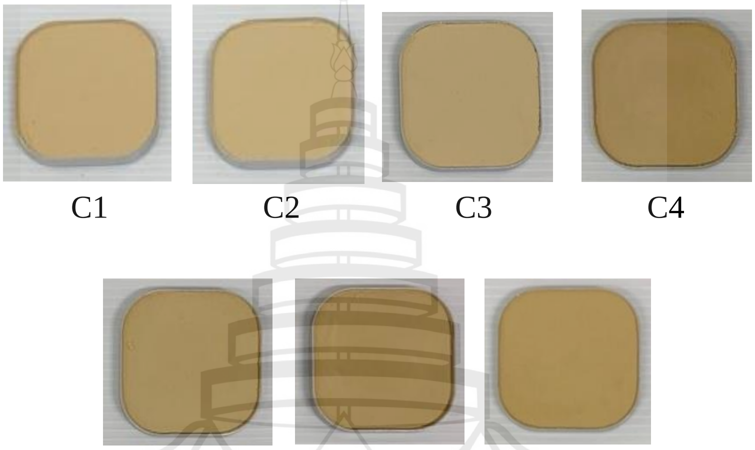
L1 L2 L3

L4 L5

**Figure 4.22** Natural Loose Powder Formulations

### 4.4.2 Formulation of Natural Compact Powder

Natural compact powder preparations were also considered by sensory evaluation, dispersion test, payoff test and breakage test. The approaching method and the final selection of natural compact powder are as table 3.2. The appearance of natural compact powder formulations is followed in Table 4.6 and Figure 4.23.


C1 C2 C3 C4

C5 C6 C7

**Figure 4.23** Natural Compact Powder Formulations

**Table 4.6** Appearance of Natural Compact Powder Formulations

| Natural Compact Powder | Appearance |
|---|---|
| C1 | Yellowish-white powder |
| C2 | Yellowish-white powder |
| C3 | Yellowish-white powder, less yellowish in color |
| C4 | Yellowish-white powder, darker in color |
| C5 | Yellowish-white powder, slightly darker in color |
| C6 | Yellowish-white powder, slightly lighter in color |
| C7 | Yellowish-white powder, more yellowish in color |

## 4.5 Evaluation of Physicochemical Property of Natural Face Powder

### 4.5.1 Sensory Evaluation

4.5.1.1 Sensory evaluation of natural loose powder: the sensory evaluation of natural loose powder was shown in Table 4.7. From L1-2, rice bran flour was increased and mung bean starch was decreased. Slip and absorbency increased. L2 was improved by increasing Thanaka powder and decreasing mung bean starch (L3). L3 increased slip, absorbency and bloom. L3 was improved by increasing Thanaka powder and decreasing rice bran flour (L4). L4 increased slip, absorbency and bloom. L4 was improved by increasing Thanaka powder and decreasing corn flour(L5). L5 increased slip, absorbency and bloom, and decreased adhesiveness. Therefore, L5 was selected for further formulation and natural preservative would be added (L5.1-Int), and would continue for stability testing (L5.1-Int).

**Table 4.7** Sensory Evaluation of Natural Loose Powder Formulations

| **Loose powder** | **Coverage** | **Slip** | **Absorbency** | **Adhesiveness** | **Bloom** |
|---|---|---|---|---|---|
| L1 | 2 | 1 | 1 | 5 | 2 |
| L2 | 2 | 2 | 2 | 5 | 2 |
| L3 | 2 | 3 | 3 | 5 | 3 |
| L4 | 2 | 4 | 4 | 5 | 4 |
| L5 | 2 | 5 | 5 | 4 | 5 |
| Commercial translucent powder | 2 | 4 | 4 | 5 | 5 |
| Commercial oil control powder | 2 | 5 | 4 | 4 | 5 |

4.5.1.2 Sensory evaluation of natural compact powder is shown in Table 4.8. From C1-2, rice bran flour was increased and mung bean starch was decreased. Slip and absorbency increased. C2, however, did not pass breakage test. C2 was improved by increasing corn flour and decreasing Thanaka powder because C2 absorbency was too good and adhesion is not good for compact powder. It increased adhesiveness, and decreased slip, absorbency and bloom, however, C3 was still not passed breakage test. C3 was improved by increasing olive oil and decreasing mung bean starch (C4). It increased adhesiveness and passed the breakage test. C4 was improved by increasing corn flour and decreasing mung bean starch (C5). C5 increased adhesiveness, slip and absorbency. C5 was improved more by reducing rice bran flour and increasing mung bean starch (C6). C6 increased smoothness and adhesiveness and decreased slip and. C6 was improved more by increasing Thanaka powder and reducing corn flour (C7). C7 increased slip, absorbency and bloom and decreased adhesiveness. Therefore, C7 was selected for further formulation and natural preservative would be added (C7.1-Int), and would continue for stability testing (C7.1-Int).

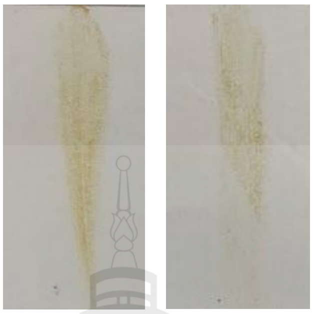
**Table 4.8** Sensory Evaluation of Natural Compact Powder Formulations

| **Compact powder** | **Coverage** | **Slip** | **Absorbency** | **Adhesiveness** | **Bloom** |
|---|---|---|---|---|---|
| C1 | 2 | 4 | 4 | 1 | 5 |
| C2 | 2 | 5 | 5 | 1 | 5 |
| C3 | 2 | 4 | 4 | 2 | 4 |
| C4 | 2 | 4 | 4 | 3 | 4 |
| C5 | 2 | 5 | 5 | 4 | 4 |
| C6 | 2 | 4 | 4 | 5 | 4 |
| C7 | 2 | 5 | 5 | 4 | 5 |
| Commercial translucent compact powder | 2 | 4 | 4 | 5 | 5 |

### 4.5.2 Dispersion Test

The dispersion of all the natural loose powders and natural compact powders were tested and examined. The dispersion of all of the natural face powders were uniform when spreading on a white paper by using a latex glove. The following Figure

4.24 was the dispersion test of selected natural loose powder (L5) and natural compact powder (C7), which dispersion were already approved (Teeratanan, 2018; Faber, 2012).

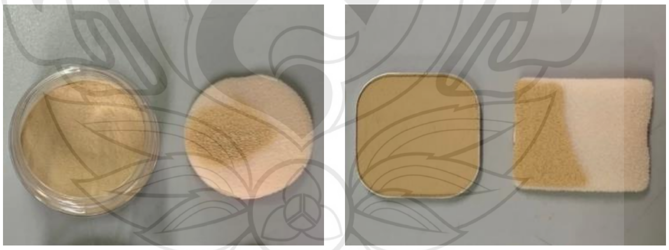


L5 C7

**Figure 4.24** Dispersion Test of L5 and C7

### 4.5.3 Payoff Test

The payoff property of all the natural loose powders and natural compact powders were tested and examined. All of the natural face powders were well adhere on the puff. The following Figure 4.25 was the payoff test of selected natural loose powder (L5) and natural compact powder (C7), which payoff were already approved (Jarupinthusophon & Anurukvorakun, 2021; Shinde et al., 2021, Teeratanan, 2018).



L5 C7

**Figure 4.25** Payoff Test of L5 and C7

### 4.5.4 Color

The appearance of developed natural loose powder and natural compact powder were as in Figure 4.26. Their *L**, *a** and *b** values were 76.39±0.01, 5.48±9.01 and 22.74±0.01 for L5.1-Int and 64.95±0.03, 7.99±±0.01 and 31.56±0.04 for C7.1-Int (Figure 4.26 and Table B1). Both of them were yellowish white color. Natural loose powder had slightly lighter color than natural compact powder because of liquid binder in compact powder, which was absorbed by natural powders and became darker.

(L5.1-Int) (C7.1-Int)

**Figure 4.26** Appearance of L5.1-Int and C7.1-Int

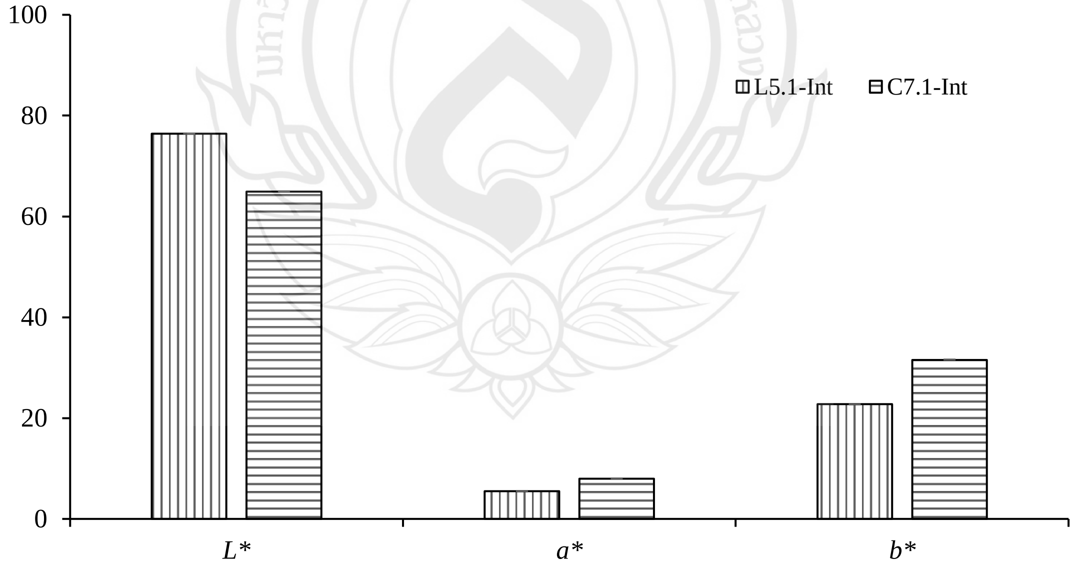


**Figure 4.27** *L**, *a** and *b** values of L5.1-Int and C7.1-Int

### 4.5.5 Sun Protection Factor Analysis

Sun protection factor analysis of both developed natural powders were shown in Table 4.9. SPF values (UVB protection) of both natural face powders were less than 15 so both of them were low protection (MacGill, 2018). Both developed natural face powder had UVA/UVB ratio between 0.60-0.79 so both of them had 3 stars for Boots star rating (UVA protection) (Tajka, 2020). Both developed natural face powders had critical wavelength of higher than 370 nm. However, to be able to claim “broad spectrum”, they must pass not only critical wavelength be higher than 370 nm but also SPF be higher than 15. Therefore, they could not be able to claim as “broad spectrum” (MacGill, 2018)

**Table 4.9** SPF values of L5.1-Int and C7.1-Int

| Natural face powder | SPF | UVA/UVB ratio | Critical Wavelength (nm) |
|---|---|---|---|
| L5.1-Int | 1.41 ± 0.00 | 0.751±0.001 | 382.67±0.58 |
| C7.1-Int | 1.54 ± 0.01 | 0.731±0.001 | 382.33±0.58 |

### 4.5.6 Fourier Transform Infrared Spectroscopy (FTIR)

The frequency of absorption bands and the relative band intensities in natural loose powder and natural compact powder (Figure 4.28) were coincident with the spectrum of its own natural ingredients. Natural loose powder and natural compact powder showed similar peaks, except some different intensities, which is the minor shifts in the amount of the functional group associated with the molecular bond (Anderson & Voskerician, 2010).

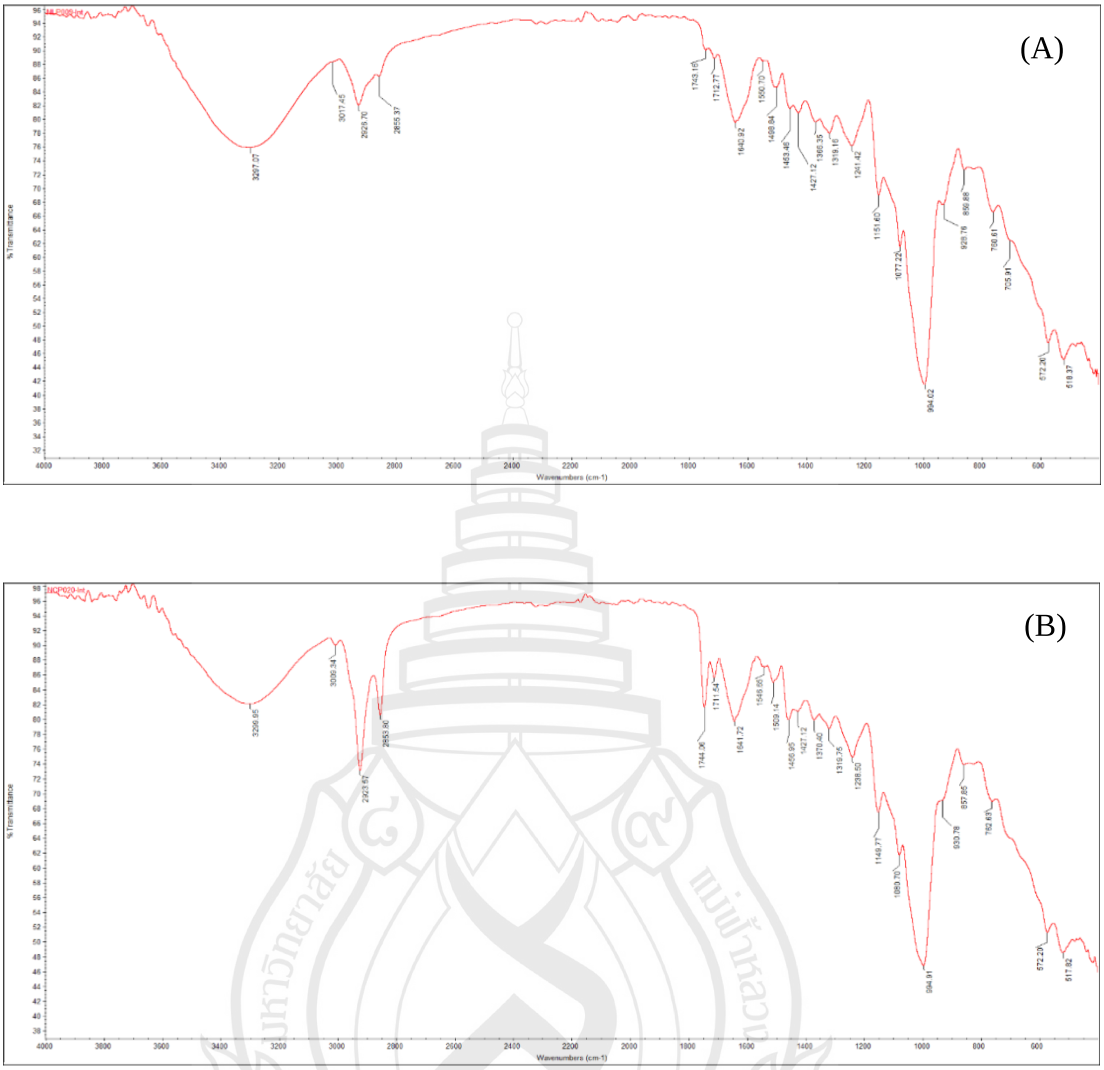


**Figure 4.28** Fourier Transform Infrared Spectroscopy (FTIR) Spectrums of L5.1-Int (A) and C7.1-Int (B)

### 4.5.7 Breakage Test

The durability of compact powder could be predicted by breakage test and the following Table 4.10 was the breakage test performed on all natural compact powders, and recorded at which time it began to break from the test. The developed natural compact powder (C7.1-Int) was broken until 6 times dropping for breakage test. Since it remained unbroken for 2-3 times, C7.1-Int could resist for normal handling by user (Jarupinthusophon & Anurukvorakun, 2020; Teeratanan, 2018).

**Table 4.10** Breakage Test of Natural Compact Powder Formulations

| **Natural compact powder** | **Broken time** |
|---|---|
| C1 | 2 |
| C2 | 2 |
| C3 | 3 |
| C4 | 4 |
| C5 | 5 |
| C6 | 6 |
| C7 | 5 |
| C7.1-Int | 6 |
| Commercial translucent compact powder | 10 |

## 4.6 Stability of Developed Natural Face Powders

### 4.6.1 Sensory Evaluation

**Table 4.11** Sensory Evaluation of Natural Face Powders under Heating-Cooling Cycling Stability Test

| **Natural face powders** | **Coverage** | **Slip** | **Absorbency** | **Adhesiveness** | **Bloom** |
|---|---|---|---|---|---|
| L5.1-Int | 2 | 5 | 5 | 4 | 5 |
| L5.1-HC | 2 | 5 | 5 | 4 | 5 |
| C7.1-Int | 2 | 5 | 5 | 4 | 5 |
| C7.1-HC | 2 | 5 | 5 | 4 | 5 |

Natural loose powder before (L5.1-Int) and after (L5.1-HC) stability test gave the similar sensory. Natural compact powder before (C7.1-Int) and after (C7.1-HC) stability test gave the same sensory. The sensory evaluation of natural face powders under heating-cooling cycling stability test is in Table 4.11.

### 4.6.2 Dispersion Test

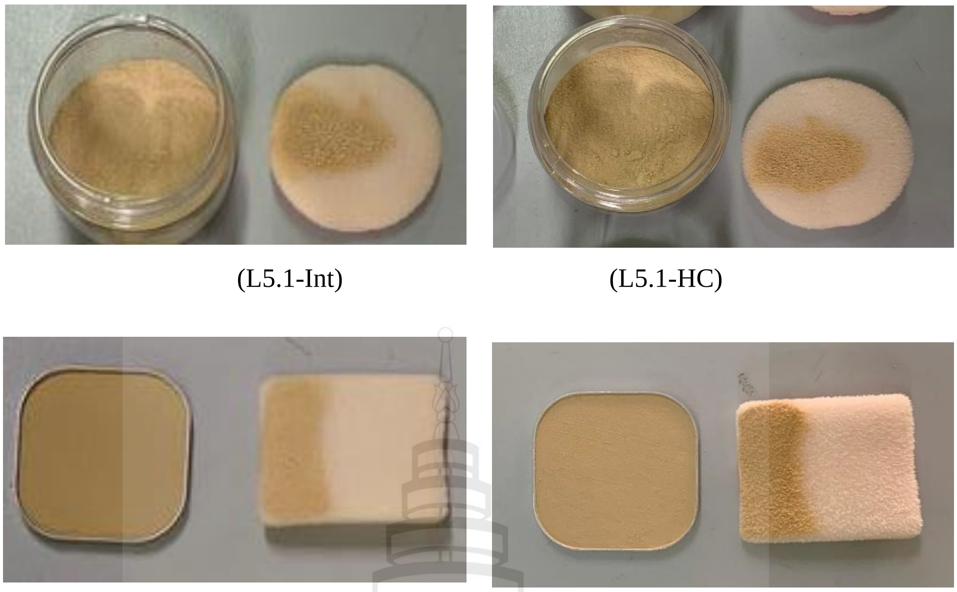
(L5.1-Int) (L5.1-HC) (C7.1-Int) (C7.1-HC)

**Figure 4.29** Dispersion Test of Natural Face Powders under Heating-Cooling Cycling Stability Test

Natural loose powder before (L5.1-Int) and after (L5.1-HC) stability test and natural compact powder before (C7.1-Int) and after (C7.1-HC) stability test were uniform when spreading on a white paper by using a latex glove. Therefore, the dispersion test of both natural face powders under stability test were approved (Teeratanan, 2018; Faber 2012). The dispersion test of natural face powders under heating-cooling cycling stability test were shown in Figure 4.29.

### 4.6.3 Payoff Test

Natural loose powders before (L5.1-Int) and after (L5.1-HC) stability test and natural compact powders before (C7.1-Int) and after (C7.1-HC) stability test were adhere well on the puff. Therefore, the payoff test of both natural face powders under stability test were approved (Jarupinthusophon & Anurukvorakun, 2021; Shinde et al., 2021, Teeratanan, 2018). The payoff test of natural face powders under heating-cooling cycling stability test were shown in Figure 4.30.

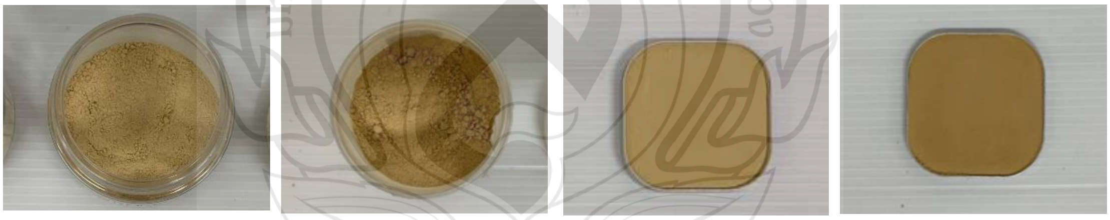


(L5.1-Int) (L5.1-HC)

(C7.1-Int) (C7.1-HC)

**Figure 4.30** Payoff Test of Natural Face Powders under Heating-Cooling Cycling Stability Test

### 4.6.4 Color



(L5.1-Int) (L5.1-HC) (C7.1-Int) (C7.1-HC)

**Figure 4.31** Appearance of Natural Face Powders under Heating-Cooling Cycling Stability Test

The appearance of natural loose powder and natural compact powder after treatment with 6-cycles of Heating-Cooling were shown in Figure 4.31. After stability test, the *L**, *a** and *b** values were 76.31±0.01, 5.09±0.01 and 22.10±0.01 for natural loose powder and 63.95±0.01, 8.12±0.03 and 31.41±0.03 for natural compact powder

(Figure 4.32 and Table B2). ΔE values for both of them were less than 1.5 (0.75±0.00 for natural loose powder and 1.02±0.03 for natural compact powder), thus the color changes could not be detected by human eyes (Lourith & Kanlayavattanakul, 2012).

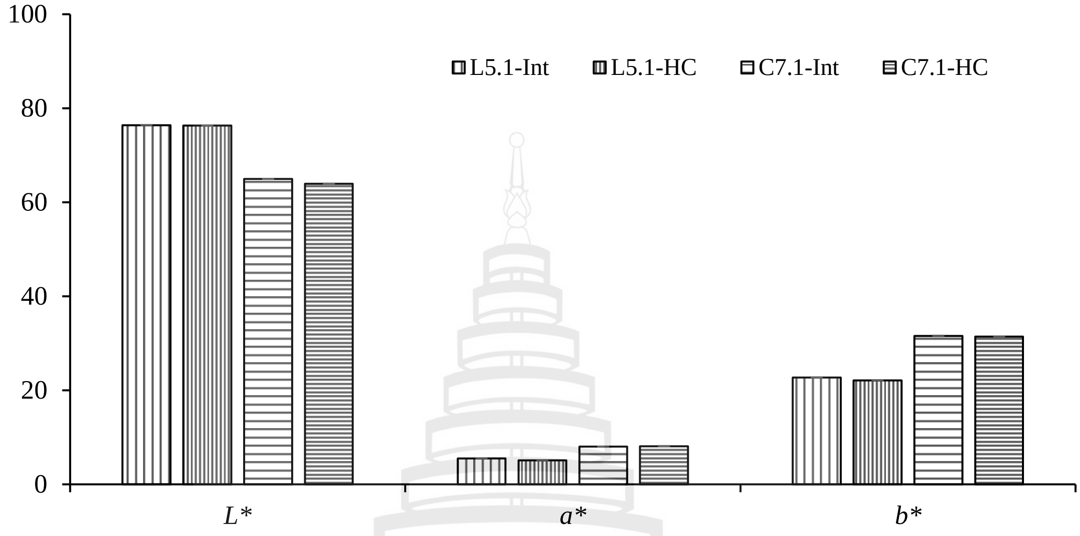


**Figure 4.32** *L**, *a** and *b** values of Natural Face Powders under Heating-Cooling Cycling Stability Test

### 4.6.5 Sun Protection Factor Analysis

For developed natural loose powder, SPF reduced 4.02±0.82%, UVA/UVB ratio and critical wavelength increased 3.24±0.28% and 0.35±0.15%, respectively, after 6 cycles of heating-cooling stability test. For developed natural compact powder, SPF, UVA/UVB ratio and critical wavelength increased 1.29±0.37%, 1.55±0.08% and 0.17±0.15%, respectively, after 6 cycles of heating-cooling stability test. However, under heating-cooling cycling stability test, their SPF values were still less than 15, UVA/UVB ratios were still between 0.60-0.79 and critical wavelengths were still higher than 370 nm. Therefore, developed natural loose powder and developed natural compact powder had minor shifts in SPF, UVA/UVB ratio and critical wavelength under heating-cooling cycling stability test (Table 4.12).

**Table 4.12** SPF values of Natural Face Powders under Heating-Cooling Cycling Stability Test

| Natural face powder | SPF | UVA/UVB ratio | Critical wavelength (nm) |
|---|---|---|---|
| L5.1-Int | 1.41 ± 0.00 | 0.751±0.001 | 382.67±0.58 |
| L5.1-HC | 1.35± 0.01 | 0.776±0.003 | 384.00±0.00 |
| C7.1-Int | 1.54 ± 0.01 | 0.731±0.001 | 382.33±0.58 |
| C7.1-HC | 1.55 ± 0.02 | 0.743±0.001 | 383.00±0.00 |

### 4.6.6 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier transform infrared spectroscopy-attenuated total reflectance gave the information of the presence of absence of specific functional groups and the chemical structure. No shifts in the frequency of absorption bands and in the relative band intensities in developed natural loose powder and natural compact powder after treatment with 6-cycles of heating-cooling (Figure 4.33) when compared, only minor shifts in intensity. This could be considered as there was no change in the chemical structure or no changes in the environment around the sample products (Anderson & Voskerician, 2010)

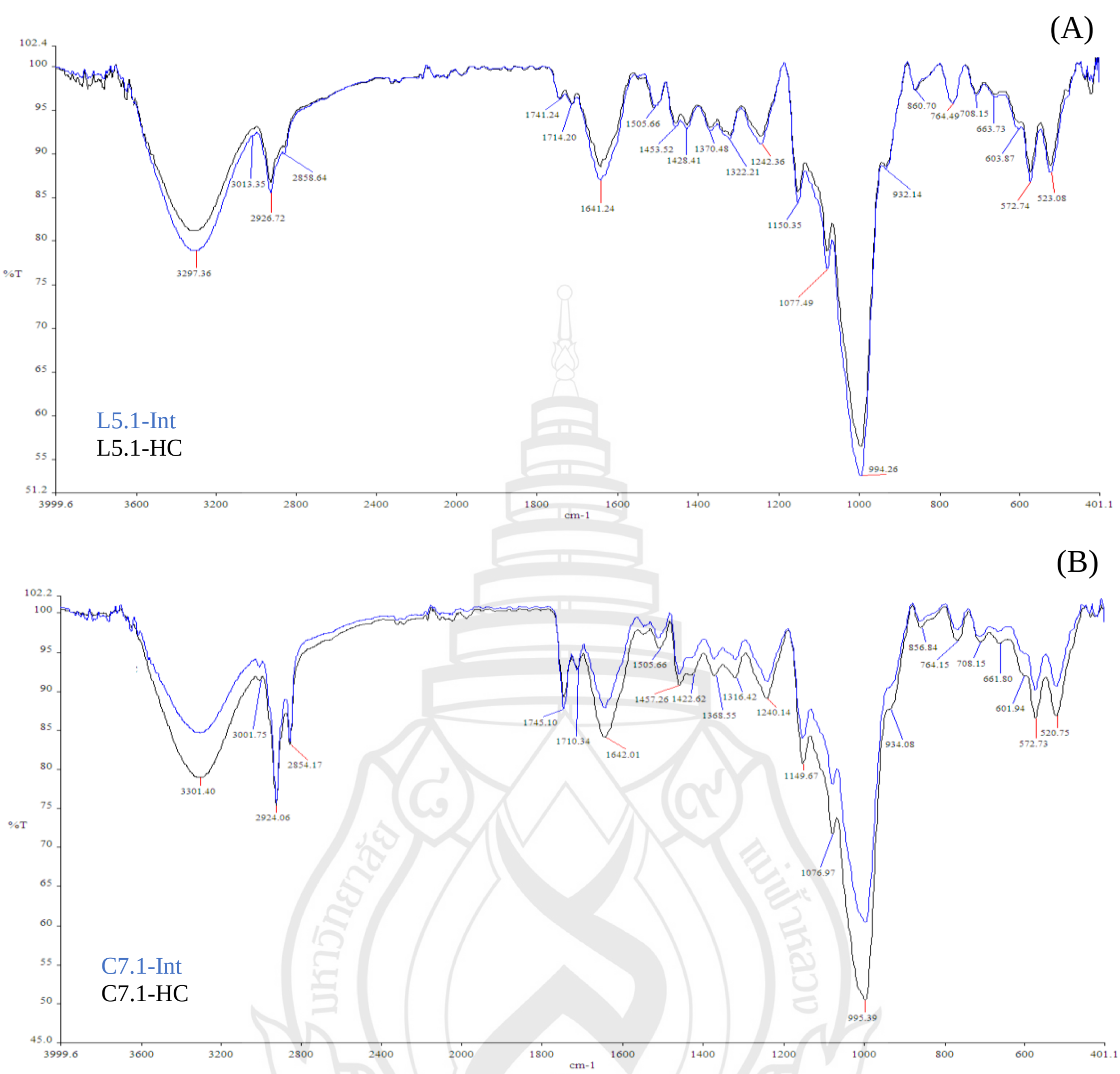


**Figure 4.33** Fourier Transform Infrared Spectroscopy (FTIR) Spectrums of Natural Loose Powder (A) and Natural Compact Powder (B) under Heating-Cooling Cycling Stability Test

### 4.6.7 Breakage Test

The durability test of compact powder after treatment with 6-cycles of heating-cooling was performed. Natural compact powder after stability test did not break until 4th time from dropping (Table 4.13), and for this reason, this natural compact powder is indicated as safe and durable after stability test (Jarupinthusophon & Anurukvorakun, 2021).

**Table 4.13** Breakage Test of Natural Face Powders under Heating-Cooling Cycling Stability Test

| **Natural Face Powders** | **Broken Time** |
|---|---|
| C7.1-Int | 6 |
| C7.1-HC | 4 |

According to Making Cosmetics (2012), when the product passes three cycles (-10 ºC and 25 ºC), it could be considered having good degree of confidence in the stability of the product. When the product passes five cycles (-10 ºC and 25 ºC), it could be considered as a really stable product. To be better prediction for considering the shelf life of the product, the product is recommended to be stored at 45 ºC for three months and when the product exhibited acceptable stability, then it should be stable at room temperature for two years (Making Cosmetics, 2012).

# CHAPTER 5

# CONCLUSION

1. Thanaka powder, corn flour, mung bean starch and rice bran flour, were prepared and stored to study the physicochemical evaluations. Prohibited four elements for cosmetic applications, such as Arsenic (As), Cadmium (Cd), Lead (Pb) and Mercury (Hg) in each natural powder did not overstep the limits by ASEAN cosmetic guidelines. The infrared spectrums of natural ingredients were conformed. The appearance and texture matched with *L**, *a** and *b** values and morphology. Low melting peak and high melting temperature of natural materials are applicable for solid dosage form. Thanaka powder had the highest crystalline region among the three others so its hardness is high and would be better stable to thermal properties. Thanaka powder and mung bean starch were good flowability, while, corn flour and rice bran flour were good cohesiveness. Thanaka powder, corn flour, mung bean starch and rice bran flour could entrap sweat, moisture and sebum form the surface of the skin and environment and be applicable for cosmetic powder products.

All natural materials under long term stability test by storing them at ambient temperature (TA) and 45 °C for 3 months were recorded and observed. The color changes couldn’t be detected by human eyes since the color difference (ΔE) did not exceed 1.5. The morphology of natural powders was also similar. Thus, the density and flowability parameters of powders should be stable in accordance with the revealed morphology. Corn flour, mung bean starch and rice bran flour’s melting temperature had some minor shifts, while Thanaka powder became increasing resistant to higher temperature after the stability test as its melting temperature became higher. Crystalline structures of natural powders were not significantly different ($p \geq 0.459$) under stability test and so their textures could be considered as similar after the stability test. Water and oil absorption capacities were also not significantly different ($p \geq 0.083$).

2. Natural loose powder and natural compact powder were formulated by sensory evaluation, and during the preparation, the selected products should pass the

dispersion test, payoff test and breakage test. The chosen products (L5.1 and C7.1) were poor for coverage, however, their slip, absorbency, adhesiveness and bloom were satisfied. Both developed natural face powders (L5.1-Int and C7.1-Int) had SPF<15 (1.41-1.55) which was UVB low protection, UVA/UVB ratios between 0.60-0.79 (0.730-0.752) which were 3 Boots star rating (UVA protection), and critical wavelengths >370 nm (381.75-383.25 nm).

The developed natural face powders under 6 cycles of heating-cooling stability test, their dispersion, payoff and durability were approved and passed, and there were similar peaks of infrared spectrums. The color difference could not be detected by eyes ($\Delta E \leq 1.05$). The SPF value of developed natural loose powder reduced 4.02±0.82%, whereas developed natural compact powder increased 1.29±0.37%. For both developed natural face powders, UVA/UVB ratios increased (1.47-3.52%) and critical wavelengths also increased (0.02-0.35%). However, their UVB protection were still low, UVA protection (UVA/UVB ratio) were still 3 stars for Boots star rating, and critical wavelength were still >370 nm. For these reasons, natural loose powder and natural compact powder were considered to be stable color cosmetic products and these formulations were regarded as successful even after the stability test evaluation.

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# APPENDICES

# APPENDIX A

## PHYSICOCHEMICAL EVALUATION AND STABILITY OF THANAKA POWDER, CORN FLOUR, MUNG BEAN STARCH AND RICE BRAN FLOUR

**Table A1** *L**, *a** and *b** values of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour

| **Natural materials** | ***L**** | ***a**** | ***b**** |
|---|---|---|---|
| Thanaka powder | 75.63±0.01 | 5.58±0.02 | 23.45±0.02 |
| Corn flour | 85.76±0.01 | -0.33±0.02 | 12.93±0.01 |
| Mung bean starch | 96.78±0.02 | -0.04±0.00 | 1.73±0.02 |
| Rice bran flour | 85.47±0.02 | 0.94±0.02 | 11.30±0.01 |

**Table A2** % Crystallinity of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour by X-ray Diffraction (XRD)

| **Natural materials** | **% crystallinity** |
|---|---|
| Thanaka powder | 38.55±1.71 |
| Corn flour | 22.52±1.87 |
| Mung bean starch | 31.44±2.05 |
| Rice bran flour | 23.92±1.91 |

**Table A3** Bulk Densities of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour

| **Natural materials** | **Bulk density** |
|---|---|
| Thanaka powder | 0.2521±0.0000 |
| Corn flour | 0.2517±0.0006 |
| Mung bean starch | 0.5254±0.0016 |
| Rice bran flour | 0.4127±0.0010 |

**Table A4** Tapped Densities of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour

| **Natural materials** | **Tapped density** |
|---|---|
| Thanaka powder | 0.2866±0.0008 |
| Corn flour | 0.3482±0.0012 |
| Mung bean starch | 0.5976±0.0021 |
| Rice bran flour | 0.5964±0.0021 |

**Table A5** Water Absorption Capacities of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour

| Natural materials | Water absorption capacity (%) |
|---|---|
| Thanaka powder | 17.31±0.77$^{a}$ |
| Corn flour | 16.89±0.57$^{a}$ |
| Mung bean starch | 7.84±0.33$^{b}$ |
| Rice bran flour | 16.27±0.69$^{a}$ |

**Table A6** Oil Absorption Capacities of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour

| Natural materials | Oil absorption capacity | | |
|---|---|---|---|
| | Soybean oil | Olive oil | Mineral oil |
| Thanaka powder | 13.39±0.29$^{a}$ | 12.65±0.28$^{a}$ | 11.98±0.30$^{a}$ |
| Corn flour | 10.81±0.57$^{b}$ | 10.89±0.57$^{b}$ | 9.80±0.42$^{b}$ |
| Mung bean starch | 5.47±0.29$^{c}$ | 5.06±0.33$^{c}$ | 5.58±0.58$^{c}$ |
| Rice bran flour | 9.84±0.41$^{d}$ | 9.98±0.28$^{d}$ | 8.63± 0.41$^{d}$ |

**Figure A1** Color of Thanaka Powder under Long Term Stability Test

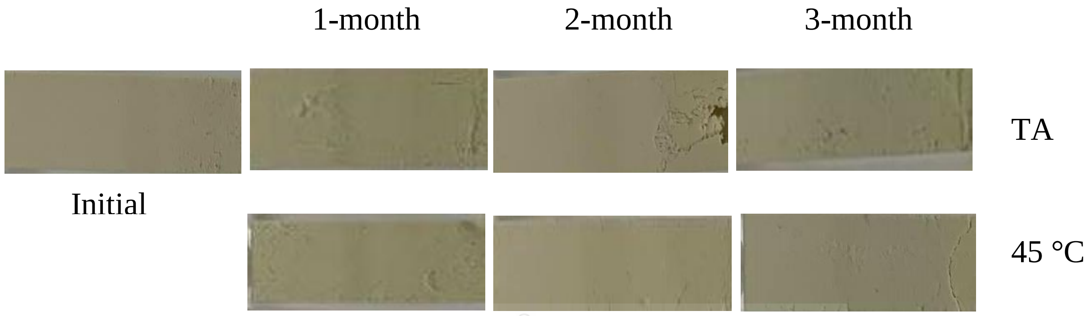


**Figure A2** Color of Corn Flour under Long Term Stability Test

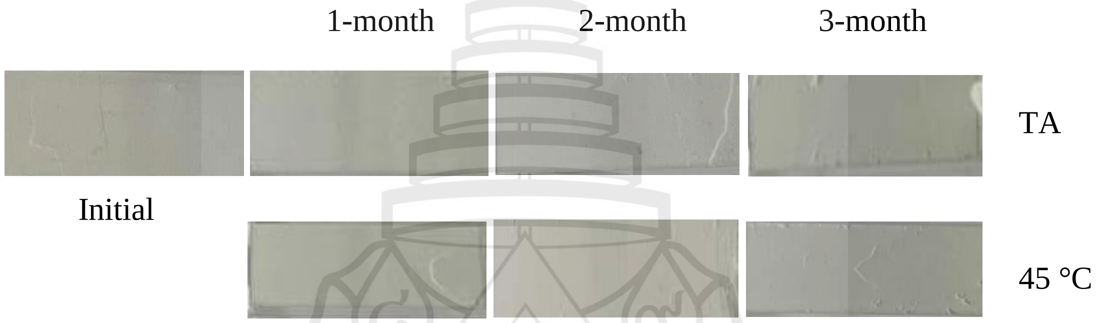
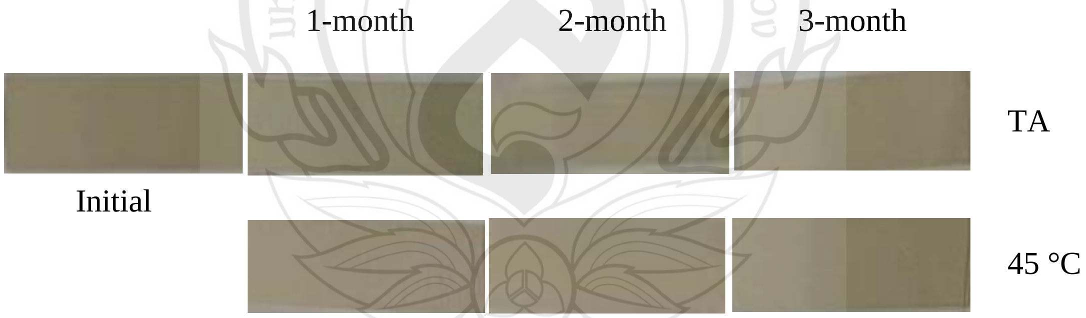


**Figure A3** Color of Mung Bean Starch under Long Term Stability Test



**A4** Color of Rice Bran Flour under Long Term Stability Test

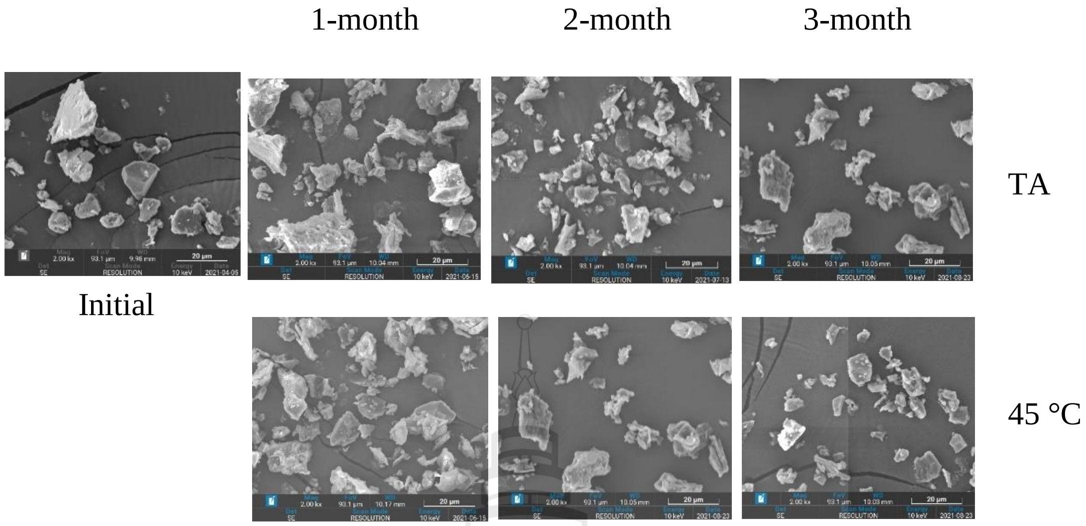


**Figure A5** Morphology of Thanaka Powder under Long Term Stability Test

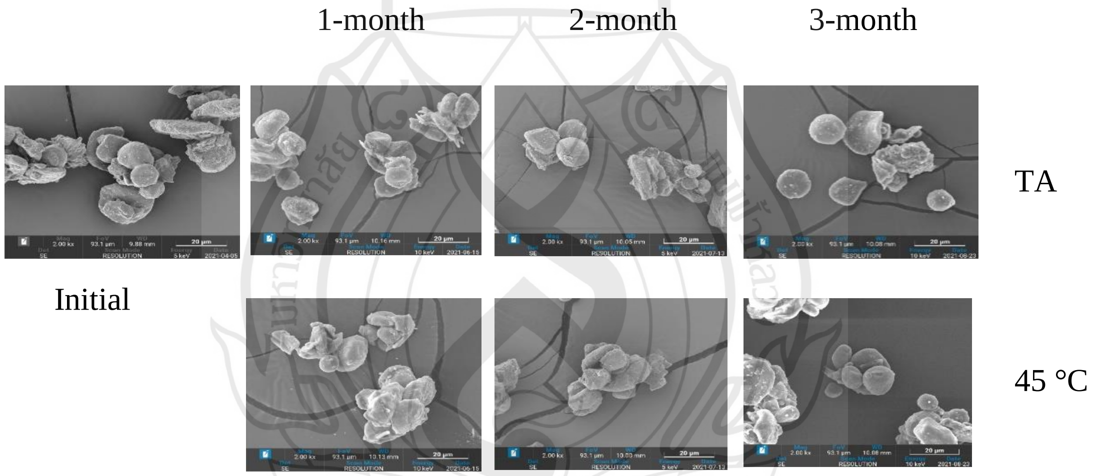


**Figure A6** Morphology of Corn Flour under Long Term Stability Test

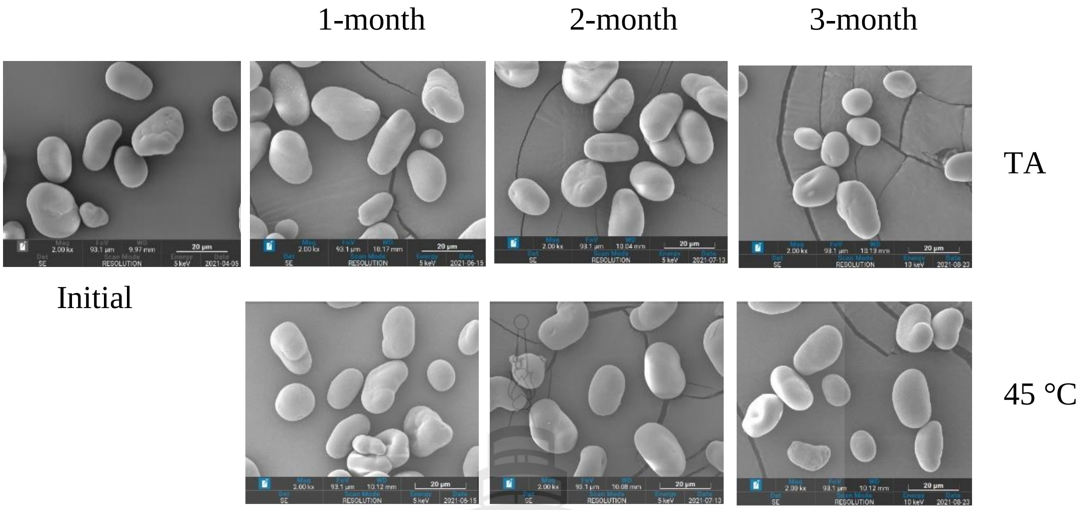


**Figure A7** Morphology of Mung Bean Starch under Long Term Stability Test

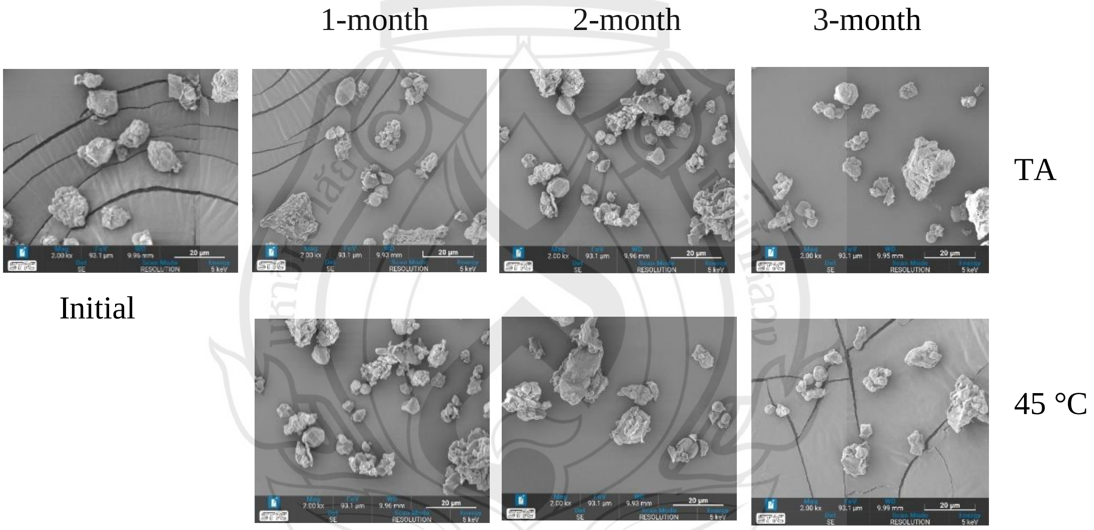


**Figure A8** Morphology of Rice Bran Flour under Long Term Stability Test

**Table A7** Color Difference of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour under Long Term Stability Test

| **Natural materials** | **Condition** | **Month** | **ΔE** |
|---|---|---|---|
| Thanaka powder | TA | 1 | 1.15±0.02 |
| | | 2 | 1.43±0.02 |
| | | 3 | 1.43±0.04 |
| | 45°C | 1 | 1.20±0.05 |
| | | 2 | 1.23±0.01 |
| | | 3 | 1.26±0.11 |
| Corn flour | TA | 1 | 0.29±0.09 |
| | | 2 | 0.40±0.03 |
| | | 3 | 0.87±0.01 |
| | 45°C | 1 | 0.69±0.10 |
| | | 2 | 0.48±0.01 |
| | | 3 | 0.41±0.01 |
| Mung bean starch | TA | 1 | 0.51±0.01 |
| | | 2 | 1.45±0.01 |
| | | 3 | 0.68±0.01 |
| | 45°C | 1 | 1.37±0.07 |
| | | 2 | 1.05±0.01 |
| | | 3 | 1.23±0.01 |
| Rice bran flour | TA | 1 | 0.46±0.08 |
| | | 2 | 1.05±0.02 |
| | | 3 | 0.86±0.02 |
| | 45°C | 1 | 0.66±0.02 |
| | | 2 | 1.40±0.01 |
| | | 3 | 1.35±0.01 |

**Table A8** Thermal Properties of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour under Long Term Stability Test

| **Natural Materials** | **Condition** | **Month** | **$T_o$ (°C)** | **$T_m$ (°C)** | **$T_e$ (°C)** |
|---|---|---|---|---|---|
| Thanaka powder | TA | 1 | 68.61 | 70.92 | 73.28 |
| | | 2 | 69.67 | 72.25 | 74.29 |
| | | 3 | 70.24 | 73.58 | 77.24 |
| | °C | 1 | 66.81 | 69.25 | 77.11 |
| | | 2 | 70.53 | 72.42 | 75.95 |
| | | 3 | 73.71 | 75.25 | 77.52 |
| Corn flour | TA | 1 | 69.99 | 75.42 | 80.53 |
| | | 2 | 69.88 | 74.33 | 77.61 |
| | | 3 | 69.88 | 74.33 | 77.61 |
| | °C | 1 | 68.93 | 74.92 | 80.43 |
| | | 2 | 69.58 | 74.92 | 80.55 |
| | | 3 | 69.82 | 75.25 | 80.89 |
| Mung bean starch | TA | 1 | 71.31 | 74.67 | 80.52 |
| | | 2 | 71.39 | 74.75 | 80.52 |
| | | 3 | 71.33 | 74.83 | 80.51 |
| | °C | 1 | 71.4 | 74.58 | 80.66 |
| | | 2 | 71.4 | 74.83 | 80.41 |
| | | 3 | 71.12 | 74.67 | 80.25 |
| Rice bran flour | TA | 1 | 66.92 | 71.25 | 74.88 |
| | | 2 | 67.88 | 70.58 | 74.04 |
| | | 3 | 65.71 | 70.92 | 75.88 |
| | °C | 1 | 67.37 | 71.58 | 75.99 |
| | | 2 | 65.83 | 72 | 75.68 |
| | | 3 | 65.79 | 70.58 | 75.75 |

**Table A9** % Crystallinity of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour under Long Term Stability Test

| **Natural materials** | **Conditions** | **Month** | **% crystallinity** | ***p*-value** |
|---|---|---|---|---|
| Thanaka powder | Initial | 0 | 38.55±1.71 | - |
| | TA | 1 | 39.27±1.82 | 0.556 |
| | | 2 | 38.61±1.24 | 0.965 |
| | | 3 | 39.46±0.92 | 0.459 |
| | 45 °C | 1 | 38.21±1.38 | 0.775 |
| | | 2 | 38.82±1.33 | 0.823 |
| | | 3 | 38.58±1.58 | 0.982 |
| Corn flour | Initial | 0 | 22.52±1.87 | - |
| | TA | 1 | 21.89±1.73 | 0.628 |
| | | 2 | 21.89±1.71 | 0.630 |
| | | 3 | 22.39±1.40 | 0.859 |
| | 45 °C | 1 | 22.25±1.87 | 0.835 |
| | | 2 | 22.47±0.98 | 0.971 |
| | | 3 | 22.69±1.10 | 0.894 |
| Mung bean starch | Initial | 0 | 31.44±2.05 | - |
| | TA | 1 | 31.42±2.19 | 0.990 |
| | | 2 | 32.48±1.26 | 0.524 |
| | | 3 | 31.41±2.21 | 0.985 |
| | 45 °C | 1 | 31.02±2.10 | 0.795 |
| | | 2 | 31.72±1.76 | 0.860 |
| | | 3 | 31.42±1.88 | 0.990 |
| Rice bran flour | Initial | 0 | 23.92±1.91 | - |
| | TA | 1 | 23.81±1.82 | 0.938 |
| | | 2 | 23.96±1.79 | 0.982 |
| | | 3 | 24.35±1.68 | 0.768 |
| | 45 °C | 1 | 23.95±1.87 | 0.984 |
| | | 2 | 24.04±1.64 | 0.936 |
| | | 3 | 24.28±1.50 | 0.807 |

**Table A10** Water Absorption Capacities of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour under Long Term Stability Test

| **Natural materials** | **Conditions** | **Month** | **Water absorption capacity** | ***p*-value** |
|---|---|---|---|---|
| Thanaka powder | Initial | 0 | 17.31±0.77 | - |
| | TA | 1 | 18.03±0.45 | 0.078 |
| | | 2 | 18.03±0.15 | 0.081 |
| | | 3 | 18.98±0.13 | 0.061 |
| | 45 °C | 1 | 17.88±0.51 | 0.159 |
| | | 2 | 17.82±0.50 | 0.204 |
| | | 3 | 18.05±0.44 | 0.071 |
| Corn flour | Initial | 0 | 16.89±0.57 | - |
| | TA | 1 | 16.35±0.33 | 0.092 |
| | | 2 | 16.96±0.08 | 0.809 |
| | | 3 | 16.54±0.41 | 0.255 |
| | 45 °C | 1 | 16.93±0.13 | 0.878 |
| | | 2 | 17.02±0.34 | 0.670 |
| | | 3 | 16.91±0.44 | 0.948 |
| Mung bean starch | Initial | 0 | 7.84±0.33 | - |
| | TA | 1 | 7.98±0.29 | 0.609 |
| | | 2 | 7.97±0.16 | 0.634 |
| | | 3 | 7.96±0.30 | 0.651 |
| | 45 °C | 1 | 7.85±0.45 | 0.971 |
| | | 2 | 7.68±0.35 | 0.568 |
| | | 3 | 7.66±0.39 | 0.522 |
| Rice bran flour | Initial | 0 | 16.27±0.69 | - |
| | TA | 1 | 16.12±0.46 | 0.697 |
| | | 2 | 16.00±0.21 | 0.481 |
| | | 3 | 16.54±0.33 | 0.491 |
| | 45 °C | 1 | 16.43±0.51 | 0.672 |
| | | 2 | 16.13±0.48 | 0.722 |
| | | 3 | 16.47±0.40 | 0.598 |

**Table A11** Soybean Oil Absorption Capacities of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour under Long Term Stability Test

| **Natural materials** | **Conditions** | **Month** | **Soybean oil absorption capacity** | ***p*-value** |
|---|---|---|---|---|
| Thanaka powder | Initial | 0 | 13.39±0.32 | - |
| | TA | 1 | 13.39±0.32 | 0.976 |
| | | 2 | 13.34±0.23 | 0.844 |
| | | 3 | 13.43±0.28 | 0.832 |
| | 45 °C | 1 | 13.58±0.06 | 0.378 |
| | | 2 | 13.30±0.20 | 0.694 |
| | | 3 | 13.56±0.35 | 0.427 |
| Corn flour | Initial | 0 | 10.81±0.57 | - |
| | TA | 1 | 10.27±0.58 | 0.124 |
| | | 2 | 10.41±0.39 | 0.250 |
| | | 3 | 10.46±0.26 | 0.304 |
| | 45 °C | 1 | 10.95±0.39 | 0.669 |
| | | 2 | 10.93±0.26 | 0.719 |
| | | 3 | 10.46±0.20 | 0.304 |
| Mung bean starch | Initial | 0 | 5.47±0.29 | - |
| | TA | 1 | 5.52±0.21 | 0.820 |
| | | 2 | 5.39±0.24 | 0.716 |
| | | 3 | 5.16±0.31 | 0.173 |
| | 45 °C | 1 | 5.64±0.06 | 0.453 |
| | | 2 | 5.61±0.25 | 0.546 |
| | | 3 | 5.24±0.37 | 0.291 |
| Rice bran flour | Initial | 0 | 9.84±0.41 | - |
| | TA | 1 | 9.79±0.35 | 0.843 |
| | | 2 | 9.82±0.11 | 0.921 |
| | | 3 | 9.92±0.03 | 0.745 |
| | 45 °C | 1 | 9.70±0.26 | 0.564 |
| | | 2 | 9.94±0.18 | 0.662 |
| | | 3 | 9.78±0.40 | 0.799 |

**Table A12** Olive Oil Absorption Capacities of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour under Long Term Stability Test

| **Natural materials** | **Conditions** | **Month** | **Olive oil absorption capacity** | ***p*-value** |
|---|---|---|---|---|
| Thanaka powder | Initial | 0 | 12.65±0.28 | - |
| | TA | 1 | 12.77±0.33 | 0.557 |
| | | 2 | 12.68±0.30 | 0.870 |
| | | 3 | 12.65±0.23 | 0.987 |
| | 45 °C | 1 | 12.58±0.24 | 0.731 |
| | | 2 | 12.63±0.07 | 0.935 |
| | | 3 | 12.53±0.16 | 0.568 |
| Corn flour | Initial | 0 | 10.89±0.57 | - |
| | TA | 1 | 10.67±0.16 | 0.503 |
| | | 2 | 10.70±0.36 | 0.555 |
| | | 3 | 10.62±0.35 | 0.415 |
| | 45 °C | 1 | 10.83±0.18 | 0.848 |
| | | 2 | 10.67±0.29 | 0.510 |
| | | 3 | 11.00±0.62 | 0.755 |
| Mung bean starch | Initial | 0 | 5.06±0.33 | - |
| | TA | 1 | 5.38±0.30 | 0.151 |
| | | 2 | 5.43±0.17 | 0.101 |
| | | 3 | 5.10±0.10 | 0.876 |
| | 45 °C | 1 | 5.46±0.32 | 0.083 |
| | | 2 | 5.13±0.24 | 0.768 |
| | | 3 | 5.06±0.25 | 1.000 |
| Rice bran flour | Initial | 0 | 9.98±0.28 | - |
| | TA | 1 | 9.79±0.29 | 0.371 |
| | | 2 | 10.11±0.25 | 0.534 |
| | | 3 | 9.98±0.43 | 1.000 |
| | 45 °C | 1 | 9.76±0.16 | 0.318 |
| | | 2 | 10.02±0.15 | 0.851 |
| | | 3 | 9.67±0.08 | 0.161 |

**Table A13** Mineral Oil Absorption Capacities of Thanaka Powder, Corn Flour, Mung Bean Starch and Rice Bran Flour under Stability Test

| Natural materials | Conditions | Month | Mineral oil absorption capacity | *p*-value |
|---|---|---|---|---|
| Thanaka powder | Initial | 0 | 11.98±0.30 | - |
| | TA | 1 | 12.01±0.24 | 0.902 |
| | | 2 | 11.99±0.41 | 0.963 |
| | | 3 | 11.98±0.08 | 0.975 |
| | 45 °C | 1 | 11.88±0.09 | 0.634 |
| | | 2 | 12.03±0.34 | 0.817 |
| | | 3 | 11.98±0.17 | 1.000 |
| Corn flour | Initial | 0 | 9.80±0.42 | - |
| | TA | 1 | 9.56±0.25 | 0.381 |
| | | 2 | 9.60±0.21 | 0.464 |
| | | 3 | 9.43±0.30 | 0.189 |
| | 45 °C | 1 | 9.64±0.41 | 0.573 |
| | | 2 | 9.58±0.35 | 0.435 |
| | | 3 | 9.52±0.29 | 0.321 |
| Mung bean starch | Initial | 0 | 5.38±0.58 | - |
| | TA | 1 | 5.23±0.48 | 0.639 |
| | | 2 | 5.10±0.35 | 0.376 |
| | | 3 | 5.27±0.15 | 0.741 |
| | 45 °C | 1 | 5.09±0.37 | 0.365 |
| | | 2 | 5.17±0.26 | 0.504 |
| | | 3 | 5.34±0.25 | 0.915 |
| Rice bran flour | Initial | 0 | 8.63±0.41 | - |
| | TA | 1 | 8.40±0.41 | 0.419 |
| | | 2 | 8.59±0.39 | 0.887 |
| | | 3 | 8.54±0.39 | 0.731 |
| | 45 °C | 1 | 8.77±0.18 | 0.628 |
| | | 2 | 8.81±0.19 | 0.540 |
| | | 3 | 8.48±0.29 | 0.579 |

# APPENDIX B

## PHYSICOCHEMICAL EVALUATION AND STABILITY OF NATURAL FACE POWDER

**Table B1** *L**, *a** and *b** values of L5.1-Int and C7.1-Int

| Natural Face Powder | L* | a* | b* |
|---|---|---|---|
| L5.1-Int | 76.39 ± 0.01 | 5.48 ± 0.01 | 22.74 ± 0.01 |
| C7.1-Int | 64.95 ± 0.03 | 7.99 ± 0.01 | 31.56 ± 0.04 |

**Table B2** *L**, *a** and *b** values of L5.1-HC and C7.1-HC

| Natural Face Powder | L* | a* | b* | ΔE |
|---|---|---|---|---|
| L5.1-HC | 76.31±0.01 | 5.09±0.01 | 22.10±0.01 | 0.75±0.00 |
| C7.1-HC | 63.95±0.01 | 8.12±0.03 | 31.41±0.03 | 1.02±0.03 |