MICROENCAPSULATION INSTANT COFFEE BY SPRAY DRYING USING HYDROLYSED KONJAC GLUCOMANNAN

Miss Desi Sakawulan

จุฬาลงกรณ์มหาวิทยาลัย
Chulalongkorn University
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ไมโครเอนแคปซูเลชันกาแฟสำเร็จรูปโดยการอบแห้งแบบพ่นที่ใช้บุกกลูโคแมนแนนไฮโดรไลเสท

นางสาวเดซี่ ซาคาวูลาน

จุฬาลงกรณ์มหาวิทยาลัย
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วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรมหาบัณฑิต
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Thesis Advisor	Assistant Professor Chaleeda Borompichaichartkul, Ph.D.
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เดชี่ ซาคาวูลาน : ไมโครเอนแคปซูเลชันกาแฟสำเร็จรูปโดยการอบแห้งแบบพ่นที่ใช้บุกกลูโคแมนแนนไฮโดรไลเสท (MICROENCAPSULATION INSTANT COFFEE BY SPRAY DRYING USING HYDROLYSED KONJAC GLUCOMANNAN) อ.ที่ปรึกษาวิทยานิพนธ์หลัก: ชาลีดา บรมพิชัยชาติกุล, อ.ที่ปรึกษาวิทยานิพนธ์ร่วม: ริชาร์ด อาร์เชอร์, 101 หน้า.

งานวิจัยนี้มีวัตถุประสงค์เพื่อศึกษาการใช้บุกกลูโคแมนแนนเป็นวัสดุผนังในการป้องกันสารต้านอนุมูลอิสระในสารสกัดจากกาแฟ และศึกษาสภาวะที่เหมาะสมในกระบวนการทำแห้งแบบพ่นเพื่อผลิตกาแฟไมโครแคปซูลสำเร็จรูป โดยการศึกษาแบ่งเป็น 2 ขั้นตอน ในขั้นตอนแรกใช้ผงบุกสองชนิด *(A. muelleri และ A. bulbifer)* และความเข้มข้นของเอนไซม์แมนนาเนสที่แตกต่างกัน เพื่อลดความหนืดของสารละลายบุกกลูโคแมนแนนไฮโดรไลเสท (KGMH) พบว่าแมนนาเนสที่ความเข้มข้น 38,000 หน่วยต่อกรัมผงบุก สามารถลดความหนืดของสารละลายบุกกลูโคแมนแนนจาก *A. muelleri* ลดลงเหลือน้อยกว่า 100 m.Pa.s ในขณะที่สารละลายบุกกลูโคแมนแนนจาก *A. bulbifer* ยังมีค่าความหนืดสูงกว่า 100 m.Pa.s ขั้นตอนที่สอง นำสารสกัดกาแฟเข้มข้น (20 ° Brix) ผสมกับ KGMH (5, 15 และ 20% w/w) ที่เป็นวัสดุเคลือบผิว และเพิ่มมอลโตเด็กตริน (MD) เพื่อปรับปริมาณของแข็งในระบบ (15, 5 % w/w) และใช้เป็นตัวควบคุม (20% w/w) จากนั้นผสมสารเคลือบผิวและสารสกัดกาแฟที่อัตราส่วน 2: 1 (w/w) แล้วทำแห้งแบบพ่นโดยใช้อุณหภูมิขาเข้าที่ 160, 170 และ 180 องศาเซลเซียส และที่อุณหภูมิขาออก 85-90 องศาเซลเซียส พบว่ากาแฟสำเร็จรูปที่ผลิตจาก KGMH 20% (โดยที่ไม่มี MD) และทำให้แห้งที่อุณหภูมิ 160 องศาเซลเซียส มีสารประกอบฟีนอลเหลืออยู่สูงสุดที่ 129% การออกฤทธิ์ต้านอนุมูลอิสระโดยวิธี DPPH เหลือที่ 131% และ การออกฤทธิ์ต้านอนุมูลอิสระโดยวิธี FRAP เหลือที่ 122% (p <0.05) ในขณะที่กาแฟสำเร็จรูปที่ผลิตจาก KGMH 20% และทำให้แห้งที่อุณหภูมิ 180 องศาเซลเซียส มีกรดคลอโรจินิกเหลืออยู่สูงสุด ดังนั้น KGMH มีผลต่อสมบัติการต้านอนุมูลอิสระและกักเก็บกาแฟในรูปของแข็งคงอยู่ในไมโครแคปซูลเกือบ 100% นอกจากนี้การเพิ่มความเข้มข้นของ KGMH เพิ่มปริมาณเส้นใยอาหารที่ละลายน้ำและไม่ละลายน้ำมีค่าเพิ่มสูงขึ้น (p <0.05) และพบว่าสมบัติทางกายภาพของกาแฟสำเร็จรูป เมื่อเพิ่มความเข้มข้นของ KGMH ส่งผลให้ผลผลิต (%yield) และค่าดัชนีการละลายน้ำลดลง แต่ค่า ดัชนีการดูดซับน้ำสูงขึ้น (p <0.05) อย่างไรก็ตามพบว่า ค่าดัชนีการละลายน้ำที่ต่ำที่สุดยังมีค่าสูงกว่า 88% ซึ่งสามารถจำแนกได้ว่าเป็นของแข็งที่ละลายได้ดี ปริมาณความชื้นและกิจกรรมของน้ำ (a_w) ของกาแฟสำเร็จรูปทุกการทดลอง มีค่า 3.37-3.75% และ 0.21-0.25 ตามลำดับ (p > 0.05) ดังนั้นจึงสรุปได้ว่ากาแฟสำเร็จรูปที่มี KGMH เพียงอย่างเดียวเป็นวัสดุเคลือบมีฤทธิ์ต่อต้านอนุมูลอิสระที่ดีกว่า และมีเส้นใยอาหารสูงกว่าไมโครแคปซูลที่มี MD อยู่ ในการศึกษาครั้งนี้แสดงให้เห็นถึงศักยภาพในการใช้สารละลาย KGMH เป็นวัตถุดิบในกระบวนการไมโครเอนแคปซูเลชันโดยใช้การอบแห้งแบบพ่นร่วมด้วย

ภาควิชา	เทคโนโลยีทางอาหาร	ลายมือชื่อนิสิต	.....
สาขาวิชา	วิทยาศาสตร์และเทคโนโลยีทางอาหาร	ลายมือชื่อ อ.ที่ปรึกษาหลัก	.....
ปีการศึกษา	2560	ลายมือชื่อ อ.ที่ปรึกษาร่วม	.....

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DESI SAKAWULAN: MICROENCAPSULATION INSTANT COFFEE BY SPRAY DRYING USING HYDROLYSED KONJAC GLUCOMANNAN. ADVISOR: ASST. PROF. CHALEEDA BOROMPICHAICHARTKUL, Ph.D., CO-ADVISOR: PROF. RICHARD ARCHER, Ph.D., 101 pp.

This research is aimed at finding a suitable wall material from konjac glucomannan to shield antioxidant compounds in coffee extract along with appropriate condition of spray drying to produce microencapsulated instant coffee. There are two steps of study. In the first step, two types of konjac powder (*A. muelleri* and *A. bulbifer*) and different concentrations of mannanase enzyme were used to investigate the viscosity reduction of konjac glucomannan hydrolysate (KGMH). It is found that mannanase at 38,000 units per gram substrate was able to lower the viscosity of *A. muelleri* solution to less than 100 m.Pa.s, while *A. bulbifer* remained above 100 mPa.s at the chosen concentration. In the second step, concentrated coffee extract (20 °Brix) was mixed with KGMH (5, 15 and 20% w/w) as coating material. Maltodextrin (MD) (15, 5 % w/w) was added to adjust total solids of coating material and used as control (20% w/w). The mixture of coating material and coffee extract at solids ratio 2.2:1 (w/w) was then spray dried at 160, 170 and 180 °C inlet temperature and at 85-90 °C outlet temperature. Instant coffee produced from 20% KGMH (without MD) and dried at 160 °C presented the highest apparent retention of phenolic compounds (129%), DPPH scavenging activity (131%), and antioxidant activity of FRAP (122%) ($p<0.05$). Meanwhile, the highest apparent retention of chlorogenic acid was provided by instant coffee of 20% KGMH and dried at 180 °C (130%). Apparently, KGMH was contributing to the antioxidant properties and the coffee solids were retained nearly 100% in microcapsule. Increasing KGMH concentration leads to higher dietary fiber both soluble and insoluble dietary fiber ($p<0.05$). Meanwhile, in physical properties, increasing KGMH concentration was correlated to lower production yield and lower water solubility index but higher water absorption index ($p<0.05$). However, it should be noted that the lowest water solubility index was still above 88% which can be classified as good solubility of powder. Moisture content and water activity of all formulations of instant coffee ranged between 3.37-3.75% and 0.21-0.25 ($p>0.05$). To sum up, this study revealed the potential use of KGMH solution as wall material in spray drying microencapsulation. Instant coffee alone as coating material presented better antioxidant activity and dietary fiber than microcapsule of MD alone.

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Department:	Food Technology	Student's Signature	.....
Field of Study:	Food Science and Technology	Advisor's Signature	.....
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CHAPTER 1
INTRODUCTION

1.1 Background

Coffee is a popular beverage that is obtained from the brewing of roasted coffee bean. According to International Coffee Organization (ICO), world coffee consumption in example 60 kg bags reached the point of 151.3 million bags in 2015/2016. Coffee consumption has been gradually rising that is reflected the increasing of coffee popularity among available beverages in the global market. Coffee has extensively studied as a special beverage with unique aromatic taste and stimulating effects. The pleasant taste and aroma came from the coffee processing especially the roasting step, where the bitter taste and aromatic volatile components are formed. Apart from its specialty pleasant flavor and aroma, many people choose coffee as their daily drink due to its effect on body health. Coffee brews are accepted as a rich source of compounds possessing antioxidant activity greater than other beverages including tea and wine (Pellegrini *et al*., 2003). Coffee is high in phenolic compounds especially chlorogenic acids and their degradation products such as caffeic, ferulic and coumatic acids, polyphenols, trigonelline and alkaloids including caffeine and also have a considerable amount of melanoidins which exhibit significant antioxidant activities (Vignoli *et al*., 2011, Brezová *et al*., 2009).

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Green coffee beans, roasted coffee beans, ground coffee and soluble or instant coffee are commercially available to the consumer. Special taste and rich in aroma contribute to fresh brewing coffee to dominate the coffee market. Nonetheless, instant coffee is also gaining more interest because of its easy-ness in preparation, longer shelf life, and higher bioactive compounds. Comparing antioxidant capacity in one cup from a different form of coffees, instant coffee is accounted for the highest amount of essential substances which expressed antioxidant capacity 3-4 times higher than ground coffee (Pérez-Hernández *et al*., 2012). During the manufacturing of instant coffee, antioxidant and other essential compounds are concentrated and resulted in enhancing of antioxidant capacity compare to ground roasted bean (Pérez-Hernández *et al*., 2012).

More advantages of instant coffee stimulate the research more deeply in its production. The conventional processes of instant coffee are spray drying and freeze drying, both techniques have some limitations regarding energy consumption and final product qualities. Freeze drying produces the best product quality in term of aroma recovery, but it uses high energy and time that affected the production cost. While, spray drying leads the high production capacity at low investment, but engender higher thermal impact and resulted in decreasing of essential compounds such as natural antioxidants. In fact, that spray drying is the most applicable process in instant coffee industry yet gives lower antioxidant and flavor compounds than other processing, thereupon considering other techniques to lowering thermal impact is proposed. Microencapsulation offered as a solution to reduce the core reactivity from essential compounds with the environmental factor. According to Gharsallaoui *et al*. (2007), microencapsulation provides a physical barrier between the core and other compounds of the product. In line with instant coffee production which mostly used spray drying technique, Frascareli *et al.* (2012) reported that spray drying is the most common procedure for microencapsulation in the food industry since it has cost-effective, flexible and produces good quality particles. Combining both techniques is expected to produce the better instant coffee product.

Wall material of microencapsulation can be selected from a wide variety of natural and synthetic polymers depend on the core material and desired characteristics in the final product. In general, carbohydrates are used as coating material for microencapsulation by spray drying since they show low viscosity at high solid content and good solubility (Silva *et al.*, 2014). In terms of instant coffee production, the wall material must be water-soluble compounds since coffee brewing employed water as solvent extraction. One of the water-soluble polysaccharides is konjac glucomannan (KGM) which extracted from *Amorphophallus konjac* tubers. Refer to Commission Regulation (EU) No. 231 (2012), KGM is authorized as food additives which are a substance that is not normally consumed as the food itself but is added to food intentionally for technological purposes. According to the United States of Food and Drug Administration (2014), KGM is classified as generally recognized as safe (GRAS) since 1997. Meanwhile, according to Food and Agriculture Organization of United

Nations and World Health Organization under Codex Alimentarius, classified konjac flour as food additive with CAS number 37220-17-0 and INS number of 425.

As a natural polysaccharide, KGM is widely used in industries such as food, pharmaceutical, chemical, and biotechnology. Konjac flour which is unrefined KGM considered as a low-calorie ingredient given by its non-digestible fiber content (Jiménez-Colmenero *et al*., 2012b). However, the application of KGM is restricted by its physical characteristic which has very high apparent viscosity if dissolved directly in water. In the current study, the enzymatic reaction was used to decrease KGM viscosity. Therefore, the aims of this study were to investigate the utilization of hydrolyzed KGM on encapsulation efficiency and to obtain the suitable spray drying condition in the production of instant coffee microencapsulation.

1.2 Objectives

- To measure the effect of hydrolysed KGM on encapsulation efficiency compared to maltodextrin (MD) for wall material
- To obtain suitable spray drying condition in the production of microencapsulation instant coffee

1.3 Scope of study

The thesis research consists two main parts:

- Selection the type of powder and enzyme concentration for KGM hydrolysis
- Optimization microencapsulation process with variation on wall material formulation and inlet temperature of spray dryer

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CHAPTER 2
LITERATURE REVIEW

2.1 Coffee

The coffee plant belongs to the family of *Rubiaceae* and genus Coffea. The fruit is known as cherries that contain seeds with which coffee beverage is made. Special taste and aroma are the keys to consumer acceptance of coffee. Moreover, people may drink coffee for some emotional reasons such as to work up their mood or relaxation. Another reason for drinking coffee might have related to rational reasons, to help to stay up while working at late night, for instance. Coffee also contributes to a public society where thousand café or coffee houses are used for friendship gathering or business meeting. As a traded commodity, coffee is the second most traded commodity in the rank international after petroleum (Borrelli et al., 2002a). A complex sequence of processes starting with planting in the field and finishing in the cup. The chains involved coffee farmer for cultivation, coffee processing, transport and marketing which supply employment to millions of people. According to Lewin *et al*. (2004), around 20-25 million smallholder family farmers in more than 50 developing countries rely their life on coffee farming and business. They estimated that approximately around 100 millions of people are directly affected by coffee trade.

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2.1.1 Basic processing of coffee

The process from coffee bean to a cup of coffee is a complex step involving many steps and reactions. In general, the process consists of cultivation and harvesting, green bean processing, storage and transport, blending, roasting, grinding, packaging, storage, and brewing. Coffee fruits are typically harvested by manually picking, stripping or mechanical harvest. Mangal (2007) explains that manually picking is a time-consuming method but tend to get good quality of coffee seeds. Meanwhile, stripping and mechanical harvesting are faster than picking method but yielded also the defects of fruits such as immature and overripe cherries as well as stones, husks, and twigs. Green and overripe cherries will affect to sour seeds that are lowering the cup quality. Harvested cherries then undergo pulp

extraction to get the only green seeds part. There are three methods in this step which are dry method, wet method, and semi-dry method. The dry method used the whole cherry which contains seeds, mucilage, and pulp and directly dried under the sun or drying machine. The dry process typically has heavier body with lower acidity (Bee *et al*., 2005). In the wet method (**Figure 2.1**), the flesh of fruit is eliminated by a specific machine, while mucilage removal can be done by natural fermentation, chemical removal or aid by machine. Wet processing produces a coffee with special sensory characteristics which alternatively used by the coffee producer to suit differs markets (Evangelista *et al*., 2015). The semi-dry process is intermediate between the natural and washed process. The coffee fruits are pulped then directly undergo drying process with beans covered by mucilage. The aim of this process is to get both dry and wet characteristics which are excellent in body with a fruity aroma.

Stored green bean either blended beans or one variety of bean then undergo roasting process. Refer to Farah (2012), complex reactions occur in the roasting process. Exothermic and endothermic reactions are started at above 160°C with expanded volume and aroma formation begins. The formation of aroma happens until the temperature of the beans reached 190°C. In this stage, Maillard and Strecker's reactions occur. Dramatically changes in chemical and biological activities took place at this stage. The natural phenolic compounds may be degraded but other essential compounds from Maillard and Strecker reaction are formed (Wang *et al.*, 2011). Roasted beans are either dispatched to market or undergoing a further process which is grinding. Pleasant aroma arising from roasted coffee are released during grinding. Coffee aroma compounds are highly volatile and unstable product, therefore grinding process contributes to the loss of volatile compounds (Akiyama *et al*., 2005). The last step in the preparation of a cup of coffee is brewing or coffee extraction. Many factors affected the composition of coffee extract including ratio ground-roasted coffee and water, temperature, the contact time between coffee and water and filter material. The most important phenomena in this step are extracting water-soluble compounds from the coffee ground. A study from Caporaso *et al*. (2014) shows that different brewing methods provide the varying

chemical composition. They conclude that high pressure increases extraction efficiency in term of total solid and antioxidant defined by ABTS method.

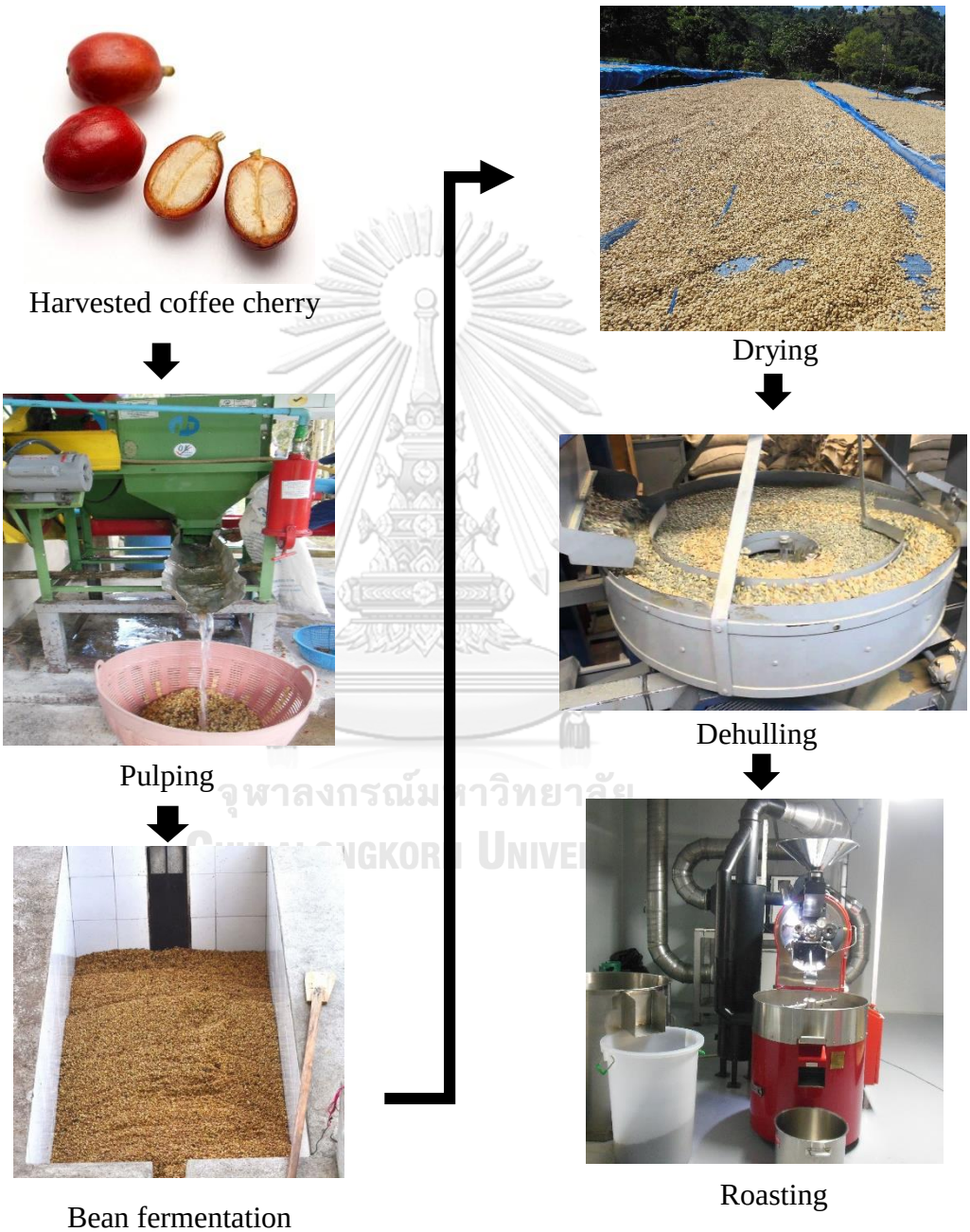

Figure 2.1 General coffee processing (wet method bean processing)

2.1.2 Potential benefits from coffee constituents

Coffee has possessed a prominent role in human society for a long time. Its consumption is gradually increasing over the years. The coffee brew itself contains a mixture of chemical components which exhibit the functionality towards human health. Coffee has been classified as a rich dietary source of antioxidants such as chlorogenic acids, hydrocynnamic acids, caffeine and Maillard reaction products, which represents the main source of antioxidant of the diet in many countries (Torres and Farah, 2010). For instance, coffee is the major source of polyphenols intake from non-alcoholic beverage consumption in Japan (Fukushima *et al*., 2009). *In vivo* study from Natella *et al*. (2002) concluded that supplementation with 200 mL of coffee beverage contributes to the increasing of plasma antioxidant capacity and it was better than tea. This finding was confirmed by Pellegrini *et al*. (2003) and concluded via *in vitro* study that among beverages, regardless of the preparation methods, coffee was the had the greatest antioxidant activity followed by citrus juice. Many dietary plant constituents, such as flavonoids and phenolic compounds that provide antioxidant activity. Interestingly, antioxidant activity from coffee might come not only from natural components but also from components that formed during coffee processing. One of predominant natural antioxidant in coffee is chlorogenic acids. Chlorogenic acids are the most abundant amongst ten identified phenolic acids in both green and roasted coffee (Somporn *et al*., 2011). This group of acids is derived from the esterification of trans-cinnamic acid such as caffeic, ferulic and *p*-coumaric with quinic acid. The major subgroup of chlorogenic acids in green coffee is caffeoylquinic acids, dicaffeoylquinic acids, feruloylquinic acids, *p*-coumaroylquinic acids and caffeoyl-feruloylquinic acids. Each of group has at least three major positional isomers. However, among these classes, caffeoylquinic acids in more specified of 5-caffeoylquinic acid is the most abundant which reported around 80% of total chlorogenic acids, therefore this compound is commonly named as chlorogenic acid (Farah, 2012). Example of chlorogenic acids benefit is as anti-obesity that also promote lipid metabolism. This work is proven by using high-fat diet-induced-obese mice supplemented by caffeic acid (Cho *et al*., 2010). Another bioactive compound that contributes to antioxidant properties in coffee is melanoidins. In contrast with chlorogenic acids, melanoidins are compounds formed

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during coffee processing. A complex physical and chemical changes including the changes of color to be very brown to dark color and formation of the coffee aroma are taking place during the roasting process. The dark color compounds represent melanoidins which contributed to 25% of dry matter in coffee beverages (Borrelli et al., 2002b). Vignoli *et al*. (2014) also reported that melanoidins are responsible for antioxidant activity of coffee measured by isolated the high molecular weight fraction from roasted coffee.

Apart from antioxidant activity, major studies dealing with health benefits and risk from coffee is related also to its stimulatory effects from caffeine. Review from Higdon and Frei (2006) mentioned that epidemiological study finds the results that coffee consumption may help to prevent some chronic diseases such as type 2 diabetes mellitus, Parkinson's disease, and liver disease. However, there is conflicting finding since caffeine also contributes to blood pressure elevation and diuresis. Most prospective cohort studies found that coffee consumption might relate to several cardiovascular disease risk factors but not significantly increased cardiovascular disease risk. Chu (2012) emphasized that moderate consumption presented more correlation with the positive impacts rather than to risks. Caffeine is widely consumed and studied psychoactive substance due to its effect to stimulate the central nervous system. Coffee is a source of caffeine which is accounted for around 0.9 to 2.5 g/100 g of green coffee depend on its variety. Arabica coffee contains around 0.9% – 1.3% of caffeine, while Robusta coffee has double concentration than Arabica (Farah, 2012). Caffeine is a non-volatile heat stable alkaloid. Therefore, caffeine amount is not decreased along coffee processing. Other than caffeine, trigonelline also classified as second most predominant alkaloid component in green bean (Viani and Horman, 1974). Trigonelline contributes to the bitterness of coffee beverages and also an important precursor for pyrroles and pyridines, volatile compounds that are formed during roasting step. Trigonelline concentration in the green bean is ranging from 0.6 to 2%. Roasting process degraded trigonelline and provide a variety volatile component. Trigonelline in roasted bean accounted for 0.3 to 1.2% depends on concentration in green bean and degree of roasting. Trigonelline has shown to have bioactive compounds

characteristics such as an antimicrobial agent for *Streptococcus mutans*, a bacterium that associated with dental caries in human, inhibit the invasion of cancer cell in the liver and a recent study shows that trigonelline has been considered as a novel of phytoestrogen (Allred *et al.*, 2009).

2.1.3 Coffee constituent problems

Coffee can deliver relatively high antioxidant compounds. However, some steps during processing such as fermentation, roasting, and drying in coffee instant process, may have a significant impact on lowering a number of bioactive components. Amid of these steps, roasting stage in coffee beans processing and drying in instant coffee production is the most intense effect, especially in thermal labile components. As for instance, phenolic compounds in coffee is affected by the roasting process. Chlorogenic acids are unstable at high temperature, thus lowering the temperature of roasting process and the addition of unroasted coffee tend to produce a final product with favorable chlorogenic acids content (Mills *et al.*, 2013). In agreement with this result, Vignoli *et al*. (2014) found the decreasing of chlorogenic acids, specifically 5-CQA, in both Robusta and Arabica coffee are correlated with the roasting degrees. Chlorogenic acids can undergo isomerization at the initial stages of roasting and degraded to become quinic acid or quinides during roasting. The chlorogenic acids degradation product, for instance, caffeic acid will further degrade along the roasting process. Trigonelline as major bioactive components in green coffee has significantly decreased during roasting due to its thermal instability characteristic. Roasting time and temperature are affected the content of trigonelline since this compound is rapidly degraded and form the volatile compounds. Trigonelline degradation enhanced when the roasting temperature reaches 200 °C and the remaining trigonelline in roasted coffee is only 5% in Arabica and 15% in Robusta at 240 °C, as a consequence the level of nicotinic acid is extremely increased more than 500% in both species (Casal *et al.*, 2000). This result is similar to a recent study which shows the declining of trigonelline content during roasting with mean decreasing up to 90% (Vignoli *et al*., 2014). Storage also contributes negatively to the essential compound of coffee especially lipid composition. A study from Toci *et al.* (2013) shows that the degree of roasting and

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storage are changing triacylglycerol and free fatty acids composition. They study with light-medium and dark-medium roast coffee beans along with air and N_2 atmosphere found that triacylglycerol is hydrolyzed and oxidized during storage in both light-medium and dark-medium samples. These reactions are in higher rate when it stored in air condition than N_2 atmosphere due to the availability of oxygen. While the hydrolysis reaction is triggered by the absorption of water to coffee bean during storage with the fact that coffee is highly hygroscopic. On the other hand, free fatty acids increased dramatically in both sample in the first three months storage. After three months storage, the content of free fatty acids decreased in both samples. The loss of free fatty acids might be correlated with the oxidation of those compounds, both changes in triacylglycerol and free fatty acids in coffee storage are associated with rancidity of coffee.

2.1.4 Instant coffee

Instant coffee is one of available product in the market that has gained more attention in recent years. Instant coffee took almost 50% of world coffee production (Esquivel and Jiménez, 2012). Simple in preparation, long shelf life and an alternative ingredient to be mixed in beverages are the reasons for instant coffee to take a place in the coffee global market. The common instant coffee production consists several steps (**Figure 2.2**)

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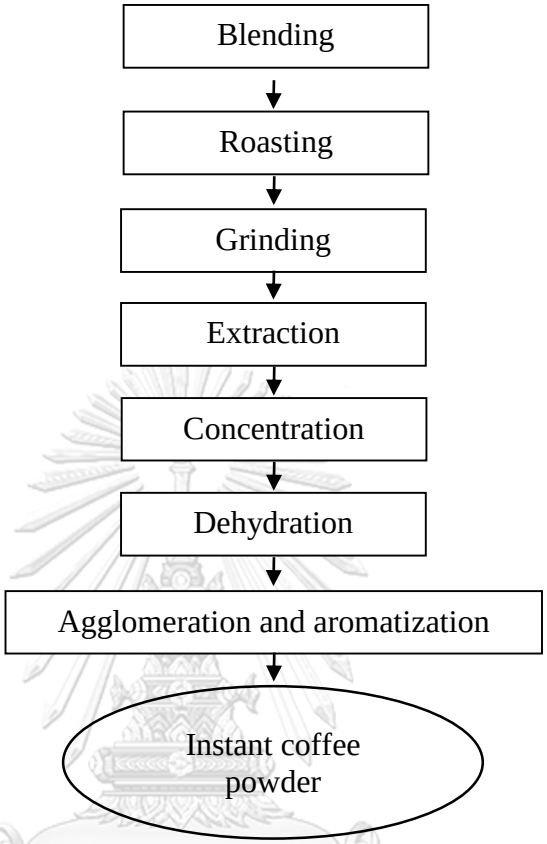

Figure 2.2 Instant coffee processing

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Various coffee beans are mixed according to company recipe until mixed evenly. Blended green beans then undergo roasting step where the flavor and aroma were released. Physical, chemical and sensorial changes are produced by pyrolysis reaction ay 160 – 260 °C (Pittia *et al.*, 2001). Huang and Zhang (2013) explain, after the targeted degree of roast is obtained, the hot roasted beans then undergo rapid cooling using water or air as a cooling medium to prevent further aroma volatilization. The roasted coffee beans then converted into coarse granule grinding process. A series of five to eight extraction columns that employed thermal and pressure are used to extract soluble compounds from grounded coffee. The water passes to hot column extractor to obtain carbohydrates before went through the colder column for flavor component extraction. By the end of extraction cycle, the concentrated coffee extract was obtained

(at least 20% of soluble solids). The step after coffee extraction is concentration with the aim of final solid extract around 40%. Most used techniques are vacuum evaporator, centrifuge inspissation and freeze concentration. The concentrated coffee extract is then undergoing dehydration process. Spray dryer and freeze dryer are commonly used in soluble coffee processing. The spray dryer is performed under feed pressure (nozzle) and high temperature (above 150 °C) to conduct rapid evaporation of water from the coffee mixture. The ability to dehydrate liquid and at the same time produce the powder in a micron scale is the key of spray drying advantages over other techniques (Woo and Bhandari, 2013). In consequence, spray drying has high productivity with low-cost operation. Meanwhile, freeze drying technique starts with freezing followed by sublimation at vacuum condition. Since it is employed very low temperature (-35 – 5 °C), this technique mostly used for preserving or drying heat-labile compounds. In instant coffee production, freeze drying is used to produce the high-quality product in term of aroma recovery. However, freeze drying requires a lot of time (90 hours) until it produces dried powder, therefore, it is consumed high energy. Different drying also will affect the physical properties of the coffee powder. Figure 2.3 shows spray drying instant coffee particle that exhibits a smooth and spherical shape, while freeze dying product is observed to be relatively less porous (Padma Ishwarya and Anandharamakrishnan, 2015). Moreover, once instant coffee powders are collected, they undergo agglomeration process to produce soluble coffee granule. Finally, aroma compounds are added to soluble coffee granule right before it is filled into the packaging. The aroma compounds were collected during the roasting and grinding process. Instant coffee as concentrated coffee extract can be used as an alternative of antioxidant intake. Instant coffee provides antioxidant 3-4 times more than coffee grounded because antioxidant and other essential compounds are concentrated during instant coffee production (Pérez-Hernández *et al*., 2012).

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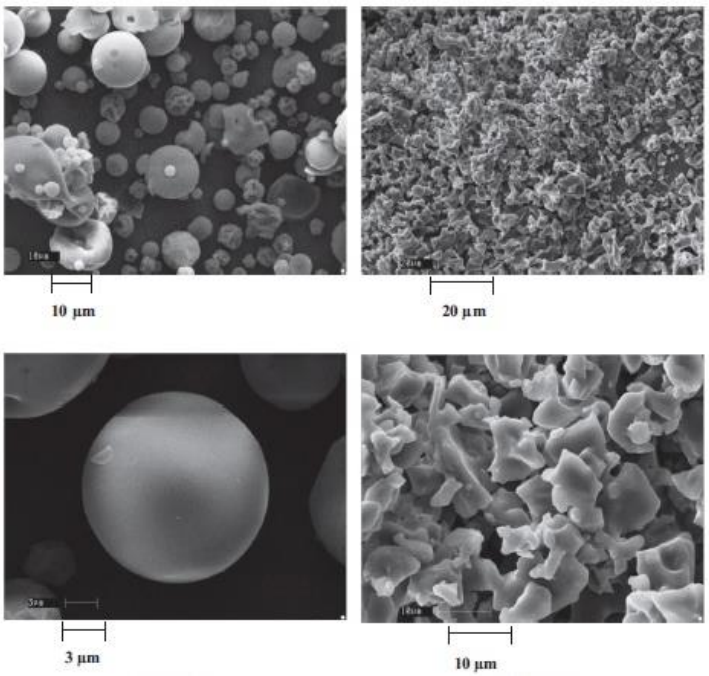

(A) (B)

Figure 2.3 SEM micrographs of instant coffee from spray drying (A) and freeze drying (B)

2.2 Microencapsulation

Bioactive compounds such as oils and phenolic compounds are mainly sensitive to oxygen, light, moisture, and heat. For example, bioactive oils mostly consist of unsaturated fatty acids and easy to be oxidized in a normal environment. Oil oxidation products including free radicals, hydroperoxides, aldehydes, hydrocarbons, ketones, and epoxides are delivered negative impact on biological tissues (Chen *et al*., 2011). In addition, some research concerning encapsulation strategies for bioactive oils concluded that the low stability of bioactive oils that impact limitation of the application can be tackled with encapsulation techniques (Rodríguez et al., 2016a). On the other hands, some plants constituents were considered technically infeasible in food processing. Unstable in feverish temperature, poor availability and unpleasant taste or strong odor are examples of food processing barrier. Microencapsulation is a promising technique that can shield bioactive compounds from light and oxygen also allowing stability, bioavailability, flavor masking and controlled release while maintaining their functional properties (Wattanaprasert *et al*., 2017, Sagalowicz and Leser, 2010,

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Gonçalves *et al*., 2016). Microencapsulation is a technique where small particles or droplets are surrounded by a wall material or embedded in the homogeneous or heterogeneous matrix and provide small capsules with many useful characteristics. Gharsallaoui *et al*. (2007) explain that microencapsulation can be used in liquid, solid and gas droplets that entrapped into thin films of a food grade microencapsulation agent.

2.2.1 Spray drying microencapsulation

Many techniques have been developed in microencapsulation, such as drying by spray or freeze drying, coacervation, in situ polymerization, extrusion, and layer-by-layer (Rodríguez et al., 2016b). Among available techniques, spray drying is the most commonly used in food industry. Example of spray drying microencapsulation in the food industry is milk powder processing. In fact, that milk is an emulsion contains milk fat as the core material, lactose and milk protein as wall materials. The benefit of this process, for instance, is formation a free-flowing powder characteristic as the amorphous and glassy lactose are formed during drying (Roos, 2002). Moreover, Chen and Mujumdar (2009) pointed out ten key advantages of spray drying:

- Predetermined characteristics such as size, density, moisture content along with the type of powder (fine powder, granules, and agglomerates) can be planed.
- The quality of powder remains constant during the whole process as long as the drying conditions are held constant.
- Powders with narrow size distribution can be produced.
- The powder is ready for packaging – no additional grinding process.
- Can be used for heat-sensitive and heat-resistive liquid compounds without significant damage to the product.
- A multipurpose process which can be used to dry a wide range of liquid materials.
- High production rates and economical process.
- The plant operation and maintenance can be run automatically.

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- Comprehensive knowledge has been established to define different phenomena of spray drying using mathematical model and computational tools.

In spray drying, water removal took place in drying chamber in a short time and very high temperature. In consequence, spray dryer produces low moisture content and low water activity of powder that is beneficial in the food industry to ensure a microbiological stability of products, reduce the storage and transport costs and obtain powder with high solubility. The application of spray dryer in microencapsulation was reviewed by Gharsallaoui *et al*. (2007), they explain that this process consists of four important stages: dispersion or emulsion preparation, homogenization of dispersion, atomization of feed mixture and dehydration of droplet particle. **Figure 2.4** shows the schematic work of spray drying. The drying process starts with atomization in which small droplets are formed from the atomizer. Small droplets increase heat-transfer contact between low-humidity hot gas and liquid and also maximized the heat and mass transfers. The contact between the water droplet and hot air takes place during atomization and stimulates the drying step. There are two types atomization, namely co-current and counter-current. Co-current process sprays the droplets in the same direction as the hot air flow, the evaporation took place instantaneously. Meanwhile, in counter-current drying, the liquid sprayed in a different direction with hot air. Consequently, the dry powder will be exposed to higher temperature compare to the co-current system. Nevertheless, it is noted that counter-current process is considered required lower energy. In drying chamber where evaporation occurs, the heat from gas makes the droplet temperature increase in constant rate and promotes droplet evaporation. The rate of water movement from the center of droplet is considered at the constant rate and equal to droplet surface vaporization. Eventually, when the moisture content at its critical value, the outer part is getting dry and drying rate extremely decrease. Theoretically finished when the particle temperature is similar to surrounded air. According to Corrigan (1995), the drying time in spray drying is done within 5-100 seconds. The dried powder then migrates to dry product and humid air separation step that happen in a cyclone. The dense powder is collected in product chamber,

while the fine particles will pass through the cyclone to be separated from the humid air.

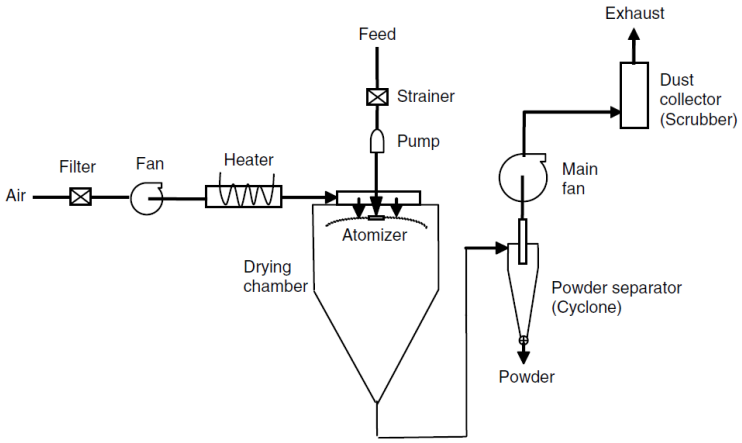

Figure 2.4 Schematic diagram of spray dying (Chen and Mujumdar, 2009)

2.2.2 Wall material

Microcapsule properties are highly depending on its wall material, core material, and microencapsulation techniques. The core material is bioactive compounds inside microcapsule, while wall material is the membrane or shell that covers bioactive in microcapsule. Wall material selection is very important in order to get high efficiency and stability. Gharsallaoui *et al*. (2007) suggested that the criteria for wall material mostly based solubility, molecular weight, glass transition, crystallinity, diffusibility, film forming, and emulsifying properties wall material can be chosen from a wide range of polymers depends on the core material property and targeted powder characteristic. Different from another technique, in spray drying microencapsulation, the wall material should a water-soluble compound. The wall solution also should have low viscosity with good properties of emulsifying or have a stable dispersion before drying. The most common polymer used in this technique is carbohydrates. Protein also classified as a good wall material since it has an amphiphilic property which required for hydrophobic core material encapsulation. Moreover, in this technique, rapid solidification is a key for core material encapsulation (Chen and Mujumdar, 2009).

Carbohydrates such as starches, maltodextrin, and gums are the most common encapsulation agent for spray drying microencapsulation. These materials have a good solubility and gelling properties. Carbohydrate also provides little affinity for hydrophobic flavor, for instance, gum Arabic provided good encapsulation efficiency in pepper oleoresin microencapsulation (Shaikh *et al*., 2006). This result was a good agreement with Yang *et al*. (2009) which found that gum Arabic can be used as orange oil encapsulation. In addition, some carbohydrates in low concentration also can act as an emulsifier. Pectin as a polymer that contains protein residue is able to produce emulsions and work sufficiently for spray drying mixture preparation (Drusch, 2007). However, the utilization of low molecular weight of carbohydrates tends to promote caking, collapse and re-crystallization of powder upon storage. Meanwhile, functional properties of protein allow them to be a good wall material for a wide range of core materials. The most commonly applied proteins for spray drying encapsulation are skim milk, gelatin, and whey proteins. A study from Aghbashlo *et al.* (2013) revealed that skim milk powder, whey protein concentrate and whey protein isolate can be used as a single wall material or mixed of it for fish oil microencapsulation. **Table 2.1** shows different wall material as an encapsulated agent with different core materials.

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Table 2.1 Spray drying microencapsulation method of bioactive compounds with different wall materials and encapsulation efficiency

Bioactive	Technology	Materials	Encapsulation efficiency (%)	Authors
Green coffee extract	Spray dryer, I: 170 °C, O: 90±1 °C	Lecithin and chitosan	86	Carvalho *et al*. (2014)
Fish oil	Spray dryer, I: 140, 160, 180 °C	Skim milk	59-86	Aghbashlo *et al.* (2013)
Rosemary essential oil	Spray dyer, I: 190 °C	Maltodextrin and modified starch	54	de Barros Fernandes *et al.* (2014)
Flaxseed oil	Spray dyer, I: 190 °C, O: 80 °C	Modified starch	30	Barroso *et al.* (2014)

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2.3 Konjac glucomannan

Konjac glucomannan is a natural polysaccharide extracted from *Amorphophallus* spp. plants. This type of plant is a perennial herbaceous herb which grows in elevated areas in subtropical and tropical regions mainly in Asian countries (Fang and Wu, 2004, Tatirat and Charoenrein, 2011). Glucomannan isolated from konjac is a high molecular weight, water-soluble and non-ionic polysaccharides. The

molecule consists a linear random copolymer of (1→4) linked β-D-mannose and β-D-glucose with an approximate ratio of mannose and glucose at 1.6:1 (Davé and McCarthy, 1997). KGM solution formed very high viscosity at low soluble solid. KGM has a high viscosity of the water-soluble adhesive, excellent thickening, gel and film agents which classified as pseudoplastic fluid (Wang *et al.*, 2012). Konjac tuber has been used for a long time for traditional uses and its special characteristics also make konjac potentially to be used for advanced technology.

2.3.1 Basic application of Konjac glucomannan

Japan and China have been used konjac tuber as food and food additives for more than 1000 years (Zhang *et al*., 2005). Konjac flour is used to make noodles, tofu and traditional snacks with springy and chewy characteristics. In Japanese cuisine, the mixture of konjac or konnyaku flour with lime and water is used as the main ingredient of konjac Japanese noodle (Shirataki) and other cuisines such as sukiyaki and udon. In traditional medicine of China, the therapeutic effects of konjac extract are associated with the function of detoxification, tumor-suppression, blood stasis alleviation and phlegm liquefaction (Niwa *et al.*, 2014). Refer to Shah *et al*. (2015), the most important KGM characteristics for human health are the ability to delay gastric emptying, aids in avoiding constipation, being a laxative to support diverticulitis management, a fermentable substrate in the colon, restricting the growth of pathogens in the gut.

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In the traditional way of konjac powder processing, harvested konjac tubers are washed before peeling, slicing and drying under sun drying or machine. Dried sliced konjac are grounded to produce konjac flour. Japan is the first country to develop konjac as commercially flour by pulverizing dried chips and used the coarse grounded konjac chip to be the raw material of traditional foods (Chua *et al.*, 2010). The naturally odorless and tasteless of konjac powder making it easy to be used in daily cooking and baking when gluten-containing flour or other glutinous powder are avoided. Konjac powder can be used as a thickening agent for sauce, glaze, soup, and stew. Nowadays, konjac flour mixed with other gelling agents can be used as fat analog or fat substitute. Konjac gel showed a good thermal water binding and have

better elasticity during heating and exhibited rheological thermal properties above 40 °C mimic pork backfat (Jiménez-Colmenero *et al*., 2012a). However, in this study also observed that konjac gel presented lower physical ability compare to pork backfat such as hardness, chewiness, penetration force, gel strength, extraction force and extrusion.

2.3.2 Advance application of Konjac glucomannan

Originally the purified flour from konjac tuber is used in food production and later also used as a food additive as a gelling and thickening agent that is permitted as a food ingredient in Europe with E-425 number (FSA, 2016). The special rheological and gelling property of KGM is also used in emulsifier and stabilizer products in food, beverages, cosmetic and pharmaceutical industries. Since 1996, KGM also approved a binder in meat and poultry products by the USDA. KGM and its derivatives have been broadly exploited in pharmaceutical science as drug delivery with controlled release ability (Wu and Shen, 2001). In biotechnology area, KGM is applied as a multipurpose edible film which stimulates the idea of the probiotic edible film. As knowledge is growing, KGM can be used also in advance technology such as encapsulation, microencapsulation, and nanoencapsulation of bioactive materials. Nussinovitch (2004) invented hydrocolloid membrane with KGM in its formulation. The thermal-stable droplet was able to encapsulate a liquid contained at least one enzyme, a cell, a biological agent, a pharmaceutical component, an immunological agent or the mixture of those compounds. This hydrocolloid membrane was able to hold the liquid containing essential compounds without bursting at temperature -20 to 90 °C. The concept of encapsulation was further observed into micro scale. The recent investigation of KGM is to use it as a coating material in microencapsulation via spray drying. Microencapsulation is a technique where small particles or droplets are surrounded by a wall material or embedded in the homogeneous or heterogeneous matrix and provide small capsules with many useful characteristics. In food technology, microencapsulation can be used in liquid, solid and gas droplets that entrapped into thin films of a food grade microencapsulation agent (Gharsallaoui *et al.*, 2007). Konjac glucomannan is a

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potential agent to be used as wall material. The explanation will be included in chapter 2.3.3.

2.3.3 Konjac glucomannan as wall material

Wall material of microencapsulation can be selected from a natural and synthetic material depend on the core material and desired characteristics in the final product. In general, carbohydrates and gums are used as coating material for microencapsulation by spray drying since they show low viscosity at high solid content and good solubility (Silva *et al.*, 2014). KGM as natural polysaccharides can be used as wall material substances. Several studies observe more deeply on the application of KGM hydrolysate (KGMH) as a coating agent in microencapsulation via spray drying (**Table 2.2**). The ability of KGM as wall material is restricted by its physical properties particularly high in apparent viscosity at low solid content. To be applicable in spray drying microencapsulation, KGM should undergo hydrolysis reaction to get lower viscosity and appropriate for nozzle spray dryer. Hydrolysis reaction can be done either by acid or enzymatic reactions. Acid hydrolysis of polysaccharides is the simplest method to convert KGM to be KGM hydrolysate. However, the reaction is time-consuming and the high acid residue at the end of reaction hinder its utilization in the food industry. For instance, Huang *et al*. (2010) used sulfuric acid 3.2 M at 40 °C for 7 days and observe the particle size (nanocrystal) after hydrolysed KGM was dried by freeze dryer. Moreover, Wang *et al.* (2015) also employed 2.8 M sulfuric acid at 40 °C for 7 days and investigate the size of KGM microcrystal. While Tanaka *et al*. (2013) used 1 M of sulfuric acid in a boiling water bath for 5 hours with gentle stirring every one hour to get fully hydrolysed KGM solutions. On the other hands, the specific work of enzyme makes enzymatic hydrolysis can be an excellent way to get the targeted and controlled hydrolysis of polysaccharides. When compared to acid hydrolysis, oligosaccharides from enzymatical hydrolysis of KGM can be used as the KGM hydrolysate stimulates the growth of lactobacilli and bifidobacteria (Al-Ghazzewi *et al*., 2007).

Moreover, the application in the food industry is more reliable to use food grade enzyme to hydrolyse the KGM than using concentrated acid. KGM structure

consists of four different ß-D-(1→4) linkages which are mannose to mannose, mannose to glucose, glucose to mannose and glucose to glucose. Therefore, the macromolecule backbone of KGM is contained in D-mannopyranosyl residue and D-glucopyranosyl residue. The linkages in KGM backbone can be hydrolysed by mannanase, endoglucanase or cellulose, and pectinase. However, mannanase is the most common enzyme to depolymerize KGM due to its high productivity compared to other enzymes. The predominant monomer of KGM is mannose and glucose at ratio 1.6:1, while mannanase breaks the main chain of ß-D-1,4-mannopyranosyl linkages. In consequence, mannanase has high productivity than another enzyme at the same time of hydrolysis. On the other hand, endoglucanases belong to the cellulose-degrading enzymes and cutting the linkage of ß-D-1,4-linkages in cellulose chain. The key point of cellulose enzyme is the linkages between glucose to mannose and glucose to glucose. Moreover, pectinase is the big group of enzymes that break the chain of glycosidic linkage, especially at 1,4-α-galactosiduronic. In addition, a report from Cescutti *et al*. (2002) stated that the presence of few short side-chines may contain galactose residues which are the target of pectinase enzyme.

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Table 2.2 Utilization of KGM and other wall materials in spray drying microencapsulation

Core material	Wall materials	Spray drying condition	Encapsulation efficiency (%)	Authors
Sweet orange oil flavor	KGM, gum Arabic, and starch sodium octenyl succinate	Inlet temperature: 160 °C	60-81	Yang *et al.* (2009)
kaffir lime oil	KGM and KGM + gum Arabic	Inlet temperature: 160 - 200 °C	56.86	Adamiec *et al.* (2012)
Andrographolide	KGM, KGM+ β-cyclodexrtin, KGM+ α-cyclodexrtin	Inlet temperature: 170 °C, oulet temperature: 85 °C	10.82-46.63	Wattanaprasert *et al.* (2017)

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2.3.4 Konjac as fiber sources

Functional fibers are non-digestible carbohydrates that have a beneficial effect on human body. According to chemical characteristics, dietary fiber was classified as soluble and insoluble fibers. Insoluble fibers mainly are cell wall of plant components such as cellulose, lignin, and hemicellulose, while soluble fiber consists non-cellulosic polysaccharides including pectin, gums, and mucilage (Kalyani Nair *et al.*, 2010). The positive effects of dietary fiber can be divided into three mechanisms including bulking, viscosity and fermentation. Generally

insoluble fiber exhibits bulking effect which can increase stool mass, alleviating constipation and maintain bowel movement. Cellulose and lignin with their particle formation and water holding ability were effectively increased fecal bulk and help in intestinal transit time, prevent and relieve constipation (Ho *et al*., 2012). Meanwhile, soluble dietary fiber which fermentable in human intestine may increase stool bulk by promoting the growth of gut microflora and produce gas and short chain fatty acids. Previous studies have a belief that soluble fibers lower cholesterol while insoluble fibers are contributed to stool texture. However, this conclusion is inconsistent since not all soluble dietary fiber has significant effect to lowering cholesterol such as wheat bran and resistant starch which classified as soluble dietary fiber but these fibers did not affect serum lipids (Jenkins *et al*., 1998, Slavin, 2005). Another terminology for fiber classification is the viscosity of fiber. A meta-analysis analyzed the effect of pectin, oat bran, guar gum and psyllium (2 to 10 gram/day) on blood lipid which showed significant decreases in total and low-density lipoprotein cholesterol concentration (Brown *et al.*, 1999).

Glucomannan is classified as a soluble, fermentable and highly viscous dietary fiber (Keithley and Swanson, 2005, Dikeman and Fahey, 2006). The review between glucomannan and its role in weight reduction was reviewed by Keithley and Swanson (2005) and it is concluded that at the doses of a 2-4 gram per day, glucomannan was acceptable and presented in significant weight loss in overweight and obese person. Glucomannan molecule in konjac powder was able to promote satiety by delaying gastric emptying and slowed small-bowel transit time. Glucomannan as soluble fiber also supports fecal energy loss by reducing fat and protein absorption (Baer *et al*., 1997) as well as inhibits carbohydrate absorption and improve glycemic parameters (Jenkins *et al*., 1994). It is noted that konjac glucomannan is also acted as prebiotic which promote human intestinal *Clostridium* and produce glucomanno-oligosaccharides following by short-chain fatty acid fermentation by gut microbiota (Nakajima *et al.*, 2002). More recently Chen *et al.* (2008) conducted a clinical study with seven constipated Chinese women by feeding them with 4.5 g/day konjac glucomannan and resulted in a significant rise in weekly defecation frequently and relieved the bowel movement. Moreover, they also found

that *Lactobacilli* and short chain fatty acids were increase resulted in lowering fecal pH. Hydrolyzed konjac glucomannan which obtained from hydrolysis reaction of konjac powder was presented similar benefits with prebiotics. Al-Ghazzewi *et al.* (2007) found that hydrolyzed konjac glucomannan able to promote the growth of probiotic strains of *Lactobacilli* and *Bifidobacteria* in a single culture. In line with this study, Connolly *et al*. (2010) noted that the ability of hydrolyzed konjac glucomannan was comparable with inulin. What is more, konjac hydrolysate also produce selective stimulating of gut microbiota and a favorable short-chain fatty acid. The evidence stated that KGM and hydrolyzed KGM provide a lot of functional properties to the human body. Regardless of direct function to body well-being, hydrolyzed KGM could also benefit in food drying as drying aid and bioactive encapsulant. However, the application of KGM in food drying scarcely to be found. Therefore, the utilization of KGM in spray drying instant coffee could be contributed to KGM application knowledge.

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CHAPTER 3
MATERIALS AND METHODS

3.1 Materials

3.1.1 Raw materials

Raw materials consist of konjac glucomannan powder (Kunming, China), medium/dark roast Arabica roasted beans (Coffee Properties Plus Co., Ltd, Bangkok Thailand), Maltodextrin DE 10-15, food grade (Chemipan Corporation Co., Ltd., Bangkok, Thailand) and Mannanase 50,000,000 IU per gram enzyme, food grade (Bosar Biotechnology, China)

3.1.2 Chemicals and instruments

Table 3.1 Chemical for sugar analysis

Name of chemicals	Company	Origin
Dinitrosalycyclic acid	Sigma-Aldrich	Switzerland
Phenol crystalline	Pancreac AppliChem	Spain
D-Mannose	Fluka/Sigma-Aldrich	United Kingdom
D-Glucose	Ajax Finechem Pty Ltd	Australia
Sulphuric acid 98%	QReC	New Zealand

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Table 3.2 Chemicals for antioxidant analysis

Name of chemicals	Company	Origin
2,2-diphenyl-1-picrylhydrazyl	Sigma-Aldrich	Germany
$FeCL_3.6H_2O$ crystalline	Pancreac AppliChem,	Spain
2,4,6-tr(2-pyridyl)-S-triazine	Sigma-Aldrich	Switzerland
Methanol	RCI Labscan Limited,	Thailand
Sodium Acetate	Lobalchemie	India
6-Hydroxy-2,5,7,8-tetramethylchromane-2-carboxylic acid	Sigma-Aldrich	China
Chlorogenic acid hydrate	TCI	Japan

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Table 3.3 Instruments and apparatus

Instrument	Model	Origin
Mini Spray Dryer	BÜCHI B-290	Switzerland
Spectrophotometry UV-VIS	Thermo scientific, Spectronic® 20 Genesys™	United States
Rotary vacuum evaporator	BÜCHI R-114	Switzerland
Oil-bath	BÜCHI B-485	Switzerland
Hot air oven	Binder ED 400	Germany
Rotational viscometer	Fungilab Premium R	Spain
Moka Pot 22.5 cm X 10 cm	Yami® YM-6007	China
Moisture analyzer	Mettler-Toledo HB43-S	Switzerland
Water activity meter	AquaLab Series3 TE	United States

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3.2 Methods

3.2.1 KGM hydrolysis

Two types of konjac powder, *A. muelleri* and *A. bulbifer*, were used at 20% (w/w) concentration for viscosity reduction study along with Mannanase enzyme at 22,000, 30,000, 38,000 and 46,000 units per gram KGM. Powder type and enzyme concentration selection were based on the reduction viscosity to the point around 100 m.Pa.s. After enzyme concentration was selected, KGM at concentration of 5, 15 and 20% (w/w) were used for the study of wall material efficiency. All KGM hydrolysis was conducted under a controlled condition at 40±1 °C and 200 rpm of an overhead stirrer for 1 hour.

3.2.2 KGMH characterization

Viscosity examination of KGMH was conducted by using rotational viscometer. A 100 ml of KGMH solutions were used for analysis. During analysis, the spindle was used according to the sample and torque percentage (20 – 70%). For instance, a sample with the lowest enzyme concentration has a very high viscosity, therefore, spindle R7 was used. While sample with low viscosity used spindle R3. Meanwhile, the analysis of total sugar and reducing sugar were conducted by Phenol-sulfuric and DNS methods, respectively (Bui *et al.*, 2016, Chen *et al*., 2013) (Appendix A1). Moreover, density and porosity of konjac powder before hydrolysis were also analyzed to confirm the viscosity results. Konjac powder was gently loaded into a 50 cm^3 cylinder until the flour reached a volume of 10 cm^3. The ratio of weight to volume was defined as a bulk particle of powder (Passos, 1999). Meanwhile, the particle density was measured by the standard liquid pycnometric (AOAC, 2006). The volume of the particle is observed from the liquid volume increase upon adding the particle into a liquid (toluene with density 0.8625 g/cm^3). The particle density was calculated by Eq. (1), where ρ_1 is the toluene density, m_0 is empty pycnometer weight, m_1 is pycnometer filled with toluene, m_s is weight of pycnometer with particle, and m_{sl} is weight of pycnometer including sample and toluene. The porosity (ε) of konjac powder was calculated by Eq. (2).

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(1) $$\rho_P = \frac{\rho_1(m_s - m_0)}{(m_1 - m_0) - (m_{sl} - m_s)}$$

(2) $$\varepsilon = \frac{1 - \rho_{bulk}}{\rho_{particle}}$$

3.2.3 Coffee extraction

The extraction process was conducted by using moka pot with 24 gram of ground coffee and 115 ml hot water. The extract then undergoing concentration process by vacuum evaporator to get 20 °Brix of concentrated coffee extract. The temperature of evaporator water bath was maintained at 40±5 °C. The concentration process was conducted around 20 minutes per batch (200 ml).

3.2.4 Spray drying microencapsulation process

The KGMH solution then was used for drying by using mini spray dryer (**Figure 3.1**). **Table 3.4** shows the combination of wall material where each formula will be spray dried at inlet temperature of 160, 170 and 180 °C. The outlet temperature was fixed at 85-90 °C by adjusting feed rate (20 – 40%).

Table 3.4 Actual ratio of soluble solids of coffee mixture before spray drying

Formulation		Weight (g)				Wall and core solid ratio (w/w)
		Raw material	Solid content	Mixture	After evaporation	
Formula 1	KGMH 20% (w/w)	200.0	40.0	300.0	200.0	2.2 : 1
	Coffee 20° brix	100.0	18.5			
	MD	0.0	0.0			
Formula 2	KGMH 15% (w/w)	200.0	30.0	310.0	200.0	2.2 : 1
	Coffee 20° brix	100.0	18.5			
	MD	10.0	10.0			
Formula 3	KGMH 5% (w/w)	200.0	10.0	330.0	200.0	2.2 : 1
	Coffee 20° brix	100.0	18.5			
	MD	30.0	30.0			
Formula 4	KGMH 0%	0.0	0.0	300.0	200.0	2.2 : 1
	Coffee 20° brix	100.0	18.5			
	MD 20% (w/w)	200.0	40.0			

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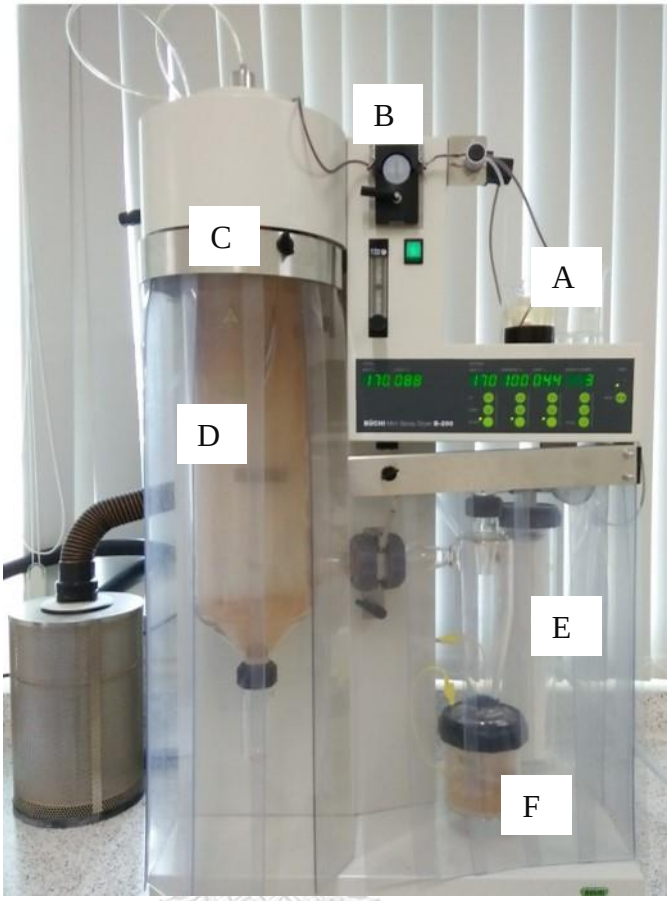

Figure 3.1 Spray Drying used in experiment; feed chamber (A), peristaltic pump (B), atomizer (C), drying chamber (D), cyclone (E), and powder collection chamber (F)

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3.2.5 Chemicals analysis

Antioxidant properties are observed including the total phenolic content (TPC), DDPH scavenging ability, ferric reducing antioxidant power (FRAP) and chlorogenic acid examination. Total phenolic contents are measured by Folin-Ciocalteu method and calculated using chlorogenic acid equivalent, meanwhile, DPPH scavenging and FRAP were using Trolox as the standard (Margarita et al., 2012, Vignoli et al., 2011). Moreover, amount of chlorogenic acid was observed by using HPLC (Varian Prostar, USA) and the spectra were detected by Prostar 335 Photodiode Array Detector at 278 nm (Appendix A2).

The retention percentage of antioxidant properties was calculated as explained by Ballesteros et al. (2017) and shown in equation 3:

(3) $$\text{Retention percentage}(\%) = \frac{A_2}{A_1} \times 100\%$$

Where:

A_2 is amount of concerned compounds in instant coffee powder (dry basis)

A_1 amount of concerned compounds in coffee extract (dry basis) (Appendix B1-B6)

Dietary fiber analysis consists soluble dietary fiber and insoluble dietary fiber measurements by using the gravimetric technique (AOAC, 2006), 991.43 (Appendix A3). Meanwhile, the proximate analysis was carried out following AOAC (2012); protein with N x 6.25 (991.20), total fat (992.06), and ash content (920.93).

3.2.6 Physical analysis

Water solubility index and water absorption index were analyzed according to the method explained by (Ahmed *et al.*, 2010), moisture content was analyzed using moisture analyzer Mettler-Toledo HB43-S, water activity was conducted using water activity meter AquaLab Series3 TE.

Production yield is calculated using equation 4:

(4) $$\text{Yield}(\%) = \frac{\text{Weight of microcapsules in dry basis}}{\text{Weight of total solid (g)}} \times 100\%$$

Where total solid is including konjac powder (g) + mannanase (g) + soluble solid of coffee extract calculated from refractometer (g).

The measurement of color was conducted using the Minolta chroma meter model C400. The instrument reads color in three-dimensional format consists of the level of brightness between black and white (L*), the balance between red and green

with 100 being red and -100 green, and the relative yellow and blue (b*). The sample was measured in form of instant coffee powder and instant coffee beverages.

3.2.7 Sensory analysis

Rating hedonic with 15 cm line scale was used to evaluate the liking acceptance of microencapsulated instant coffee at overall liking, color, aroma, taste or flavor, and viscosity. For the taster (panelist) screening, first, the taster should people who can drink black coffee. The screening also was based on the ages (18 – 39 years old) which represent the young ages and early adult that contributed to 50 and 63% of the coffee consumption in the US. Moreover, young and adult are the target for the functional product of coffee. For sample preparation, 3.6 grams of microencapsulated instant coffee was diluted in 100 ml boiling water, while the coffee reference was used at 2 grams at the same volume of coffee beverage. The coffee brews were served in a hot condition to the taster.

The purchase intent with 5 categories (definitely would not buy, probably would not buy, may or may not buy, probably would buy, and definitely would buy), was used to analyze the willingness of the consumer to buy the instant coffee. The purchase intent was asked in three questions, first is the purchase intent for the tasted sample, second is the purchase intent for the given packaging picture with the explanation that the product contains a considerable amount of dietary fiber and antioxidant activity, and purchase intent for the given coffee packaging picture with explanation of fiber and antioxidant and possess the taste with the tasted coffee.

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3.2.8 Statistical analysis

All experiment and analysis were conducted in three replications, except it stated. The statistical analysis was carried out by complete randomized design via One-way ANOVA and Duncan test. Sensory analysis was analyzed by independent *t-test.* All the test is run in IBM SPSS 22 program.

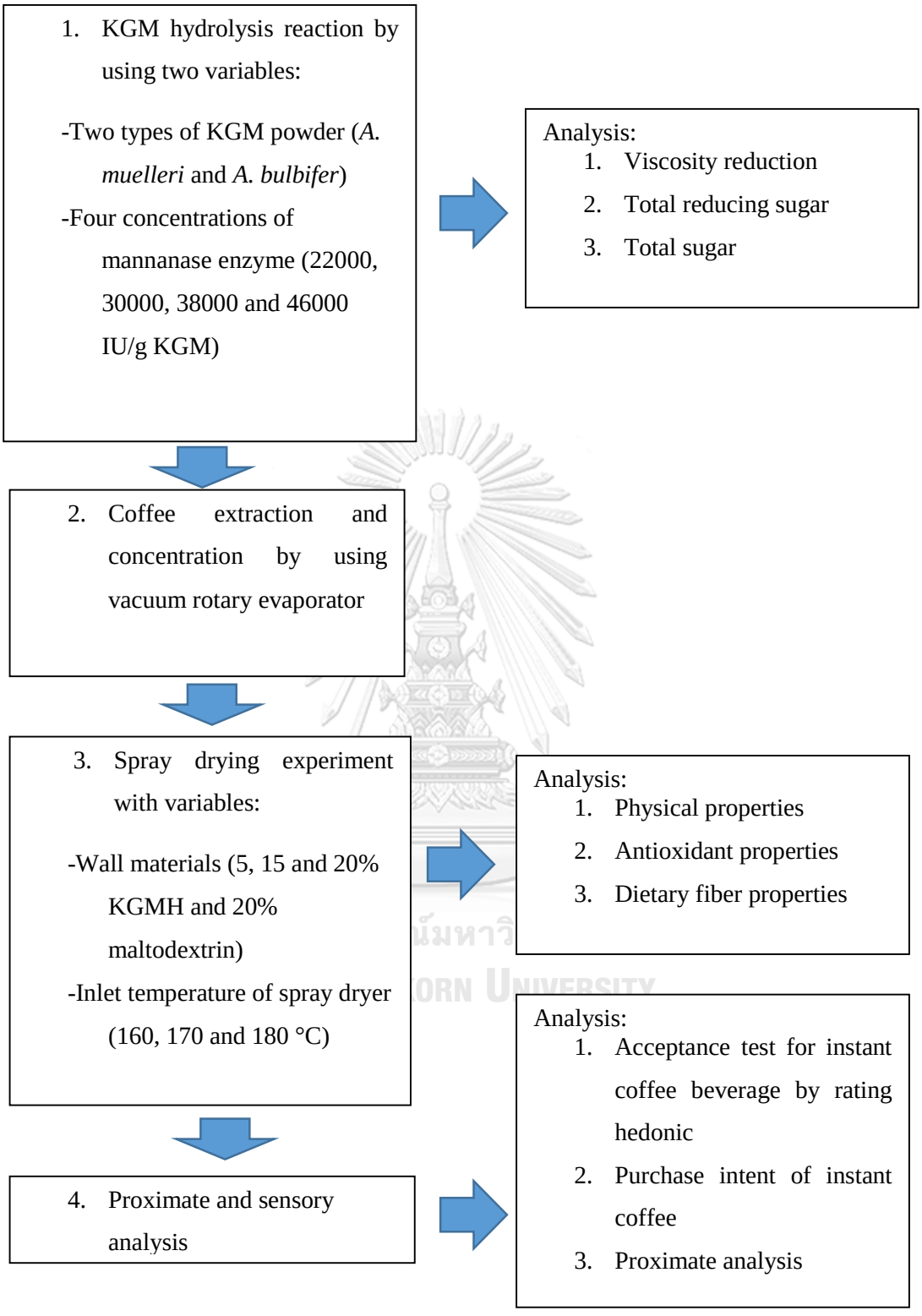

Figure 3.2 Flow chart of the study

CHAPTER 4
RESULTS AND DISCUSSION

4.1 Effect of enzyme concentration and type of powder

4.1.1 Viscosity of KGMH

The aim of hydrolysis reaction was to reduce the viscosity of KGM solution to be around 100 mPa.s as the value limit for spray drying (Iida *et al.*, 2008). Enzymatic hydrolysis was used to breakdown ß-(1→4) linked D-mannose and D-glucose in KGM main chain. Previous studies have shown that mannanase worked effectively to lower KGM viscosity than other carbohydrate-based enzymes such as endoglucanase, hemicell, finizym and pectinase (Li, 2004, Mikkelson *et al.*, 2013, Wattanaprasert *et al.*, 2017). As the enzyme works specifically to the substrates, the current study used two types of KGM powder that produced from different species, *A. muelleri* and *A. bulbifer*, and four different concentration of enzyme which are 22,000, 30,000, 38,000 and 46,000 IU/g KGM powder. The changes of viscosity of 20% (w/w) KGMH solution are shown in **Figure 4.1**.

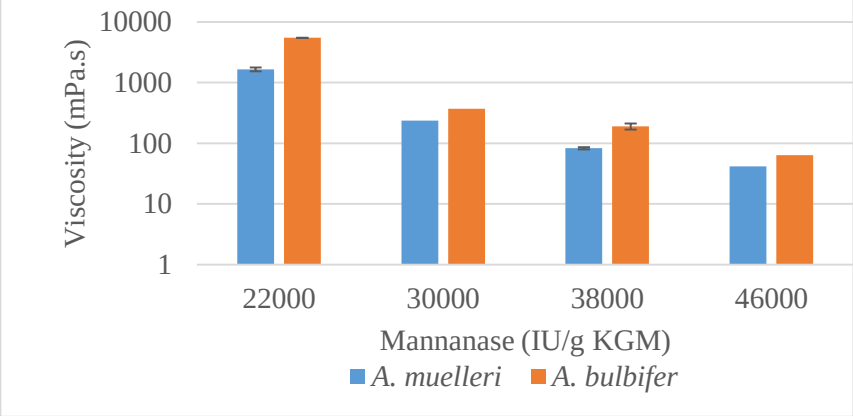

Figure 4.1 Effect of enzyme concentration on viscosity of KGMH from *A. muelleri* and *A. bulbifer* powders

The two types of the powder showed different responses to given concentration of enzyme. At 22,000 IU/g KGM, *A. muelleri* hydrolysate showed 1,658±123 mPa.s while *A. bulbifer* was recorded at 5,481±38 mPa.s. The sharp decline was observed when the concentration was increased to be 30,000 IU/g KGM

and showed 235.5±3.54 and 370.3±21.64 mPa.s for *A. muelleri* and *A. bulbifer*, respectively. Meanwhile, the viscosity decreasing was in slightly changes when the enzyme concentration was increased to 38,000 and 46,000 IU/g KGM which showed 82.5±4.84, 189.5±3.53 and 41.5±4.94, 63.5±0.71 mPa.s, respectively. In general, KGMH solutions from *A. muelleri* powder presented lower viscosity than *A. bulbifer* in each treatment. Impaprasert *et al.* (2014) explained that viscosity of KGM solution was in strong relation to its powder porosity. The high porosity of powder tends to have high ability to absorb water and resulted in higher viscosity. **Table 4.1** shows the comparison of means value and its standard deviation of bulk density, particle density, and bulk porosity of *A. muelleri* and *A. bulbifer* powders. Bulk density is defined by comparing the powder mass and its bulk volume, while particle density is the density when the pores of powder were excluded and measured by the mass of the sample and its solid volume.

Table 4.1 Bulk density, particle density, and bulk porosity of KGM powders

Sample	Bulk density (g/cm^3)	Particle density (g/cm^3)	Bulk porosity
A muelleri	$1.33±0.01^a$	$0.57±0.00^a$	$0.20±0.00^a$
A bulbifer	$1.45±0.00^b$	$0.72±0.00^b$	$0.36±0.06^b$

Values in a column followed by different letters are significantly different ($p<0.05$)

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The results showed that *A. bulbifer* tends to have higher bulk density, particle density, and bulk porosity than *A. muelleri* ($p<0.05$). The porous characteristics of bulk of *A. bulbifer* particles caused this powder to have more channel in bulk form and will absorb more water than *A. muelleri* at the same time of hydration. To sum up, since the aim of this part of the study was to get the optimum condition for KGM hydrolysis prior spray drying with the requirement of viscosity at around 100 mPa.s, thus the concentration of enzyme at 38,000 IU/g of KGMH from *A. muelleri* was chosen to be used for the next experiment. The decision was also supported by the effectiveness of enzyme and to avoid the hydrolysis of KGM polymer become fully monosaccharide that will provide a negative effect on spray drying microencapsulation.

4.1.2 Total sugar, total reducing sugar, and degree of polymerization of KGMH

Depolymerization process which converted glucomannan polymer to its monomers or mixture of monomers and glucomanno-oligosaccharides, was occurred during hydrolysis reaction. Glucomanno-oligosaccharides have been reported to have the ability in bioactive compounds protection during spray drying microencapsulation (Wattanaprasert *et al.*, 2017). Chen *et al*. (2013) reported that total reducing sugar can be used as a signal to represent the volumes of glucomanno-oligosaccharides released after the reaction. **Figure 4.2** shows the result of sugars measurement from the enzymatic hydrolysis of KGM with different enzyme units per gram KGM. Total reducing sugar of all KGMH solutions was slightly increased when the enzyme concentration was increased from 22,000 IU/g KGM to 46,000 and showed the value at 13.5 to 15 mg/ml of *A. muelleri* hydrolysate and 9 to 13 mg/ml of *A. bulbifer* hydrolysate. Meanwhile, total sugar was averagely decreased but inconsistent and the values were hanging around 17 mg/ml for *A. muelleri* and 19 mg/ml for *A. bulbifer* hydrolysates. The ratio of total sugar and total reducing sugar is used for prescribed the average degree of polymerization and the results showed that increasing of enzyme concentration was resulted in slightly declining of degree polymerization which dropped from 2 to 1 in both hydrolysates. However, these results were statistically similar among the treatments and concluded that designed enzyme concentration not affected the total reducing sugar, total sugar and degree of polymerization. This result was in good agreement with Chen *et al.* (2013) which observed the effect of mannanase enzyme in sugar profiles KGMH after went through enzymatic hydrolysis at 40 °C, pH 6 for 60 minutes and concluded that increasing the ratio of enzyme to substrate was resulted in slightly increase of total reducing sugar and total sugar but not in linear correlation. Similar studies by Mikkelson *et al.* (2013) and Wattanaprasert *et al.* (2017) which employed mannanase with enzyme to substrate ratios at 0.002 and 0.05% have reported that enzymatic hydrolysis was marked to release reducing sugar and produced glucomanno-oligosaccharides at concentration 17% and 30%, respectively for both studies. Lastly, with the fair amount of reducing sugar and low average degree of polymerization obtained, glucomanno-oligosaccharides were expected to be produced after hydrolysis.

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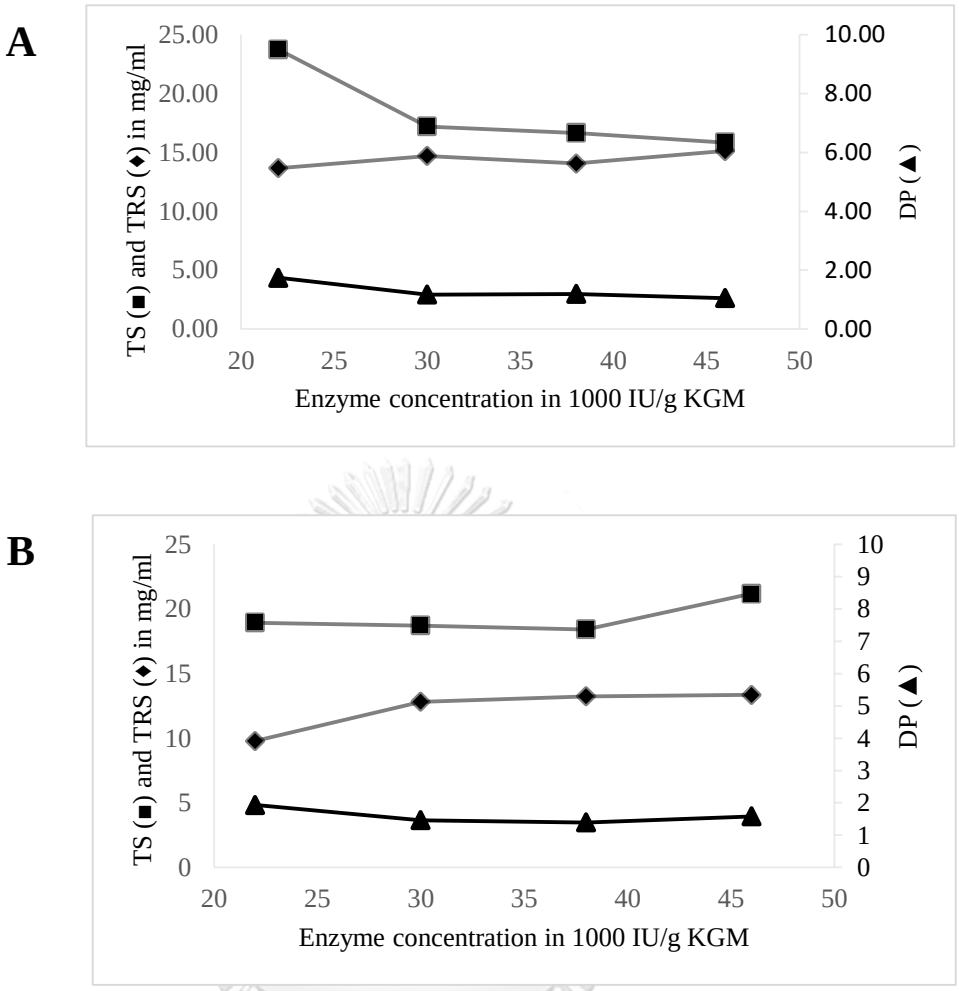

Figure 4.2. Total reducing sugar, total sugar, and degree of polymerization of *A. muelleri* (A) and *A. bulbifer* (B) hydrolysates

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4.2 Effect of wall material and spray drying condition

4.2.1 Antioxidant properties of microencapsulated instant coffee

Instant coffee was analyzed for antioxidant ability by FRAP and DPPH methods, total phenolic content (TPC) by the Folin-Ciocalteau method. All antioxidants values were compared to its coffee extract to define retention percentage. Moreover, the correlation between antioxidant experiment and chlorogenic acid as the predominant polyphenols was observed.

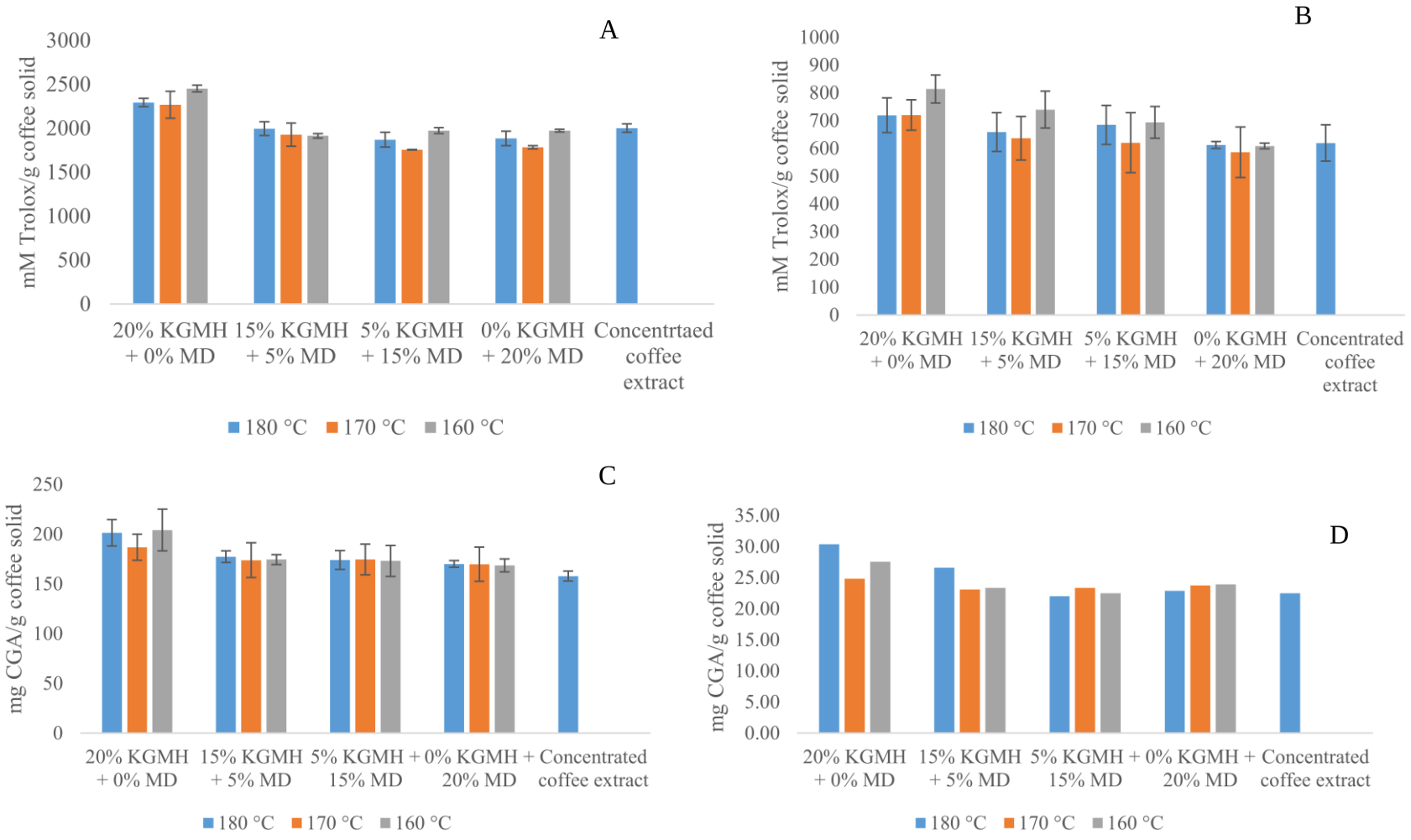

Figure 4.3 Antioxidant activity (FRAP) (A) and DPPH, total phenolic content (C), and chlorogenic acid content (D)

Figure 4.3 presented the number of phenolic compounds and antioxidant activity based on coffee solid in coffee extract and instant coffee powder. The results have shown that spray drying microencapsulation process, especially when using KGMH as wall material, was able to shield phenolic and antioxidant compounds. Moreover, some experiments (especially TPC measurement) presented an increase of phenolic compounds and antioxidant activity in the final product than coffee extract before spray drying and resulted in excessive of retention percentage. There is a chance that KGMH also contributed to antioxidant activity in the final product since it contains reducing sugar that can act as reducing agent and positively recorded as phenolic compounds.

In TPC measurement, instant coffee powder with 20% (w/w) KGMH and spray dried at 180 and 160 °C were presented higher values than other treatments with 204.04±20.98 and 201.33±13.15 mg CGA/g coffee solid (p<0.05), while the coffee extract presented 157.87±4.95 mg CGA/g coffee solid. Therefore, the retention percentage was over 100%. Prior *et al.* (2005) explain that Folin-Ciocalteau method in TPC measurement relies on the reduction-oxidation reaction between molybdotungstate reagent and phenolic compounds. There is controversy over the positive result in this method since not only phenols that might be detected but also reducing agent and metal chelators. The high values in the current study might come from reducing sugar of wall material and coffee extract. It is noted that 20% (w/w) KGMH solution exhibited the reducing sugar content at around 15 mg/ml hydrolysate or 7.5% (w/w). Excessive retention percentage might also come from the new products that formed during spray drying via thermal reaction. For example, in instant coffee of 20% (w/w) MD, increasing inlet temperature from 160, 170 and 180 °C was lead to slightly increase of TPC (168.62±6.42, 169.79±17.13, and 170.06±3.40 mg CGA/g coffee solid). However, the pattern is inconsistent in higher KGMH concentration formulation where 170 °C inlet temperature showed lower value. This result showed that degradation and formation of phenolic compounds are occurred during drying and in some points the degradation was higher than formation of antioxidant compounds. Nevertheless, in average, KGMH alone was presented better TPC retention.

To confirm TPC result, a specific phenolic compound which is chlorogenic acid was observed by HPLC. Chlorogenic acid derived from esterification of *trans*-cinnamic acids and quinic acid. The nature, number of cinnamic substituents and esterification position make chlorogenic acid to have many isomers and derivatives (Farah, 2012). In the current study, concentrated coffee extract had 22.49±1.35 mg/g coffee solid, while instant coffee powder contained 22-30.60 mg/g coffee solid. Among the treated instant coffee powders, 20% (w/w) KGMH instant coffee powder produced at 180 °C presented the highest chlorogenic content with retention percentage 129.53%. In general, increasing KGMH concentration and inlet temperature lead to the higher chlorogenic acid content. Dawidowicz and Typek (2011) concluded that chlorogenic acid undergoes transformations such as isomerization, esterification, and hydrolysis during its water extraction. Moreover, high temperature during spray drying leads to releasing of chlorogenic acid from bound to free molecules. In the roasting process of coffee bean, chlorogenic acid is incorporated into Maillard reaction products. This incorporated molecules will further degrade into lower molecular weight components during spray drying in instant coffee production and might release some chlorogenic acids. Zhang *et al*. (2016) conclude that during thermal processing, the availability of 5-Hydroxymethylfurfural (Maillard reaction product) was highly correlated with the presence of chlorogenic acid. The result was in line with Michalska *et al.* (2018) conclude that during vacuum drying of cranberry extract, increasing temperature leads to the increasing of chlorogenic acid content. They also stated that the amount of chlorogenic acid was highly correlated with HMF content in the final product. The instant coffee mixture contains high amount of Maillard products that bring the black color, therefore there is a chance that during spray drying chlorogenic acid might increase. In conclusion, in TPC and chlorogenic acid measurements, the conversion and releasing of phenolic compounds during spray drying are expected to contribute to the higher value of phenolic content in the final product. However, still, thermal degradation is occurred since in some formulation the chlorogenic acid retention were below 100%.

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On the other hands, DPPH scavenging ability results have shown a similar pattern with TPC experiment where the 20% (w/w) KGMH instant coffee spray dried at 160 °C was presented higher value than the other formulation (813.30±100.89 mM Trolox/g coffee solid), while the extract presented 619.21±130.91 mM Trolox/g coffee solid. During spray drying, high temperature might trigger some reactions such as phenolic degradation and Maillard reaction which can create new compounds that possess higher antioxidant capacity. It is noted that in coffee mixture, antioxidant activity is contributed by phenolic compounds and melanoidins (non-phenolic). Since DPPH method has a wide range of mechanism, the new compounds created during drying could possibly be detected and express antioxidant capacity. Furthermore, in FRAP method, the similar formulation in TPC and DPPH methods was noted to have the highest value which accounted for 2,453.23±78.23 mM Trolox/g coffee solid (p<0.05). Other formulations were accounted for around 1,756.19 to 2,293.53 mM Trolox/g coffee solid, at the same time when its extract have 2,002.56±94.03 mM Trolox/g coffee solid. Similar reason for FRAP result where the high antioxidant activity in final product might come from the new product formed during drying.

Table 4.2 shows the retention percentage of phenolic content and antioxidant activity over its extract. In TPC, all instant coffee powder presented an increase of phenolic compounds. In other experiments, some formulation, especially instant coffee with MD as wall material have presented the retention below 100% which indicates that thermal degradation still occurred and MD could not retain all the antioxidants components. In conclusion, with the same amount total solid of the coffee mixture prior spray drying, 20% (w/w) KGMH as coating material was work better to shield phenolic and antioxidant compounds than MD and its combination with MD. It appears that KGMH also was contributing to antioxidant evaluation. KGMH solution contained a fair number of oligosaccharides that is marked by reducing sugar release during the reaction. These oligosaccharides provided an intermolecular binding between KGMH molecule and bioactive components from the coffee extract. Bioactive compounds were enfolded during mixture stirring prior spray drying, once the solution encounter rapid dehydration at spray drying chamber,

the binding force between oligosaccharides molecules were built and resulted in particle encircle (Wattanaprasert et al., 2017). In fact that many studies also used MD as wall material and present satisfaction results since MD also have matrix forming properties which essential for spray drying microencapsulation, however it is also reported that MD did not provide a good retention especially for volatile compounds during spray drying because of their poor film-forming ability (Chronakis, 1998). To sum up, from antioxidant profile examination results, the best condition for instant coffee production was KGMH alone as wall material and inlet temperature at 160 °C.

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Table 4.2 Retention percentage of total phenolic content, chlorogenic acid, and antioxidant activity by DPPH and FRAP

Inlet temperature (°C)	Wall material formulation	Retention percentage (%)			
		Total phenolic	Chlorogenic acid	DPPH scavenging ability	Antioxidant activity (FRAP method)
160	20% (w/w) KGMH	129.25	117.56	131.35	122.50
	15% (w/w) KGMH+5% (w/w) MD	110.51	99.51	119.41	95.64
	5% (w/w) KGMH+15% (w/w) MD	109.62	95.88	111.96	98.60
	20% (w/w) MD	106.81	101.94	98.25	98.52
170	20% (w/w) KGMH	118.33	115.29	116.26	113.27
	15% (w/w) KGMH+5% (w/w) MD	110.09	107.30	102.70	96.29
	5% (w/w) KGMH+15% (w/w) MD	110.60	108.43	100.12	87.70
	20% (w/w) MD	107.55	110.28	94.54	89.09
180	20% (w/w) KGMH	127.53	129.53	116.11	114.53
	15% (w/w) KGMH+5% (w/w) MD	112.28	113.40	106.33	99.67
	5% (w/w) KGMH+15% (w/w) MD	110.26	93.85	110.51	93.40
	20% (w/w) MD	107.72	97.51	98.78	94.14

Table 4.3 Correlation between chlorogenic acid content and TPC, DPPH, and FRAP methods

Correlation	r (Pearson correlation)
Chlorogenic acid vs TPC	0.848
Chlorogenic acid vs FRAP	0.754
Chlorogenic acid vs DPPH	0.473
FRAP vs TPC	0.908
DPPH vs TPC	0.783

Correlation is significant at the 0.01 level (2-tailed)

The correlation between each experiment was examined using Pearson correlation. The results in **Table 4.3** shows that chlorogenic acid was involved more in TPC and FRAP mechanisms which have 0.848 and 0.754 correlation. Contrarily, the correlation between chlorogenic acid and DPPH method was poor which only 0.473. A high correlation was found between FRAP and TPC (0.908) but lower correlations were showed between DPPH and TPC (0.783). This result showing that in FRAP experiment, the most components was in a group of phenolic compounds including chlorogenic acid. Meanwhile, the component that contributed most to DPPH scavenging might antioxidant compounds of phenolic and non-phenolic groups. The previous study has shown the similar result which presented that chlorogenic acid and DPPH method have correlation at 0.430. They found a strong correlation between DPPH methodology and melanoidins which showed 0.860 (Margarita et al., 2012). Therefore, in DPPH method, the antioxidant component that contributed might not only from phenolic compounds but also from non-phenolic such as melanoidins. Moreover, FRAP and TPC (Folin Ciocalteu) have a similar mechanism which is mostly by single electron transfer (SET). Prior *et al.* (2005) stated that SET mechanism could not detect the antioxidant compounds from thiols and nitrogenous compounds, hence, melanoidins that are nitrogenous compounds would not be detected by FRAP and TPC methods. Regardless, DPPH which involved single electron transfer and hydrogen atom transfer mechanisms might also implicate melanoidins in the antioxidant mechanism.

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4.3 Dietary fiber content of microencapsulated instant coffee

According to Codex Alimentarius Commission (2009) dietary fiber is a group of carbohydrate polymer with at least 10 monomeric units which are not hydrolysed in the small intestine of human. Dietary fiber can be obtained naturally and modification by physical, chemical or enzymatic reactions. In the European Union (EU), oligosaccharides with 3-9 monomeric units are defined as dietary fiber as well. An investigation by Nakajima *et al.* (2002) concluded that KGM was fermented in the large intestine which marked by the production of endo-1,4-β-mannanase and other degrading enzyme and short-chain fatty acid. In the current study, KGM was undergoing hydrolysis reaction and expected to produce some oligosaccharides which have dietary fiber properties. According to its solubility in water, dietary fiber was divided by soluble dietary fiber (SDF) and insoluble dietary fiber (IDF). **Table 4.4** presented dietary fiber profile from microencapsulated instant coffee. It is obviously observed that increasing KGMH concentration was contributed to higher dietary fiber both soluble and insoluble (p<0.0.5). In SDF, increasing inlet temperature leads to higher SDF content where the highest content (22.15%±3.04) was from 20% (w/w) KGMH spray dried at 180 °C. Combination of wall material of 15% (w/w) KGMH and 5% (w/w) MD as well as 5% (w/w) KGMH and 15% (w/w) MD showed the value around 16 and 10% in all inlet temperature. Meanwhile, instant coffee from MD alone only exhibited around 2% of SDF. High inlet temperature contributed to the better drying performance especially in physical properties such as production yield. Hence, it produces SDF more. IDF content was exhibited similar response where increasing KGMH as wall material was contributed to IDF content. The inlet temperature did not significantly influence IDF content. The highest value was presented by instant coffee 20% (w/w) KGMH spray dried at 170 °C. Besides, in all inlet temperature, combination wall material of 15% (w/w) KGMH and 5% (w/w) MD as well as 5% (w/w) KGMH and 15% (w/w) MD exhibited IDF content at around 6 and 3%, respectively. Meanwhile MD instant coffee presented around 2% of IDF.

On the whole, increasing KGMH concentration was related to the rise of both SDF and IDF. Al-Ghazzewi et al. (2007) concluded that KGM in form of hydrolysate was acted as dietary fiber and exhibited prebiotic ability. KGMH as a prebiotic showed comparable ability with inulin (Connolly et al., 2010). What is more, coffee solid might

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also contribute to dietary fiber content, therefore, formulations without KGMH still contained a few amounts of fiber. Most of the fiber in coffee is SDF that consists high-molecular-weight polysaccharides such as galactomannan and type II arabinogalactans (Chu, 2012).

Taking into account the fiber content and compare to product in the market, KGMH instant coffee was presented higher dietary fiber. For instance, one brand of instant coffee which claim as slimming instant coffee contained 1% of natural dietary fiber. Meanwhile, 20% (w/w) KGMH instant coffee presented around 25 – 28% of total dietary fiber. Moreover, The Dietary Reference Intakes recommend a daily intake of dietary fiber is 14 g per 1000 calories or 38 gr/day for men and 25 gr/day for women adults 50 years and younger (Trumbo *et al*., 2002). Despite the serving suggestion is less than 5 gram per day, still, the high content of dietary fiber in KGMH instant coffee can be the alternatives for dietary fiber intake.

Table 4.4 Total soluble dietary fiber and insoluble dietary fiber of instant coffee

Inlet temperature	Wall material	SDF (g/100 g solid sample)	IDF (g/100 g solid sample)
160 °C	20% (w/w) KGMH	18.31 ± 3.04^{de}	6.91 ± 3.70^{bc}
	15% (w/w) KGMH + 5% (w/w) MD	16.84 ± 3.87^{de}	6.79 ± 2.07^{bc}
	5% (w/w) KGMH + 15% (w/w) MD	10.46 ± 2.69^{abc}	2.82 ± 0.85^{ab}
	20% (w/w) MD	4.41 ± 3.53^{ab}	2.13 ± 0.76^{a}
170 °C	20% (w/w) KGMH	20.92 ± 2.49^{e}	7.57 ± 1.48^{c}
	15% (w/w) KGMH + 5% (w/w) MD	16.30 ± 3.87^{de}	3.86 ± 1.48^{abc}
	5% (w/w) KGMH + 15% (w/w) MD	10.61 ± 3.96^{bcd}	2.85 ± 0.61^{ab}
	20% (w/w) MD	2.57 ± 0.63^{a}	1.98 ± 1.12^{a}
180 °C	20% (w/w) KGMH	22.15 ± 3.04^{e}	6.71 ± 1.92^{bc}
	15% (w/w) KGMH + 5% (w/w) MD	15.66 ± 3.87^{cde}	6.07 ± 2.04^{abc}
	5% (w/w) KGMH + 15% (w/w) MD	8.76 ± 2.69^{bc}	3.86 ± 0.44^{abc}
	20% (w/w) MD	1.61 ± 3.53^{ab}	2.38 ± 1.10^{a}

Values in a column followed by different letters are significantly different ($p<0.05$)

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4.4 Physical properties

The effects of spray drying conditions and wall material solutions on physical properties of microencapsulated instant coffee were observed including yield production, moisture content, water activity, water solubility index (WSI), water absorption index (WAI) and surface morphology. The results in **Table 4.5** pointed out that increasing KGMH concentration while decreasing MD concentration was resulting to produce a lower yield. The increasing of inlet temperature was contributed to better production yield, but it was inconsistent. The highest yield was produced by wall material of 20% (w/w) MD at 160 and 180 °C of inlet temperature (p<0.05) which accounted for 65.27%±4.67 and 64.00%±8.03. In all inlet temperature, 20% (w/w) KGMH alone was exhibited comparable results with the mixture of KGMH and MD as wall materials. This result was in line with Tolun *et al*. (2016) which have a similar wall material design with current study and concluded that MD (DE 17-20) alone produced the highest yield at 52.77%, while when MD was mixed with gum Arabic the results had a contrary influence on the yield. Instant coffee with KGMH was had more loses because of KGMH properties. During spray drying, KGMH solution is stickier than MD solution, therefore, the amount of adhered sample in drying chamber after drying was found higher in KGMH instant coffee sample.

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Table 4.5 Product yield, moisture content, and water activity of instant coffee

Inlet temperature (°C)	Wall material	Product yield (%)	Moisture content (% w.b.)^{ns}	Water activity^{ns}
160	20% (w/w) KGMH	53.66±4.81^{a}	3.73±0.52	0.24±0.02
	15% (w/w) KGMH + 5% (w/w) MD	52.55±1.88^{a}	3.42±0.34	0.22±0.05
	5% (w/w) KGMH + 15% (w/w) MD	62.86±2.17^{bcd}	3.75±0.09	0.23±0.00
	20% (w/w) MD	65.27±4.67^{d}	3.64±0.53	0.23±0.02
170	20% (w/w) KGMH	54.30±0.15^{ab}	3.52±0.36	0.21±0.01
	15% (w/w) KGMH + 5% (w/w) MD	53.07±1.77^{a}	3.66±0.22	0.23±0.01
	5% (w/w) KGMH + 15% (w/w) MD	58.31±3.10^{abcd}	3.72±0.04	0.23±0.02
	20% (w/w) MD	62.26±6.31^{cd}	3.58±0.28	0.22±0.03
180	20% (w/w) KGMH	55.27±2.37^{abc}	3.37±0.29	0.25±0.03
	15% (w/w) KGMH + 5% (w/w) MD	53.24±2.91^{a}	3.68±0.30	0.25±0.03
	5% (w/w) KGMH + 15% (w/w) MD	61.39±2.65^{bcd}	3.40±0.23	0.24±0.01
	20% (w/w) MD	64.00±1.19^{d}	3.54±0.10	0.24±0.01

Values in a column followed by different letters are significantly different (p<0.05)

Ns: non-significant

When all formulation was evaluated, the powder has moisture content around 3.3 to 3.7% in wet basis (**Table 4.5**). Either inlet temperature or coating material formulation was not affected instant coffee moisture content (p>0.05). In spray drying technique, the water content and total solid of feed mixture had a great effect on the final moisture content of powder (Oberoi and Sogi, 2015). The fact that all formulation before spray drying process was in the same total soluble solid concentration and the

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range of inlet temperature was not extremely far may result in the averagely same value of powder moisture content. The same explanation goes to water activity where all formulation and the given processing treatment were having similar water activity values. All formulation was having a very low moisture content which beneficial for the storage of powder. Low moisture content and water activity promote higher T_g and make the powder more stable during storage. However, it should be noted that KGMH solution contained a fair amount of reducing sugar (7.5% w/w) that is contributed to powder hygroscopicity. In addition, MD is a material with low hygroscopy (Rodríguez-Hernández *et al.*, 2005) and Tonon *et al*. (2008) reported that MD concentration during spray drying was contributed to lower hygroscopy of powder. In consequence, even though the moisture content of all formulation was in similar value, but appropriate storage condition should be considered to lower the incidence of water absorption by the powder, especially, in KGMH instant coffee powder.

Table 4.6 Water solubility index and water absorption index of instant coffee

Inlet temperature °C	Wall material	WSI (%)	WAI (%)
160	20% (w/w) KGMH	88.95±5.16^{a}	8.94±1.51^{c}
	15% (w/w) KGMH	91.89±4.10^{abc}	3.07±0.76^{a}
	5% (w/w) KGMH	96.54±0.88^{cd}	3.13±1.36^{a}
	20% (w/w) MD	94.08±3.57^{cd}	3.20±0.72^{a}
170	20% (w/w) KGMH	89.03±3.68^{a}	9.01±1.54^{c}
	15% (w/w) KGMH	91.90±3.06^{abc}	4.59±1.49^{ab}
	5% (w/w) KGMH	96.00±0.79^{bcd}	3.83±1.60^{ab}
	20% (w/w) MD	96.80±2.34^{cd}	3.27±1.33^{a}
180	20% (w/w) KGMH	89.38±3.61^{a}	8.80±1.89^{c}
	15% (w/w) KGMH	90.35±3.54^{abc}	5.91±1.19^{b}
	5% (w/w) KGMH	92.47±5.78^{abcd}	5.41±0.64^{ab}
	20% (w/w) MD	98.98±0.87^{d}	3.58±0.73^{ab}

Values in a column followed by different letters are significantly different (p<0.05)

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WSI is an important property to repeal powder behavior in aqueous and its reconstitution ability. **Table 4.6** presented the WSI of microencapsulated instant coffee powder from different spray drying condition and formulation. In general, the value of WSI was in contrary to the amount of KGMH used but correlated with increasing of inlet temperature. It is observed that from wall material with KGMH alone and MD alone were presented the lowest and the highest WSI. The most solubilize powder was come from instant coffee produced from 20% (w/w) MD at 180 °C (98.98±0.87%) while the lowest WSI was 20% (w/w) KGMH powder at inlet temperature 170 and 160 °C (89.03±3.68 and 88.95±5.16%) (p<0.05). Similar finding from Jafari *et al.* (2017) who found that juice powder with 25-45% (w/w) MD as drying aids have WSI above 90%, while Adamiec *et al.* (2012) noted that by using combination KGMH and gum Arabic at wall material concentration at 9% (w/w) presented WSI at 67-69%. Even though KGMH instant coffee exhibited the lowest WSI, the value of WSI was still above 88% which mean that all instant coffee powder still has a good solubility.

In the opposite value with WSI, increasing KGMH concentration was promoted the WAI value. Moreover, the rising of inlet temperature also was associated with increasing WAI. The WAI percentage of microencapsulated instant powder has exhibited the highest value in 20% (w/w) KGMH of wall material in all inlet temperature, whereas the lowest was found at 20% (w/w) MD at 170 °C and all formulation contained KGMH that spray dried at 160 °C. The noticeable variation of WAI between KGMH and MD might be due to the differences strength of bonding between a water molecule and hydroxyl groups in both materials. Xiao *et al*. (2015) found that KGM flour able to absorb 38.8 g water per dry mass of KGM, therefore, KGMH has been known to have a good water holding capacity.

The surface morphology of instant coffee powder was evaluated (**Figure 4.4** - **Figure 4.6**). The micrographs are obtained from SEM with 700 and 2000 times magnification exhibited that instant coffee powder from different drying condition has shown similar morphology which has a round-ball shape with an irregular surface. The microstructure of all instant coffee indicated that the drying process was conducted successfully since the particles were not exhibited an extreme structural collapse or breaking. The instant coffee powder consists of different sizes of a spherical particle with diameter around 10-40 μm and surrounded by the smaller spherical particle.

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KGMH instant coffee has more size distribution and decreasing inlet temperature leads to higher size particle distribution. In contrary, MD instant coffee powder presented relatively similar size of particle. In general, MD powder has smaller size than KGMH powder. This result was in agreement with Ballesteros *et al.* (2017) which observe phenolic microcapsule with MD and gum Arabic as wall material. They found that spray dried microcapsule from MD was presented smaller size than gum Arabic microcapsule. Moreover, both microcapsules were had similar size which around 30 µm.

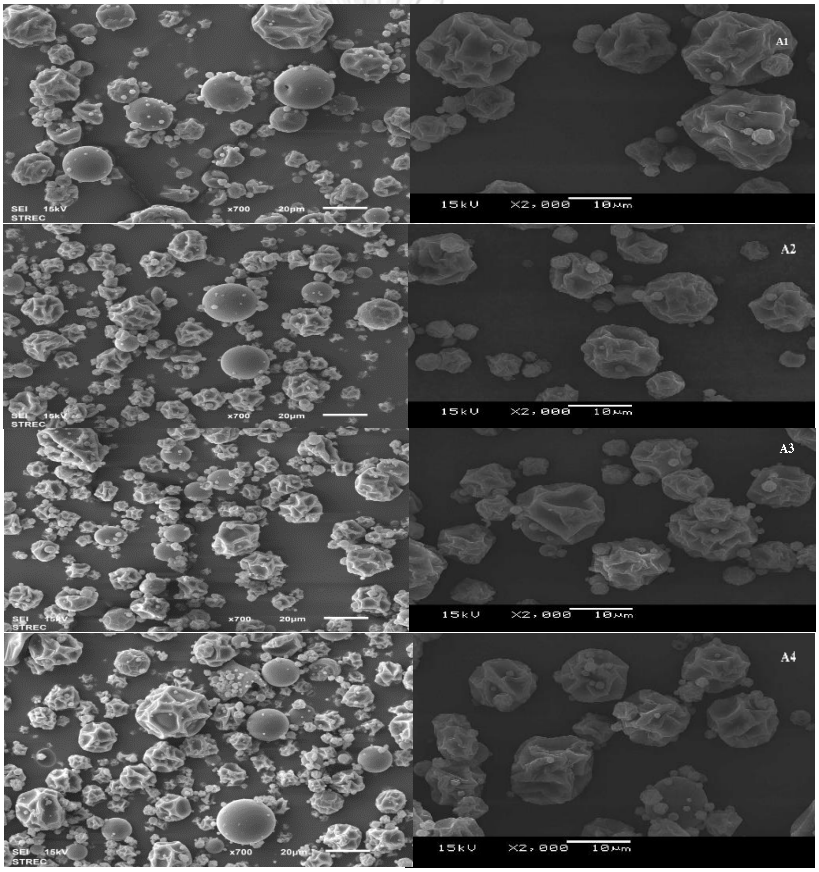

Figure 4.4 Surface morphology of instant coffee (700 and 2,000 magnification) with wall material 20% (w/w) KGMH (A1), 15% KGMH + 5% (w/w) MD (A2), 5% KGMH + 15% (w/w) MD (A3) and 20% (w/w) MD (A4) at 180 °C

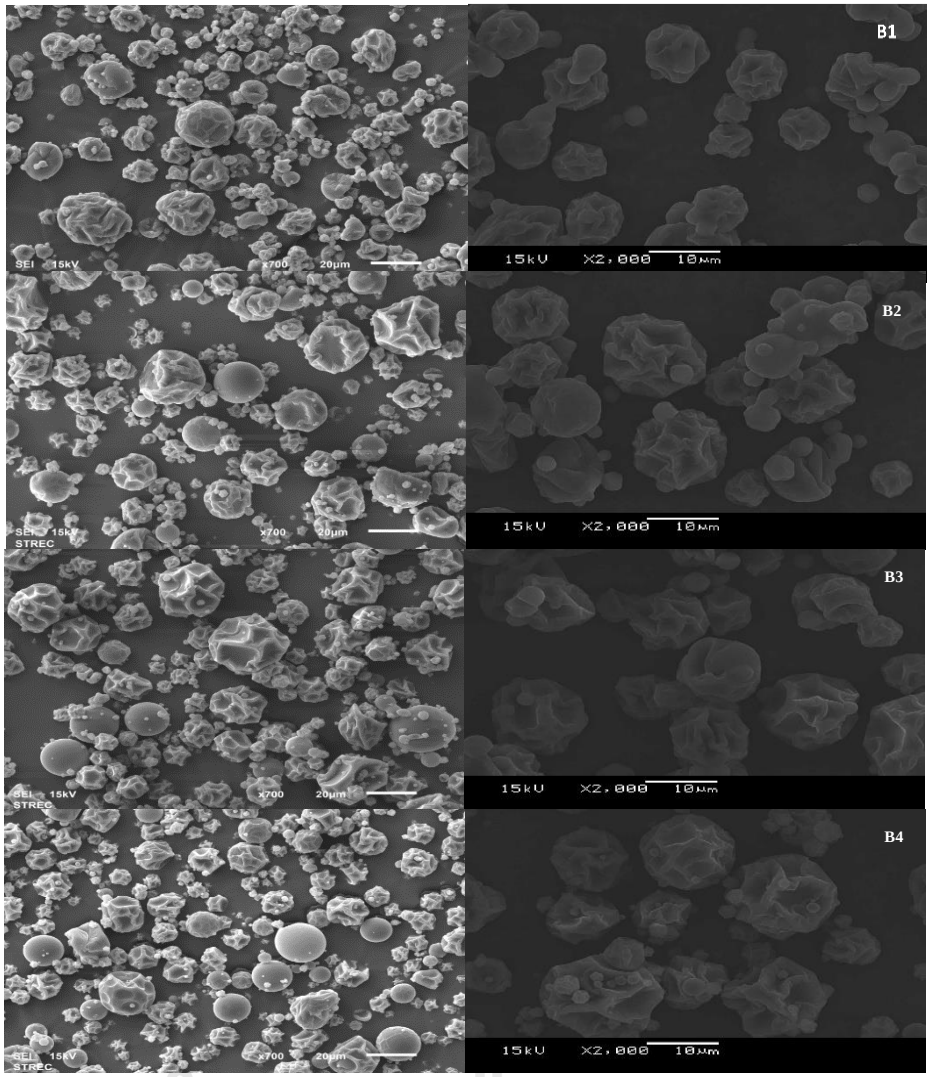

Figure 4.5 Surface morphology of instant coffee (700 and 2,000 magnification) with wall material 20% (w/w) KGMH (B1), 15% KGMH + 5% (w/w) MD (B2), 5% KGMH + 15% (w/w) MD (B3) and 20% (w/w) MD (B4) at 170 °C

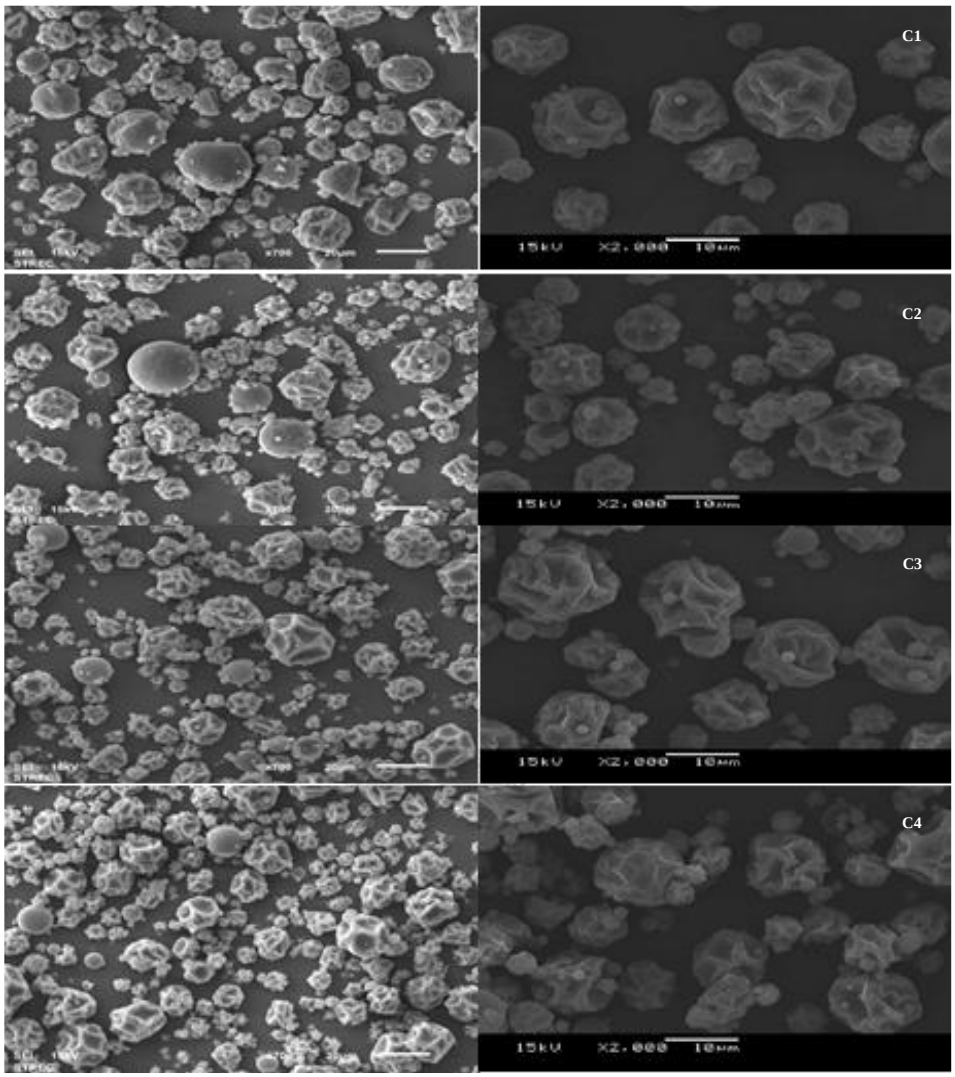

Figure 4.6 Surface morphology of instant coffee (700 and 2,000 magnification) with wall material 20% (w/w) KGMH (B1), 15% KGMH + 5% (w/w) MD (B2), 5% KGMH + 15% (w/w) MD (B3) and 20% (w/w) MD (B4) at 160 °C

4.5 Best formulation and suitable spray drying condition

From the results of the experiments, it is noted that wall material and inlet temperature influenced chemical and physical properties of instant coffee powder. Chemical properties of instant coffee powder exhibited a trend where increasing KGMH concentration and lowering inlet temperature were lead to the better antioxidant and dietary fiber profiles. Meantime, physical properties are mostly in the similar pattern each other yet all powder still exhibited good powder characteristics. Therefore, based on chemical properties, instant coffee powder made from 20% (w/w) KGMH and dried at 160 °C is the best formulation among the treatments.

4.6 Proximate analysis of best formulation

Table 4.7 Chemical content of 20% (w/w) KGMH instant powder produced at 160°C

Composition	Chemical content g/100 g sample
Carbohydrates	53.04
Dietary fiber	25.22
Protein	8.34
Ash	8.01
Moisture	3.84
Crude fat	1.55

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Table 4.7 showed the composition of the best formulation (KGMH instant coffee). It is found that carbohydrates are a predominant component in KGMH instant coffee powder where oligosaccharides and some sugars from KGMH solution and concentrated coffee extract contributed to higher carbohydrates content. Protein and ash are other major components in instant coffee powder. Interestingly, the crude fat is 1.55% and it is reflected that only small amount of oil that was extracted. Coffee oil has a role in increasing the aromatic potential of the soluble coffee and coffee beverages (Frascareli *et al*., 2012). On the other hands, in roasted beans, lipids as major components assisted to loss of sensorial quality because of lipid oxidation (Toci *et al*., 2013). Therefore, the low content of oil in instant coffee powder could lower the

incidence of lipid oxidation, even though the aroma of instant coffee might not be appealing as the fresh brew coffee.

The instant coffee composition then is calculated to kilocalorie and reference instant coffee is used to compare the amount of calorie. The reference instant coffee is the most famous instant coffee brand in Thailand. It is noted that one serving size of reference instant coffee (2 gram) provides 5 Kcal. After calculation, KGMH instant coffee provides 5.4 Kcal per 2 gram of powder. However, the serving size of KGMH instant coffee is double than reference instant coffee. Thereof, one serving size KGMH instant coffee will give 10.97 Kcal.

4.7 Sensory analysis

Rating hedonic with 15 cm line scale was conducted to evaluate the liking acceptance microencapsulated instant coffee and was compared with a top brand instant coffee in Thailand. The raw data of sensory test was presented in the Appendix Table B.14-B.17. The result from independent *t-test* showed that KGMH instant coffee had lower mean value at all attribute liking. However, the value of liking acceptance was somewhat equal with reference instant coffee (p>0.05) (**Figure 4.7**). What is more, higher gap of values was found in the attribute flavor and aroma. In consequence, the improvement of product can be focused in flavor and aroma. The purchase intent of KGMH instant coffee is ‘probably would not buy’ to ‘may or may not would buy’ (2.8), this purchase intent increased become ‘may or may not would buy’ to ‘probably would buy’ (3.5) when the tasted product was given an additional explanation of functional properties (high antioxidant and dietary fiber). Meanwhile, the reference product presented higher purchase intent with the value of 3.08 for the common product, while for the additional functional properties exhibited the similar value with KGMH instant coffee (3.5). In addition, both KGMH and reference instant coffee have shown the similar value of the expected price of regular instant coffee (± 90 THB) and functional instant coffee (± 116 THB). However, these values were high variation since many panelists did not know the average price of instant coffee in the market. Nevertheless, when the difference of the price was calculated, both KGMH and reference instant coffee were presented same value which 26 THB increased for functional instant coffee.

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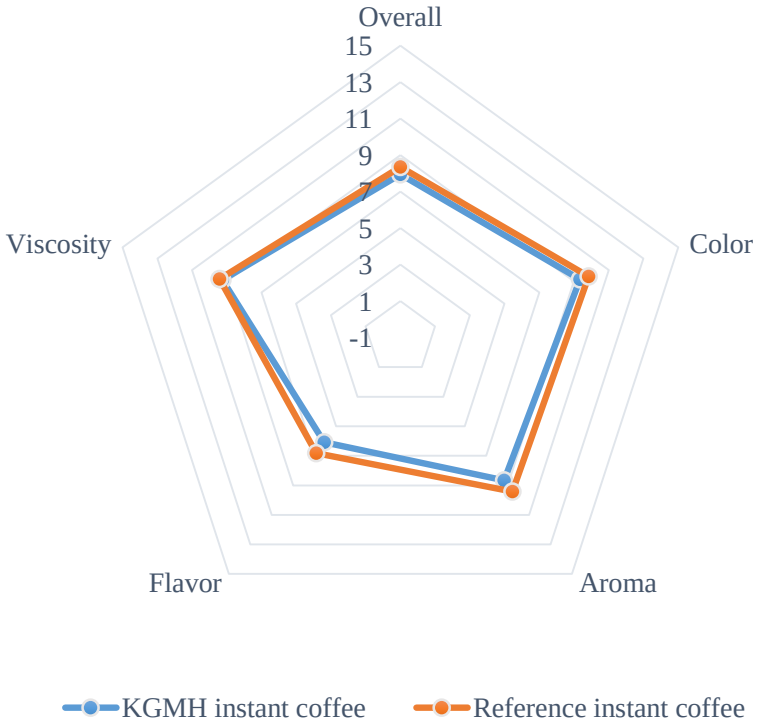

Figure 4.7 Liking acceptance score from hedonic test with 15 cm line scale

4.8 Color of KGMH instant coffee powder

The measurement of color was conducted using chromameter that reads color in three-dimensional format. The instrument provides the level of brightness between black and white (L*), the balance between red and green with 100 being red and -100 green, and the relative yellow and blue (b*).

Figure 4.8 Instant coffee powder and beverages

KGMH instant coffee has lighter color than reference product (**Figure 4.8** and **Table 4.8**). The reference product powder has two times darker, slightly redder, and twice time less yellow than KGMH instant coffee. The coffee liquid (2 gr in 100 ml water) was presented the same results where the reference product has darker color. In sensory evaluation, the equi-strength of color was conducted by using difference test with 20 tasters. It is noted that, 3.6 gr of 20% (w/w) KGMH was equilibrium with 2 gr of reference product in 100 ml water (**Figure 4.8** on right side). Regardless human evaluation have shown similar color, but by using chroma meter the coffee liquid still presented different color which is lighter color.

Table 4.8 The color of instant coffee powder and beverages

Instant coffee sample	L*	a*	b*
Reference product	24.48±0.27	6.56±0.16	8.88±0.38
20% KGMH	51.53±0.08	5.40±0.04	22.78±0.26
Reference product liquid (2 gr in 100 ml water)	23.30±0.02	0.43±0.00	2.89±0.04
20% KGMH liquid (2 gr in 100 ml water)	21.90±0.00	-0.33±0.09	1.32±0.01
20% KGMH liquid (3.6 gr in 100 ml water)	21.75±0.02	-0.40±008	1.69±0.06

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CHAPTER 5
CONCLUSION AND RECOMMENDATION

5.1 Conclusion

KGMH can be used as an appropriate natural wall material in spray drying microencapsulation of instant coffee. KGMH concentration affected antioxidant and other physicochemical properties. Increasing KGMH concentration leads to the better antioxidant properties including TPC, antioxidant capacity by DPPH and FRAP and chlorogenic acid content. The wall material was able to shield almost all antioxidant component and resulted in the excessive retention percentage. The application of KGMH alone as wall material exhibited the best retention percentage compare to microcapsule made from KGMH-MD in all ratio and MD alone.

The inlet temperature of spray drying was showing an important role in antioxidant properties. The increasing of inlet temperature was correlated with lower retention of antioxidant properties except for chlorogenic acid that was increased. In general, the lowest inlet temperature (160 °C) produced better antioxidant properties than spray drying at 170 and 180 °C.

The best condition of producing instant coffee from this study is 20% (w/w) KGMH as wall material at 160 °C inlet temperature of spray drying. The optimum condition produces microencapsulated instant coffee with the retention percentage about 129, 131, 122, and 130%, respectively for TPC, DPPH, FRAP and chlorogenic acid experiments. It appears that KGMH is being detected by the antioxidant experiments and approximately 100% of coffee solid was being trapped inside the microcapsule.

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5.2 Recommendation

In the present study, KGMH can be used effectively as a coating material for coffee extract and the ability was better than MD. However, there are some limitation in the current study and need further research to provide a better understanding. The further research should focus on the following points:

- Improvement of coffee extract to obtain more bioactive compounds

- Measurement of antioxidant activity for wall materials alone to know their contribution to the keys tests.
- Comparison and or combination with other wall material could be tried to get better powder properties

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APPENDIX

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APPENDIX A

Analysis Procedures

A.1 Total sugar and total reducing sugar measurements

Total sugar was performed using a phenol-sulfuric acid method by reacting 1 ml of diluted KGMH with 1 ml of 5% (w/w) phenol solution and 5 ml of concentrated H_2SO_4. The mixture was incubated at 25 °C for 20 minutes. An aqueous solution of mannose and glucose in the ratio of 1.6:1 (w/w) was used as a standard solution. The reaction result was measured with UV-VIS spectrophotometer with 490 nm. Meanwhile, total reducing sugar was determined by dinitrosalycyclic (DNS) acid assay. The reaction took place by mixing 2 ml of diluted sample with 2 ml of DNS reagent. The mixture then immersed in boiling water for 10 minutes and quickly cooled in cold water before spectrophotometry measurement at 570 nm. The same standard solution in different concentration was used for total reducing sugar determination.

A.2 Antioxidant experiments

The total phenolic content was performed by Folin-Ciocalteu and calculated by chlorogenic acid equivalent. Dissolved sample (50 µl) was mixed with 3 ml water and 250 µl Folin Ciocalteu reagent. Then the mixture was incubated for 5 minutes before added with 750 µl 20% (w/w) Na_2CO_3 and 950 µl water and incubate for 60 minutes. Phenolic compounds were spectrophotometrically examined at 765 nm (Margarita et al., 2012). Antioxidant activity was analyzed by DPPH method at 515 nm of spectrophotometric analysis. A volume of 0.1 ml diluted sample and 3.9 ml DPPH-methanolic solutions (0.025 mg/ml methanol) were mixed and incubated at the dark room for 30 minutes prior measurement (Margarita et al., 2012). Ferric reducing antioxidant power (FRAP) also used to prescribe antioxidant activity. The FRAP reagent was obtained from the mixture of 10 mM TPTZ solution in 40 mM HCl, 20 mM $FeCL_3.6H_2O$ solution and 0.3 mM acetate buffer pH 3.6 with the ratio 1:1:10, respectively. The FRAP reagent was warmed at 37 °C for 30 minutes, then a diluted

sample (30 µl) was mixed with 2.7 ml FRAP reagent. Water was added to adjust the solution to the volume of 3 ml and incubated in water bath 37 °C for 30 minutes. The measurement was done by spectrophotometry assay at 595 nm (Vignoli et al., 2011). The chlorogenic acid determination was done by injected 20 µl of the sample or standard solutions in HPLC (Varian Prostar, USA) with mobile phase water, acetic acid and methanol (799, 1 and 200 ml), column reverse phase ODS 250 and detected by Prostar 335 Photodiode Array Detector at 278 nm.

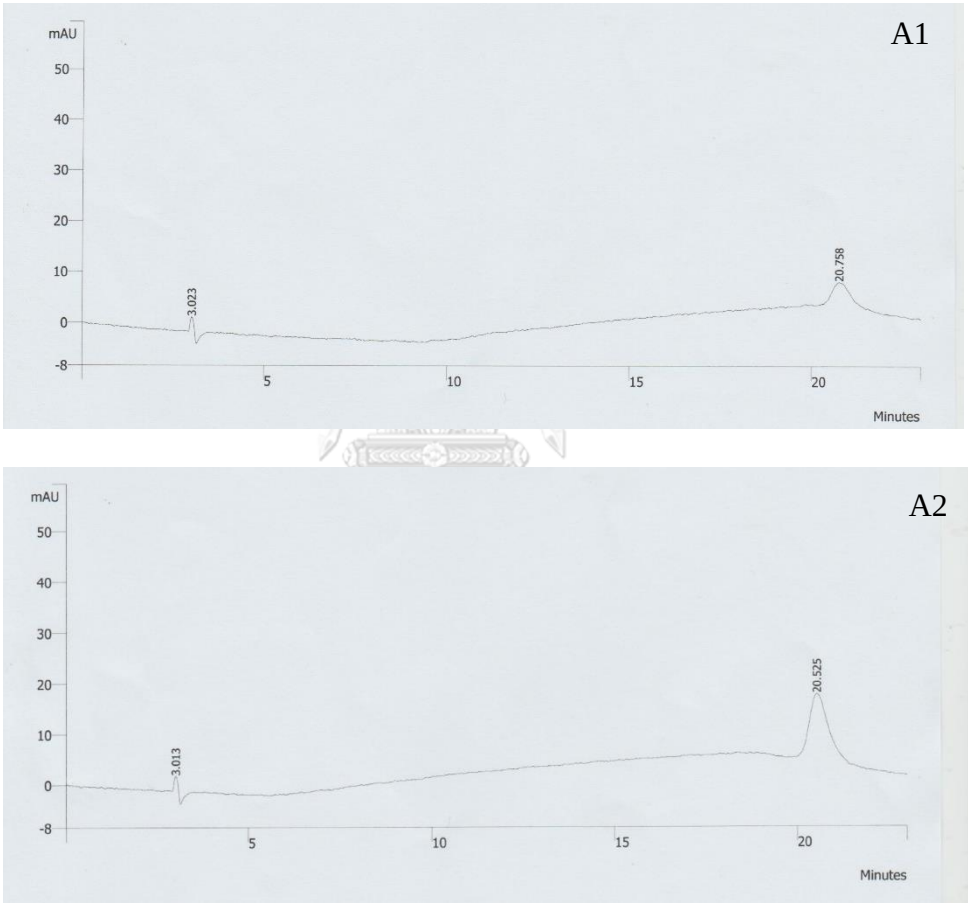

Figure A2. 1 Chlorogenic acid standard solution 10 ppm (A1) and 25 ppm (A2)

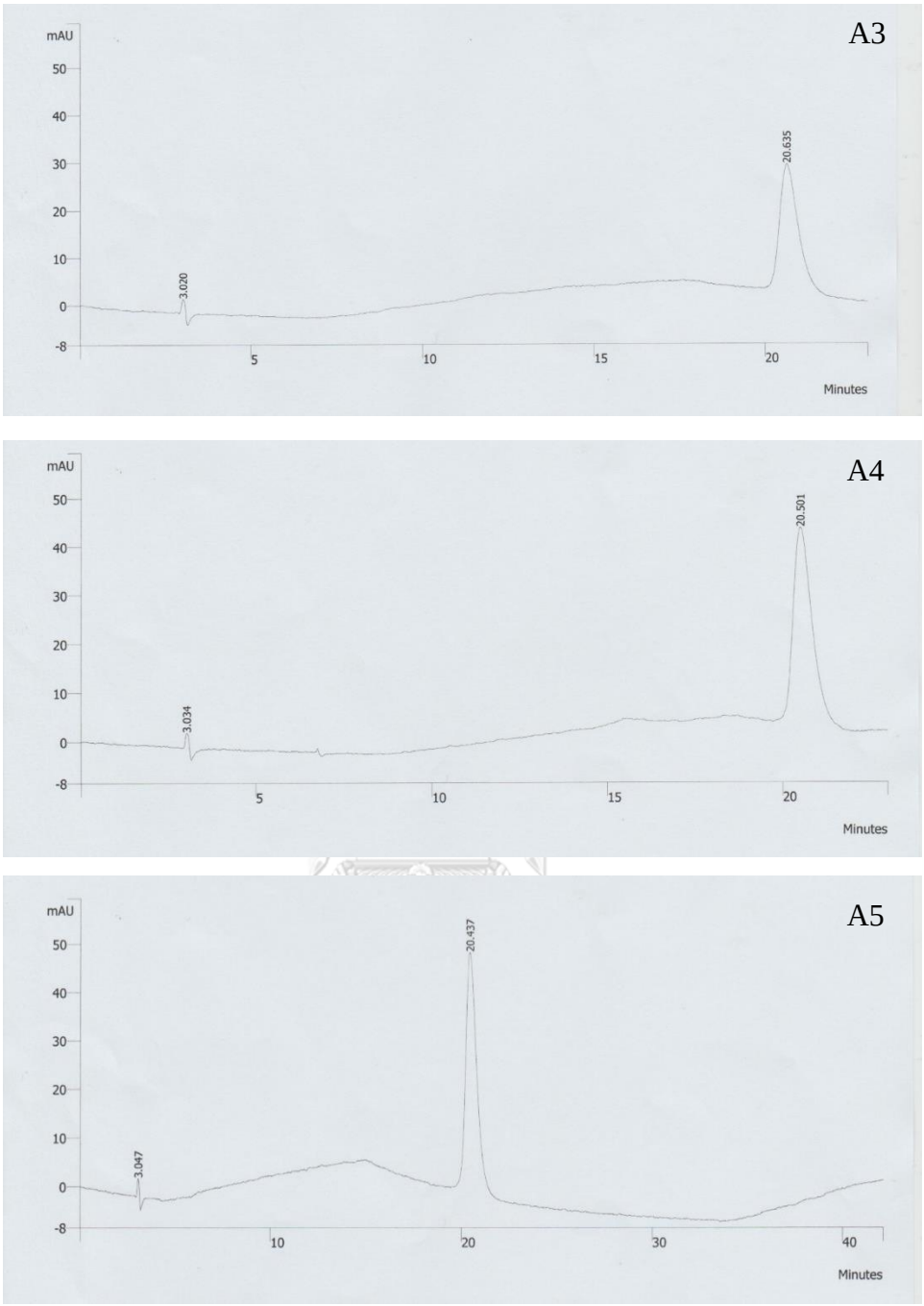

Figure A2. 2 Chlorogenic acid standard solution 50 ppm (A3), 75 ppm (A4) and 100 ppm (A5)

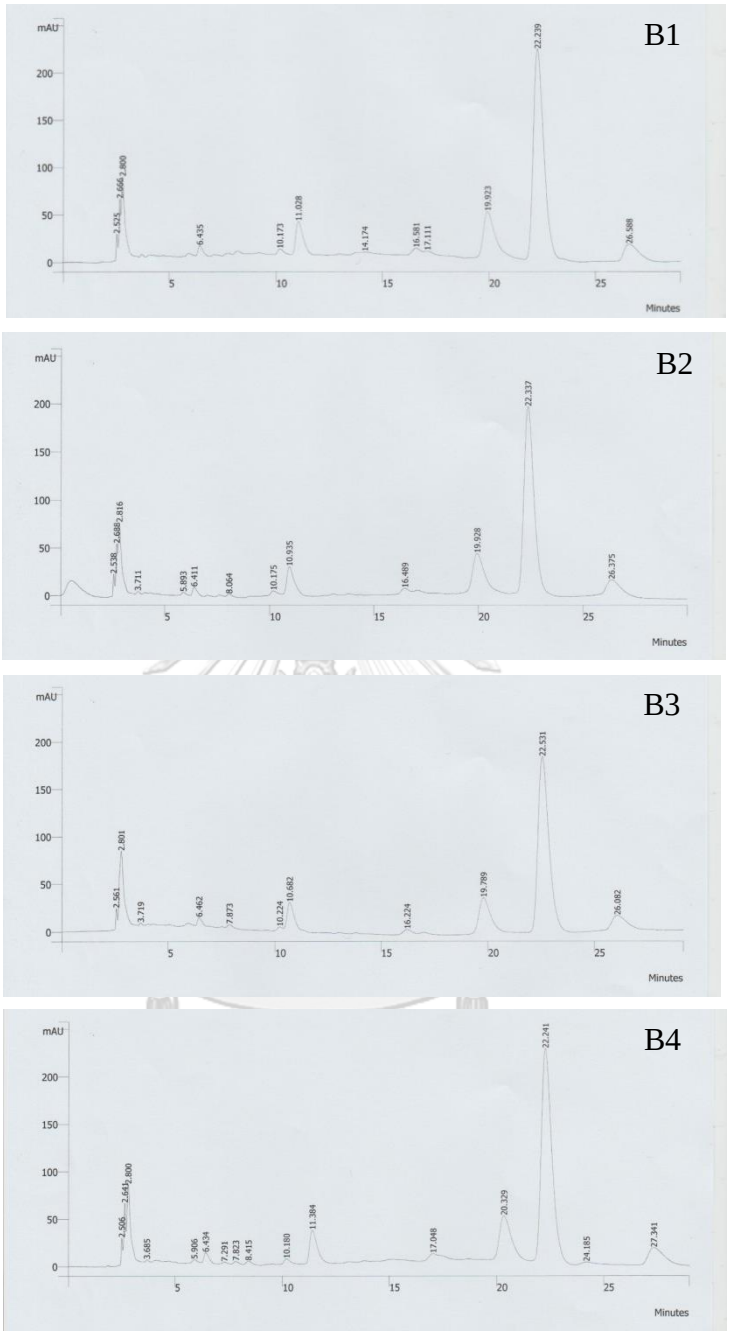

Figure A2. 3 Chlorogenic acid content in instant coffee produced at 180 °C inlet temperature with 20% KGMH (B1), 15% KGMH (B2), 5% KGMH (B3) and 20% maltodextrin (B4)

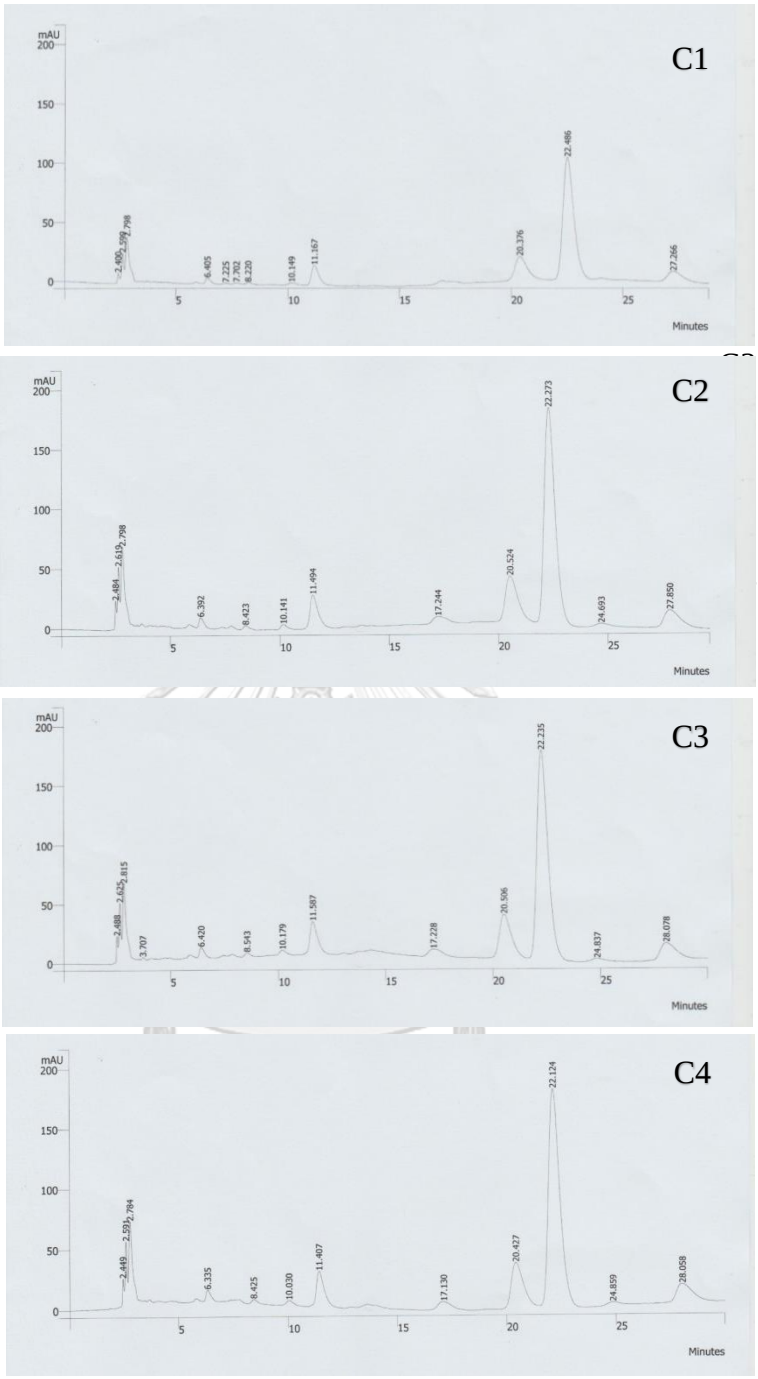

Figure A2. 4 Chlorogenic acid content in instant coffee produced at 170 °C inlet temperature with 20% KGMH (C1), 15% KGMH (C2), 5% KGMH (C3) and 20% maltodextrin (C4)

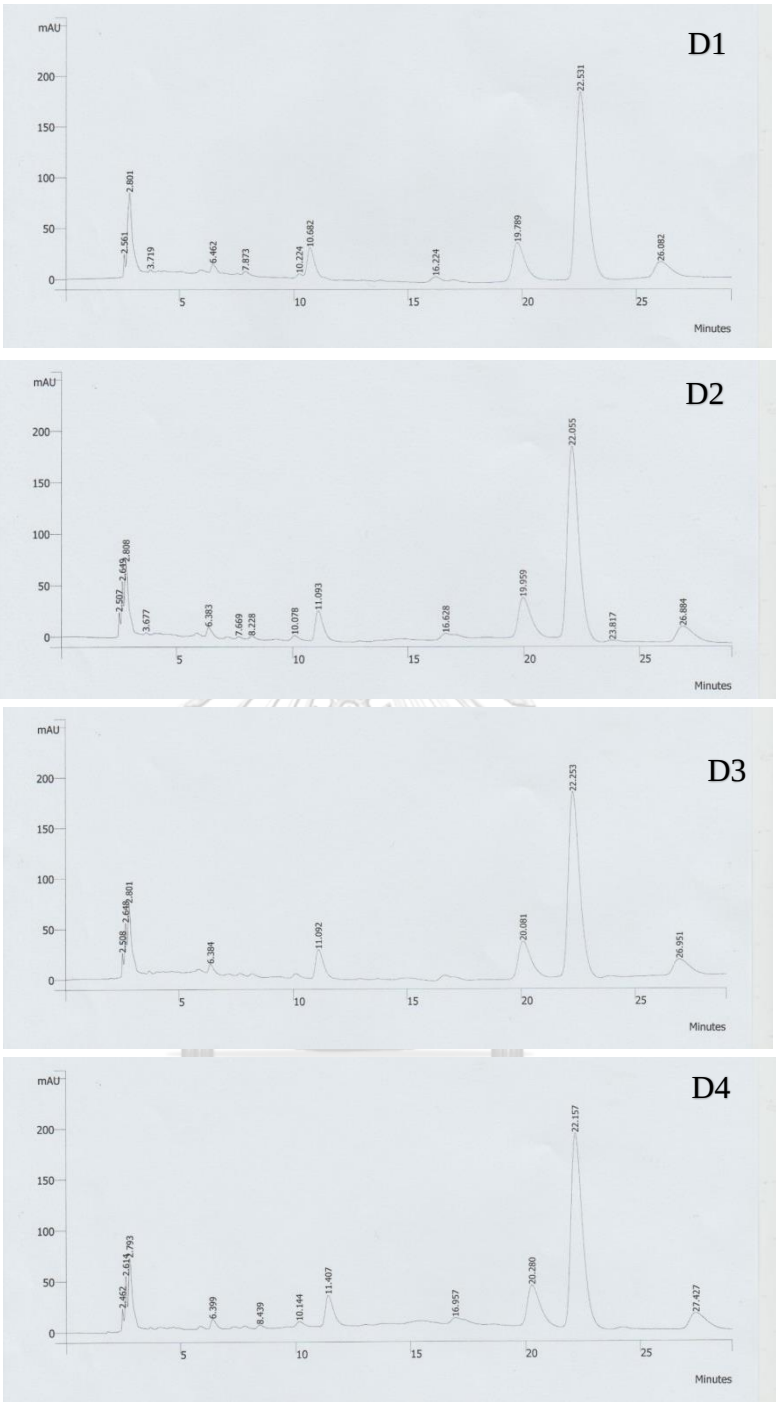

Figure A2. 5 Chlorogenic acid content in instant coffee produced at 160 °C inlet temperature with 20% KGMH (D1), 15% KGMH (D2), 5% KGMH (D3) and 20% maltodextrin (D4)

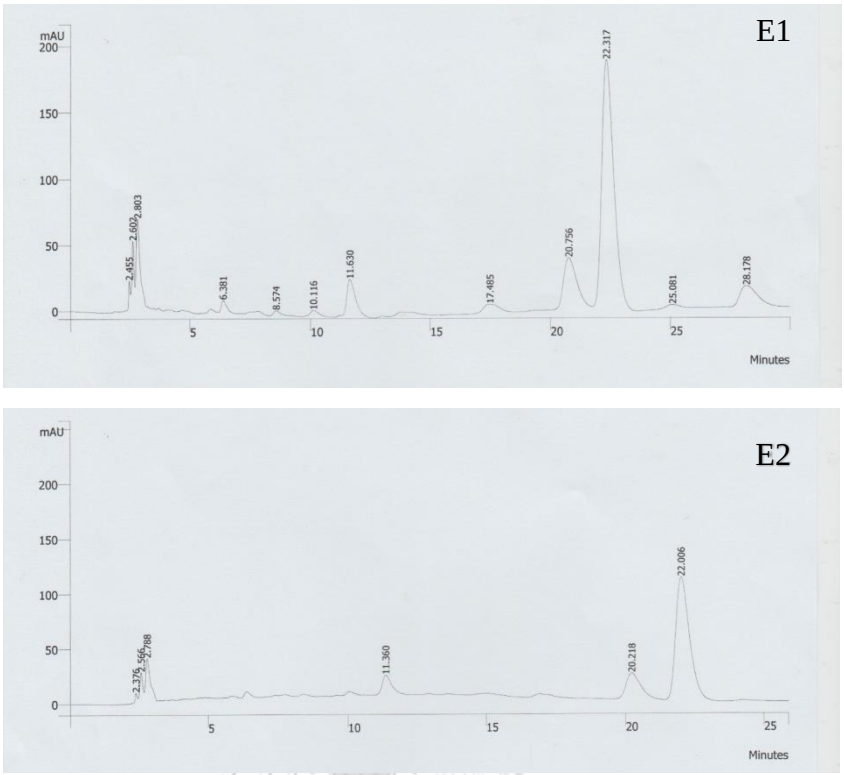

Figure A2. 6 Chlorogenic acid content in coffee extract at 170 °C (E1) and 160 and 180 °C (E2)

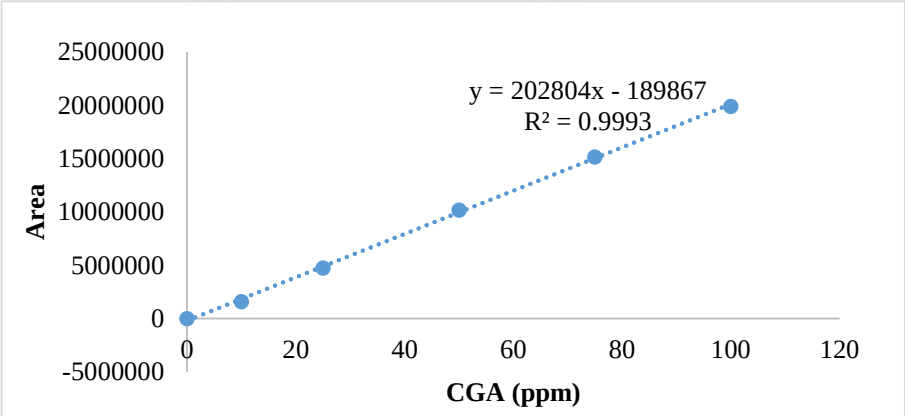

Figure A2. 7 Chlorogenic acid content in coffee extract at 170 °C (E1) and 160 and 180 °C (E2)

A3. Dietary fiber determination

Determination of soluble and insoluble dietary fiber was based on AOAC Method 991.43 by using dietary enzyme kits from megazyme (Ireland). Sample (1.00 gram) was accurately weighed into 400 ml tall-form beaker glass. 40 ml MES-TRIS buffer solution (pH 8.2) was added to a beaker and mixed by magnetic stirrer until completely diluted. 50 µl of thermal-stable α-amylase was added while stirring at low speed. The glass was covered with aluminum foil and placed in shaking water bath at 98±2 °C. After that, samples were incubated while shaking at 120 shake/minutes for 30 minutes. Samples were removed from the water bath and let it cool to 60 °C before added with 100 µl protease. Recover the glass with aluminum foil and placed into shaking water bath 60 °C. After 30 minutes of protease incubation, samples were removed and 5 ml of 0.561 N HCl was dispensed into sample while stirring until the sample was cooling down. The solutions should have pH at range 4.1-4.8 (adjusting can be done using 5% (w/w) NaOH or 5% (w/w) HCl). 200 µl amyloglucosidase was added to the solution and stirring by a magnetic stirrer, replace the aluminum cover and incubate the samples in shaking water bath at 60 °C.

Whatman paper number 1 with diameter 90 mm was drying at 103 °C for 6 hours then the weight is noted as the weight of the dried paper filter. Placed the paper to Buchner funnel, wet and redistribute the paper using 3 mL distilled water, apply suction to filter flask and pour the sample solution into the filter system. Then, 10 mL pre-heated distilled water was added to wash the residue in beaker glass and paper filter. Save the filtrate for soluble dietary fiber determination. After the filtrate was separated, wash the residue on the paper filter is washed with 95% ethanol and acetone. The paper filter containing insoluble dietary fiber was overnight dried in hot air oven at 103 °C. For soluble dietary fiber determination, filtrate solutions were added with 4 times volume of ethanol pre-heated at 60 °C. The solutions then were incubated at room temperature for 60 minutes to precipitate the fiber. Put another dried paper into Buchner funnel and wet it by using 78% ethanol. Suction was applied to draw the paper onto Buchner funnel and the incubated solutions were poured to vacuum filter system. 15 mL of 78% ethanol, 95% ethanol and acetone were used for washing the residue. The

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paper filter containing soluble dietary fiber was overnight dried in hot air oven at 103 °C. Fiber calculation was based on gravimetric method over the initial dry sample.

A4. Water solubility index (WSI) and water absorption index (WAI)

Sample powder (2.5 g) and 30 ml distilled water were mixed in a 50 ml centrifuge tube. The diluted sample was incubated at 30 °C for 30 minutes before centrifuge at 2,090xg for 15 minutes. Supernatant and pellet were separated in different pre-weight petri dish glasses and oven-drying overnight at 103 °C. WSI was calculated from dried supernatant divided by initial dried sample, while WAI was measured from solid pellet over the dry sample.

A5. Sensory evaluation

Coffee beverage taste test	
Taster ID.:_____	Sample code:_____

Instruction
Put a vertical mark on the given scale line that best describes your opinion of each attribute. Please rinse your mouth with water after you finish evaluate the product.
Write down your response truthfully and as detailed as possible for open-ended questions.
Choose an option for multiple-choice questions.

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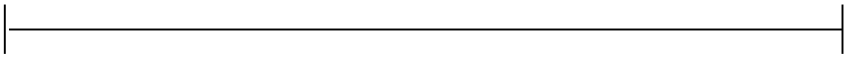
1. Overall, how much do you like this sample?

   Your overall impression after you smell and taste the coffee.

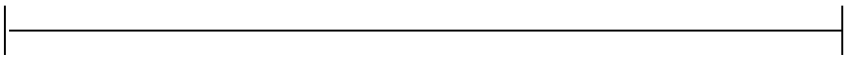


Dislike extremely Like extremely

1b. Which aspect of this sample need to be improved: _____
_____
_____
_____

1c. Which aspect of this sample that you like the most (give 3 answers):
_____
_____

2. How much do you like the color of the sample?



Dislike extremely Like extremely

2b. Why?

_____

_____

3. How much do you like the aroma of the sample?

Aroma is an odor detected by nasal or an olfactory sensation in your nose.

Dislike extremely Like extremely

3b. Why?

_____

_____

4. How much do you like the taste/flavor?

Taste/flavor is the overall experience from both taste & smell after the sample entering your mouth.

Dislike extremely Like extremely

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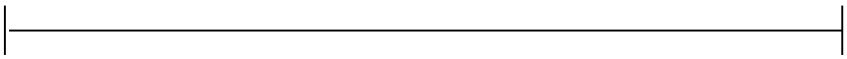
4b. For this sample, how do you want the taste/flavor of this coffee beverage to be improved: _

_____

_____

5. How much do you like the thickness/viscosity of the coffee?

Thickness/Viscosity is the feeling of the viscosity of the coffee liquid.



Dislike extremely Like extremely

5b. For this sample, how do you want the viscosity of this coffee beverage to be improved:

_____

_____

6. How much are you willing to buy this product? (choose 1 choice)

Definitely would not buy	Probably would not buy	May or May not buy	Probably would buy	Definitely would buy

7. The picture below is a package of a regular instant coffee. From one package (90 grams), you can make around 25 cups of instant coffee beverage. With this information, how much are you willing to pay for one package?

		Answer:_____Baht

8. If the same product in question 7 provides high antioxidant with around 10% of cholorogenic acid and also high of dietary fiber (contained more than 6 grams of fiber per 100 g product).
    - Chlorogenic acid is known to protect body cells, have anti-aging benefits, prevent cancer and degenerative diseases
    - Dietary fiber is known to help maintain gastrointestinal tract health, lower cholesterol levels, control blood sugar level
9. How much are you willing to buy this product with these benefits? (Choose 1 choice)

Definitely would not buy	Probably would not buy	May or May not buy	Probably would buy	Definitely would buy

How much do you want to pay for the product in question 8.

Answer: _____Baht

10. If the product in question 8 has the same taste as the product you just tasted, how much are you willing to buy this product with the benefits in question 8? (Choose 1 choice)

Definitely would not buy	Probably would not buy	May or May not buy	Probably would buy	Definitely would buy

Thank you, you have finished this ballot.
Please call a staff member to assist you.

==========

A6. Scanning Electron Microscope (SEM)

The surface morphology of instant coffee powder was analyzed by using SEM JEOL JSM-6480LV. The sample was spread on a brass stub using double-sided tape and coated with a thin layer of gold under vacuum to render the electrical conductivity. The photographs were taken at excitation voltage of 15 kV.

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APPENDIX B

Raw Data

Table B.1 Viscosity of KGMH after hydrolysis with different concentration of enzyme

Enzyme concentration (IU/g KGM)	Replication	Viscosity (mPa.s)	
		A. muelleri	***A. bulbifer***
22000	1	1746	5508
	2	1571	5454
30000	1	238	386
	2	233	355
38000	1	86	192
	2	79	187
46000	1	38	64
	2	45	63

Table B.2 Total reducing sugar of KGMH solutions

Enzyme concentration (IU/g KGM)	Replication	Total reducing sugar (mg/ml)	
		A muelleri	*A bulbifer*
22000	1	13.64	9.79
	2	13.39	9.94
	3	13.99	9.59
30000	1	14.65	12.80
	2	14.94	13.01
	3	14.55	12.66
38000	1	14.15	13.19
	2	14.06	13.56
	3	13.97	12.97
46000	1	15.09	12.94
	2	15.37	14.25
	3	14.91	12.89

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Table B.3 Total sugar of KGMH solutions

Enzyme concentration (IU/g KGM)	Replication	Total sugar (mg/ml)	
		A muelleri	*A bulbifer*
22000	1	23.73	18.80
	2	23.83	17.13
	3	23.69	20.91
30000	1	18.57	18.19
	2	16.55	18.15
	3	16.50	19.82
38000	1	16.37	19.00
	2	16.37	18.45
	3	17.28	17.83
46000	1	15.72	21.68
	2	15.67	19.74
	3	16.17	22.16

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Table B.4 Total phenolic content of instant coffee powder and coffee extract

Inlet temperature °C	Wall material	Total phenolic compounds (mg CGA/g coffee solid)		
		Replication 1	Replication 2	Replication 3
160	20% (w/w) KGMH	228.14	189.89	194.09
	15% (w/w) KGMH+5% (w/w) MD	179.52	174.30	169.56
	5% (w/w) KGMH+15% (w/w) MD	191.12	163.46	164.57
	20% (w/w) MD	176.02	165.34	164.49
170	20% (w/w) KGMH	195.44	193.38	171.60
	15% (w/w) KGMH+5% (w/w) MD	182.95	184.69	153.75
	5% (w/w) KGMH+15% (w/w) MD	184.38	182.68	156.77
	20% (w/w) MD	189.58	160.06	159.74
180	20% (w/w) KGMH	216.51	193.72	193.77
	15% (w/w) KGMH+5% (w/w) MD	183.82	172.76	175.22
	5% (w/w) KGMH+15% (w/w) MD	184.86	169.88	167.47
	20% (w/w) MD	173.84	169.07	167.27
Coffee extract		163.60	163.6	155.01

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Table B.5 Antioxidant capacity of instant coffee powder and coffee extract (DPPH method)

Inlet temperature °C	Wall material	Antioxidant activity (mM Trolox/g coffee solid)		
		Replication 1	Replication 2	Replication 3
160	20% (w/w) KGMH	721.69	796.79	921.43
	15% (w/w) KGMH+5% (w/w) MD	629.67	701.37	887.13
	5% (w/w) KGMH+15% (w/w) MD	619.91	635.51	824.30
	20% (w/w) MD	610.73	586.84	627.55
170	20% (w/w) KGMH	641.36	674.26	844.13
	15% (w/w) KGMH+5% (w/w) MD	507.28	589.73	810.72
	5% (w/w) KGMH+15% (w/w) MD	424.97	583.08	851.75
	20% (w/w) MD	412.69	567.60	775.86
180	20% (w/w) KGMH	651.57	641.80	863.61
	15% (w/w) KGMH+5% (w/w) MD	606.46	552.68	816.06
	5% (w/w) KGMH+15% (w/w) MD	635.96	574.20	842.63
	20% (w/w) MD	582.55	629.59	622.88
Coffee extract		506.87	587.79	762.98

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Table B.6 Antioxidant capacity of instant coffee powder and coffee extract (FRAP method)

Inlet temperature °C	Wall material	Antioxidant activity (mM Trolox/g sample)		
		Replication 1	Replication 2	Replication 3
160	20% (w/w) KGMH	2362.86	2498.41	2498.41
	15% (w/w) KGMH+5% (w/w) MD	1909.47	1866.06	1970.23
	5% (w/w) KGMH+15% (w/w) MD	2052.24	1919.75	1951.54
	20% (w/w) MD	2001.23	1976.31	1941.52
170	20% (w/w) KGMH	1915.79	2410.34	2478.82
	15% (w/w) KGMH+5% (w/w) MD	1762.59	2230.00	1792.22
	5% (w/w) KGMH+15% (w/w) MD	1752.29	1760.06	1756.22
	20% (w/w) MD	1800.32	1741.15	1810.51
180	20% (w/w) KGMH	2186.70	2362.15	2331.72
	15% (w/w) KGMH+5% (w/w) MD	1816.10	2078.10	2093.88
	5% (w/w) KGMH+15% (w/w) MD	2055.94	1827.45	1727.77
	20% (w/w) MD	2069.06	1834.88	1751.60
Coffee extract		1894.15	2061.93	2051.6

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Table B.7 Soluble dietary fiber content of instant coffee

Inlet temperature °C	Wall material	Soluble dietary fiber (g/100 g solid sample)		
		Replication 1	Replication 2	Replication 3
160	20% (w/w) KGMH	19.25	14.91	20.78
	15% (w/w) KGMH+5% (w/w) MD	19.05	12.37	19.10
	5% (w/w) KGMH+15% (w/w) MD	13.41	8.15	9.82
	20% (w/w) MD	7.53	5.11	0.58
170	20% (w/w) KGMH	18.39	20.99	23.37
	15% (w/w) KGMH+5% (w/w) MD	16.33	20.15	12.41
	5% (w/w) KGMH+15% (w/w) MD	13.07	11.99	6.15
	20% (w/w) MD	3.08	1.87	2.76
180	20% (w/w) KGMH	22.70	16.04	27.70
	15% (w/w) KGMH+5% (w/w) MD	16.17	13.07	17.73
	5% (w/w) KGMH+15% (w/w) MD	9.01	11.66	5.68
	20% (w/w) MD	1.87	2.00	0.96

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Table B.8 Insoluble dietary fiber content of instant coffee

Inlet temperature °C	Wall material	Insoluble dietary fiber (g/100 g solid sample)		
		Replication 1	Replication 2	Replication 3
160	20% (w/w) KGMH	12.73	3.24	4.76
	15% (w/w) KGMH+5% (w/w) MD	10.00	4.88	5.49
	5% (w/w) KGMH+15% (w/w) MD	2.55	2.13	3.77
	20% (w/w) MD	1.89	1.51	2.98
170	20% (w/w) KGMH	11.46	5.67	5.56
	15% (w/w) KGMH+5% (w/w) MD	4.98	2.18	4.44
	5% (w/w) KGMH+15% (w/w) MD	2.18	2.98	3.40
	20% (w/w) MD	1.01	1.70	3.22
180	20% (w/w) KGMH	7.82	7.85	4.48
	15% (w/w) KGMH+5% (w/w) MD	8.35	5.45	4.41
	5% (w/w) KGMH+15% (w/w) MD	3.35	4.19	4.03
	20% (w/w) MD	3.51	1.32	2.30

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Table B. 9 Yield production of spray drying

Inlet temperature °C	Wall material	Yield production (%)		
		Replication 1	Replication 2	Replication 3
160	20% (w/w) KGMH	53.29	58.65	49.05
	15% (w/w) KGMH+5% (w/w) MD	50.87	54.57	52.19
	5% (w/w) KGMH+15% (w/w) MD	65.36	61.70	61.51
	20% (w/w) MD	70.62	63.19	62.00
170	20% (w/w) KGMH	54.40	54.38	54.12
	15% (w/w) KGMH+5% (w/w) MD	51.18	54.69	53.34
	5% (w/w) KGMH+15% (w/w) MD	58.20	61.46	55.26
	20% (w/w) MD	69.26	60.53	57.00
180	20% (w/w) KGMH	57.03	56.20	52.58
	15% (w/w) KGMH+5% (w/w) MD	55.24	54.57	49.90
	5% (w/w) KGMH+15% (w/w) MD	63.07	62.77	58.33
	20% (w/w) MD	63.31	65.37	63.32

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Table B. 10 Moisture content of instant coffee powder

Inlet temperature °C	Wall material	Moisture content (% wet basis)		
		Replication 1	Replication 2	Replication 3
160	20% (w/w) KGMH	4.12	3.14	3.93
	15% (w/w) KGMH+5% (w/w) MD	3.03	3.55	3.67
	5% (w/w) KGMH+15% (w/w) MD	3.75	3.84	3.67
	20% (w/w) MD	3.75	4.11	3.06
170	20% (w/w) KGMH	3.43	3.92	3.21
	15% (w/w) KGMH+5% (w/w) MD	3.91	3.47	3.61
	5% (w/w) KGMH+15% (w/w) MD	3.73	3.67	3.75
	20% (w/w) MD	3.73	3.75	3.26
180	20% (w/w) KGMH	3.44	3.06	3.62
	15% (w/w) KGMH+5% (w/w) MD	3.63	3.41	4.01
	5% (w/w) KGMH+15% (w/w) MD	3.5	3.13	3.56
	20% (w/w) MD	3.5	3.65	3.47

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Table B. 11 Water activity of instant coffee powder

Inlet temperature °C	Wall material	Water activity		
		Replication 1	Replication 2	Replication 3
160	20% (w/w) KGMH	0.230	0.235	0.229
	15% (w/w) KGMH+5% (w/w) MD	0.245	0.245	0.245
	5% (w/w) KGMH+15% (w/w) MD	0.232	0.182	0.232
	20% (w/w) MD	0.252	0.219	0.219
170	20% (w/w) KGMH	0.24	0.243	0.219
	15% (w/w) KGMH+5% (w/w) MD	0.162	0.24	0.26
	5% (w/w) KGMH+15% (w/w) MD	0.241	0.228	0.25
	20% (w/w) MD	0.246	0.219	0.21
180	20% (w/w) KGMH	0.256	0.208	0.274
	15% (w/w) KGMH+5% (w/w) MD	0.211	0.226	0.206
	5% (w/w) KGMH+15% (w/w) MD	0.247	0.245	0.219
	20% (w/w) MD	0.22	0.244	0.278

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Table B. 12 Water solubility index of instant coffee powder

Inlet temperature °C	Wall material	Water solubility index (%)		
		Replication 1	Replication 2	Replication 3
160	20% (w/w) KGMH	91.17	83.05	92.63
	15% (w/w) KGMH+5% (w/w) MD	95.02	93.41	87.25
	5% (w/w) KGMH+15% (w/w) MD	96.02	96.04	97.55
	20% (w/w) MD	98.20	92.02	92.02
170	20% (w/w) KGMH	92.30	85.04	89.75
	15% (w/w) KGMH+5% (w/w) MD	94.92	91.97	88.80
	5% (w/w) KGMH+15% (w/w) MD	95.56	96.91	95.55
	20% (w/w) MD	99.51	95.45	95.45
180	20% (w/w) KGMH	93.13	85.92	89.07
	15% (w/w) KGMH+5% (w/w) MD	94.43	88.58	88.04
	5% (w/w) KGMH+15% (w/w) MD	85.87	96.66	94.87
	20% (w/w) MD	99.98	98.48	98.48

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Table B. 13 Water absorption index of instant coffee powder

Inlet temperature °C	Wall material	Water absorption index (%)		
		Replication 1	Replication 2	Replication 3
160	20% (w/w) KGMH	8.34	10.66	7.83
	15% (w/w) KGMH+5% (w/w) MD	3.81	3.10	2.29
	5% (w/w) KGMH+15% (w/w) MD	4.67	2.08	2.64
	20% (w/w) MD	2.37	3.59	3.65
170	20% (w/w) KGMH	7.35	9.28	10.40
	15% (w/w) KGMH+5% (w/w) MD	4.06	3.44	6.28
	5% (w/w) KGMH+15% (w/w) MD	4.51	2.00	4.97
	20% (w/w) MD	2.51	2.51	4.81
180	20% (w/w) KGMH	6.65	10.17	9.59
	15% (w/w) KGMH+5% (w/w) MD	4.79	5.77	7.16
	5% (w/w) KGMH+15% (w/w) MD	5.06	6.15	5.03
	20% (w/w) MD	3.16	3.16	4.42

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Table B.14 Sensory data of overall, color and aroma

Taster No.	Overall		Color		Aroma	
	1	2	1	2	1	2
1	7.4	9.6	6.7	7.5	9.5	10.6
2	11.6	7.5	7.8	10.5	11	7.7
3	5.4	2.8	9.8	10	3.6	12.3
4	8.3	9.7	11.3	10.3	12	12.7
5	6.5	12	13.5	13.2	3	12.2
6	9.8	7.5	11	8	10.8	7.2
7	9.6	12.5	12.5	12.5	12.5	12.2
8	7.5	10	11.3	11.3	5.5	12
9	10	12	11.8	14.5	7.3	2.8
10	9.2	6	12	12	10	12
11	8	10.5	5	10.3	8.3	3.5
12	10.3	11.5	11	11.5	8.8	11.7
13	11	8.7	6.7	9	11.7	8.8
14	5.6	10	10	10.3	4.7	10.7
15	8.5	3.5	9	7	13.5	2.5
16	5.5	7.8	7.2	11.3	6.4	6.7
17	5.5	8.3	7.5	7	9	9.8
18	11.7	14	4.7	14	10.7	14
19	7.7	6.8	13	10	14.5	10.3
20	10.6	13.5	11	8.4	5.6	12
21	6.5	4.2	13.4	8	11	8.5
22	8.7	12.5	13	15	14.7	15
23	10.3	6.4	12.5	12	9.2	9.5
24	5.5	9.4	10	7.7	4.5	8.1
25	8.4	6.7	7.3	8.5	10.3	9.5
26	6.7	9	3.7	5.3	6.4	8.7
27	8.8	11	11.3	7.5	4	10
28	8.5	6.6	13	13.3	9	7.2
29	6.7	3.4	6.5	3.7	3.5	3.5
30	7.7	5.5	10.5	7.2	11.5	11.3
31	7.4	6	6.5	7.2	5.5	5.4
32	4.8	4	3.5	6	6	6
33	4	4.5	3.3	7	7.5	13.5
34	9	10.5	10.5	11	12	10.7
35	6.5	7.4	11.6	12.3	6	12.7
36	9.5	10	8.7	14.7	10.5	12.2
37	5.4	8	8	9	11	5.5

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Table B.15 Sensory data of flavor and viscosity

Taster No.	Flavor		Viscosity	
	1	2	1	2
1	4.5	9	8.5	7.3
2	4	9	4.3	10.5
3	4	2.5	10.5	10.6
4	8	9.7	10.2	11
5	8.7	10	13.3	13.5
6	4	4.5	10.5	4.7
7	9.4	11.5	9.5	11.7
8	11	11.7	8	11.7
9	7	12.3	14.5	14.7
10	7	3.4	5	11.5
11	7.7	5.4	8.3	4.1
12	10.3	11	10.3	10.5
13	8.7	9	9.5	9.3
14	6.5	9.6	5	6.5
15	3.4	3	6.5	3
16	6	8.3	6	5.6
17	3	4.5	11.3	7
18	2.5	2	5	9
19	3	9.3	11.5	8
20	6	2.4	11	9
21	6.6	5.4	14.5	13.4
22	6.3	15	6.7	15
23	9.4	4.5	14.2	12
24	1	2.5	11	4
25	6.7	8.3	9.5	9
26	6.3	3	5.5	5.2
27	6.3	9.2	9	9.5
28	7.2	6	14	14.2
29	4.5	1.6	9	10
30	5	7.6	10.5	10
31	6.7	4.5	8.3	7.7
32	2	1.8	2	6.2
33	4.5	3.6	11.3	11.2
34	6.7	6.5	5.5	9
35	7	8.5	13.8	13
36	10.4	12.5	13	14.8
37	4	4	5.5	5.5

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Table B.16 Expected price for normal and functional of instant coffee

Taster No.	KGMH instant coffee (THB)		Reference instant coffee (THB)	
	Normal	Functional	Normal	Functional
1	50	90	60	100
2	60	100	60	100
3	70	80	70	75
4	50	65	50	70
5	90	120	90	120
6	100	180	100	120
7	150	175	150	200
8	30	50	30	50
9	230	300	200	250
10	50	60	55	65
11	60	70	60	75
12	70	100	70	110
13	110	120	110	120
14	89	79	89	99
15	60	60	60	60
16	70	120	70	120
17	125	160	125	170
18	70	85	70	85
19	60	80	60	80
20	40	60	40	60
21	200	250	200	250
22	50	80	80	100
23	100	150	100	150
24	75	100	75	100
25	45	55	45	55
26	60	70	55	60
27	150	170	200	210
28	55	79	50	79
29	59	79	59	79
30	150	180	150	180
31	120	120	139	139
32	79	129	79	125
33	100	150	120	180
34	45	65	45	69
35	50	75	50	75
36	199	250	200	250
37	120	100	120	120

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VITA

Miss Desi Sakawulan was born on December 3, 1991, in Subang, Indonesia. She got her B.Sc degree in Food Science and Technology from Bogor Agricultural University, Indonesia, in 2014. She got a full scholarship to pursue master degree at Chulalongkorn University from Government of Republic of Indonesia through Indonesia Endowment Fund for Education (Lembaga Pengelola Dana Pendidikan).

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