ผลของขนาดอนุภาคต่อการกำจัดไขมันออกจากผงเนื้อในเมล็ดมะขามด้วยตัวทำละลายอินทรีย์ในถังกวน

นาย สุชาติ ฉัตรเศรษฐกูล

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิศวกรรมศาสตรมหาบัณฑิต
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EFFECT OF PARTICLE SIZE ON FAT REMOVAL FROM TAMARIND KERNEL POWDER USING ORGANIC SOLVENT IN MIXING TANK

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A Thesis Submitted in Partial Fulfillment of the Requirements
for the Degree of Master of Engineering Program in Chemical Engineering
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Faculty of Engineering
Chulalongkorn University
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Thesis Title	EFFECT OF PARTICLE SIZE ON FAT REMOVAL FROM TAMARIND KERNEL POWDER USING ORGANIC SOLVENT IN MIXING TANK
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สุชาติ ฉัตรเศรษฐกูล : ผลของขนาดอนุภาคต่อการกำจัดไขมันออกจากผงเนื้อในเมล็ดมะขามด้วยตัวทำละลายอินทรีย์ในถังกวน (EFFECT OF PARTICLE SIZE ON FAT REMOVAL FROM TAMARIND KERNEL POWDER USING ORGANIC SOLVENT IN MIXING TANK) อ.ที่ปรึกษาวิทยานิพนธ์หลัก : รศ.ดร. จิรกานต์ เมืองนาโพธิ์, 103 หน้า.

โพลีแซคคาไรด์จากผงเนื้อในเมล็ดมะขามเป็นสารที่มีประโยชน์ซึ่งสามารถนำไปประยุกต์ใช้ในอุตสาหกรรมอาหาร เครื่องสำอาง และเภสัชกรรม โดยใช้เป็นสารให้ความหนืด สารรักษาความคงตัว เป็นต้น ผงเนื้อในเมล็ดมะขามมีองค์ประกอบของไขมันซึ่งส่งผลทำให้เกิดกลิ่นหืนและสีของผลิตภัณฑ์เปลี่ยนไป งานวิจัยนี้จึงมีวัตถุประสงค์เพื่อทำการทดลองหาภาวะที่เหมาะสมของชนิดตัวทำละลาย อัตราส่วนระหว่างผงเนื้อในเมล็ดมะขามต่อตัวทำละลาย ขนาดของอนุภาค และเวลาในการสกัดต่อร้อยละการกำจัดไขมัน และศึกษาเปรียบเทียบประสิทธิภาพการกำจัดไขมันในถังกวนที่ทำการขยายขนาดจากปริมาตร 0.5 ลิตร เป็น 10 ลิตร จากการวิเคราะห์องค์ประกอบของผงเนื้อในเมล็ดมะขามที่ใช้เป็นวัตถุดิบ พบว่า มีปริมาณโพลีแซคคาไรด์ร้อยละ 70.47 โปรตีนร้อยละ 18.92 ไขมันร้อยละ 8.05 และองค์ประกอบอื่นๆอีกร้อยละ 2.56 และความหนืดของวัตถุดิบที่ความเข้มข้นร้อยละ 2 กรัมต่อมิลลิลิตร มีค่าเท่ากับ 540 เซนติพอยส์ จากผลการทดลองพบว่าตัวทำละลายร่วมระหว่างเอทานอลร้อยละ 95 กับไอโซโพรพานอลร้อยละ 99.9 ที่อัตราส่วน 1:1 ปริมาตรต่อปริมาตร มีประสิทธิภาพในการกำจัดไขมันใกล้เคียงกับไอโซโพรพานอลร้อยละ 99.9 ซึ่งเป็นตัวทำละลายที่มีประสิทธิภาพสูงในการกำจัดไขมัน เมื่อพิจารณาถึงอัตราส่วนของผงเนื้อในเมล็ดมะขามต่อตัวทำละลายพบว่าเมื่อเพิ่มปริมาณของตัวทำละลาย ร้อยละการกำจัดไขมันจะมีค่าสูงขึ้นเนื่องจากมีความแตกต่างของความเข้มข้นเพิ่มสูงขึ้นทำให้การโอนถ่ายมวลมากขึ้น อย่างไรก็ดีการเพิ่มปริมาณตัวทำละลายส่งผลต่อการเพิ่มค่าใช้จ่าย ดังนั้นอัตราส่วนจำนวนน้ำหนักเป็นกรัมของผงเนื้อในเมล็ดมะขามต่อปริมาตรเป็นมิลลิลิตรของตัวทำละลายที่ 1:3 จึงเหมาะสมในการกำจัดไขมัน นอกจากนี้ยังพบว่าเมื่อใช้ไอโซโพรพานอลสามารถนำกลับมาใช้สกัดใหม่ได้ 6 ครั้งในขณะที่ตัวทำละลายร่วมนำกลับมาใช้ใหม่ได้ 2 ครั้งเท่านั้น การกำจัดไขมันจะมีประสิทธิภาพสูงเมื่อใช้ผงเนื้อในเมล็ดมะขามขนาดเล็กซึ่งมีพื้นที่ผิวจำเพาะในการโอนถ่ายมวลสารสูง แต่ผงเนื้อในเมล็ดมะขามขนาดเล็กต้องใช้พลังงานในการลดขนาดสูง ดังนั้นผงเนื้อในเมล็ดมะขามขนาดกลางจึงเหมาะสมสำหรับการกำจัดไขมัน เมื่อทำการศึกษาเปรียบเทียบการกำจัดไขมันในถังกวนขนาดใหญ่และถังกวนขนาดเล็ก พบว่าไม่มีการสูญเสียประสิทธิภาพในการกำจัดไขมัน ภาวะที่เหมาะสมในการกำจัดไขมัน คือ ตัวทำละลายร่วมระหว่างเอทานอลร้อยละ 95 กับ ไอโซโพรพานอลร้อยละ 99.9 ที่ 1:1 ปริมาตรต่อปริมาตร อัตราส่วนจำนวนเป็นกรัมของผงเนื้อในเมล็ดมะขามต่อปริมาตรเป็นมิลลิลิตรของตัวทำละลายที่ 1:3 อนุภาคขนาดกลาง (0.553-0.598 มิลลิเมตร) และใช้เวลาในการสกัด 30 นาที ภายใต้ภาวะการทดลองนี้สามารถกำจัดไขมันได้ร้อยละ 74.49 ผลิตภัณฑ์ที่ได้มีปริมาณโพลีแซคคาไรด์ โปรตีน ไขมัน และองค์ประกอบอื่นๆเท่ากับร้อยละ 76.19 17.36 2.05 และ 4.40 โดยน้ำหนักตามลำดับ และค่าความหนืดของผลิตภัณฑ์มีค่าเท่ากับ 665 เซนติพอยส์

ภาควิชา ……….วิศวกรรมเคมี………….. ลายมือชื่อนิสิต……………………………………

สาขาวิชา ……...วิศวกรรมเคมี………….. ลายมือชื่อ อ.ที่ปรึกษาวิทยานิพนธ์หลัก………………

ปีการศึกษา …….2551…………………..

4970640021 : MAJOR CHEMICAL ENGINEERING
KEYWORDS : TAMARIND KERNEL POWDER / FAT REMOVAL / ETHANOL / ISO-PROPANOL / CO-SOLVENT / PARTICLE SIZE

SUCHART CHATSETTAKUL : EFFECT OF PARTICLE SIZE ON FAT REMOVAL FROM TAMARIND KERNEL POWDER USING ORGANIC SOLVENT IN MIXING TANK. ADVISOR : ASSOC. PROF. CHIRAKARN MUANGNAPOH, Dr.Ing., 103 pp.

Polysaccharide from Tamarind Kernel Powder (TKP) is a useful material in many industries such as food, cosmetics, and pharmaceutical industries for thickening and stabilizing. The fat in TKP makes product low quality because of rancidity and change of product color. This study investigated the effects of solvent type, TKP to solvent ratio, particle size, and extraction time on fat removal percentage and determined the suitable condition for fat removal. Moreover, the extraction performance of laboratory scale with the working volume of 0.5 liter was compared to that of semi-pilot scale with the working volume of 10 liter. It was found that the TKP as raw material consisted of 70.47% polysaccharide, 18.92% protein, 8.05% fat, and 2.56% other ingredients. The viscosity of raw material at 2% g/ml was 540 cp. Regarding to solvent types, co-solvent between 95% ETOH and 99.9% IPA at the ratio of 1:1 v/v provided a high fat removal percentage which was almost similar to that of an effective solvent 99.9% IPA. Among the TKP to solvent ratios, the fat removal percentage increased with the TKP to solvent ratio decreased because a large bulk volume of solvent gave high concentration difference which favored mass transfer. However, high solvent utilization has an impact on operating costs. Thus, the use of TKP to solvent ratio at 1:3 g/ml was suitable. Moreover, it was also found that we could reuse IPA 6 times while the reusable of co-solvent was only 2 times. The extraction was effective with small particles due to their high specific surface area. To avoid high energy consumption for size reduction, the suitable particle size was found to be medium size. Fat removal results obtained from semi-pilot scale were found to be in good agreement with those of laboratory scale without any loss of extraction efficiency. The suitable condition for fat removal was 30 min extraction time, 1:3 g/ml TKP to solvent ratio, medium-size TKP (0.553-0.598 mm), and 1:1 v/v 95% ETOH:99.9% IPA co-solvent. Under this condition, the fat removal percentage was 74.49 % and the product viscosity was 665 cp. The composition of this product were 2.05% fat, 76.19% polysaccharide, 17.36% protein, and 4.40% others. A high-quality, low-fat and high-viscosity product was obtained and potentially used in food industry as a viscosity enhancer.

Department : ……Chemical Engineering Student's Signature : ....

Field of Study : …Chemical Engineering Advisor's Signature : ....

Academic Year : ..2008………………..

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NOMENCLATURE

ETOH	Ethanol
IPA	Iso-propanol
k_La	Mass Transfer Coefficient (s^{-1})
L	Large Size Tamarind Kernel Powder (0.843-0.886 mm)
M	Medium Size Tamarind Kernel Powder (0.553-0.598 mm)
S	Small Size Tamarind Kernel Powder (0.029-0.032 mm)
TKP	Tamarind Kernel Powder
TSP	Tamarind Seed Polysaccharide

CHAPTER I

INTRODUCTION

1.1 Motivation

Tamarind (*Tamarindus indica* Linn.) is a common and the most important tree of Thailand and South East Asian countries. The fruit pulp is often freshly eaten and has many other culinary uses such as jam, candy, juice, and drink. The tamarind seed is a by-product of tamarind pulp industry. Tamarind kernel powder (TKP) is separated and subsequently refined from tamarind seed in order to produce a useful product named tamarind seed polysaccharide (TSP), an expensive natural gum. Tamarind seed constitutes about 30-34% of the whole fruit. However, 80,000 tons per year is discarded (Kumar and Bhattacharya, 2008). In Thailand, tamarind cultivating area is 570,000 rais resulting in producing 280,000 tons of tamarind fruit per year (Department of Agricultural Extension, Thailand, 2004), thus, tamarind seeds are immense.

The TKP is commercially available product produced by flaking off tamarind seed coat and grinding kernel into fine powder. In Thailand, G.M. Ichihara Co., Ltd. is the TKP production company. The ground tamarind seed was sieved by mesh using mesh size of 200. Many Japanese companies use this TKP under the trade name of MAKAM 200 (G.M. Ichihara, (Thailand) Co., Ltd.) as a raw material to produce TSP by means of removing protein and fat from TKP. It is interesting to produce TSP in Thailand instead of selling TKP aboard. Therefore, this research was done in cooperation with G.M. Ichihara, (Thailand) Co., Ltd under TRF-MAG funding. TSP provides a variety of utilizations in many fields. In food and pharmaceutical industries, it was applied to be thickening, stabilizing, gelling, and emulsifying agent (Goyal et al., 2006). Ultra pure TSP is used as a viscosity enhancer in pharmaceutical formulations for ophthalmic use in artificial tears (Saettone et al., 1998). Additionally, TSP is capable of forming gel over a wide range of pH, including neutral and basic conditions.

The compositions of TKP are 65.1–72.2% polysaccharide, 3.9–8.0% fat, 15.0–20.9% protein, 2.5–8.2% crude fiber, 11.4–22.7% moisture, and 2.4–4.2% total ash (Shankarachaya, 1998). The presence of some non-polysaccharides, which are protein, fat, and fiber, are undesirable. Furthermore, protein in TKP may generate foam during dispersion in aqueous solution. Their denaturization causes insoluble precipitation problem. The water-insoluble fiber in TKP may cause the shut-down of process equipment for fiber removal. The effect of fat in TKP makes low quality product such as sticky, rancid, and non–free flowing TKP during storage (Jones et al., 1976). The tamarind kernel oil contains a large proportion of unsaturated fatty acids which are easily oxidized. Its major fatty acids are linoleic acid, oleic acid, and palmitic acid. Therefore, elimination of fat at the beginning of product storage results in high quality TKP.

From the above reasons, this research will be emphasized on fat removal from TKP. The extraction of fat from TKP was reported by using organic solvents such as hexane, chloroform, and methanol (Andriamanantena et al., 1983), ethylene dichloride (Jones et al., 1978), and acetone (Teraoka et al., 1990). However, the disadvantages of these solvents and other organic solvents currently used in industry are toxicity, flammability, and restriction to governmental regulations. If these solvents are not properly removed from the product, they will harmful to the consumers' health. To avoid these problems, alternative solvents such as ethanol (ETOH) and isopropanol (IPA) are considered to use as the extractant in fat removal from TKP. From literature reviews, ETOH and IPA are also applied for oil extraction from natural agricultural crops such as ground corn (Kwiatkowski et al., 2002), cottonseed (Zhang et al., 2002), soybean (Gandhi et al., 2003), rice bran (Proctor et al., 1996) and rosehip seed (Franco et al., 2006) and used in many consumer products including food, drug, and cosmetics. Therefore, food grade ETOH and IPA have a good potential in extracting fat from TKP. Due to high cost of IPA, ETOH collaborate with IPA as a co-solvent is developed to reduce solvent cost. In TKP production, the energy consumption of seed milling step is very high. It is necessary to investigate the suitable particle size which will give high product yield and high fat removal at lower milling energy consumption. In fact, the fat in small particle with large specific area can be easily and rapidly extracted. But it poses not only the difficulty in operation due to slow drainage rates at high pressure drop during filtration but also high milling energy consumption.

In this study, the effects of particle size, solvent type, TKP to solvent ratio and extraction time on fat removal from TKP were investigated.

1.2 Objective

To evaluate the effect of particle size on fat removal from TKP using organic solvents in mixing tank.

1.3 Working scopes

1.3.1 Fat extraction from TKP conditions were fixed at 35ºC and 400 rpm stirring speed. The investigating parameters were as followed.
   (i) solvent type: 95% ETOH, 99.9% ETOH, 99.9% IPA, and 1:1-4:1 v/v ratio of 95% ETOH to 99.9% IPA
   (ii) TKP to solvent ratio: 1:3-1:6 g/ml
   (iii) particle size: 0.029-0.032, 0.553-0.598, and 0.843-0.886 mm
   (iv) extraction time: 1-120 min

1.3.2 Evaluation of the suitable condition for fat removal from TKP by considering the percentage of fat removal, product yield, polysaccharide and protein content in product, and product viscosity.

1.3.3 Scale up the mixing tank from 0.5 liter to 10 liter and apply the suitable condition from 1.3.2 in 10-liter mixing tank.

1.4 Expected benefits

1.4.1 Provide the suitable condition for fat removal from TKP.

1.4.2 Provide a high quality (low fat and high viscosity) and value-added product.

1.4.3 Provide a prototype of fat removal system that is useful for production scale.

CHAPTER II

BACKGROUND AND LITERATURE REVIEW

2.1 Tamarind

Tamarind (*Tamarindus indica* L.) is a tropical fruit tree which grows in dry/monsoonal climates. It belongs to the family Leguminosae (Fabaceae). It grows in more than 50 countries of the world. The major areas of production are in Asian countries like India, Bangladesh, Sri Lanka, Thailand, and Indonesia, and in the African and the American continents (Kumar and Bhattacharya, 2008). Every part of the tamarind tree is useful, especially the fruit. The sweetish acidic pulp of the fruit is a commercial product. It is the chief souring agent for curries, sauces, and certain beverages. The tender leaves and flowers are used as vegetable.

Figure 2.1 *Tamarindus indica* Linn. (www.sfns.net)

2.1.1 Description of plant (Kaur et al., 2006)

Foliage**:** The bright green and pinnate foliage is dense and feathery in appearance, making an attractive shade tree with an open branch structure. The leaves are normally evergreen but may be shed briefly in very dry areas during the hot season. There are usually as many as 10 to 20 nearly sessile 0.5-1 inch, pale green leaflets per leaf.

Flowers: The inconspicuous, inch wide, five petaled flowers are borne in small racemes and are yellow with orange or red steaks. The flower buds are pink due to the outer color of the 4 sepals which are shed when the flower opens.

Fruit: The 3-8 inch long, brown, irregularly curved pods are borne in abundance along the new branches. As the pods mature, they fill out somewhat and the juicy, acidulous pulp turns brown or reddish brown. When fully ripe, the shells are brittle and easily broken. The pulp dehydrates to a sticky paste enclosed by a few coarse stands of fiber. The pods may contain from 1 to 12 large, flat, glossy brown, obviate seeds embedded in the brown, edible pulp. The pulp has a pleasing sweet/ sour flavor and is high in both acid and sugar. It is also rich in Vitamin B and high in calcium.

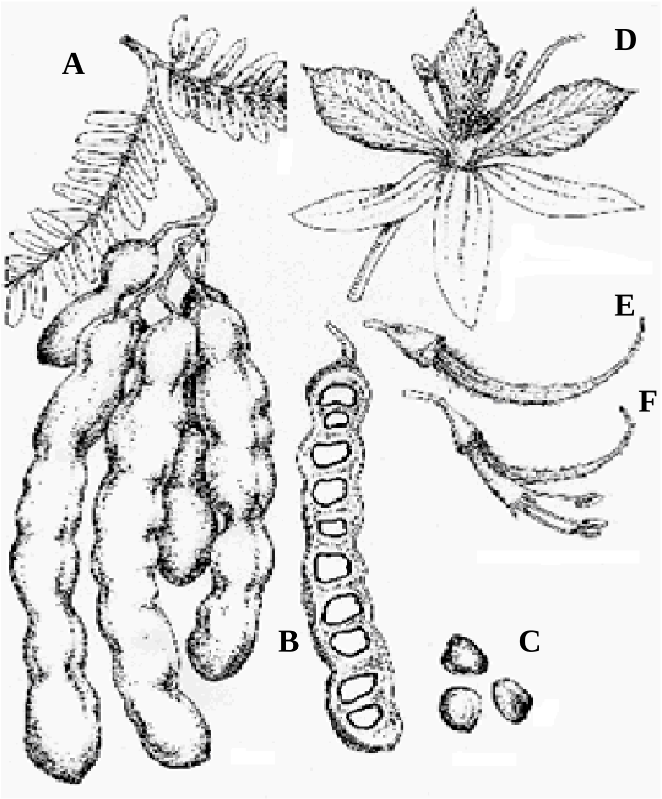


Figure 2.2 Flower and pods of tamarind showing the pistil, stamens and seed A.) Cluster of mature fruits. B.) Vertical section through a single pod/ fruit to show seed arrangement. C.) seeds. D.) Flower. E.) Detail of pistil and stigma. F.) Detail of corolla tube, pistil and stamens. (El-Siddig et al, 2006)

2.2 Tamarind kernel powder (TKP)

Tamarind kernel powder is creamy white powder and is obtained from tamarind seed. TKP will develop rancid smell and brown color on storage, whereas the defatted TKP remains white and odorless.

The rheological behavior of TKP solution behaves like non-Newtonian fluid (pseudo-plastic). An increase in TKP concentration increases the viscosity of solution and the non-Newtonian behavior. The viscosity decreases exponentially with temperature.

The tamarind seed comprises of the testa or seed coat (20–30%) and the endosperm or kernel (70–80%). The compositions of the whole seed, the kernel, and testa are shown in Table 2.1. Their compositions vary widely with variety and the extent of maturity (Kumar and Bhattacharya, 2008).

Table 2.1 Composition of tamarind seed, kernel, and testa (Kumar and Bhattacharya, 2008)

Constituent	Whole Seed (%)	Seed kernel (%)	Seed coat (%)
Moisture	9.4-11.3	11.4-22.7	11.0
Protein	13.3-26.9	15.0-20.9	-
Fat/oil	4.5-16.2	3.9-16.2	-
Crude fiber	7.4-8.8	2.5-8.2	21.6
Carbohydrates	50.0-57.0	65.1-72.2	-
Total ash	1.60-4.2	2.4-4.2	7.4
Nitrogen free extract	59.0	-	-
Yield of TKP	50.0-60.0	-	-
Calories/100g	340.3	-	-
Total sugar	11.3-25.3	-	-
Reducing sugar	7.4	-	-
Tannin	-	-	20.2

2.2.1 Tamarind seed polysaccharide (TSP) or jellose

As mention before, TSP was obtained from TKP purification process. The characteristic of TSP is a white fine-powder. TKP contains at least 50-60% of polysaccharide which is the active hydrocolloid principle. TSP has ability to form gel in the presence of sugar or alcohol. In this respect, it has properties and characteristics similar to that of fruit pectin to form acid-sugar jelly and can be used to form pectin-like gel in products such as jam, jelly, and marmalade. But unlike fruit pectin, TSP can form gels over a wide pH range, including neutral and basic conditions. It is also not affected by boiling in neutral aqueous solutions, even if boil for long periods. Fruit pectin undergoes degradation on boiling and falls to one-third of their original value after one hour of boiling. Therefore, TSP can be used as a gel formation agent and may be substituted for fruit pectin. TSP does not contain galacturonic acid and methyluronate and is therefore not regarded as a true pectin; it is termed ‘‘jellose’’ (Siddig et al., 2006). At the same concentration, the viscosity of TSP solution is greater than that of acacia gum, tragacanth, MC 1500, and locust bean gum but it is less than that of alginate and guar gum (Suttananta, 1997).

The polysaccharide in tamarind kernel is a xyloglucan which has (1→4)-β-D-glucan backbone substituted with side chains of α-D-xylopyranose and β-D-galactopyranosyl (1→2)-α-D-xylopyranose linked (1→6) to glucose residues (Goyal et al., 2006). The tamarind xyloglucan composed of three units of xyloglucan oligomer with heptasaccharide (Glu_4Xyl_3), octasaccharide (Glu_4Xyl_3Gal), and nonasaccharide ($Glu_4Xyl_3Gal_2$) as shown in Figure 2.3 (Yamanaka et al., 2000). The ratio of D-galactose/D-xylose/D-glucose is 1:2.25:2.9 and the constituent ratio of xyloglucan heptamer, octamer, and nonamer is reported to 13:39:48. The molecular weight of polysaccharide in the range of 115-2,500 kDa is reported (Marathe et al., 2001).

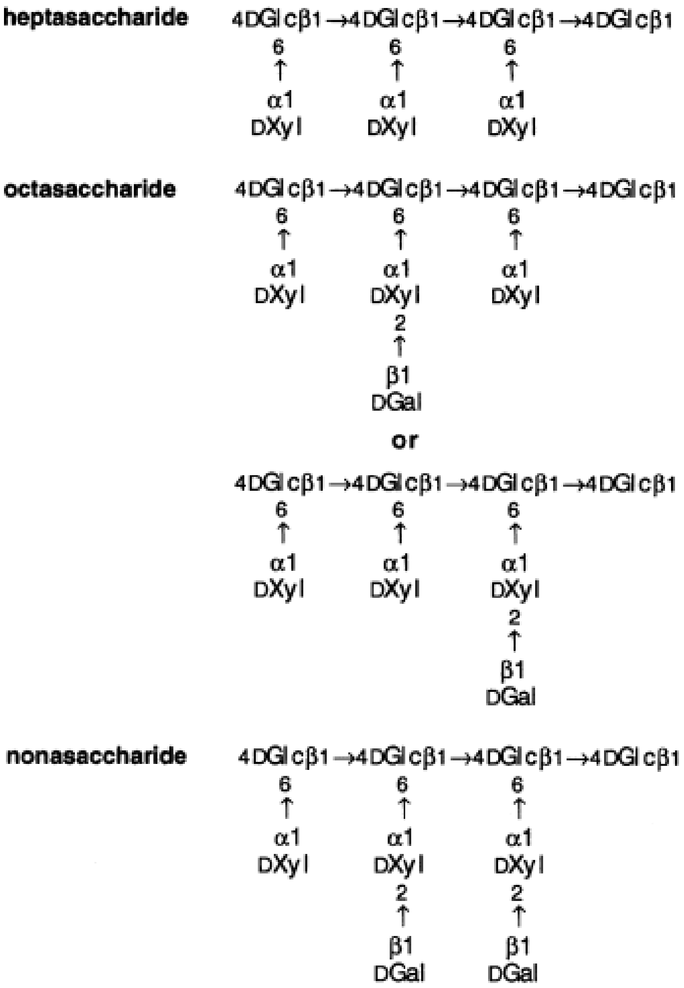


Figure 2.3 Three units of TSP xyloglucan oligomer (Yamanaka et al., 2000)

2.2.2 Tamarind kernel protein (Bhattacharya et al., 1994)

Tamarind kernel proteins are divided into four groups according to the solubility-fractionation. The proteins are fractionated into 19.6% albumin, 21.7% globulin, 3.9% prolamine, and 16.1% glutelins according to their solubility in distilled water (pH 7.0), 5% NaCl solution (pH 7.0), 70% ethanol, and 0.25% NaOH (pH 10.0) solution, respectively. A total of 61.3% protein can be extracted by these solvents, so that the sum of non-protein nitrogen and insoluble proteins accounted for the remaining 38.7%. The amino-acid composition of the tamarind seed protein is showed in Table 2.2. The proportions of hydrophobic amino acids (alanine, valine, leucine, isoleucine, and phenylalanine) and hydrophilic amino acids (lysine, histidine, aspartic acid, glutamic acid, and arginine) are 28.2% and 42.3%, respectively.

Table 2.2 Amino-acid composition of the tamarind seed protein (Bhattacharya et al., 1994)

Amino acid	mg/16g N
Lysine	5.96
Histidine	2.01
Arginine	4.20
Aspartic acid	11.59
Threonine	3.75
Serine	7.71
Glutamic acid	18.53
Proline	6.19
Glycine	9.12
Alanine	6.96
Cysteine	0.30
Valine	4.60
Methionine	0.33
Isoleucine	4.12
Leucine	8.21
Tyrosine	1.99
Phenylalanine	4.33

2.2.3 Tamarind kernel oil

The tamarind kernel oil is amber in color, free of smell, and sweet to taste. It can be used for making varnishes, paints, and burning in oil lamps. Tamarind kernel oil contained a relatively large proportion of unsaturated fatty acids (75%) with linoleic acid (56.10%) as the predominant fatty acid. The fatty acids composition and physico-chemical properties of tamarind kernel oil are tabulated in Table 2.3 and 2.4 (Shankaracharya, 1998).

Table 2.3 Composition of fatty acids of tamarind kernel oil (Shankarachaya, 1998)

Fatty acid	(%)
Lauric (12:0)	trace-28.2
Myristic (14:0)	trace-0.4
Palmitic (16:0)	8.7-20.0
Stearic (18:0)	Trace-7.0
Oleic (18:1)	15.0-27.0
Linoleic (18:2)	7.5-56.1
Linolenic (18:3)	1.1-5.6
Arachidic (20:0)	1.9-12.2
Behenic (22:0)	3.0-12.2
Decosenoic (22:1)	0.1
Decosa-tetraenoic (22:4)	5.6
Lignoceric (24:0)	3.0-22.3

Table 2.4 Characteristics of tamarind seed kernel fatty acids (Shankarachaya, 1998)

Characteristics	Value
Saponification value	183.4-192.0
Iodine value	99.1-140.0
Titre	45.8 ^{0}C
Bellier’s turbidity temp.	52 ^{0}C
Specific gravity	0.9194-0.9324
Refractive Index	1.4510-1.4770
Acid value	0.10-12.70
Unsaponifiable matter, %	1.60-3.70
Ester value	164
Sterols	
β-Sitosterol	66-72% of total sterols
Campesterol	16-19% of total sterols
Stigmasterol	11-14% of total sterols

2.2.4 Production of tamarind kernel powder

Tamarind seed consists of an outer hard brown seed coat and seed kernel. The seed coat is completely removed from the kernel by roasting at 320ºC for 30 min and followed by decortications. After roasting, the thermal properties and behavior of the seed coat and the kernel are different resulting in different extent of expansion and contraction that helps in separating the coat from the seed. Temperature controlling and duration of heating should be minimal to prevent color development, decrease in molecular weight, and the consequent decrease in viscosity of the isolated gum or jellose. The decorticated seed is then ground by crushing in an impact grinder followed by pulverizing to the required fineness. TKP tends to deteriorate during storage under humid conditions. Hence, storage in a dry place in moisture proof containers is important. Mixing with 0.5% of sodium bisulphite before packing will prevent enzymatic deterioration.

2.3 Literature reviews

2.3.1 Polysaccharide purification

Deguchi et al. (1966) showed a process for obtaining tamarind seed polysaccharide or jellose. Firstly, tamarind seed was ground into 3 to 16 mesh particles and then bleached by 0.25% sulfurous acid solution. Next, the sulfurous acid solution was filtered off and the ground seed was transferred to 25-fold amount of water. The mixture was heated to 95-100ºC for 2 hours and then hot-filtered. The desired jellose was coagulated from mixture solution by adding sulfate salt such as sodium sulfate, ammonium sulfate, magnesium sulfate, and aluminum sulfate (critical concentration of sulfate: 25% w Na_2SO_4, 46% w $(NH_4)_2SO_4$, 23% w $MgSO_4$, and 26% w $Al_2(SO_4)_3$). After adding sulfate salt, the coagulate rose to the surface of solution as fluffy flakes. The coagulated jellose was then separated by sieving and dried. Finally, the desired jellose, white or grayish color, nor odor, and non-toxic, was obtained.

Gordon et al. (1968) studied a process for recovering polysaccharide from tamarind seed kernel. This process was divided into two extraction steps. In first extraction, TKP was extracted with IPA and extraction conditions were fixed at 78ºC and TKP to solvent ratio of 1:5. After first extraction completed, the slurry mixture was separated by filtration. The recovered filter cake was dried at low temperature to prevent the degradation of polysaccharide and was then used for the second extraction. The dried TKP from the first step was extracted with 25 to 30 times of water. The slurry was warmed to 85ºC for one hour. After that, the dissolved polysaccharide was separated from undissolved TKP by filtration. The pure polysaccharide was then recovered from filtrate solution by either roll drying or alcohol precipitation.

Jones et al. (1978) revealed a process for purifying polysaccharide from tamarind seed kernel. This purification process containing three steps were solvent defatting, grinding, and air classifying. In solvent defatting step, TKP was extracted with ethylene dichloride at room temperature for two hours. Defatted TKP was recovered by centrifugation and allowed to dry. Fine grinding of defatted TKP was performed in a hammer mill capable of reducing the particle size to about 100 microns or less. The powdered TKP was then air classified. The ground TKP can be classified into three fractions, the first fraction being rich in protein, the second fraction being rich in polysaccharides, and the third fraction being rich in mechanical impurities. The purified product, 54% of product yield, contained 86.35% polysaccharide and 3.5% protein.

Sandford et al. (1984) investigated the clarification method of TKP. The production of clarified TKP containing three steps, were strong base treatment, neutralization, and isolation, respectively. Firstly, TKP was mixed for about two hours with 50% NaOH to form aqueous slurry. The pH of the slurry was controlled in range of pH 10 to 14. The slurry was diluted with five volumes of water and stirred for two hours. The diluted slurry was then neutralized with concentrated HCl and the polysaccharide was precipitated with three volumes of absolute IPA. The precipitate was dried for about three hours at 60ºC and milled. If neutralization was omitted, the precipitated product was in the alkali metal salt form.

Jaruwattanayon (2000) produced TSP by using filtration technique. At the beginning, adherence of protein to polysaccharide was found. Thus, ultrasonic wave was applied to TKP in ETOH solution in order to separate protein from polysaccharide. Before pre-treating with ultrasonic, average particle size of powder was 39.67 micrometer (in 50% w/w ethanol solution). After treating with ultrasonic, protein and polysaccharide were dispersed and average particle size of powder decreased to 30.15 micrometer. Finally, protein was removed from polysaccharide by filtration with wiper. Obtained TSP had polysaccharide, protein, and fat of 93.62, 4.84, and 1.54 % w/w, respectively.

Rattanaporn (2001) separated protein from TKP by using rotating filter. Various factors affecting protein separation from TKP by using rotating filter were the concentration of suspension, the pressure across the filter, rotating speed, and the distance between the filter surface and the internal surface of the outer cylinder. The optimal conditions for the filtration of the TKP suspension by using rotating filter were as follows; the suspension concentration of 20 g/l, the filter rotating speed of 1700 rpm, the pressure across the filter of 0.16 bar, and the distance between the filter surface and the internal surface of the outer cylinder of 0.008 m. Under these conditions, the following results were obtained; the filtrate flux of 9354.41 l/m^{3}hr, protein removal of 74.36%, and polysaccharide loss of 40.31%, protein, polysaccharide, and fat content in the product of 9.70%, 66.41%, and 5.10%, respectively and product yield of 29.03% w.

Sangyont (2002) studied protein separation from TKP using protease (neutrase) and ETOH. The suitable condition for enzyme activity, which was at the working temperature of 45ºC, TKP concentration of 40 g/l, and the viscosity of the kernel powder not exceeding than 50 cP, the enzyme was most active at the pH value of 8 and its activity was not affected by agitation speed (200, 400, and 600 rpm). On the contrary, its activity was substantially denatured more than 10% when it exposed to 5% v/v up of ethanol solution. It was found that at agitation speed of 600 rpm, reaction time of 20 minutes, pH of 8, and in the presence of enzyme gave the highest protein separation of 97.051% (cooking basis) or 100.369% (non- cooking basis). The suitable enzyme (43.4 unit/ml) dosage was at 0.01% v/v for 40 g/l of TKP and in 20 min incubation time. Protein separation, comparing with the other researches, the use of enzyme could also reduce the polysaccharide loss in the range of 40-60% due to unchanging in size of the most polysaccharide particle.

Poommarintaravarakul (2003) showed the application of diafiltration and concentration for protein separation from TKP using rotating filter. The production of polysaccharide from TKP suspension in which the neutrase enzyme was added was studied by consecutively applying the concentration and diafiltration techniques. By applying the concentration and diafiltration factors of 1.25 and 5, respectively, the purity of produced polysaccharide, the percentage polysaccharide loss, and the percentage protein removal were 96.71, 62.06, and 97.47%, respectively.

2.3.2 Natural oil extraction

Andriamanantena et al. (1983) revealed the oil content from six samples of tamarind kernel. The seed kernel was extracted with hexane and a mixture of chloroform and methanol and the yield of the oil were 6.0-6.4% and 7.4-9.0%, respectively. Investigation by gas liquid chromatography revealed 15 fatty acids, mainly palmitic (14-20%), stearic (6-7%), oleic (15-27%), linoleic (36-49%), arachidic (2-4%), behedic (3-5%), and lignoceric (3-8%) acids. Seven sterols were separated and quantitatively analyzed by gas liquid chromatography. The main sterols were β-sitosterol (66-72%), campesterol (16-19%), and stigmasterol (11-14%).

Proctor et al. (1996) reported on extraction of rice bran oil with hexane and IPA at ambient temperature. Oil yield obtained by 10 min hexane extraction was 15.21% and 16.28% for IPA. Free fatty acid levels were 2-3% in both solvents. The oil extracted with IPA was significantly more stable to heat-induced than hexane-extracted oil. Antioxidants that were more easily extracted by IPA than hexane may be responsible for the increased stability. Thus, IPA was effective solvent, comparing with hexane, for rice bran oil extraction.

Akaranta et al. (1996) extracted castor oil from castor seeds with a bioresource solvent. The solvents used were feint, a liquid industrial waste from the distillery containing C_2 to C_4 alcohols and water and commercial hexane. The extraction at 69ºC for 6 hours, hexane with a boiling range of 68-70ºC gave a lower yield (84.5%) when compared with yield for dry feint (92.5%) of boiling range 78-94ºC. It was found that oil yield percentage increased with increase in temperature. At higher extraction temperature the solvent viscosity becomes lower and diffusivities of the solute (oil) and solvent become larger resulting in high extraction rate. Results of the chemical and physical properties of the extracted oils indicated that feint could serve as an effective substitute solvent for commercial hexane in the extraction of castor oil.

Kuk et al. (1998) studied cottonseed oil extraction with a new solvent system: isohexane and alcohol mixtures. A solvent system, consisting of isohexane and 5 to 25% alcohol, either ETOH or IPA, was examined for oil extraction from cottonseed. The experimental results indicated that the percentage of extracted oil obtained by isohexane and alcohol mixtures extraction was around 26.2-29% and 27% for absolute *n*-hexane. From these results, the addition of alcohols to isohexane had not affected the extraction capability of solvent mixtures for extracting cottonseed oil. Therefore, this new solvent system was effective solvent in extracting oil same as *n*-hexane.

Zhang et al. (2002) showed extraction of cottonseed collets with IPA. Extractability of collets with 95% IPA was better than that of flakes. The residual oil content and solvent hold-up of the collets were 1.6 and 33.0% compared to 4.5 and 53.2% for the flakes, respectively. The aqueous IPA extraction proceeded more slowly and showed a lower oil carrying capacity than that of hexane. The residual oil content of the cottonseed collets extracted with hexane was 1.2% while the solvents containing 97, 93, and 88% IPA resulted in 1.5, 1.9, and 2.4%, respectively. Reduction in water content improved the extraction efficiency of IPA.

Kwiatkowski et al. (2002) extracted corn oil from whole ground corn using ethanol as the solvent. The yield of oil was measured as a function of temperature, extraction time, solvent-to-solids ratio, and ethanol concentration. Optimal conditions were a solvent-to-solids ratio of 4 ml/g corn, an absolute ETOH, an extraction time of 30 min, and a temperature of 50ºC. The extraction efficiency under these conditions was 70%, equivalent to 3.3 g oil/100 g corn. A three-stage extraction, where the same corn was extracted with fresh ETOH, resulted in oil recovery of 93%, equivalent to 4.5 g/100 g corn. When anhydrous ETOH was used to repeatedly extract fresh corn, moisture was absorbed linearly by ETOH from the corn in successive stages, which, in turn, decreased oil yield and increased nonoil components in the extract.

Gandhi et al. (2003) reported on alternative solvents for the extraction of soybean oil. It was found that *n*-heptane, *n*-propanol, IPA, and ETOH were equally effective in the extraction of soybean oil when compared with n-hexane. The aqueous solvent mixtures were also efficient in extracting the oil. The advantage with the higher aqueous content solvent was the lower amount of organic solvent required, however, more energy would be required to remove the water from solvent. At 8 hours of extraction, the aqueous solvent systems containing either 90:10 and 80:20 *n*-propanol, IPA or ETOH gave oil extraction percentage in range of 92.0-97.8% and 98.0-99.5% for pure solvents.

Amarasinghe et al. (2004) investigated the pre-treatment methods for rice bran oil stabilization and the effect of pellet size on rice bran oil extracted yield by using hexane as the solvent. The pre-treatment method composed of hot air drying, steaming, refrigeration, sun drying, fluidized bed drying, and chemical stabilization. Compared with the oil extracted from untreated bran, all the pre-treatment methods showed a reduction in free fatty acid content of the oil. It was found that steaming was the most suitable method of bran pre-treatment since the method controlled the free fatty acid content and enhanced the oil extractability. In fact, steaming releases the oil in the grain and results in outward migration of the oil. The extraction process was more efficient with small size pellets due to the increase in surface area available for mass transfer. The mass transfer coefficient for rice bran oil in hexane using 5 mm height x 8 mm diameter of rice bran was 4.36 x 10^{-4} ms^{-1}.

Franco et al. (2007) studied extraction of oil from rosehip seed (*Rosa rubiginosa*) with ETOH as solvent. The influence of temperature and water content on the oil solubility was also analyzed. Oil solubility dramatically decreased with the water present in ETOH (from 92% to 99.9%). The influence of temperature was high for absolute ETOH (99.9%), the values varying between 40 g/l at 20ºC and 140 g/l at 60ºC, whereas decreased with ETOH percentage, being 15 g/l at 20ºC and 45 g/l at 60ºC for 96%. Although solubility highly increases with temperature, it always will be higher in hexane, being necessary a high liquid to solid ratio in ETOH extraction to achieve the same performance. The extraction with ETOH can get other advantages like those environmental.

Durling et al. (2007) studied effects of particle size, temperature, extraction time, solvent-to-sage ratio and the ethanol–water ratio on the extraction of essential oil from dried sage (*Salvia officinalis*). The extractions were carried out in a stirred tank. The optimal extraction conditions were a particle diameter size of 1 mm, an extraction temperature of 40ºC, a solvent-to-sage ratio of 6:1 (v/w), a hydroalcoholic solution of 69% wt ETOH, and an extraction time of one hour. Under these conditions gave 42% recovery of essential oil. The stirred tank extraction process was scaled up by a factor of 100 (1 kg of sage was extracted per trial) at optimal conditions. Extraction yield result at an industrial scale was found to be in good agreement with those at a laboratory scale that was 39.8% recovery.

Liauw et al. (2008) investigated neem oil extraction from Neem seed (*Azadirachta indica A. Juss*) with *n*-hexane and ETOH. Effects of particle size, temperature, and type of solvent on oil yield and oil quality were studied. The maximum yields obtained from neem oil extraction were 44.29% for *n*-hexane and 41.11% for ethanol at 50°C and 0.425-0.71 mm particle size. Based on psycho-chemical characteristics analysis, increasing temperature decreased iodine value but increased saponification, acid, and peroxide value which means increasing temperature increased neem oil yield but decreased neem oil quality.

CHAPTER III

THEORY

3.1 Liquid-solid leaching (Geankoplis, 1995)

Many biological, inorganic, and organic substances occur in a mixture of different components in a solid. In order to separate the desired solute constituent or remove an undesirable solute component from the solid phase, the solid is contacted with a liquid phase. The two phases are intimate contact and the solutes can diffuse from the solid to the liquid phase, which causes a separation of the components originally in the solid. This process is called “liquid-solid leaching or leaching”. It is an extensively used to recover many important food and biological components such as sucrose in cane or beets, lipids from oilseeds, proteins in oilseed meals, phytochemicals from plants, and functional hydrocolloids from algae, among others. Leaching may also be used to remove undesirable contaminants and toxins presented in foods and feeds.

3.1.1 Preparation of solids for leaching

Biological materials are cellular in structure and the soluble constituents are generally found inside the cells. The rate of leaching may be comparatively slow because the cell walls provide another resistance to diffusion. However, to grind the biological material sufficiently small to expose the contents of individual cells is impractical. Sugar beets are cut into thin wedge-shaped slices for leaching so that the distance required for the water solvent to diffuse to reach individual cells is reduced. The cell walls of soybeans and many vegetable seeds are largely ruptured when the original materials are reduced in size to about 0.1 mm to 0.5 mm by rolling or flaking. Cells are smaller in size, but the walls are ruptured and the vegetable oil is easily accessible to the solvent. In figure 3.1 (Aguilera, 2003) shows the architectural arrangements of cells associated with four pretreatments. The basic structure considers that all cells are intact and completely surrounded by a thick cell wall (A). Enzymatic treatment with a cocktail of cell wall–degrading enzymes may pierce the wall of outer cells at different places, leaving openings that connect the cytoplasm directly to the solvent (B). A third situation may be that induced by blanching, resulting in degradation of the cell wall membrane (and possibly the cell wall itself), diminishing the overall internal resistance for transport of solute as most cells become interconnected (C). Another conceivable architecture is that of a totally connected cytoplasm and broken outer cells exposed to the solvent due to extensive physical destruction of the tissue (D). In all cases, the proportion of cell wall material to cytoplasm is the same.

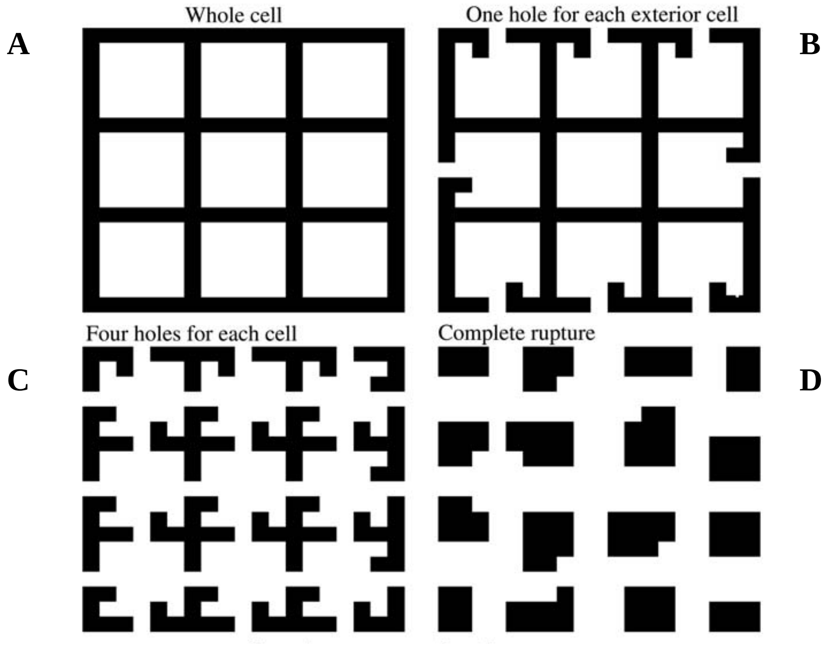


Figure 3.1 Architectural arrangement of cells of pickled cucumbers subject to different treatments (Aguilera, 2003)

3.1.2 The main steps in leaching

In the leaching of soluble materials from inside a particle by a solvent, a series of phenomenological steps have to occur during the period of interaction between solute-containing particles and solvent affecting the separation as represented schematically in figure 3.2. These include:

1. The solvent transfers from the bulk solvent solution to the surface of the solid.
2. The solvent penetrates or diffuses into the solid.
3. The solute dissolves into the solvent.

4. The solute then diffuses through the solid solvent mixture to the surface of the particle.
5. The solute is transferred to the bulk solution.

In general, the transfer rate of the solvent from the bulk solution to the solid surface is quite rapid, and the transfer rate of the solvent into the solid can be somewhat rapid or slow.

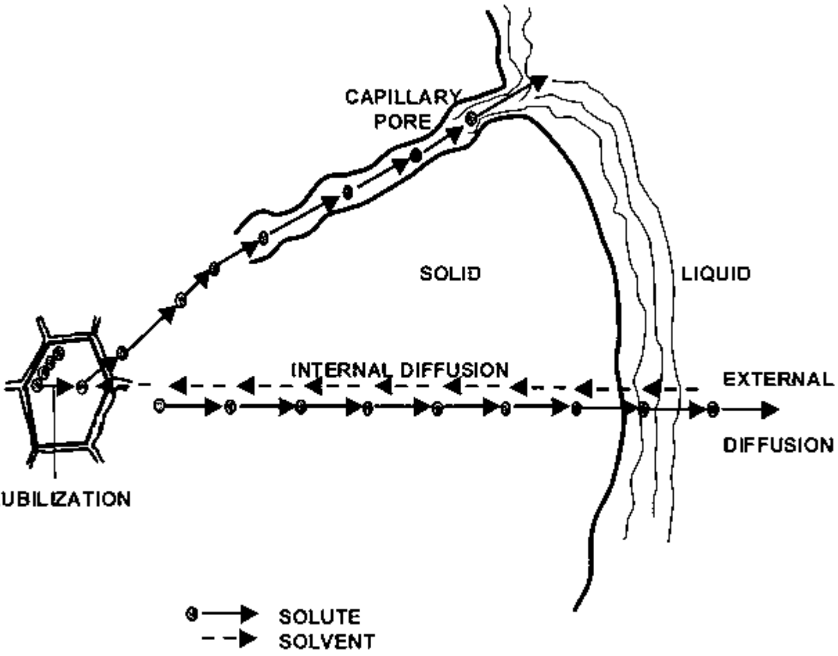


Figure 3.2 Scheme of the main steps in solvent extraction of solid food particles (Aguilera, 2003)

The diffusion rate of the solute through the solid and solvent to the solid surface is often the controlling resistance in an entire leaching process and can depend on a number of different factors. If the solid is made up of an inert porous solid structure with the solute and solvent in pores in the solid, the diffusion through the porous solid can be described by an effective diffusivity. The void fraction and tortuosity are needed. The resistance to mass transfer of the solute from the solid surface to the bulk solvent is in general quite small compared to the resistance to diffusion within the solid itself.

3.1.3 Extraction as a diffusion process (Aguilera, 2003)

Molecular diffusion is the process by which molecules are transported from one part of the system to another by random movement as a result of a concentration gradient. In leaching the interior of the solid cannot be agitated and turbulence is unlikely to occur in small capillaries and pores, leaving molecular diffusion as the main transport mechanism within the solid phase. Solvent extraction may be considered as a diffusion process in the liquid (fluid) state since solute transfers, even inside a solid, exists as a dilute solution. In some cases, solvent influx may occur due to pressure gradients due to capillary forces or by mechanical relaxation of the cellular matrix.

Fick's first law is useful for defining a diffusion coefficient or diffusivity (*D*). It simply establishes that under steady-state conditions. The unidirectional flux of solute A in the *r* direction is directly proportional to the diffusivity of the solute, to the area traversed by the flux and to the gradient of solute concentration between two points, expressed in terms of absolute concentration (*dc/dr*) or fraction (*dx/dr*). Fick's first law describes diffusion referred to a fixed coordinate system and for the unidirectional case, is expressed in Eq. 3.1.

$$N_A = \frac{\overline{N}_A}{A} = -cD\frac{dx_A}{dr} = -D\frac{dc_A}{dr} \qquad (3.1)$$

where N_A = the flux in mass per unit time and unit area

$\overline{N_A}$ = the rate of extraction (mass/time)

r = the direction of flow

A = the area across which diffusion occurs

c = the total concentration (mass/volume)

In practical situations and for short times, unsteady or nonstationary conditions exist and the concentration of solute varies with time (*t*) and position (*r*) inside the solid. In such cases, Fick's second law (or the diffusion equation) is applied and expressed in Eq. 3.2.

$$\frac{dc_A}{dt} = D\frac{\partial^2 c_A}{\partial^2 r^2} \qquad (3.2)$$

The flow of solute is directly proportional to the change of the concentration gradient with position. So, when the gradient is constant (i.e., linear concentration profile), then $\partial c_A/\partial t = 0$, meaning that steady-state conditions exist and Eq. 3.1 is applied.

3.1.4 Rate of leaching when dissolving a solid

When a material is being dissolved from the solid to the solvent solution, however, the rate of mass transfer from the solid surface to the liquid is the controlling factor. There is essentially no resistance in the solid phase if it is a pure material. The equation for this can be derived as follows for a batch system. The following can also be used for the case when diffusion in the solid is very rapid compared to the diffusion from the particle. The rate of mass transfer of the solute A being dissolved to the solution of volume V is

$$\frac{\overline{N_A}}{A} = k_L(c_{AS} - c_A) \tag{3.3}$$

where $\overline{N_A}$ = the rate of solute A extraction (kg/s)

A = surface area of particles (m^2)

k_L = a mass-transfer coefficient (m/s)

c_{AS} = the saturation solubility of the solid solute A in the solution (kg/m^3)

c_A = the concentration of A in the solution at time (kg/m^3)

By a material balance, the rate of accumulation of A in the solution is equal to Eq. 3.3 times the area A.

$$\frac{Vdc_A}{dt} = \overline{N_A} = Ak_L(c_{AS} - c_A) \tag{3.4}$$

Integrating from t = 0 and $c_A = c_{A0}$ to t = t and $c_A = c_A$,

$$\int_{c_{A0}}^{c_A} \frac{dc_A}{c_{AS} - c_A} = \frac{Ak_L}{V} \int_{t=0}^{t} dt \tag{3.5}$$

$$\frac{c_{AS} - c_A}{c_{AS} - c_{A0}} = e^{-(k_L A/V)t} \tag{3.6}$$

3.1.5 Solvent selection

Solvent selection is based on several properties:

1. Solubility of the specific compound (or compounds) in the solvent.
2. Recovery, since the solvent will be reused in subsequent extractions. If distillation or evaporation is used, the solvent should not form azeotropes and the latent heat of vaporization should be small. Removal of the solvent from the miscella (and from the spent solids) can pose serious problems if the residual level of the solvent must be minimized.
3. Interfacial tension and viscosity. The solvent should be capable of wetting the solid matrix and its viscosity should be sufficiently low so it can flow easily. Wettability is also important if the solvent must penetrate through pores and capillaries in the solid matrix.
4. Ideally, the solvent should be nontoxic, stable, nonreactive, nonflammable, harmless to the environment, and cheap.

3.2 Agitation and mixing

Agitation is means whereby mixing phases can be accomplished and by which mass and heat transfer can be enhanced between phases or external surfaces. The operation of agitation, which includes mixing as a special case, is now well established as an important and in a wide variety of chemical processes. Specifically, agitators are applied to three general classes of problems:

1. To produce static or dynamic uniformity in multicomponent multiphase systems.
2. To facilitate mass or energy transfer between the parts of a system not in equilibrium.
3. To promote phase changes in multicomponent system with or without a change in composition.

Mixing in tanks is an important area when one considers the number of processes, which are accomplished in tanks. Essentially, any physical or transport process can occur during mixing in tanks. Qualitative and quantitative observations, experimental data, and flow regime identifications are needed and should be emphasized in any experimental pilot studies in mixing. Fluid mechanics and geometry are key points to understand mixing. The fluid mechanics transports the material about the tank, whereas the geometry determines the fluid mechanics. In fact, the geometry is so important that the processes can be considered geometry specific. Solid suspension is very much dependent upon the shape of the tank bottom; liquid-liquid dispersion depend upon the geometry of the impeller; blending, upon the relative size of the tank to the impeller; and power draw, upon the impeller geometry. Mixing efficiency in a stirred tank is affected by various numbers of parameters such as baffles, impeller speed, impeller type, clearance, tank geometry, solubility of substance, eccentricity of the impeller. The standard mixing tank is shown in figure 3.3. The geometric proportions of the agitation system which are considered as a typical "standard" are shown below.

$$\frac{D_a}{D_t} = 0.3 - 0.5 \qquad \frac{H}{D_t} = 1 \qquad \frac{C}{D_t} = \frac{1}{3} \qquad \frac{W}{D_a} = \frac{1}{5} \qquad \frac{J}{D_t} = \frac{1}{12}$$

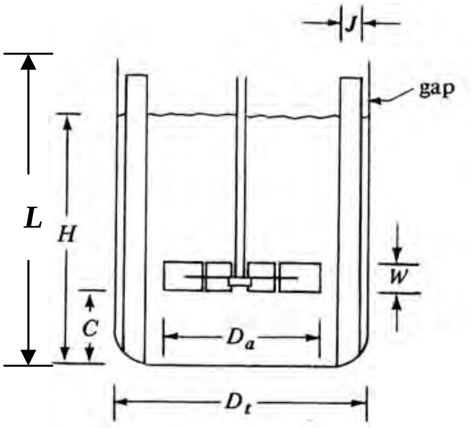


Figure 3.3 Dimensions of mixing tank (Geankoplis, 1995)

A vortex is produced owing to centrifugal force acting on the rotating liquid. If vortex reaches the impeller severe air entrainment occurs. The depth and the shape of the vortex depend on impeller and vessel dimensions as well as on rotational speed. Baffles are flat vertical strips set radially along the tank wall. Baffles avoid vortex formation. In baffled tanks, a better concentration distribution throughout the tank and therefore improvement in the mixing efficiency is achieved. The larger the width of the baffles, the better is the mixing to some extend. In the un-baffled vessel with the impeller rotating in the center, centrifugal force acting on the fluid raises the fluid level at the wall and lowers the level at the shaft. Flow patterns can be changed according to the type of impellers, and fall into three categories: axial, radial and tangential flow. The flow patterns are shown in figure 3.4.

Axial Flow: Axial flow discharge coincides with the axis of impeller shaft, so when the impeller operates in a down pumping mode, the flow impinges on the bottom of the tank and spreads out in all directions toward the wall. The flow rises along the walls up the liquid surface and is pulled back to the impeller. Since axial flow impeller produce only one loop, fluids mix faster and blend time is reduced compared to radial flow impellers. The fluid does not take sharp turns near impellers and because of this, power consumption is less than that of radial flow impellers at the same speed and same the diameter.

Radial Flow: Radial Flow discharge is parallel to the impeller radius toward to the vessel wall. If a radial impeller is not positioned close to the surface or the tank bottom, the flow will split into two streams upon impinging on the tank wall. Each flow loop will continue along the wall and then return to impeller.

Tangential Flow: A tangential flow pattern is naturally induced by swirling or vortexing flow that fluids assume in an unbaffled tank. Tangential flow patterns offer very little mixing because the velocity gradients are very small.

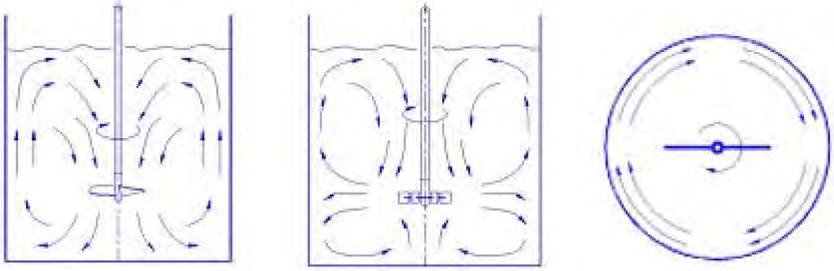


Figure 3.4 Flow patterns (www.wellman.co.th)

3.2.1 **Agitator scale-up** (Geankoplis, 1995)

In the process industries experimental data are often available on a laboratory-size or pilot-unit-size agitation system and it is desired to scale-up the results to design a full-scale unit. Geometric similarity is important and simplest to achieve. Kinematic similarity can be defined in terms of ratios of velocities or of times. Dynamic similarity requires fixed ratios of viscous, inertial, or gravitational forces.

Although geometric similarity is achieved, dynamic and kinematic similarity cannot often be obtained at the same time. Therefore, it is often up to the designer to rely on judgment and experience in the scale-up. The details of scale-up procedure are shown below.

1. Calculate the scale-up ratio R. Assuming that the original vessel is a standard cylinder with $D_{T1} = H_1$, the volume V_1 is

   $$V_1 = \left(\frac{\pi D_{T1}^2}{4}\right)(H_1) = \left(\frac{\pi D_{T1}^3}{4}\right) \tag{3.7}$$

   Then the ratio of the volumes is

   $$\frac{V_2}{V_1} = \frac{\pi D_{T2}^3 / 4}{\pi D_{T1}^3 / 4} = \frac{D_{T2}^3}{D_{T1}^3} \tag{3.8}$$

   The scale-up ratio is then

   $$R = \left(\frac{V_2}{V_1}\right)^{1/3} = \frac{D_{T2}}{D_{T1}} \tag{3.9}$$

2. Using this value of R, apply it to all of the standard dimensions to calculate the new dimensions. For example,

   $$D_{a2} = RD_{a1} \text{ , } J_2 = RJ_1$$

3. Then a scale-up rule must be selected and applied to determine the agitator speed N_2 to use to duplicate the small-scale results using N_1. This equation is as follows:

   $$N_2 = N_1\left(\frac{1}{R}\right)^n = N_1\left(\frac{D_{T1}}{D_{T2}}\right)^n \tag{3.10}$$

   When n = 1 for equal liquid motion
   n = 3/4 for equal suspension of solids
   n = 2/3 for equal rates of mass transfer (which is equivalent to equal power per unit volume).

4. Knowing N_2, the power required can be determined using Eq. 3.11.

   $$N_p = \frac{P}{D_a^5 \, N^3 \rho} \tag{3.11}$$

CHAPTER IV

MATERIALS AND METHODS

4.1 Chemicals

1. Tamarind kernel powder: G.M. Ichihara Co., Ltd., Thailand.
2. Tamarind seed polysaccharide or Sobigum: G.M. Ichihara Co., Ltd., Thailand.
3. 95% V/V food grade ethanol: SR. Lab, Thailand.
4. 99.9% V/V isopropanol: QRec.
5. 99.9% V/V ethanol: MERCK.
6. Sodium hydroxide: J.T. Baker.
7. Sulfuric acid: J.T. Baker.
8. Boric acid: QRec.
9. Phenol: Panreac.
10. Selenium reagent mixture: MERCK.
11. Petroleum ether: Fisher Scientific.

4.2 Equipments

1. Spectrophotometer: Spectronic 20 Genesys, Spectronic instruments, USA.
2. Scanning electron microscopy: JSM-5410LV, JOEL, Japan.
3. Distillation Unit Nitrogen analyzer: BUCHI 339
4. Gas chromatography: 7890S, Agilent Technologies, USA.
5. Rotary evaporator: N-1000, EYELA, Japan.
6. Water bath: Julabo, Labartechnik GMBH, Germany.
7. Viscometer: B8L, Toki, Japan.
8. Particle size analyzer: LS 230, COULTER CORPORATION, USA.
9. Peristaltic pump: 505U, WATSON MARLOW, England.
10. Closed mixing tank 0.5 and 10 liter. (Figure 4.1 and 4.2)
11. Soxhlet apparatus.

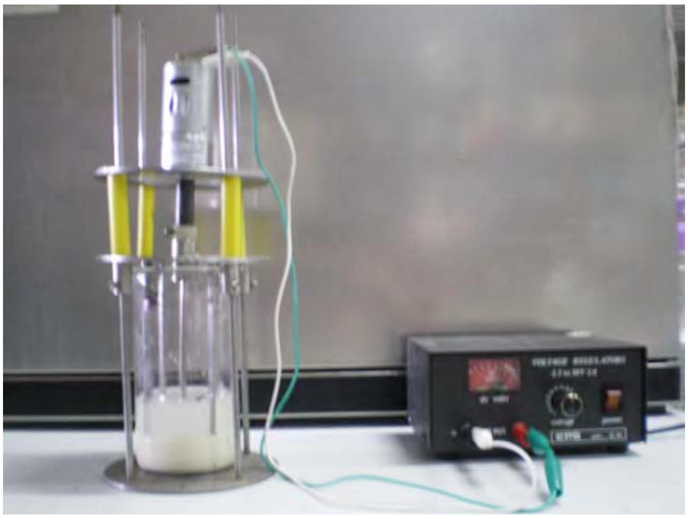


Figure 4.1 0.5-liter closed mixing tank

Figure 4.2 10-liter closed mixing tank

4.3 Methods

4.3.1 Methods for batch extraction

Fresh TKP was added to solvent in closed system mixing tank with working volume 0.5 liter. The mixing tank was put into the water bath with temperature controlling at 35 ^{0}C and stirring at 400 rpm. The series of experiments were conducted and each trial was terminated at various extraction times. The defatted TKP was separated from solvent by a vacuum filter using the Whatman filter paper No.1 and subsequently allowed to dry at room temperature. Filtrate was collected and concentrated with rotary evaporator to acquire the tamarind kernel oil. The acquired tamarind kernel oil was further dried in oven dryer to remove residual solvent and the fatty acid composition of oil was analyzed by gas chromatography. The content of fat, polysaccharide, and protein in final product were analyzed. Diagram for fat extraction was shown in figure 4.3.

The suitable condition for fat removal was evaluated by considering the percentage of fat removal, product yield, polysaccharide and protein content in product and product viscosity.

Several extraction experiments were carried out to determine the effect of solvent type, TKP to solvent ratio, particle size of TKP, and extraction time on fat removal and product quality. The conditions tested were summarized in Table 4.1.

Table 4.1 Extraction process variables to be studied

Variables	**Condition**
Solvent type	95% ETOH, 99.9% ETOH, 99.9% IPA, and 1:1-4:1 v/v ratio of 95% ETOH to 99.9% IPA
TKP to solvent ratio	1:3-1:6 g/ml
Particle size of TKP	0.029-0.032, 0.553-0.598, and 0.843-0.886 mm
extraction time	1-120 min

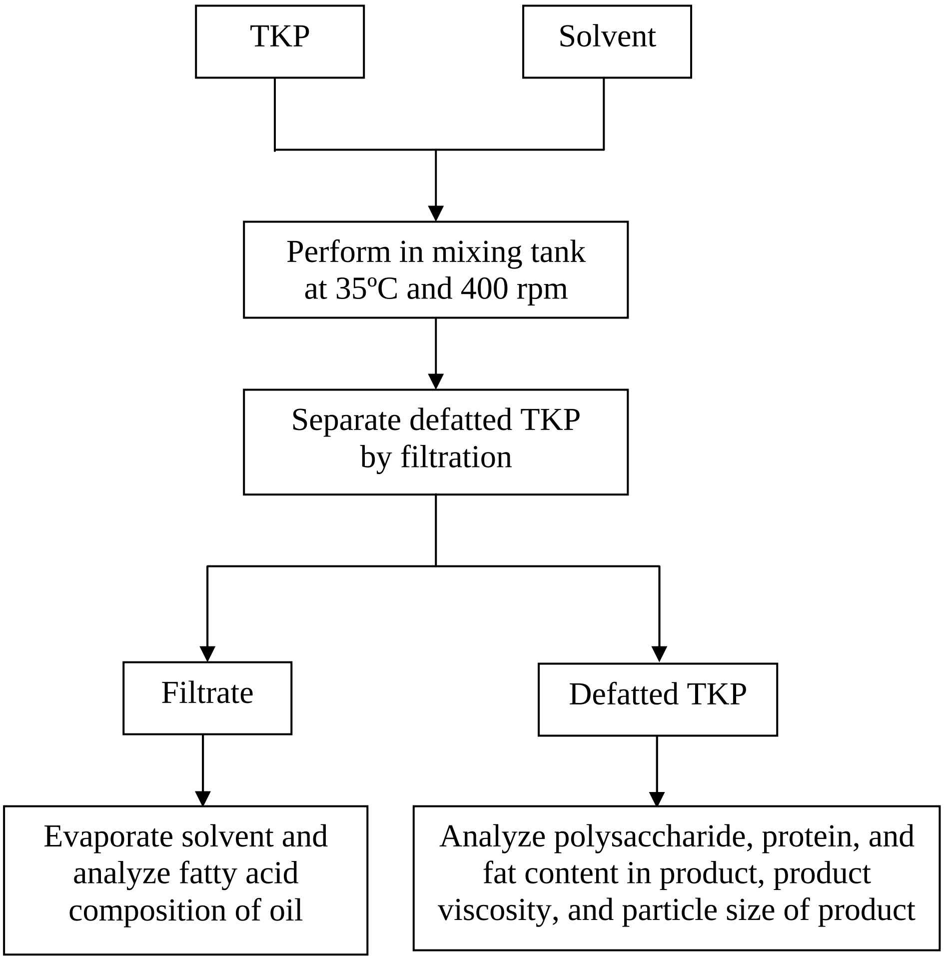


Figure 4.3 Diagram for fat extraction from TKP

4.3.2 Methods for multiple-batch extraction using recycled solvent and fresh TKP

Multiple-batch extraction was performed in closed mixing tank with working volume of 0.5 liter. The filtrate from the first extraction stage was reused to extract fresh TKP in next stage. The amount of TKP for each stage was maintained at 1:3 g/ml TKP to solvent ratio. Diagram for multiple batch extraction was shown in figure 4.4.

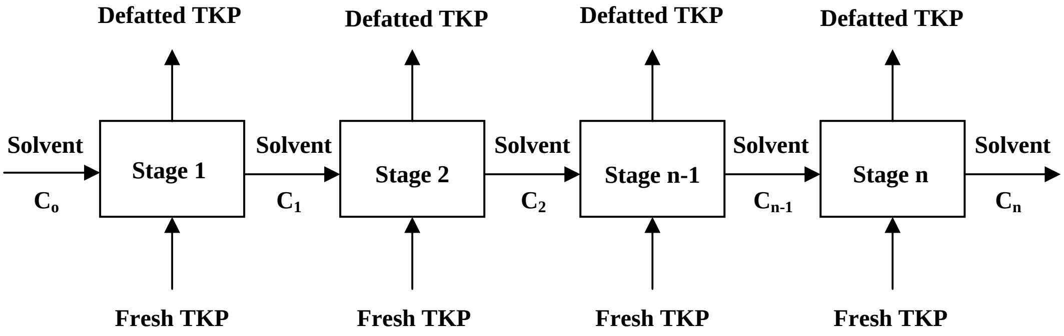


Figure 4.4 Diagram for multiple-batch extraction

4.3.3 Scale-up of mixing tank

The mixing tank of extraction process was scaled up by a scale-up ratio of 2.71. For power per unit volume constant, agitation speed for 10-liter mixing tank was 205 rpm. The temperature of system was controlled at 35ºC by the circulating water through tank jacket. The suitable condition of each TKP size from laboratory scale was applied for extraction in semi-pilot scale with working volume of 10 liter. The product from extraction was harvested through port at tank bottom. The fat removal yield resulted from semi-pilot scale were compared with laboratory scale.

4.4 Methods for sample analysis

4.4.1 Protein analysis

Protein content in TKP was determined by Kjeldahl method (AOAC.920.87). Samples were digested with concentrated sulfuric acid by boiling. After that, distillation and titration steps were performed in distillation unit nitrogen analyzer (BUCHI 339).

4.4.2 Polysaccharide analysis

The quantity of polysaccharide in TKP was measured by Phenol-sulfuric method through a corresponding standard curve. 5% w/v phenol and concentrated sulfuric acid was added to sample. After the reaction was stopped, the color intensity was measured by spectrophotometer (Spectronic 20 Genesys, Spectronic instruments, USA) at 483 nm.

4.4.3 Fat analysis

The amount of fat in TKP was analyzed by Soxhlet extraction method (AOAC. 920.39).

4.4.4 Viscosity analysis

The viscosity of dispersed sample in water at 2% w/v was measured by viscometer (B8L, Toki, Japan) at 25ºC.

4.4.5 Particle size analysis

The particle size of TKP samples was measured by particle size analyzer (LS 230, COULTER CORPORATION, USA).

4.4.6 Gas chromatography analysis of tamarind kernel oil extracts

The fatty acid composition of extracted tamarind kernel oil was analyzed by using gas chromatography (GC). Agilent 7890S gas chromatograph equipped with column HP-88 100 m x 0.25 mm x 0.20 µm (J&W) and FID detector was used for the analysis. Tamarind kernel oil was converted into its methyl esters. The amount of each sample injected was 1.0 µl. Helium, at a constant flow rate of 0.8 ml/min, was used as the carrier gas and a spilt/spiltless injector was used with the split ratio of 100:1. The injector temperature was 250 ^{0}C and the detector temperature was 285 ^{0}C. The column temperature program was as follow: the initial column temperature was 100 ^{0}C, which was maintained for 4 min, then increased from 100 ^{0}C to 240 ^{0}C at 3 ^{0}C/min, and held for 20 min at 240 ^{0}C.

4.4.7 Scanning electron microscopy (SEM) analysis

After the extraction of fat, the TKP was collected and dried in oven dryer for SEM analysis. Sample particles were fixed on the rod brass and sputtered with gold. The microstructure and the surface character of TKP samples were observed and recorded on the scanning electron microscope (JSM-5410LV, JOEL, Japan).

4.5 Calculation parameters

- Percentage of fat removal (dry basis)

$$Fat\ removal\,(\%) = \frac{initial\ fat\ (g\,/\,g\,TKP) - residual\ fat\ (g\,/\,g\,TKP)}{initial\ fat\ (g\,/\,g\,TKP)} \times 100$$

- Percentage of product yield (dry basis)

$$Yield\,(\%) = \frac{product\,(g)}{raw\ material(g)} \times 100$$

CHAPTER V

RESULTS AND DISCUSSION

This chapter presents the experimental results dealing with fat extraction from TKP using organic solvent in mixing tank. Firstly, the composition of TKP as raw material and its properties were investigated. Secondly, the effects of solvent types, TKP to solvent ratios, particle sizes, and extraction times on fat removal were investigated in 0.5-liter mixing tank to determine the suitable extraction condition. Additionally, multiple-batch extraction using reused solvent and fresh TKP was also carried out in order to determine the saturation solubility of fat in each solvents and number of stage of reusable solvent. Thirdly, the fat extraction of three particle sizes was carried out in 10-liter mixing tank. Moreover, the performance of laboratory scale extraction was compared to that of semi-pilot scale extraction. Finally, the composition of tamarind kernel oils was determined.

5.1 Properties of TKP

TKP as raw material was obtained from G.M. Ichihara Co., Ltd., Thailand and also analyzed for the initial composition. The composition of TKP in dry basis was shown in Table 5.1. TKP was rich in polysaccharide as a viscosity enhancer and contained a little fat. Although, the fat content in TKP was low but it poses the problem of product sticky and rancidity. Therefore, it should be removed firstly.

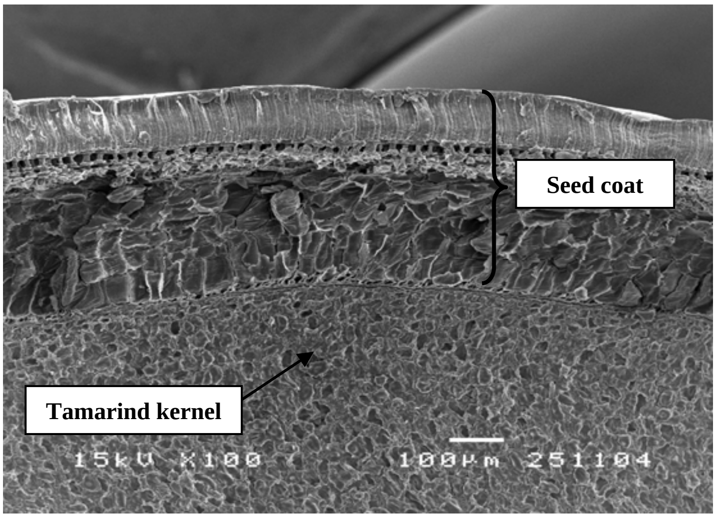
Table 5.1 Composition of TKP

Constituents	Percentages by weight
Polysaccharide	70.47
Protein	18.92
Fat	8.05
Others	2.56

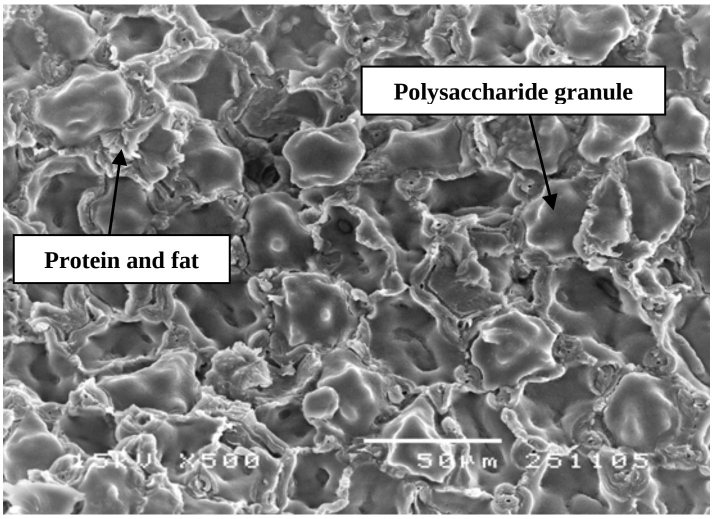
The viscosity of raw material TKP measured at 2% w/v and 25ºC gave the viscosity of 540 cp. Total porosity of tamarind kernel was 6.41% and it had the pore diameter of 0.008 micron. The picture of TKP particles which used in this study was shown in figure 5.1. The diameter of small, medium, and large TKP particles were 0.029-0.032, 0.553-0.598, and 0.843-0.886 mm, respectively.

Figure 5.1 Tamarind kernel powder (a) small-size TKP (b) medium-size TKP and (c) large-size TKP

The microstructure of cross section of tamarind seed was shown in figure 5.2. Tamarind kernel was enclosed by the seed coat (Figure 5.2 a) and constituted of the polysaccharide granules embedded in protein and fat matrix (Figure 5.2 b).



(a)



(b)

Figure 5.2 Microstructure of cross-section of raw material seed

(a) at 100x (b) at 500x

5.2 Effects of solvent types on fat removal

The extraction performance of solvent was generally determined by the solubility characteristics between an extracting agent and a solute. Figure 5.3 shows the fat removal percentage of different solvents that obtained from small size TKP at 3 min. The results showed that the fat removals obtained from 99.9% IPA and 95% ETOH were around 97.62 and 41.58%, respectively. To find the explanation, the extraction using 99.9% ETOH was also carried out in order to compare fat solubilization in different solvents. The result showed that absolute ETOH had the same performance as absolute IPA. This means that the presence of 5% water in ETOH solution affected the solubility characteristics of fat in solvent due to the high polarity of water. The water content in ethanol reduced extraction efficiency because the fat can be solubilized lower in aqueous ethanol. This is in good agreement with previous work data published by Kwiatkowski et al., (2002), Zhang et al., (2002), and Franco et al, (2007) which showed a drastic loss of extraction efficiency for ETOH and IPA in the case of water presence. Absolute ETOH and IPA were effective solvents but absolute ETOH was more harmful. If this solvent remains in product, it has effects on consumer’s health. Focusing on the price of solvents, absolute IPA is more expensive than 95% ETOH (APPENDIX D). Therefore, the co-solvent between 95% ETOH and 99.9% IPA was used in order to reduce solvent cost. Moreover, figure 5.3 also shows the effect of ETOH:IPA ratios on fat removal. The percentages of fat removal obtained from 1:1, 2:1, 3:1, and 4:1 v/v of ETOH:IPA ratios were 96.53, 79.26, 66.90, and 57.97%, respectively. The ratio of ETOH:IPA at 1:1 v/v gave higher fat removal percentage than other ratios due to the effect of water content in ethanol solution. Fat removal percentage yielded from ETOH:IPA at 1:1 v/v extraction was about 1% lower than that obtained from 99.9% IPA. The test results indicated that this new solvent system not only was an effective solvent in removing fat from kernel but also could reduce solvent cost. Thus, the use of co-solvent for extraction was more preferable than that used single alcohol. The alternative solvents, 99.9% IPA, 95% ETOH, and ETOH:IPA at 1:1 v/v, were chosen for next experiments.

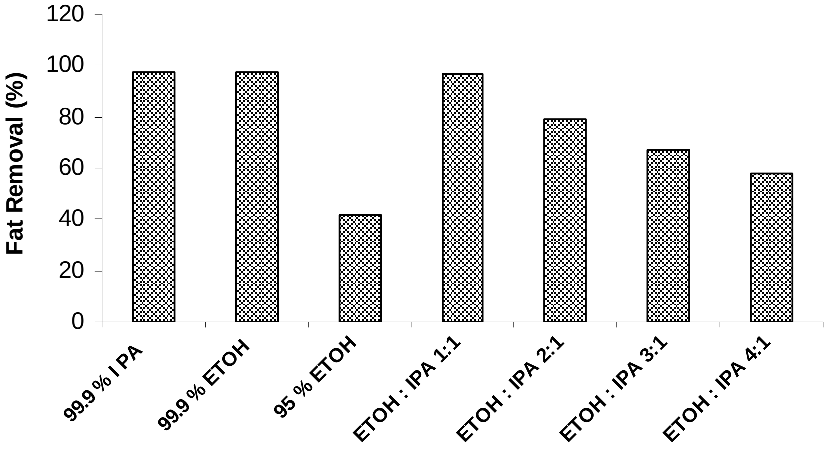


Figure 5.3 Effects of solvent types on fat removal percentages at 35ºC, 400 rpm, 3 min, and TKP:solvent ratio of 1:3 g/ml

5.3 Effects of TKP to solvent ratios on fat removal

The driving force for extraction is the concentration gradient of the solute on solid surface and that in bulk solution. The more solvent volume usage caused the more fat removal from TKP due to favorable mass transfer. In this section, the percentage of fat removal as a function of TKP to solvent ratio was studied by using small-size TKP. Thus, the suitable TKP to solvent ratio for each of solvent was determined in order to eliminate the problem of solubility limitation. The results from figure 5.4 indicate that during the first 3 min of extraction, the initial extraction rate increased as the amount of ETOH increased due to highly different concentration. In case of IPA and 1:1 v/v co-solvent extractions (Figure 5.5 and 5.6), the initial extraction rate at 1:3, 1:4, 1:5, and 1:6 g/ml TKP to solvent ratio gave the similar trend of extraction and gave higher initial extraction rate than ETOH. Therefore, an increase in amount of IPA or 1:1 v/v co-solvent could not affect the fat removal while an increase in amount of ETOH affects the fat removal. This is due to fat solubility in ETOH is lower than IPA and 1:1 v/v co-solvent.

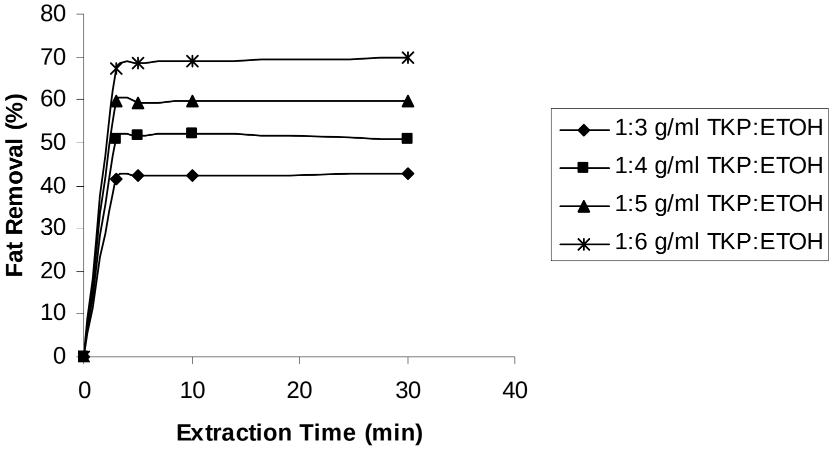


Figure 5.4 Effects of TKP to 95% ETOH ratios and extraction times on fat removal percentages at 35°C, 400 rpm, and small-size TKP

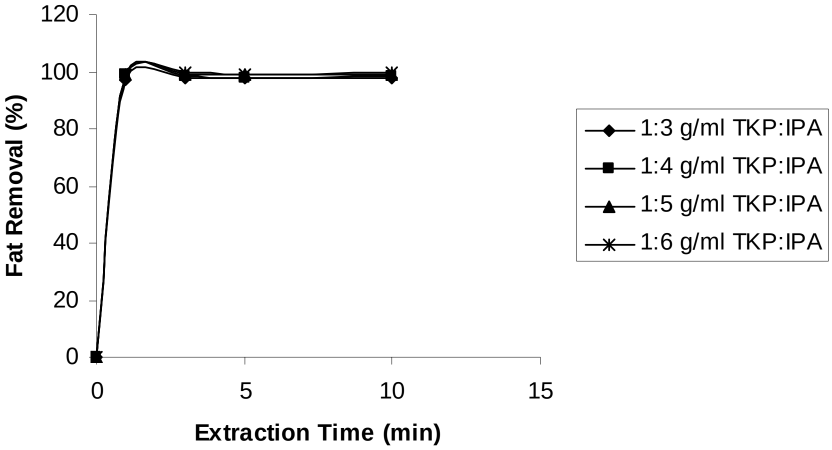


Figure 5.5 Effects of TKP to 99.9% IPA ratios and extraction times on fat removal percentages at 35°C, 400 rpm, and small-size TKP

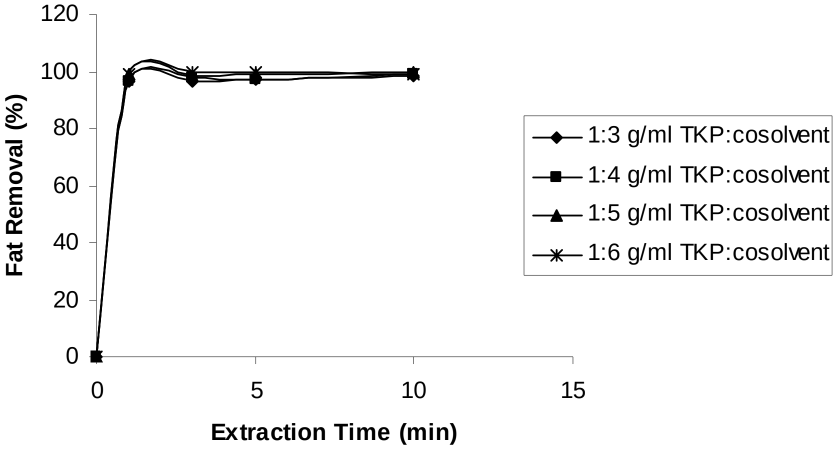


Figure 5.6 Effects of TKP to 1:1 v/v co-solvent ratios and extraction times on fat removal percentages at 35ºC, 400 rpm, and small-size TKP

The comparison of fat removal as a function of TKP to solvent ratio was studied by using small-size TKP at 3-min extraction time as shown in figure 5.7. The results show that TKP to solvent ratios made different in fat removal percentages. The absolute IPA and the co-solvent at 1:1 v/v showed an almost constant trend while 95% ETOH showed the sharply upward trend as the TKP to solvent ratio decreased. For all of these three solvents, the maximum fat removal percentage occurred at 1:6 g/ml. The fat removal yielded from IPA, co-solvent, and ETOH under this ratio were 99.74, 99.54, and 67.23%, respectively. It means that solutes were extracted in proportion to the amount of solvent used. This effect was also confirmed by Franco et al., (2007) who reported that lower solid to solvent ratios promoted an increasing concentration gradients between solvent and solid for the rose hip seed oil extraction with ETOH. However, high solvent utilization has an impact on operating costs; both solvent cost and energy cost associated with the evaporation of solvent. Therefore, the extraction at 1:2 g/ml was also carried out in order to reduce a quantity of solvent used. Under this ratio, TKP particle in solvent was not randomly distributed during mixing. Thus, the use of TKP to solvent ratio at 1:3 g/ml was more suitable than other ratios and it was chosen for next investigations.

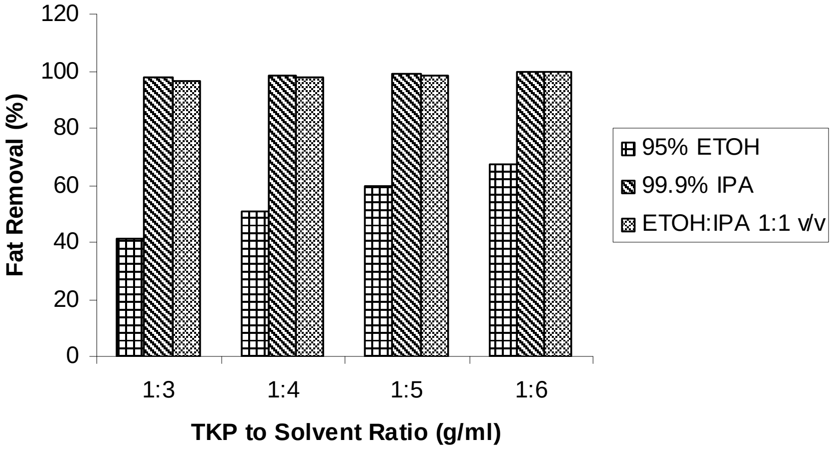


Figure 5.7 Effects of TKP to solvent ratios on fat removal percentages at 35ºC, 400 rpm, and 3-min extraction time

Product yields as a function of TKP to solvent ratios was shown in figure 5.8. It can be seen that ETOH extraction gave slightly higher product yield than IPA and co-solvent extraction because the fat in TKP could be removed lower in ETOH extraction. Thus, the residual fat of TKP in ETOH extract was higher than in other solvents. The product yields of three solvents at various TKP to solvent ratios were above 91%. Polysaccharide and protein contents in product were shown in figure 5.9 and 5.10, respectively. The polysaccharide content in product was higher than in raw material which increased from 70.47% for raw material to about 78.25% for product. The protein content in product was slightly the same as raw material TKP. Only 1% protein was extracted because small protein molecules might be solubilized in alcohol. The results of product viscosity were shown in figure 5.11. The products gave higher viscosity compared with raw material because the amount of polysaccharide in product increased. The viscosity increased from 540 cp for raw material to around 625-810 cp for products.

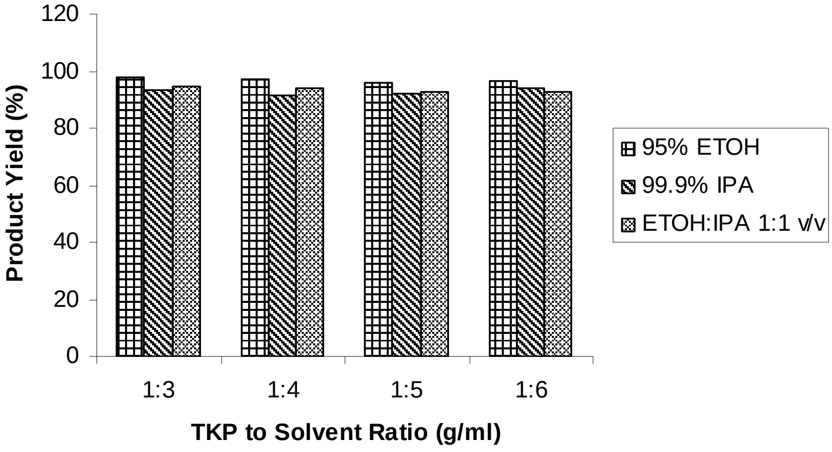


Figure 5.8 Effects of TKP to solvent ratios on product yields at 35ºC, 400 rpm, and 3-min extraction time

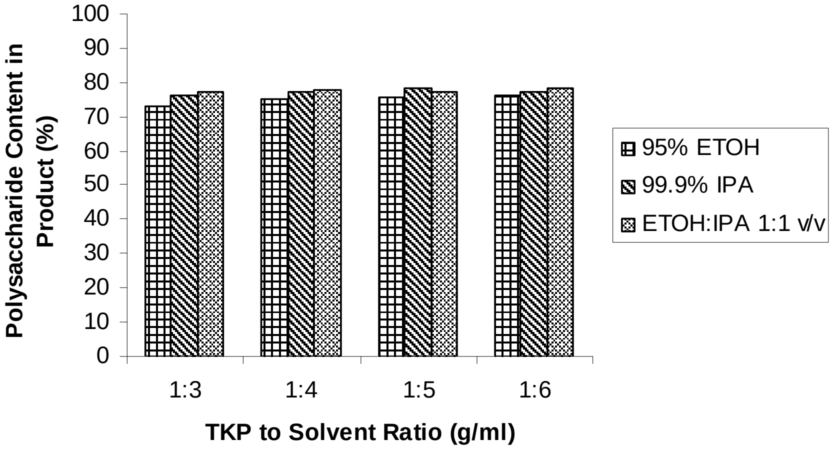


Figure 5.9 Effects of TKP to solvent ratios on polysaccharide levels at 35ºC, 400 rpm, and 3-min extraction time

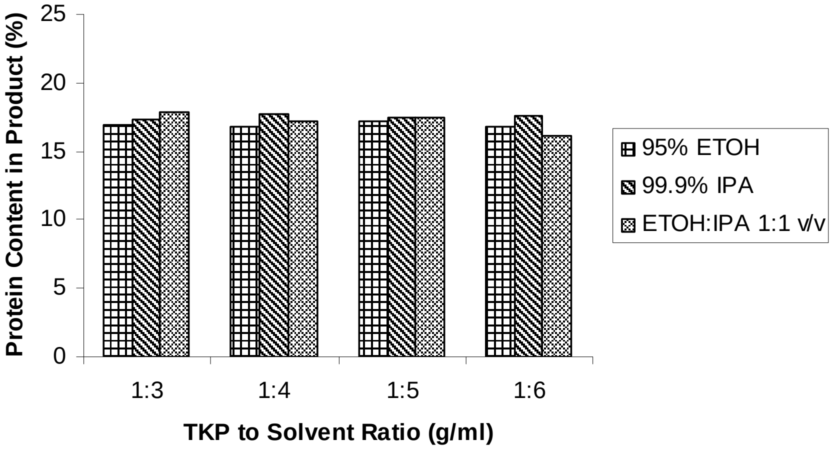


Figure 5.10 Effects of TKP to solvent ratios on protein levels at 35ºC, 400 rpm, and 3-min extraction time

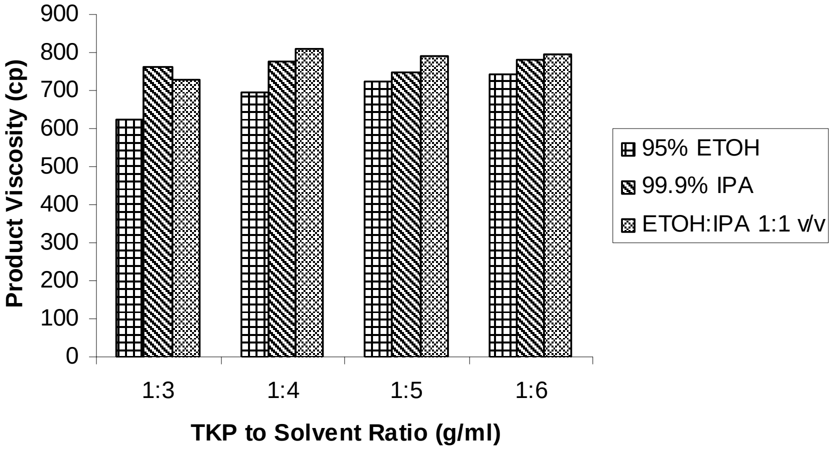


Figure 5.11 Effects of TKP to solvent ratios on viscosities at 35ºC, 400 rpm, and 3-min extraction time

5.4 Multiple-batch extraction using reused solvent and fresh TKP

Multiple-batch extractions were performed in order to determine the saturation solubility of fat or oil in solvent and number of stage of reusable solvent. The data from these experiments were useful for continuous extraction. Figure 5.12 shows the stage of reused solvent with small size of fresh TKP in each successive stage and the TKP to solvent ratio was maintained at 1:3 g/ml. The oil in TKP was transferred to solvent in each stage until it reached the saturation point which had no driving force for mass transfer. It can be seen that absolute IPA gained higher oil concentration than 1:1 v/v co-solvent and ETOH, respectively. The oil concentration was constant at the TKP to solvent ratio higher than 2 g/ml (6 stages of solvent reusable), which was the saturation solubility of oil in IPA. In the case of co-solvent and ETOH, the saturation point occurred at TKP to solvent ratio of 0.67 g/ml for co-solvent (2 stages of solvent reusable) and 0.50 g/ml for ETOH (1.5 stages of solvent reusable). The saturation solubility of oil in IPA, co-solvent, and ETOH were 0.105, 0.037, and 0.015 g/ml, respectively. It meant that the oil could be solubilized higher in IPA compared with co-solvent and ETOH. For multiple batch extraction, the volume of solvent decreased in each stage because of the absorption of solvent by TKP and filter paper. The percentage of solvent loss for IPA, co-solvent, and ETOH were 16.64, 17.37, and 30.18, respectively. ETOH extraction had almost twice higher solvent loss than IPA and co-solvent extraction. The ETOH containing 5% water could form hydrogen bond with the hydroxyl group of polysaccharide, therefore; more ETOH was absorbed in TKP.

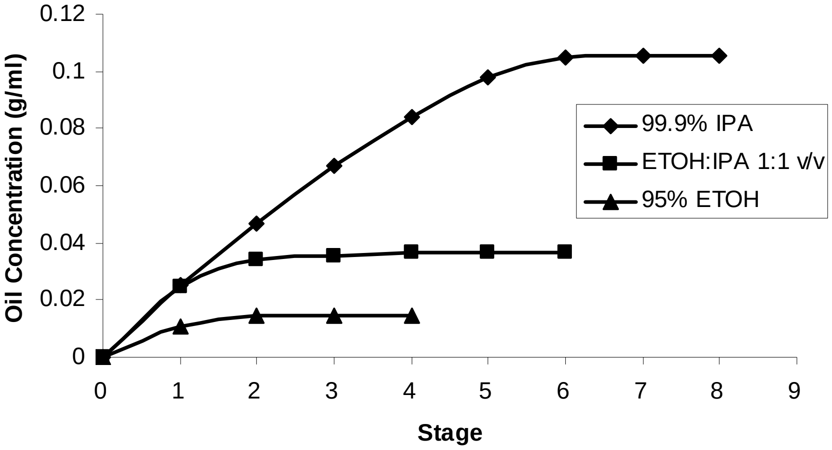


Figure 5.12 Multiple-batch extraction using reused solvent and fresh TKP at 35ºC, 400 rpm, 3 min extraction time and at 1:3 TKP:solvent ratio

5.5 Effects of particle sizes and extraction times on fat removal

Effects of particle sizes and extraction times on fat removal were studied by using 95% ETOH, 99.9% IPA, and co-solvent at 1:1 v/v, respectively and TKP to solvent ratio was maintained at 1:3 g/ml. From the ETOH extraction result (figure 5.13), the percentages of fat removal obtained from small and medium TKP extraction were about 42-45% for the same period of time. The exhaustive extraction was restricted by solubility limitations of the fat in ETOH. This reason was in good agreement with the results from figure 5.7, as the extraction was limited by solubility with ETOH solvent used. In the case of larger size, fat removal was around 38-40% for all extraction time periods which was relatively lower than that of small ones due to its lesser surface area.

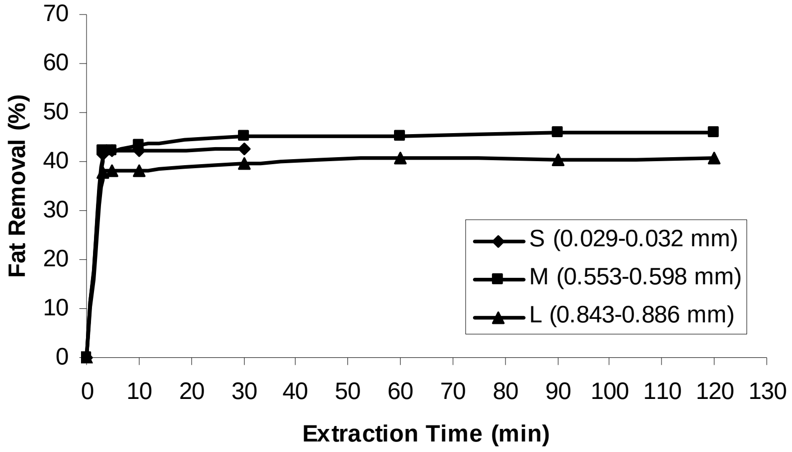


Figure 5.13 Effects of particle sizes and extraction times on fat removal percentages using 95% ETOH at 35ºC, 400 rpm, and TKP to solvent ratio of 1:3 g/ml

Figure 5.14 and 5.15 show the percentages of fat removal as a function of particle sizes and extraction times using absolute IPA and co-solvent at 1:1 v/v extraction, respectively. It was found that the percentages of fat removal decreased when the particle sizes increased. The small-size TKP gave the highest extraction rate and the highest fat removal (98% for IPA and 96% for co-solvent extraction) within a few minutes of extraction time because it has higher specific surface area than the large ones. In small particles, the cell wall was destroyed in a grounding step resulting in the exhibiting of high fat content at the particle surface. Therefore, the fat can be easily and rapidly extracted without internal mass transfer limitations. The extraction time of 3 min was suitable for small-size TKP extraction.

Regarding to medium and large TKP, at the initial period of extraction time, all of fat on TKP surface was extracted first resulting to maintain constant extraction rate. After that the diffusion of fat inside of TKP particle plays an important role of extraction. Therefore, the rate of extraction decreased with time and reached zero. During the first 5 min of extraction, the extraction of medium and large TKP in IPA and co-solvent gave high extraction rate and high fat removal yield which were around 60-66% for medium particles and 51-54% for large ones. These results indicated that a roasting step at 320ºC in TKP production released the fat in the grain and resulted in the outward migration of fat. As the temperature rose, the fatty acid could diffuse out of the inside hydrophobic cavity of amylose single helix. The molecular modeling of amylose-fatty acid complexes was proposed by Buléon et al., (1998) as shown in figure 5.16. Thus, TKP had more fat on the particle surface. The SEM images of cross-section tamarind kernel were illustrated in figure 5.17 and supported the above reason. Comparing the unroasted kernel surface (figure 5.17 (a)) with the roasted kernel surface (figure 5.17 (b)), the unroasted kernel did not have fat covering on surface resulting in clear surface but in the case of roasted kernel, the surface of tamarind kernel was covered by fat.

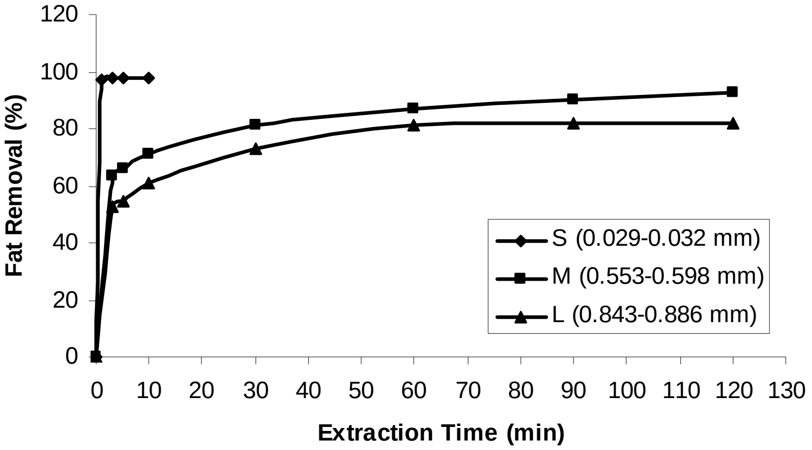


Figure 5.14 Effects of particle sizes and extraction times on fat removal percentages using 99.9% IPA at 35°C, 400 rpm, and TKP to solvent ratio of 1:3 g/ml

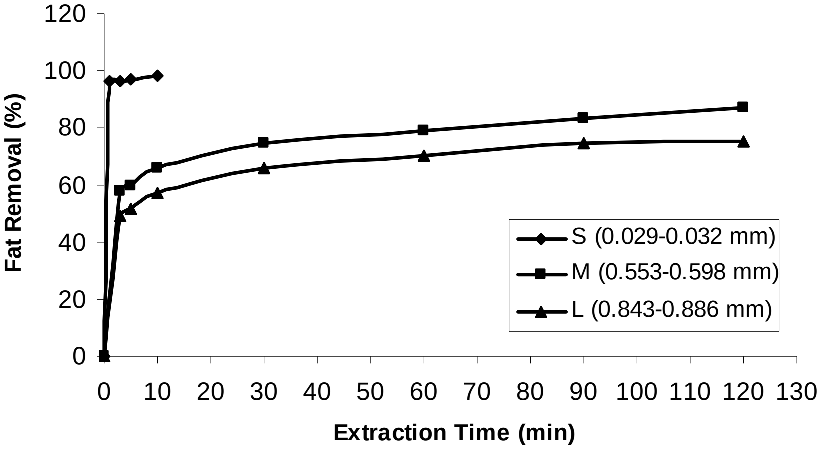


Figure 5.15 Effects of particle sizes and extraction times on fat removal percentages using co-solvent 1:1 v/v at 35ºC, 400 rpm, and TKP to solvent ratio of 1:3 g/ml

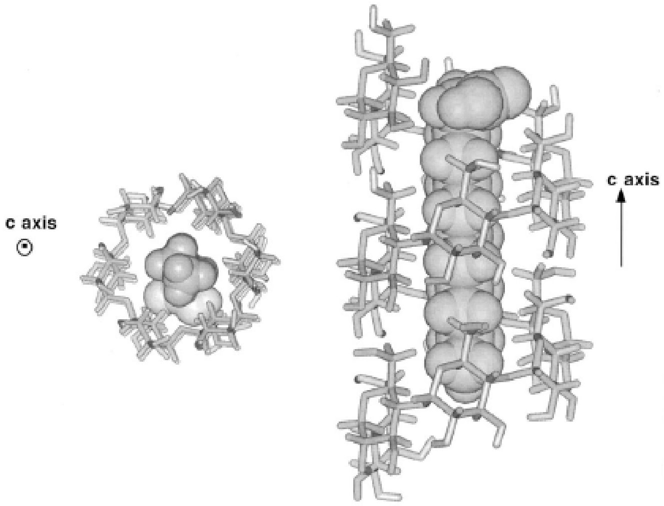


Figure 5.16 Molecular modeling representation of amylose–fatty acid complexes (Buléon et al., 1998)

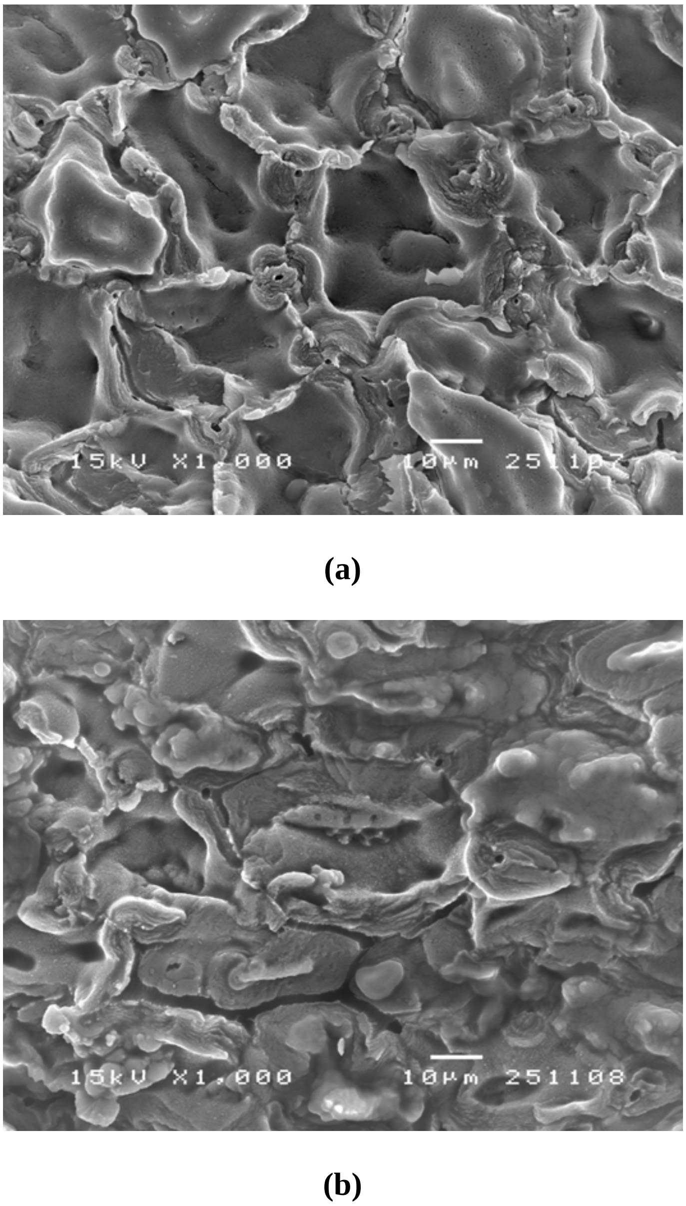


Figure 5.17 Cross-section picture of tamarind kernel

(a) Unroasted tamarind kernel at 1,000x

(b) Roasted tamarind kernel at 1,000x

When times proceed, the fat leaching was controlled by diffusion of solvent into and out of the particle which caused the increase of particle porosity. The pore diameter and the total porosity of product extracted with various solvents at 90 min and at TKP to solvent ratio of 1:3 g/ml were shown in Table 5.2. The total porosity from ETOH extraction was lower than from IPA and co-solvent extraction. ETOH gave low extraction efficiency due to the limitation of solvent used, resulting in low fat diffusion from the interior of TKP particle into solvent bulk. Considering medium and large particles, the extraction of medium TKP gave higher total porosity than that obtained from large ones. Because smaller particles had high specific surface area, the solvent penetration path lengths decreased resulting in more fat diffusion from the inner of particle to solvent.

Table 5.2 Pore diameter and porosity of raw material and products

TKP	**Pore diameter (μm)**	**Total porosity (%)**
Raw material	0.008	6.41
Medium /ETOH	0.297	11.63
Large/ETOH	0.399	9.42
Medium/IPA	0.207	14.94
Large/IPA	0.219	14.88
Medium/co-solvent	0.218	15.57
Large/co-solvent	0.211	14.91

The extraction rate of fat from TKP particle is governed by the following stages. Firstly, the fat dissolves in solvent. After that the fat in solution diffuses to the surface of TKP particle. Finally, the fat moves from the surface of the TKP, where it is assumed to be in the form of saturated solution, into the bulk of the solution. With the presence of the correct solvent, solution of the fat takes place rapidly and does not greatly influence the overall extraction rate. Because of the complex structure, the rate of movement within the TKP particle is difficult to quantify. For batch extraction, the rate of mass transfer from the saturated layer at the TKP surface to the bulk solvent is equal to Eq. 3.4. To find the mass transfer coefficient (k_La), the plot of $\ln c_{AS}/(c_{AS}-c_A)$ versus time was performed and determined from the slope (k_LA/V or k_La) of the graph. The mass transfer coefficients from the experimental data were summarized in Table 5.3. The results indicated that mass transfer coefficient increased with the particle size diminution. Among these particle sizes, the small particle provided the highest mass transfer coefficient. This implied that the fat in small particle was rapidly transferred from the particle surface to the bulk solvent. Due to the small particle that possess the high specific area enhanced the mass transfer. The surface area of small particle was 19 folds higher than medium particle. In case of medium particle was 1.6 folds higher surface area than large particle. Thus, the difference in mass transfer coefficient between small and large ones was obvious.

Table 5.3 Mass transfer coefficient of fat from TKP extraction

Solvent	**k_La (s^{-1})**		
	Small	**Medium**	**Large**
ETOH	8.30×10^{-3}	7.40×10^{-3}	6.00×10^{-3}
IPA	4.60×10^{-3}	9.00×10^{-4}	8.00×10^{-4}
Co-solvent	1.9×10^{-2}	2.90×10^{-3}	2.40×10^{-3}

Figure 5.18-5.20 shows effects of particle sizes and extraction times on product yields. The experimental results indicated that the product yields of ETOH, IPA, and co-solvent extraction at various time were in range of 92.94-97.52, 88.78-93.25, and 89.60-94.95%, respectively. ETOH extraction gave higher product yield than other solvents because ETOH was low-extraction-efficiency solvent causing high residual fat in product. The polysaccharide content in product obtained from IPA and co-solvent extraction was slightly high compared with ETOH extraction (Figure 5.21-5.23). Because IPA and co-solvent could remove fat in high yield, this resulted in an increase in the polysaccharide content. As seen in figure 5.24-5.26, the protein in TKP was partially extracted. Additionally, the protein content in raw material slightly decreased from 18.92% to around 16.31-18.03% in the product. The viscosity of

product as a function of particle sizes and extraction times was shown in figure 5.27-5.29. The small particles gave higher product viscosity than the large ones. For small particles, the fat in TKP was highly extracted and resulted in an increase amount of polysaccharide in product. Thus, the product viscosity increased with amount of polysaccharide. The product viscosities obtained from small, medium, and large by using ETOH as extracting solvent were in the range of 620-640, 614-636, and 545-590 cp, respectively. For IPA extractions, the product viscosity obtained from small and medium TKP were 705-774 and 674-688 cp, respectively, and 542-605 cp for large ones. The viscosity of small, medium, and large TKP product which extracted with co-solvent at 1:1 v/v were 775-790, 660-677, and 551-670 cp, respectively.

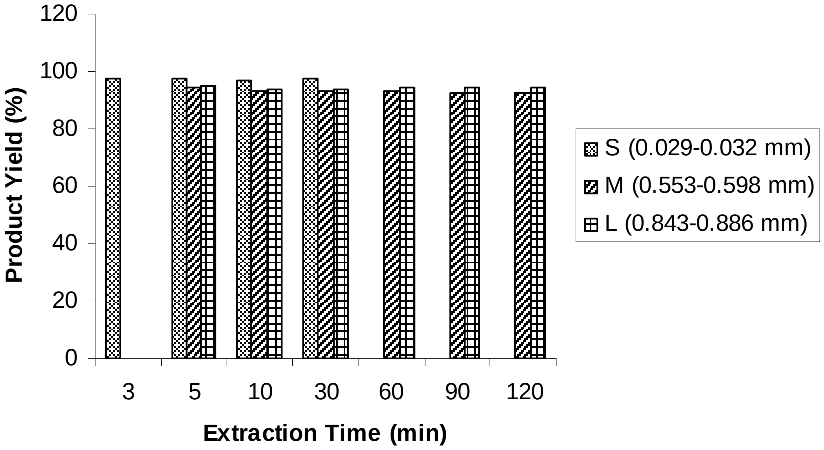


Figure 5.18 Effects of particle sizes and extraction times on product yields using 95% ETOH at 35ºC, 400 rpm, and TKP to solvent ratio of 1:3 g/ml

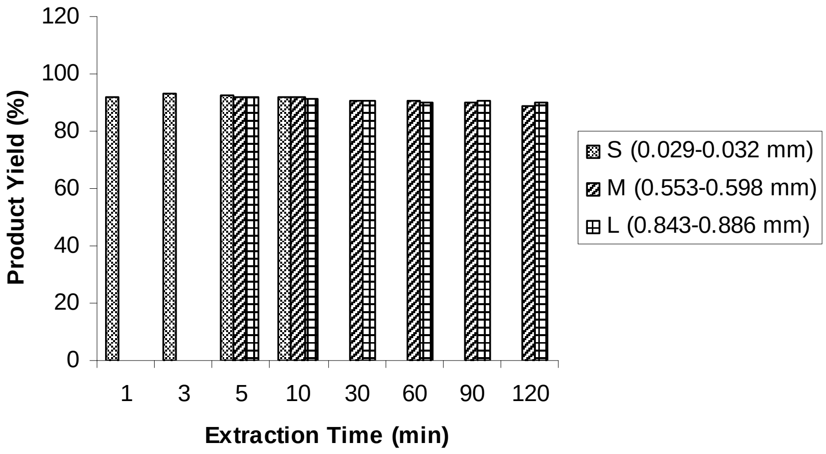


Figure 5.19 Effects of particle sizes and extraction times on product yields using 99.9% IPA at 35ºC, 400 rpm, and TKP to solvent ratio of 1:3 g/ml

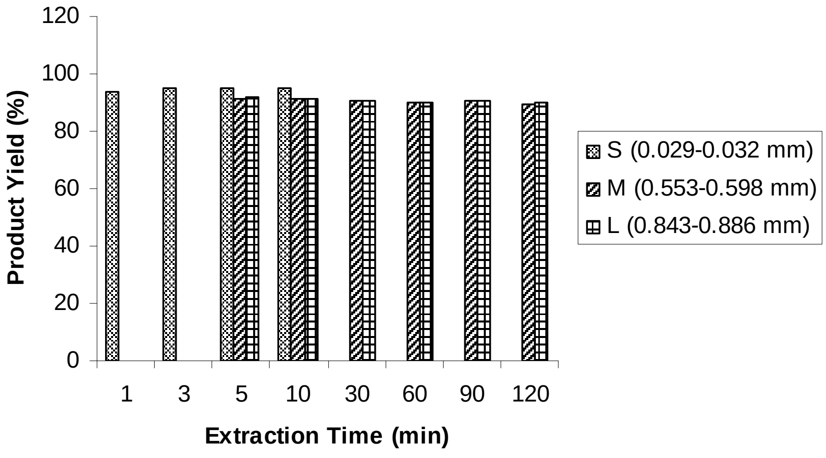


Figure 5.20 Effects of particle sizes and extraction times on product yields using co-solvent 1:1 v/v at 35ºC, 400 rpm, and TKP to solvent ratio of 1:3 g/ml

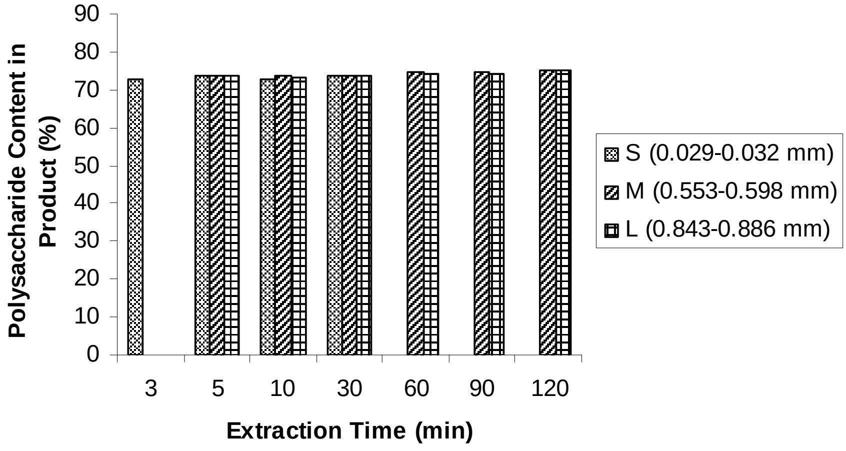


Figure 5.21 Effects of particle sizes and extraction times on polysaccharide levels using 95% ETOH at 35ºC, 400 rpm, and TKP to solvent ratio of 1:3 g/ml

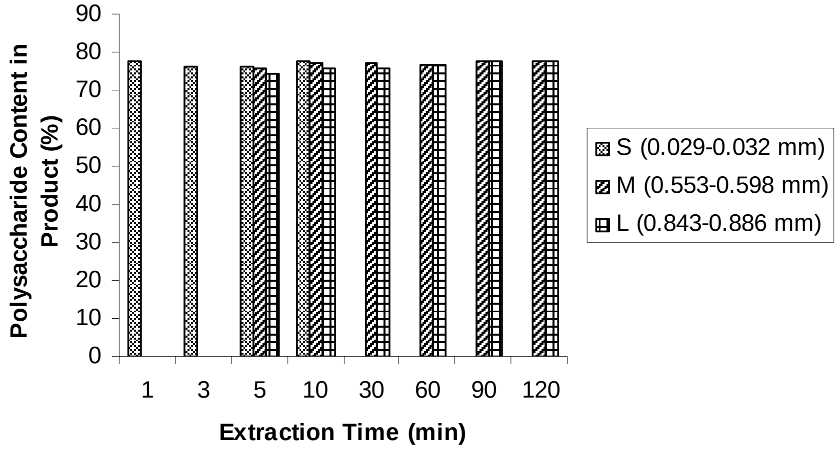


Figure 5.22 Effects of particle sizes and extraction times on polysaccharide levels using 99.9% IPA at 35ºC, 400 rpm, and TKP to solvent ratio of 1:3 g/ml

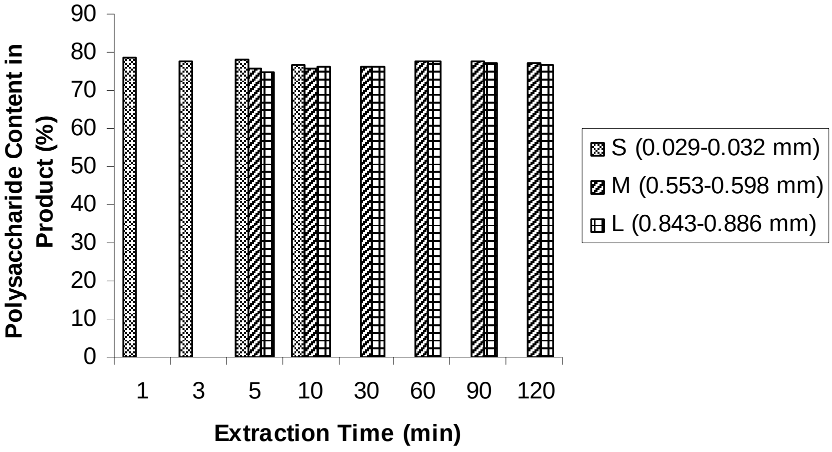


Figure 5.23 Effects of particle sizes and extraction times on polysaccharide levels using co-solvent 1:1 v/v at 35ºC, 400 rpm, and TKP to solvent ratio of 1:3 g/ml

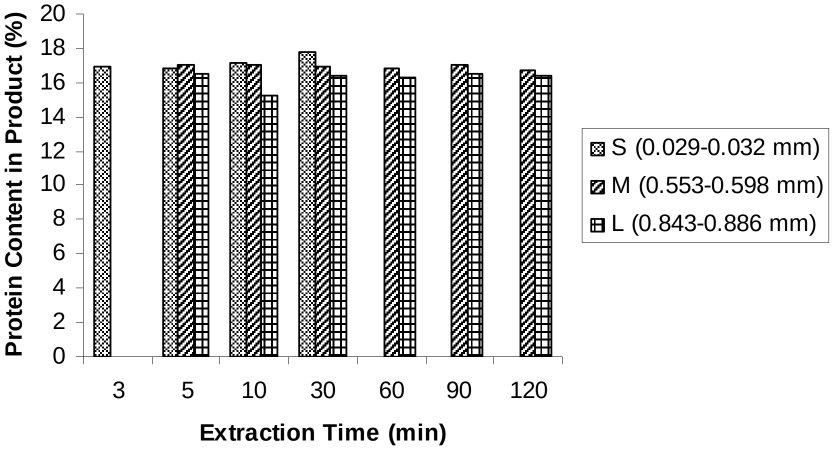


Figure 5.24 Effects of particle sizes and extraction times on protein level using 95% ETOH at 35ºC, 400 rpm, and TKP to solvent ratio of 1:3 g/ml

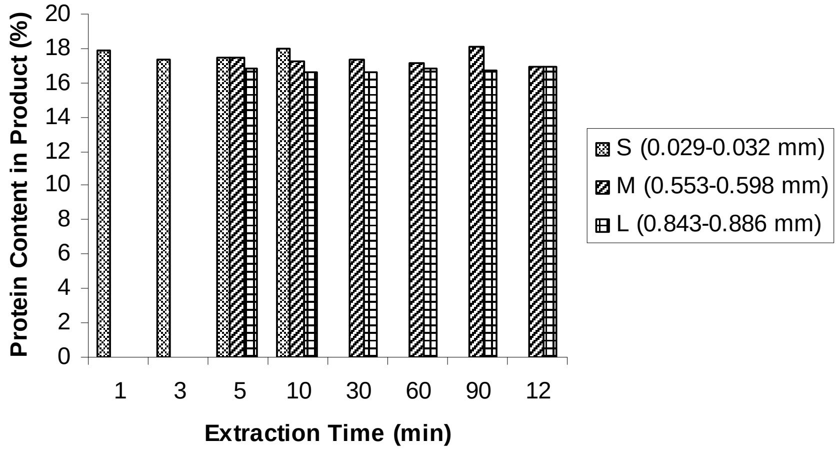


Figure 5.25 Effects of particle sizes and extraction times on protein levels using 99.9% IPA at 35ºC, 400 rpm, and TKP to solvent ratio of 1:3 g/ml

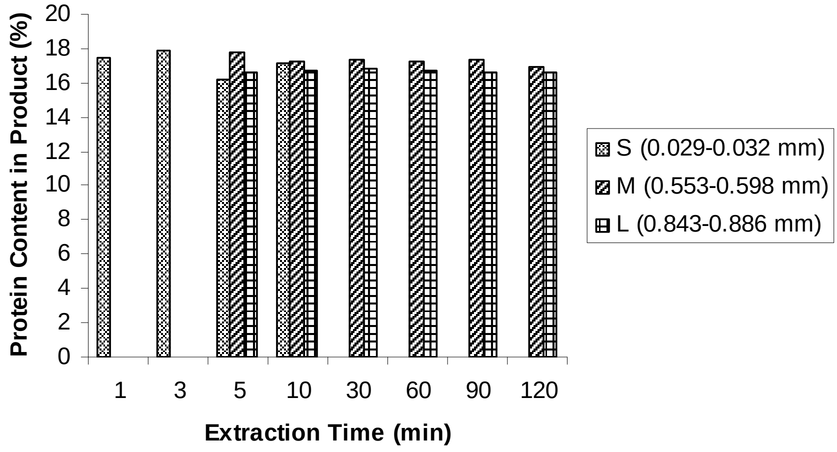


Figure 5.26 Effects of particle sizes and extraction times on protein levels using co-solvent 1:1 v/v at 35ºC, 400 rpm, and TKP to solvent ratio of 1:3 g/ml

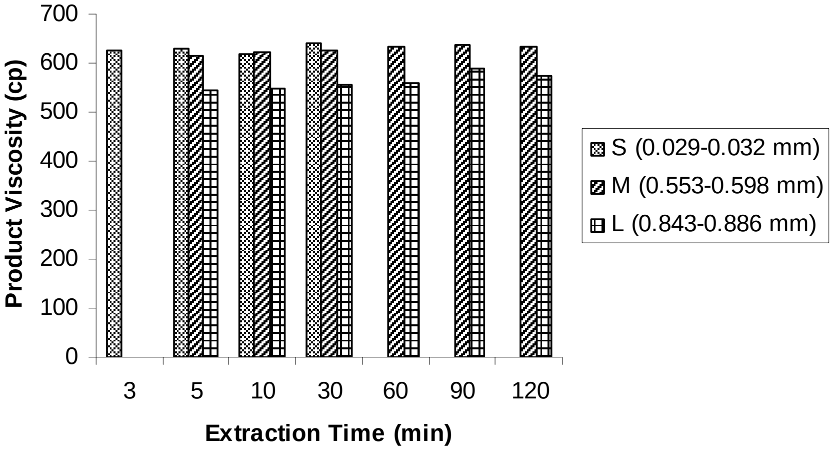


Figure 5.27 Effects of particle sizes and extraction times on viscosities using 95% ETOH at 35ºC, 400 rpm, and TKP to solvent ratio of 1:3 g/ml

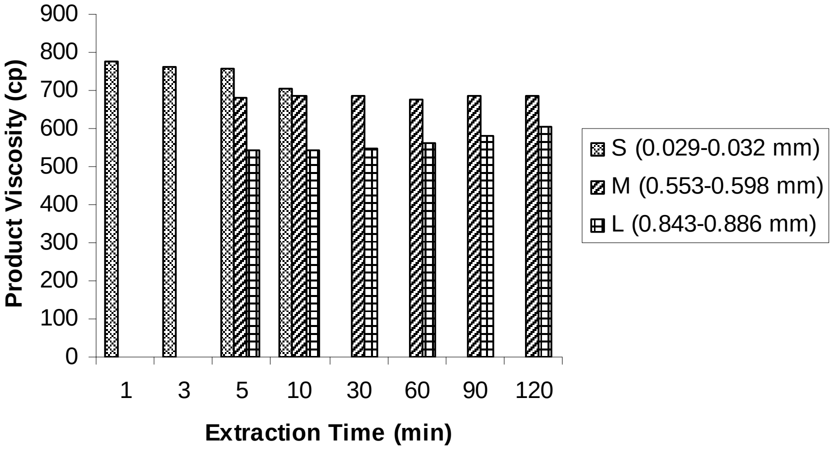


Figure 5.28 Effects of particle sizes and extraction times on viscosities using 99.9% IPA at 35ºC, 400 rpm, and TKP to solvent ratio of 1:3 g/ml

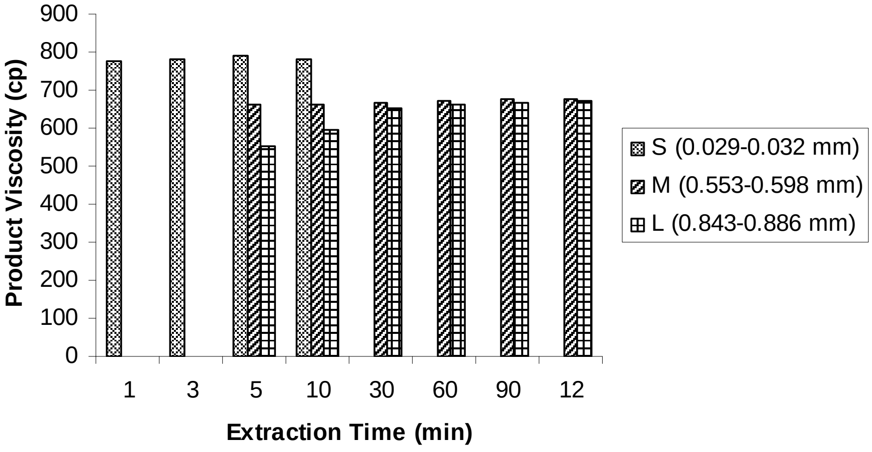


Figure 5.29 Effects of particle sizes and extraction times on viscosities using co-solvent 1:1 v/v at 35ºC, 400 rpm, and TKP to solvent ratio of 1:3 g/ml

To find the suitable solvent, fat removal from co-solvent and IPA were compared in order to reduce solvent cost. The previous experimental results show the use of co-solvent gave a slightly low percentage of fat removal compared with IPA extraction. However, co-solvent is less expensive than IPA. Therefore, co-solvent at 1:1 v/v was chosen for fat leaching in scale-up experiments.

5.6 Scale up of mixing tank

The extractions of three particle sizes were carried out in scaled-up mixing tank at the temperature of 35ºC with the TKP to solvent ratio of 1:3 g/ml using co-solvent at 1:1 v/v as extracting solvent. The fat removals from these experiments were compared with the data from laboratory scale in order to study extraction performance in semi-pilot scale (figure 5.30). The mixing tank was scaled up by a scale up ratio of 2.71 (2.5 kg of TKP was extracted per trial) and stirring speed was fixed at 205 rpm. The flow in the tank of both scales was in the range of turbulent regime with the impeller Reynolds number (N_{Re}) of 8,511 for experimental scale and 32,296 for semi-pilot scale. The extraction profiles from laboratory scale were almost equal to those obtained from semi-pilot scale.

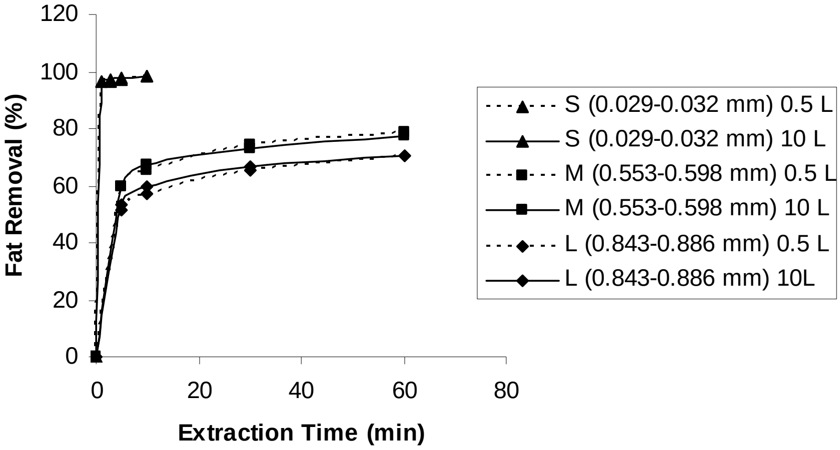
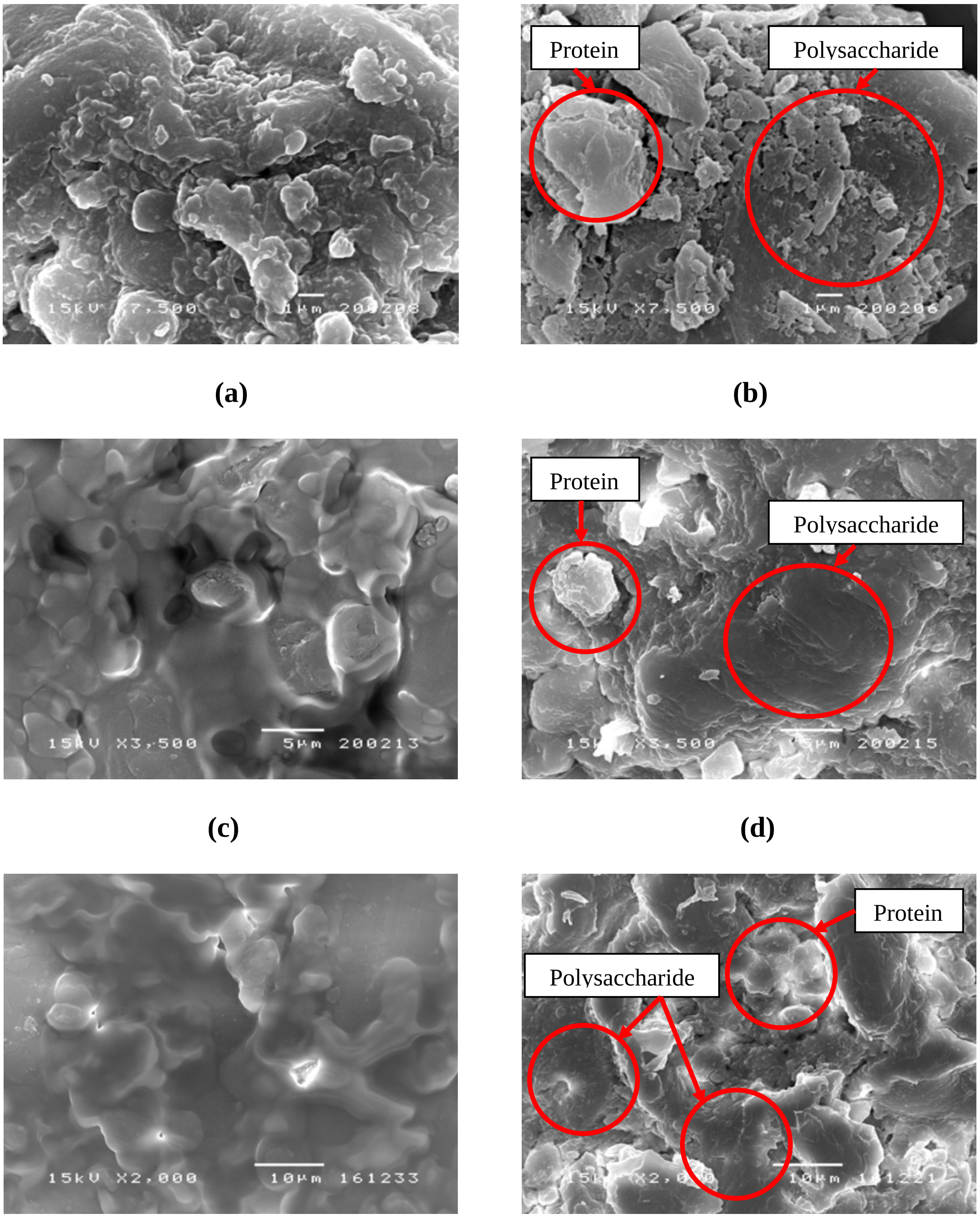


Figure 5.30 Fat removals from experimental and semi-pilot scale

at various particle sizes

These results are consistent with that reported by Amarasinghe et al., (2004) and Stanisavljević et al., (2007), reporting an enhancement of extraction efficiency subjected to the extraction of small particles of rice bran and tobacco seed, respectively. Although the small-size TKP is effective for extraction, they pose the problems of huge energy consumption in size reduction and processing difficulties such as dust and heat generation during grinding; and blocked filters during extraction at a large scale. Therefore, the medium or large particles should be chosen to avoid the above problems. At 30-min extraction, the fat removal from medium particles was 8% higher than that obtained from large ones. Thus, in the industrial point of view, the use of medium-size TKP for extraction was more preferable than large-size TKP. The suitable condition for fat removal from TKP was found to be medium-size TKP, the TKP to solvent ratio of 1:3 g/ml, co-solvent at 1:1 v/v, and extraction time of 30 min which gave 74.49% fat removal and product viscosity of 665 cp. The composition of product under this condition were 2.05% fat, 76.19% polysaccharide, 17.36% protein, and 4.40% others. A high-quality, low fat and high-viscosity product was obtained and potentially used in food industry as a viscosity enhancer.

SEM investigations were carried out to clarify the characteristics of TKP for treated and untreated with co-solvent. The SEM images of the untreated TKP are shown in figure 5.31. There was a significant amount of fat on the surface of materials (figure 5.31(a), (c), and (e)). After 3 min of small size extraction, the majority of fat on the surface of material had gone (fat removal yield 96.53%) as shown in figure 5.31(b). After 30 min of medium and large particle extractions, most of the fat from TKP had been extracted (fat removal yield 74.49% for medium particles and 65.70% for large ones) as shown in figure 5.31(d) and (f). These visual observations demonstrated that the treated TKP had clear surface. Polysaccharide and protein molecules could be obviously seen after the treatment which had a much less fat content.



(a) **(b)**

(c) **(d)**

(e) **(f)**

Figure 5.31 Surface characteristics of TKP

Untreated (a) and treated (b) small TKP with co-solvent 3 min at 7,500x

Untreated (c) and treated (d) medium TKP with co-solvent 30 min at 3,500x

Untreated (e) and treated (f) large TKP with co-solvent 30 min at 2,000x

5.7 Fatty acid composition of extracted oils

The color of both tamarind kernel oils extracted with IPA and co-solvent at 1:1 V/V were bright yellow and dark yellow for ETOH extraction as shown in figure 5.32. Both oils from IPA and co-solvent extraction were liquid at room temperature but oil from ETOH formed wax. The oil extracted with ETOH was more viscous than those obtained from IPA and co-solvent extraction. This could be ascribed to its more saturated nature. The fatty acid composition of oils from ETOH, IPA, and co-solvent was summarized in Table 5.4. For all of the three extracted oil samples, the major fatty acids were linoleic acid (51.7-52.1%), oleic acid (17.1-19.3%), and palmitic acid (9.4-13.0%). These data corresponded to those reported by Andriamanantena et al. (1983) who did the extraction of tamarind kernel by hexane and a mixture of chloroform and methanol. The result showed that the polyunsaturated fatty acid content of extracted tamarind kernel oils was about 52% while the monounsaturated fatty acid content and the saturated fatty acid content were about 19 and 27%, respectively. The oils contained a relatively large proportion of unsaturated fatty acids with linoleic acid (51-52%) at the highest concentration. Small differences were observed in the fatty acid composition of three extracted oil samples. Palmitic acid, a saturated fatty acid with lower carbon atom, was significantly higher in ethanol extracts than in isopropanol and co-solvent extracts. This result could affect the physical characteristics of oil from ETOH extraction that it formed wax at room temperature. The unsaturated fatty acid was easily oxidized by oxygen in air at the location of double bond. Auto-oxidation was the cause of oil rancidity resulting in low-quality the TKP. However for rancidity reduction, unsaturated fatty acid should be removed. Co-solvent could extract linoleic acid, an unsaturated fatty acid with two double bonds, at high level (51.8%). This yield was almost equal to that of ETOH extraction (52.1%). Therefore, the use of co-solvent met our goal.

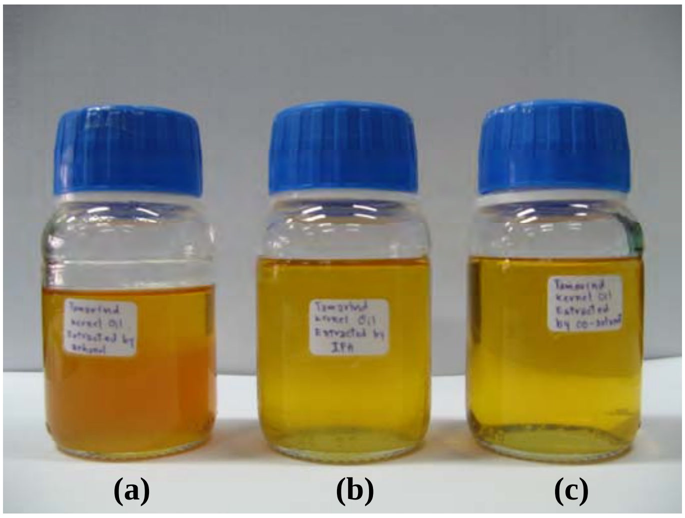


Figure 5.32 Tamarind kernel oil extracts

(a) Tamarind kernel oil extracted with 95% ETOH

(b) Tamarind kernel oil extracted with 99.9% IPA

(c) Tamarind kernel oil extracted with co-solvent at 1:1 v/v

Table 5.4 Fatty acid composition of tamarind kernel oils

Fatty acid composition	Total fatty acid (%)		
	Oil extracted with IPA	Oil extracted with ETOH	Oil extracted with Co-solvent[a]
Myristic acid (14:0)	0.1	0.1	0.1
Palmitic acid (16:0)	9.4	13.0	10.1
Heptadecanoic acid (17:0)	0.1	0.1	0.1
Stearic acid (18:0)	4.6	4.9	4.7
Oleic acid (18:1)	17.1	19.3	17.7
Vaccenic acid (18:1)	0.3	0.3	0.3
Linoleic acid (18:2)	51.7	52.1	51.8
Arachidic acid (20:0)	2.5	1.8	2.4
Eicosenoic acid (20:1)	1.0	0.9	1.0
Eicosadienoic acid (20:2)	0.1	0.1	0.1
Eicosatrienoic acid (20:3)	0.1	-	-
Eicosapentaenoic acid (20:5)	0.1	0.1	0.1
Behenic acid (22:0)	4.2	2.5	3.8
Tricosanoic acid (23:0)	0.2	0.1	0.2
Lignoceric acid (24:0)	7.4	3.5	6.5
Others	1.1	1.2	1.1

[a] 95% ETOH:99.9% IPA at 1:1 v/v

CHAPTER VI

CONCLUSIONS AND RECOMMENDATIONS

6.1 Conclusions

The main objective of the present work was to evaluate the effect of particle size on fat removal from TKP using organic solvents in mixing tank. The fat removal parameters; solvent type, TKP to solvent ratio, particle size, and extraction time were studied in 0.5-liter mixing tank. Subsequently, the mixing tank with 0.5-liter working volume was scaled up to 10 liter. Furthermore, the extraction efficiencies of both mixing tanks were compared. From the experimental results, it can be concluded as follows:

1. 99.9% IPA was more effective than 95% ethanol for fat removal. The water content in ETOH reduced extraction efficiency because the fat could be solubilized lower in aqueous ETOH.
2. The new solvent system was found to be 95% ETOH:99.9% IPA at 1:1 v/v, provided almost the same extraction performance as 99.9% IPA.
3. The suitable TKP to solvent ratio for fat removal from TKP was found to be at the ratio of 1:3 g/ml.
4. The saturation solubility of oil in IPA, co-solvent, and ETOH were 0.105, 0.037, and 0.015 g/ml, respectively.
5. The extraction was more effective with small particle due to their high specific surface area. To avoid high energy consumption for size reduction and operation problems, the suitable particle size was medium size (0.553-0.598 mm).
6. Extraction yield results obtained from semi-pilot scale extraction were found to be in good agreement with those of laboratory scale without any loss of extraction efficiency.

7. The suitable condition for fat removal was at 30 min extraction time, 1:3 g/ml TKP:solvent ratio, medium-size TKP, and 1:1 v/v co-solvent. Under this condition, the fat removal percentage was 74.49 % and the product viscosity was 665 cp. The composition of this product were 2.05% fat, 76.19% polysaccharide, 17.36% protein, and 4.40% others.

6.2 Recommendations

1. For further study, the factors that affected the extraction efficiency such as extraction temperature should be considered to improve the efficiency of the method.
2. The prototype of mixing tank with 10-liter working volume should be considered to scale up for use at an industrial scale.

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APPENDICES

APPENDIX A

Analytical methods

1. Protein analysis

The amount of protein in TKP was determined by Kjeldahl method (AOAC.920.87).

- **Chemicals**

1. 6.25 M Sodium hydroxide (NaOH)
2. 2% w/v Boric acid (H_3BO_3)
3. Concentrated sulfuric acid (H_2SO_4)
4. Selenium reagent mixture

- **Method**

The Kjeldahl method was divided into three main steps:

Digestion – 0.5 g of selenium reagent mixture was added as a catalyst to 0.2 g of TKP sample. Concentrated sulfuric acid solution was then added for digestion by boiling for 90 min. After that, a homogeneous sample in concentrated sulfuric acid solution was obtained. An ammonium sulfate solution was formed as the end result.

Distillation – Excessive 6.25 M NaOH was added to the acid digestion mixture to convert NH_4^+ to NH_3, followed by boiling and condensation of the NH_3 gas in a receiving solution.

Titration – The amount of ammonia in the receiving solution was determined by titration with standard sulfuric acid.

The percentage of protein in TKP was calculated through:

$$\text{Protein (\%)} = \frac{1.4007 \times 6.25 \times N_{H_2SO_4} \times V_{H_2SO_4}}{Wt_{sample}}$$

$N_{H_2SO_4}$ = Normality of standard sulfuric acid

$V_{H_2SO_4}$ = Volume of sulfuric acid for titration

Wt_{sample} = Weight of sample

2. Polysaccharide analysis

The amount of polysaccharide in TKP was determined by Phenol-sulfuric method (Dubois et al., 1956).

• Chemicals

1. 5% w/v Phenol (C_6H_5OH)

2. Concentrated sulfuric acid (H_2SO_4)

• Method

TKP sample solution was prepared by having polysaccharides in the range of 10-70 µg/ml. 1 ml of phenol and 5 ml of concentrated sulfuric acid were added into 1 ml of sample solution. Solution was mixed by vortex and placed to form a reaction for 10 min. After that, the reaction was stopped by placing in water bath at 25-30ºC for 10-20 min. The color intensity was measured by spectrophotometer at 483 nm. The measured absorbance was recorded and calculated for the amount of polysaccharide in TKP through standard calibration curve of TSP as shown in figure A-1.

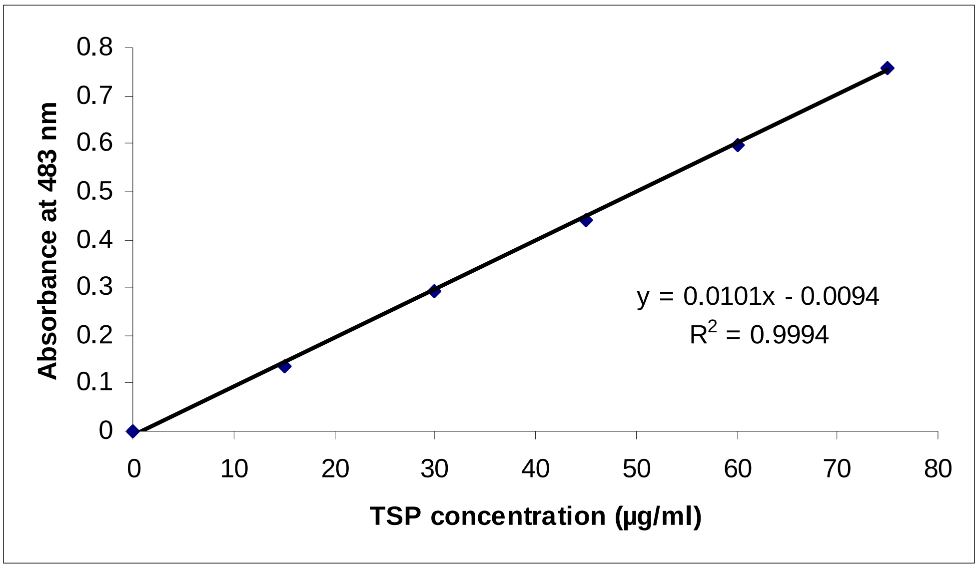

Figure A-1 Standard calibration curve of tamarind seed polysaccharide (TSP)

3. Fat analysis

The amount of fat in TKP was determined by soxhlet extraction method (AOAC. 920.39).

- **Chemicals**

1. Petroleum ether

- **Method**

Two grams of tamarind kernel powder was placed into a thimble of a soxhlet apparatus and extracted with 150 ml petroleum ether. Extraction was carried out for 3 hours. After that, solvent was poured into weighed evaporating flask. Solvent was evaporated by a rotary-evaporator and the last traces were removed by placing the flask with the extract in an oven at 110ºC for one hour. The flask was cooled in a dessicator and weighed.

4. Viscosity analysis

The viscosity of sample at 2% w/v was determined by viscometer (B8L, Toki, Japan) at 25ºC.

- **Method**

Four grams of TKP was dispersed in 200 ml water and boiled at 80ºC for 30 min. After that, the slurry was placed in water bath while controlling temperature at 25ºC. The viscosity of TKP slurry was measured by viscometer as maintaining the temperature of slurry at 25ºC.

APPENDIX B

Dimensions of mixing tank

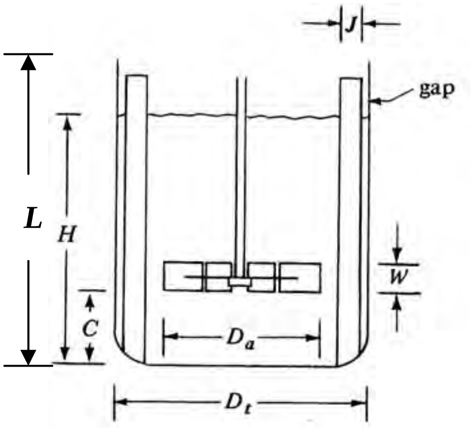

Figure B-1 Dimensions of mixing tank **(**Geankoplis, 1995)

Table B-1 Geometric proportion of mixing tank with working volume 0.5 and 10 liter

Dimension	**0.5 liter**	**10 liter**
Height of tank (L)	12.3 cm	38.9
Height of liquid (H)	8.6 cm	23.4
Diameter of tank (D_t)	8.6 cm	23.4
Diameter of impeller (D_a)	4.3 cm	11.7
Width of impeller (W)	0.9 cm	2.3
Height of impeller above tank bottom (C)	2.9 cm	7.8
Width of baffle (J)	0.7 cm	1.9
Type of impeller	Propeller	Propeller
Tank material	Glass	Stainless steel 304
Temperature controlling	Water bath	Water jacket

Figure B-2 10-liter closed mixing tank

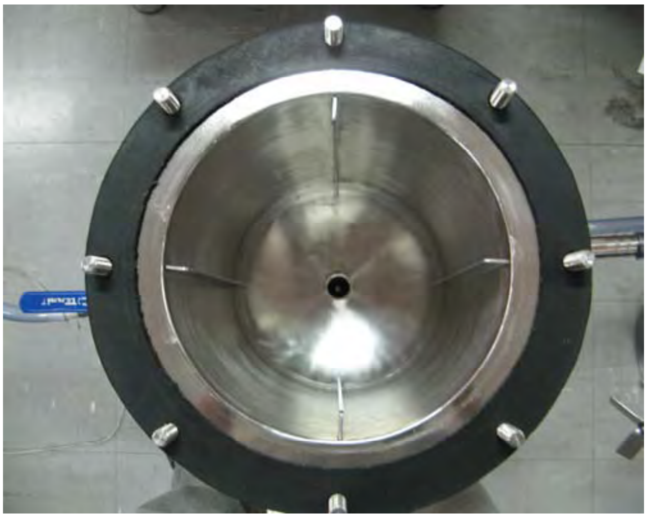

Figure B-3 Inner 10-liter closed mixing tank

Figure B-4 Impeller

APPENDIX C

Experimental results

Table C-1 Experimental results from 95% ETOH extraction in 0.5-liter mixing tank

ETOH	Time (min)	Fat removal (%)	Content (dry basis)				Yield (%)	Viscosity (cp)
			Polysaccharide	Protein	Fat	Others		
S size								
1:3 S/L*	3	41.585	72.900	16.978	4.702	5.419	97.523	625
	5	42.269	73.749	16.846	4.647	4.758	97.481	630
	10	42.238	73.004	17.159	4.650	5.188	96.896	620
	30	42.612	73.564	17.754	4.619	4.062	97.279	640
S size								
1:4 S/L	3	50.898	75.282	16.856	3.952	3.910	97.324	693
	5	51.822	74.025	16.854	3.878	5.243	97.477	564
	10	51.931	74.823	16.979	3.869	4.329	97.627	595
	30	50.972	74.184	17.199	3.947	4.671	97.492	609
S size								
1:5 S/L	3	59.490	75.413	17.154	3.261	4.172	95.862	724
	5	59.074	75.591	17.348	3.294	3.766	96.521	610
	10	59.623	75.060	17.053	3.250	4.636	96.588	605
	30	59.720	75.710	17.212	3.242	3.836	97.465	784
S size								
1:6 S/L	3	67.233	76.044	16.845	2.638	4.473	96.581	745
	5	68.369	76.068	17.064	2.546	4.322	96.936	585
	10	68.973	75.169	17.210	2.498	5.123	96.528	575
	30	69.956	76.443	17.541	2.418	3.598	96.550	567
M size								
1:3 S/L	5	42.232	73.501	17.019	4.650	4.830	95.857	614
	10	43.485	73.529	17.086	4.549	4.836	93.167	624
	30	45.327	73.837	16.960	4.401	4.803	93.063	626
	60	45.238	74.466	16.832	4.408	4.293	92.943	632
	90	45.816	74.544	17.067	4.362	4.027	92.712	636
	120	45.290	75.013	16.742	4.404	3.841	92.765	635
L size								
1:3 S/L	5	38.201	73.785	16.455	4.975	4.785	94.777	545
	10	38.252	73.477	15.253	4.970	6.299	94.011	547
	30	39.673	73.619	16.435	4.856	5.091	93.985	555
	60	40.946	74.312	16.310	4.754	4.625	94.211	560
	90	40.505	74.271	16.461	4.789	4.479	94.333	590
	120	40.824	75.331	16.407	4.763	3.499	94.241	575

*Solid to solvent ratio

Table C-2 Experimental results from 99.9% IPA extraction in 0.5-liter mixing tank

IPA	Time	Fat removal	Content (dry basis)				Yield	Viscosity
	(min)	(%)	Polysaccharide	Protein	Fat	Others	(%)	(cp)
S size 1:3 S/L[*]	1	97.291	77.435	17.867	0.218	4.480	91.886	774
	3	97.943	76.047	17.370	0.166	6.418	93.255	760
	5	97.225	76.135	17.478	0.223	6.164	92.726	755
	10	97.281	77.701	18.038	0.219	4.043	91.947	705
S size 1:4 S/L	1	98.834	78.182	17.417	0.094	4.307	92.200	805
	3	98.365	77.481	17.768	0.132	4.619	91.701	775
	5	97.678	76.537	17.981	0.187	5.295	92.295	720
	10	98.380	77.014	17.524	0.130	5.332	91.082	750
S size 1:5 S/L	1	99.226	76.435	17.076	0.062	6.427	92.257	787
	3	99.359	78.250	17.405	0.052	4.293	91.937	750
	5	99.229	77.062	18.798	0.062	4.077	91.930	700
	10	99.228	76.726	17.962	0.062	5.250	92.267	702
S size 1:6 S/L	1	99.291	76.951	17.684	0.057	5.308	93.729	740
	3	99.742	77.399	17.567	0.021	5.014	93.869	780
	5	99.340	78.228	17.687	0.053	4.032	91.775	740
	10	99.606	77.040	17.353	0.032	5.576	92.503	750
M size 1:3 S/L	5	66.252	75.831	17.469	2.717	3.983	91.706	682
	10	71.392	76.946	17.214	2.303	3.538	91.822	688
	30	80.989	77.019	17.344	1.530	4.106	90.369	686
	60	87.253	76.699	17.189	1.026	5.086	90.410	674
	90	90.105	77.842	18.146	0.796	3.215	89.877	684
	120	92.794	77.664	16.923	0.580	4.833	88.777	685
L size 1:3 S/L	5	54.640	74.250	16.868	3.651	5.230	92.007	542
	10	61.041	75.738	16.571	3.136	4.555	91.084	545
	30	72.773	75.949	16.609	2.192	5.250	90.493	550
	60	81.534	76.693	16.823	1.486	4.997	90.166	561
	90	81.671	77.765	16.721	1.475	4.038	90.580	582
	120	82.196	77.784	16.916	1.433	3.867	90.239	605

[*] Solid to solvent ratio

Table C-3 Experimental results from co-solvent at 1:1 v/v extraction in 0.5-liter mixing tank

Cosolvent	Time	Fat removal	Content (dry basis)				Yield	Viscosity
	(min)	(%)	Polysaccharide	Protein	Fat	Others	(%)	(cp)
S size								
1:3 S/L*	1	96.930	78.607	17.511	0.247	3.635	93.456	775
	3	96.528	77.412	17.858	0.279	4.451	94.810	779
	5	97.161	77.917	16.142	0.229	5.713	94.951	790
	10	98.203	76.865	17.172	0.145	5.819	94.816	780
S size								
1:4 S/L	1	96.623	78.565	16.931	0.272	4.232	93.766	890
	3	97.814	77.517	17.200	0.176	5.107	93.876	810
	5	97.171	76.061	17.243	0.228	6.468	94.574	765
	10	98.953	77.348	17.396	0.084	5.172	91.997	710
S size								
1:5 S/L	1	98.875	78.830	17.237	0.091	3.842	92.522	771
	3	98.411	77.468	17.436	0.128	4.968	92.564	790
	5	99.071	77.194	17.388	0.075	5.343	91.927	750
	10	99.533	77.354	17.468	0.038	5.141	91.622	750
S size								
1:6 S/L	1	99.073	78.567	17.436	0.075	3.923	91.940	778
	3	99.539	78.082	16.161	0.037	5.720	92.570	796
	5	99.671	77.559	15.561	0.026	6.853	92.960	700
	10	99.346	77.016	17.652	0.053	5.279	92.283	705
M size								
1:3 S/L	5	59.650	75.488	17.797	3.248	3.466	91.106	660
	10	65.689	75.484	17.227	2.762	4.527	91.255	663
	30	74.486	76.192	17.358	2.054	4.396	90.367	665
	60	78.660	77.519	17.243	1.718	3.521	89.727	670
	90	83.059	77.412	17.378	1.364	3.846	90.569	674
	120	87.114	77.234	16.938	1.037	4.791	89.605	677
L size								
1:3 S/L	5	51.378	74.723	16.606	3.914	4.757	93.504	551
	10	57.231	75.966	16.719	3.443	3.872	92.042	597
	30	65.698	76.218	16.869	2.761	4.152	91.246	654
	60	70.246	77.469	16.685	2.395	3.451	91.503	660
	90	74.678	77.216	16.642	2.038	4.104	91.350	667
	120	75.269	76.651	16.657	1.991	4.702	91.241	670

*Solid to solvent ratio

Table C-4 Experimental results from co-solvent at 1:1 v/v extraction in 10-liter mixing tank

Cosolvent	Time (min)	Fat removal (%)	Content (dry basis) Polysaccharide	Protein	Fat	Others	Viscosity (cp)
S size							
1:3 S/L[*]	1	96.712	78.103	17.421	0.265	4.211	770
	3	97.001	77.934	17.648	0.241	4.177	778
	5	97.760	77.987	17.236	0.180	4.597	782
	10	98.107	77.876	17.195	0.152	4.777	796
M size							
1:3 S/L	5	59.606	75.477	17.674	3.252	3.597	664
	10	67.453	75.481	17.341	2.620	4.558	668
	30	73.111	76.432	17.237	2.164	4.167	671
	60	77.209	77.217	17.349	1.835	3.599	677
L size							
1:3 S/L	5	53.125	74.429	16.916	3.773	4.882	575
	10	59.392	75.875	16.875	3.269	3.981	593
	30	66.711	76.512	16.789	2.680	4.019	661
	60	70.404	77.138	16.884	2.382	3.596	669

[*] Solid to solvent ratio

APPENDIX D

Mass transfer coefficient and solvent prices

- **Mass transfer coefficient**

The mass transfer coefficient (k_La) for fat removal from TKP was determined from slope of the graph which plotted between $\ln c_{AS}/(c_{AS}-c_A)$ and time.

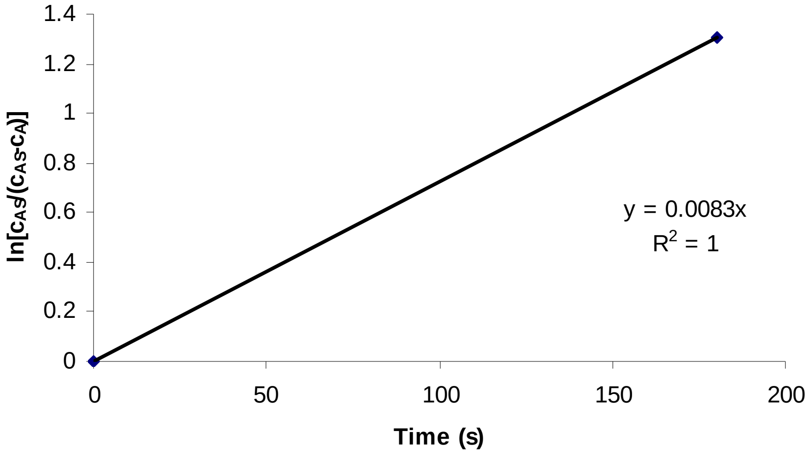

Figure D-1 Small-size TKP extraction with 95% ETOH

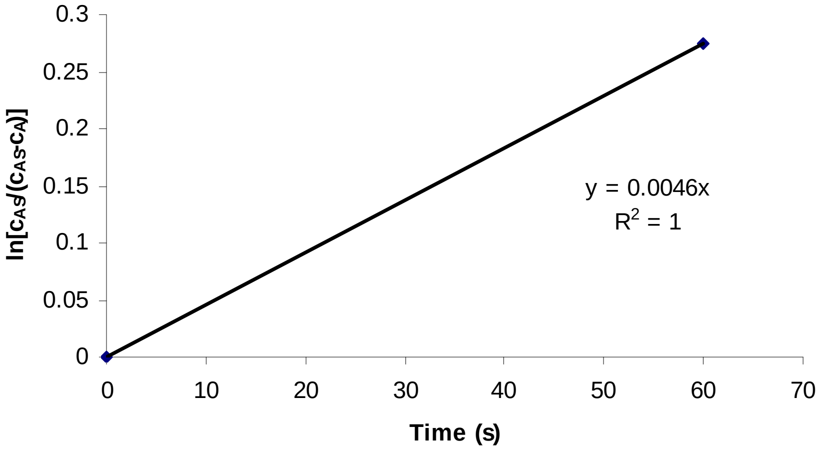

Figure D-2 Small-size TKP extraction with 99.9% IPA

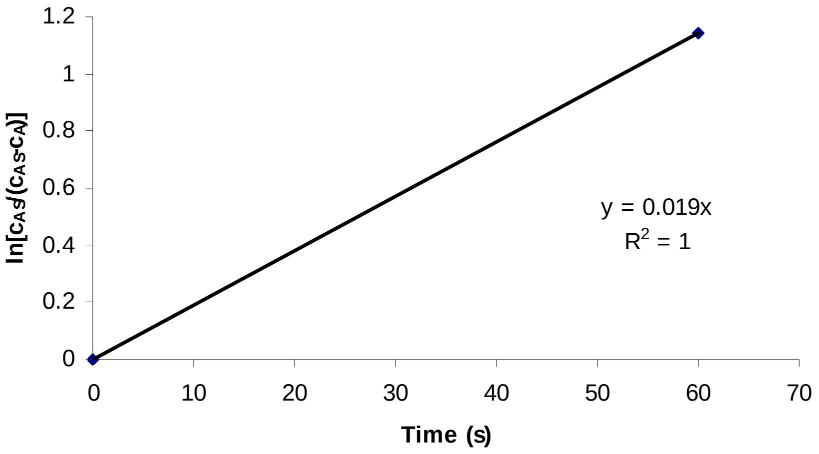

Figure D-3 Small-size TKP extraction with co-solvent at 1:1 v/v

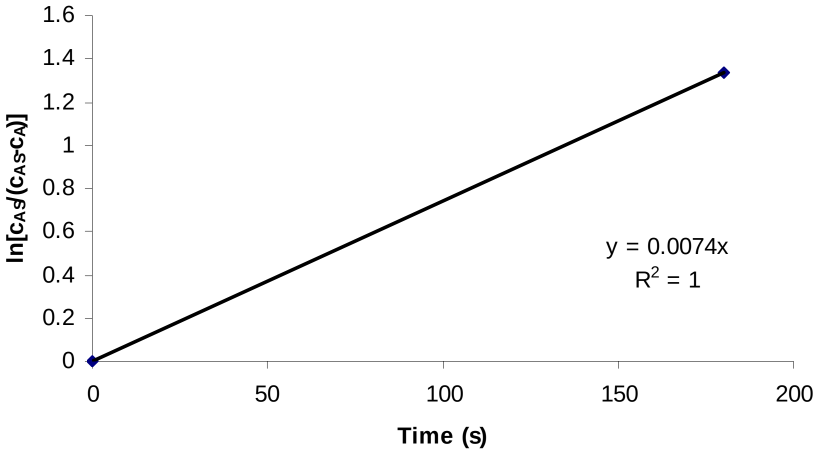

Figure D-4 Medium-size TKP extraction with 95% ETOH

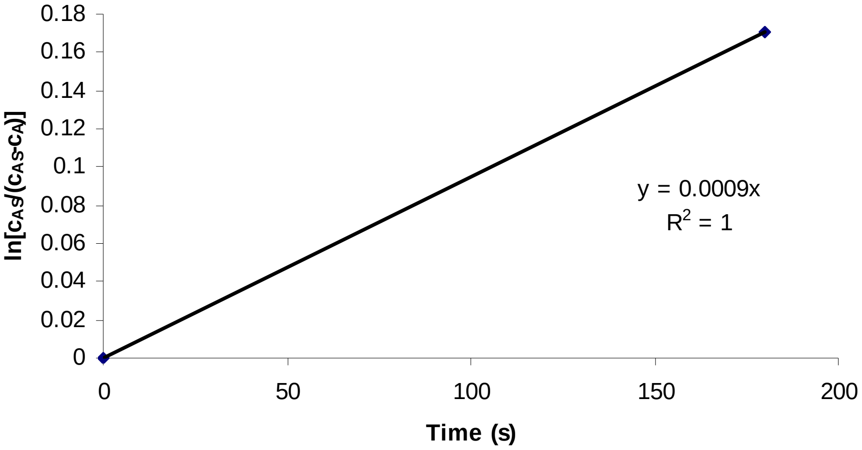

Figure D-5 Medium-size TKP extraction with 99.9% IPA

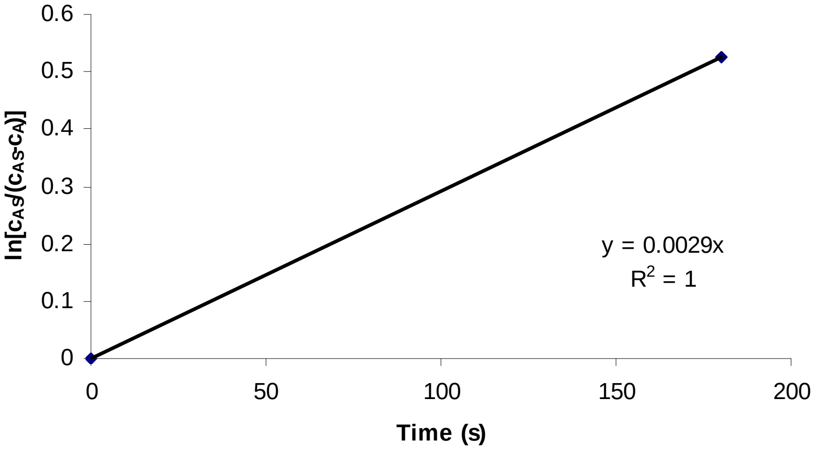

Figure D-6 Medium-size TKP extraction with co-solvent at 1:1 v/v

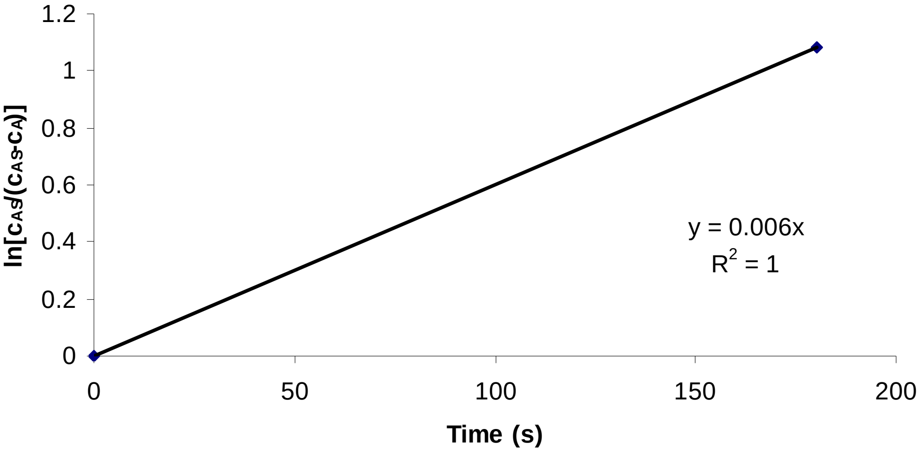

Figure D-7 Large-size TKP extraction with 95% ETOH

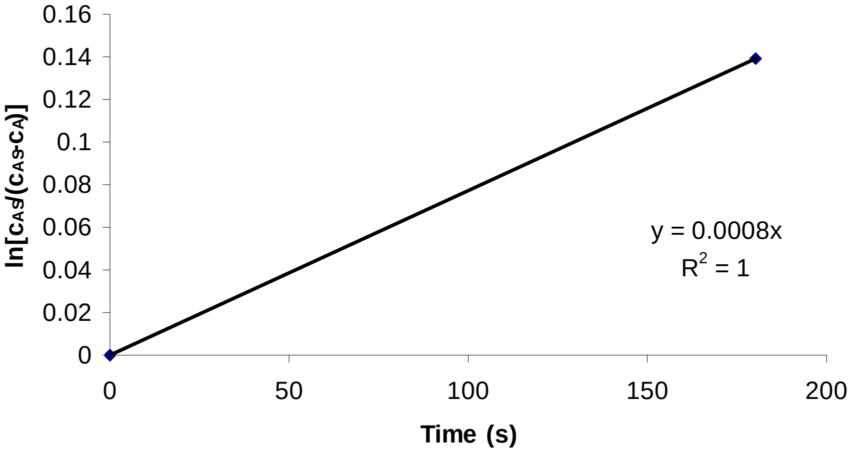

Figure D-8 Large-size TKP extraction with 99.9% IPA

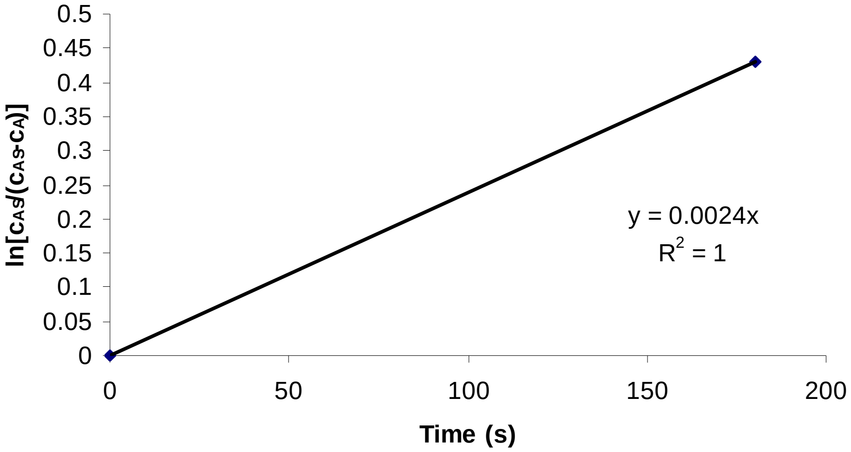

Figure D-9 Large-size TKP extraction with co-solvent at 1:1 v/v

- **Solvent prices**

Table D-1 Solvent prices

Solvent	Price/liter (Bath)
95% ETOH	17.18[a]
99.9% IPA	72.22[b]
1:1 v/v Co-solvent	44.70

Source: [a]Energy Policy and Planning Office, Ministry of Energy, Thailand
[b]SR.Lab, Thailand (April 27th, 2009)

APPENDIX E

List of publication

The 12th Asian Pacific Confederation of Chemical Engineering Congress
(APPChE 2008)

August 4th-6th, 2008
Dalian World Expo Center, Dalian, China

Fat removal from tamarind kernel powder using ethanol-isopropanol co-solvent in mixing tank

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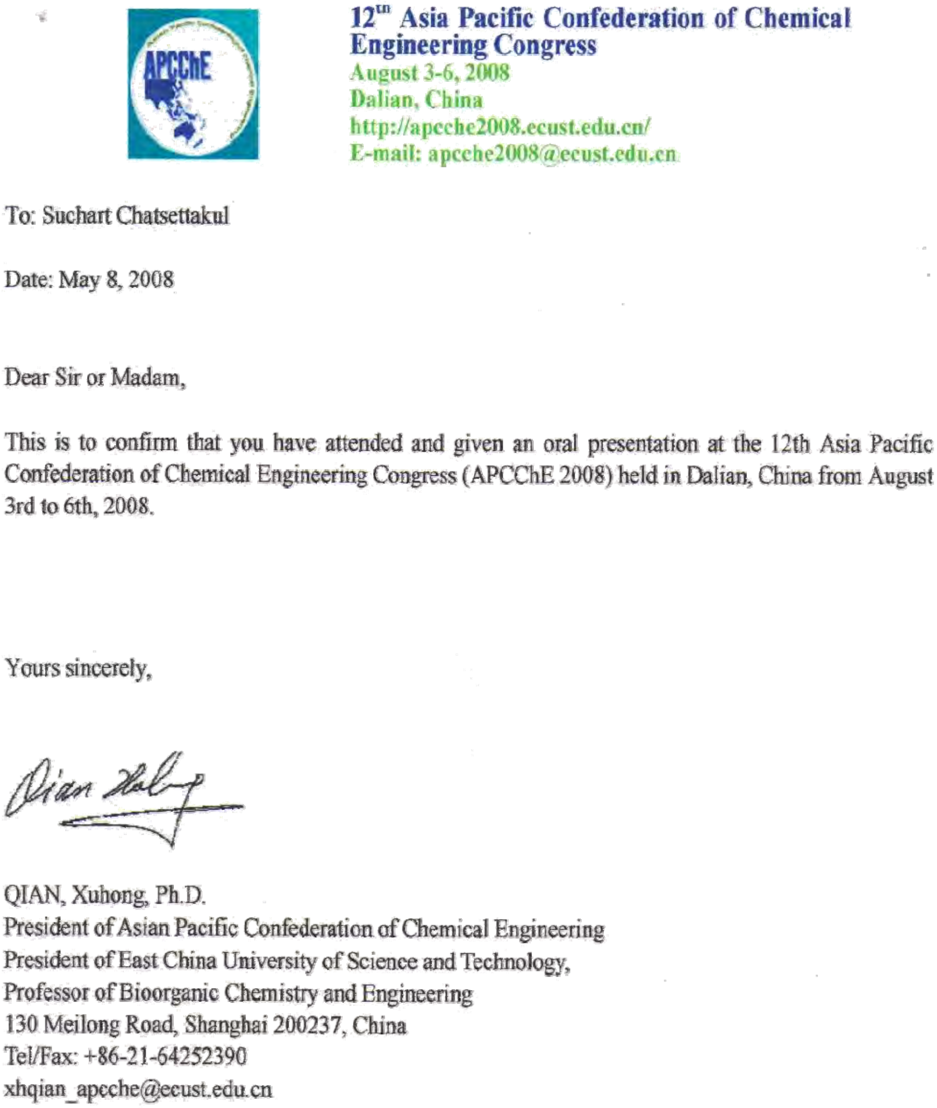

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To: Suchart Chatsettakul

Date: May 8, 2008

Dear Sir or Madam,

This is to confirm that you have attended and given an oral presentation at the 12th Asia Pacific Confederation of Chemical Engineering Congress (APCChE 2008) held in Dalian, China from August 3rd to 6th, 2008.

Yours sincerely,

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12thAPCChE Congress
Aug 4~6,2008, Dalian,China

Fat removal from tamarind kernel powder using ethanol-isopropanol co-solvent in mixing tank

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Abstract: Polysaccharide from Tamarind Kernel Powder (TKP) is the useful material in many industries such as for a thickening, stabilizing and emulsifying in food and pharmaceutical industry. The compositions of TKP compose of polysaccharide, protein, fat, fiber, and moisture. The effect of fat in TKP makes the product sticky and rancidity. Alcohol such as food grade ethanol and isopropanol is one of an alternative solvent for fat removal process instead of other harmful solvents that used in industry. Comparing to hexane and other solvents, ethanol, and isopropanol are less toxic, less flammable and free from restrictive governmental regulations. Thus, in this study, fat removal from TKP using ethanol-isopropanol co-solvent in mixing tank is investigated to determine the most suitable conditions that gave maximum % fat removal. The effects of TKP to solvent ratio (1:3, 1:4, and 1:5 g/ml) and extraction time (3, 5, 10, and 30 minutes) in fat removal were observed. The results showed that during this time period and all of TKP to solvent ratio studied using 95% wt ethanol extraction gave fat removal in the range of 40-60% while 99.9% wt isoproponal gave higher fat removal (97%). Finally, 95% wt ethanol mixed with 99.9% wt isopropanol as co-solvent at ratio of 1:1, 2:1, and 3:1(V/V) were used to extract fat from TKP using 1:3 TKP to solvent ratio for 3 minutes. The results indicated that the use of co-solvent is more preferable than single alcohol. Fat removal from 95% wt ethanol to 99.9% wt isopropanol ratio of 1:1, 2:1, and 3:1(V/V) were at 96, 88, and 79%, respectively.

Keywords: tamarind kernel powder, fat removal, ethanol, isopropanol, co-solvent

1. Introduction

Tamarind (*Tamarindus indica* Linn.) is in the *Caesalpiniaceae* Family, a common and commercial tree of Thailand and South East Asia countries. Tamarind seed is a by-product of the tamarind pulp industry which is abandoned about 20 thousand tons per year. From tamarind seed, Tamarind kernel powder (TKP) is separated and refined to produce very useful product namely, Tamarind seed polysaccharide (TSP) which can be utilized in many industries such as thickening, stabilizing, gelling agent, and emulsifying in food and pharmaceutical industry [1]. TSP is capable of foaming gels over a wide range of pH, including neutral and basic conditions. The compositions of TKP are polysaccharide 65.1–72.2%, fat 3.9–16.2%, protein 15.0–20.9%, crude fiber 2.5–8.2%, moisture 11.4–22.7%, and total ash 2.4–4.2% [2]. The effect of fat in TKP makes the product low quality such as sticky, rancidity, and non–free flowing during storage. Therefore, it is necessary to eliminate fat first. Andriamanantena et al. [3] investigated on yield of oil from six samples of tamarind seed. The kernel was extracted with hexane and a mixture of chloroform and methanol, the oil yield were 6.0–6.4% and 7.0–7.4%, respectively. Furthermore, the disadvantages of these solvents and other organic solvents used in industry are toxic, flammable, and restrictive to governmental regulations. Kwiatkowski et al. [4] used ethanol to extract oil from ground corn. The optimal conditions were at solvent to solid ratio of 4 mL/g corn, an ethanol concentration of 100%, extraction time of 30 min, and a temperature of 50 ^{0}C. Under these conditions, the extraction efficiency of single batch extraction and three-stage extraction were 70 and 93%, respectively. Zhang et al. [5] used isopropanol (IPA) to extract oil from cottonseed. The residual oil content of the cottonseed extracted with 97, 93, and 88% isopropanol were 1.5, 1.9, and 2.4% respectively. The reduction in water content improved the extraction efficiency of isopropanol. Thus, in this study, food grade ethanol and isopropanol are used for the extraction of fat from TKP in mixing tank.

The objectives of this study are to investigate the effect of solvent type, TKP to solvent ratio, co-solvent composition and extraction time on fat removal from TKP in mixing tank in order to determine the most suitable conditions for extraction.

2. Materials and methods

2.1 Materials

Tamarind kernel powder, 200 mesh sieving, was obtained from GM Ichihara Co., Ltd., Thailand as raw material. The 95% wt food grade ethanol (instead of 99.9% wt ethanol which is more toxic) and 99.9% wt isopropanol were used as the solvent for fat removal. The petroleum ether used in this study was analytical grade.

2.2 Methods

2.2.1 Batch extraction

The experimental batch extraction conditions were as followed (i) solvent type : 95% wt ethanol, 99.9% wt isopropanol and ethanol-isopropanol co-solvent; (ii) TKP:solvent ratio : 1:3 (TKP 40 g:solvent 120 ml), 1:4 (TKP 32 g:solvent 128 ml), 1:5 (TKP 27 g:solvent 135 ml); (iii) 95% wt ethanol to 99.9% wt isopropanol ratio as co-solvent (V/V) : 1:1, 2:1, 3:1; (iv) extraction time (min) : 3, 5, 10 and 30.

Batch extraction, fresh TKP (fat containing of 7.78%) was added to solvent in closed system mixing tank while stirring at 350 rpm and temperature controlling in water bath at 35°C. The series of experiments were conducted and each trial was terminated at different times. The defatted TKP was separated from solvent by a vacuum filter using Whatman filter paper No.1 and subsequently allowed to dry at room temperature. The final product was analyzed for the amount of fat by soxhlet extraction method (AOAC. 920.39) and calculated for the fat removal percentage. The fat removal percentage (dry basis) was express in (1).

$$Fat\ removal(\%) = \frac{initial\ fat\ (g/gTKP) - residual\ fat\ (g/gTKP)}{initial\ fat\ (g/gTKP)} \times 100 \qquad (1)$$

2.2.2. Fatty acid composition analysis

The fatty acid composition of extracted tamarind kernel oil in this study was analyzed using gas chromatography (GC). Agilent 7890S gas chromatograph equipped with column HP-88 100 m x 0.25 mm x 0.20 µm (J&W) and FID detector (285 °C) was used for the analysis. The operating conditions were temperature program : 100 °C hold 4 min, 3 °C/min to 240 °C hold 20 min, flow rate of carrier gas : 0.8 ml/min (Helium), and injection volume : 1 µl (split 1:100, 250 °C).

3. Results and discussion

3.1 Effect of solvent type and extraction time on fat removal

Figure 1. shows the fat removal obtained from the extraction of TKP with 99.9% wt isopropanol and 95% ethanol at TKP:solvent ratio 1:3 g/ml. for 3-30 minute. The experimental results show that at all of the extraction time period fat removal obtained from 99.9% wt isopropanol and 95% wt ethanol were all about 97 and 41% respectively. The extraction time did not make a significant difference to the % fat removal. Due to one or two minutes operation are difficult to handle, therefore, three minutes of extraction was chosen in our experiments. Regarding to the solvent type, 99.9% wt isopropanol has twice higher fat removal than 95% wt ethanol. To find the explaination, 99.9% wt ethanol was also carried out in order to compare fat solubilization in solvent. The result shows similar trend to 99.9% wt isopropanol (data not shown). This means that 5% wt water in ethanol solution plays an important role to fat solubilization in solvent. The water content in ethanol made low extraction efficiency because the fat can be solubilized lower in aqueous ethanol. This is in good agreement with previous works data published by Kwiatkowski et al. [4], Zhang et al. [5], and Franco et al. [6] which showed a drastic loss of extraction efficiency for ethanol and isopropanol in case of water presence

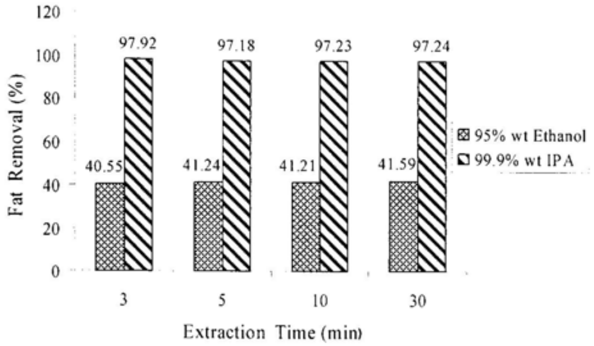

FIG.1 The effect of solvent type and extraction time on fat removal at 35 °C, 350 rpm, and TKP:solvent ratio 1:3.

3.2 Effect of TKP:solvent ratio on fat removal

Figure 2. shows the effect of TKP:solvent ratio on fat removal. The TKP:solvent ratio made a difference in the % fat removal. The 99.9% wt isopropanol showed a slightly upward trend but the 95% wt ethanol showed a highly upward trend as the TKP:solvent ratio was increased. The maximal % fat removal (56.81% for ethanol and 99.36% for isopropanol) occurred at TKP:solvent ratio of 1:5 g/ml in

both solvents. It means that concentration gradient of fat between TKP surface and bulk solvent play an important role over than extraction time. However, high solvent utilization has an impact on operating cost both recovery and raw material expenses. Thus, the suitable TKP:solvent ratio should be 1:3 g/ml. Regarding to the price of solvent, isopropanol is more expensive than ethanol. Therefore, in next investigation, co-solvent between ethanol and isopropanol was carried out in order to reduce raw material costs.

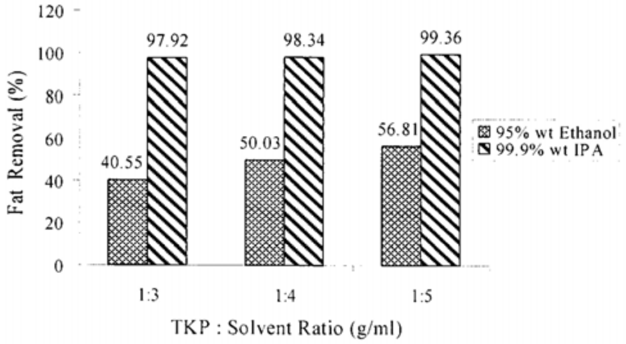

FIG.2 The effect of TKP:solvent ratio on fat removal at 35 ^{0}C, 350 rpm, and extraction time at 3 min.

3.3 Effect of co-solvent ratio on fat removal

The effect of co-solvent ratio (ethanol:IPA) on fat removal was illustrated in figure 3. As seen in Fig.3, the ratio of ethanol:IPA at 1:1 (V/V) gave higher % fat removal than 2:1 and 3:1 due to the effect of water content in ethanol solution. % Fat removal yielded from ethanol:IPA at 1:1 was 96.47 which was 1.5% lower than that obtained from 99.9% isopropanol. Thus, the use of co-solvent for extraction is more preferable than single alcohol.

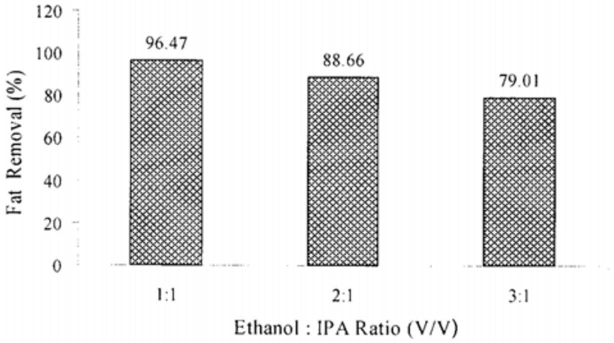

FIG.3 The effect of co-solvent (V/V) ratio on fat removal at 35 ^{0}C, 350 rpm, TKP:solvent ratio 1:3, and extraction time of 3 min.

The fatty acid composition of tamarind kernel oil extracted by 95%wt ethanol, 99.9% isopropanol, and co-solvent were shown in Table 1. At all of three extracted oil samples, the major fatty acids were linoleic acid, oleic acid, and palmitic acid. The oils contained a relatively large proportion of unsaturated fatty acids with linoleic acid (51-52%) in highest concentration. A small differences were observed in fatty acid composition of three extracted oil samples. Palmitic acid as saturated fatty acid with lower carbon atom was a significantly higher in ethanol extracts than in isopropanol and co-solvent extracts. However for rancidity reduction, unsaturated fatty acid should be removed. Therefore, the use of co-solvent meets our goal.

Table 1. Fatty acid composition of tamarind kernel oil

Fatty acid composition	% of total fatty acid Oil extracted by Isopropanol	 Oil extracted by Ethanol	 Oil extracted by Co-solvent[a]
Myristic acid (14:0)	0.1	0.1	0.1
Palmitic acid (16:0)	9.4	13.0	10.1
Heptadecanoic acid (17:0)	0.1	0.1	0.1
Stearic acid (18:0)	4.6	4.9	4.7
Oleic acid (18:1)	17.1	19.3	17.7
Vaccenic acid (18:1)	0.3	0.3	0.3
Linoleic acid (18:2)	51.7	52.1	51.8
Arachidic acid (20:0)	2.5	1.8	2.4
Eicosenoic acid (20:1)	1.0	0.9	1.0
Eicosadienoic acid (20:2)	0.1	0.1	0.1
Eicosatrienoic acid (20:3)	0.1	-	-
Eicosapentaenoic acid (20:5)	0.1	0.1	0.1
Behenic acid (22:0)	4.2	2.5	3.8
Tricosanoic acid (23:0)	0.2	0.1	0.2
Lignoceric acid (24:0)	7.4	3.5	6.5
Others	1.1	1.2	1.1

[a] 95% wt Ethanol:99.9% wt Isopropanol 1:1 V/V

Table 2. the percentage of fat, yield and viscosity of raw material and product.

TKP	% fat	% yield	Viscosity (cp) at 2% wt/V
Raw material	7.78	-	524
Product[a]	0.28	93.40	967

[a] TKP extracted by co-solvent 1:1 V/V

Table 2. shows the percentage of fat, yield and viscosity of raw material and product. Under the suitable condition for fat removal, the fat in TKP sharply decreased from 7.78 to 0.28% so that higher product quality are obtained. The product had almost twice higher viscosity than raw material

(524 cp. for raw material and 967 cp. for product) due to the fat in TKP was removed. The high quality product as low fat and high viscosity had a potential for using in food industry.

4. Conclusions

It can be concluded that 99.9% isopropanol was more effective than 95% ethanol for fat removal. The reduction in water content improved the extraction efficiency of co-solvent. The suitable condition for fat removal from TKP was extraction time of 3 min, TKP:solvent ratio 1:3 g/ml, and co-solvent 1:1 V/V. At this condition, the fat removal was 96%. High quality product as low fat and high viscosity had a potential for using in food industry.

Acknowledgements

This work is supported by TRF-Master Research Grants. The authors thank Mr.Chai Sukhochaiyakit, GM Ichihara (Thailand) Co., Ltd., for his support in Tamarind kernel powder as raw material.

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Fat Removal from Tamarind Kernel Powder using Ethanol-isopropanol co-Solvent

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The Certificate of Participation

to

Mr. Suchart Chatsettakul

for presenting

Fat Removal from Tamarind Kernel Powder
using Ethanol-isopropanol co-Solvent

in

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Abstract

Polysaccharide from Tamarind Kernel Powder (TKP) is the useful material in many industries such as food and pharmaceutical industries for a thickening, stabilizing and emulsifying agent. The compositions of TKP are polysaccharide, protein, fat, fiber, moisture, and ash. Fat in TKP makes the product sticky and rancidity; therefore, it should be removed firstly. Alcohol such as food grade ethanol and isopropanol is one of an alternative solvent for fat removal process instead of other harmful solvents that currently used in industry. As a result, in this study, fat removal from TKP using ethanol collaborate with isopropanol as a co-solvent in mixing tank is investigated to determine the most suitable condition. Regarding to solvent type, co-solvent ETOH:IPA at 1:1 V/V gave a high % fat removal (96%) which is similar to 99.9 % V/V IPA (97%). The yield of fat removal decreased with the increase of the water content in solvent. Concerning to particle size, medium size showing 74% fat removal in 30 min, is suggested to use in this process. The suitable condition for 74 % fat removal and 685 cp of product viscosity was at extraction time of 30 min, TKP:solvent ratio 1:3 g/ml, medium particle TKP (0.5 mm), and co-solvent 1:1 V/V. At this condition, high quality product, low fat and high viscosity, was obtained and potentially used in food industry as viscosity enhancer.

Keywords: tamarind kernel powder, fat removal, ethanol, isopropanol, co-solvent

1. Introduction

Tamarind (*Tamarindus indica* Linn.) is belonged to the family *Leguminosae* (*Fabaceae*), a common and commercial tree of Thailand and South East Asia countries. The tamarind seed is a by-product of tamarind pulp industry. Tamarind kernel powder (TKP) is separated and subsequently refined from tamarind seed in order to produce a very useful product named Tamarind Seed Polysaccharide (TSP), an expensive natural gum. TSP provides a variety of utilizations in many fields. In food and pharmaceutical industries, it was applied to be thickening, stabilizing, gelling, and emulsifying agent [1]. In addition, TSP is capable of forming gel over a wide range of pH, including neutral and basic conditions. The compositions of TKP are 65.1–72.2% polysaccharide, 3.9–8.0% fat, 15.0–20.9% protein, 2.5–8.2% crude fiber, 11.4–22.7% moisture, and 2.4–4.2% total ash [2]. The effect of fat in TKP makes the product low quality such as sticky, rancidity, and non–free flowing TKP during storage. Therefore, it is necessary to eliminate fat first. Generally, organic solvents such as hexane and a mixture of chloroform and methanol are used for the oil extraction from TKP [3]. However, the disadvantages of these solvents are toxic, flammable, and restrictive to governmental regulations. From our reviews, ethanol (ETOH) and isopropanol (IPA) are also applied for oil extraction from natural agricultural crops such as ground corn [4], cotton seed [5], soybean [6], and rosehip seed [7] and are used in many consumer products including food, drug, and cosmetics. Therefore, food grade ethanol and IPA give good potential in trial to extract fat from TKP. In TKP production, the energy consumption of seed milling step is very high. It is necessary to investigate the suitable particle size which will give high product yield and high fat removal at lower milling energy consumption. In fact, the fat in small particles can be easily and rapidly extracted. But it poses not only the difficulty in operation due to slow drainage rates at high pressure drop during filtration but also high milling energy consumption.

The objectives of this study are to investigate the effect of solvent type, particle size of TKP, and extraction time on fat removal from TKP in mixing tank in order to determine the most suitable condition for extraction.

2. Experimental

2.1 Raw material

Tamarind kernel powder was obtained from GM Ichihara Co., Ltd., Thailand as raw material. The 95% V/V food grade ethanol (instead of 99.9% V/V ethanol which is more toxic and expensive) and 99.9% V/V isopropanol were used as the solvent for fat removal. The petroleum ether used in this study was analytical grade.

2.2 Batch extraction

Batch extraction conditions were as followed (i) solvent type: 95% V/V ethanol, 99.9% V/V isopropanol and ethanol-isopropanol co-solvent (ii) particle size: 0.028, 0.5, and 1.0 mm (iii) extraction time (min): 5, 10, 30, 60, 90, and 120.

Batch extraction, 40 g of fresh TKP (fat containing of 8.05%) was added to 120 ml of solvent in closed system mixing tank while stirring at 400 rpm and temperature controlling in water bath at 35°C. The series of experiments were conducted and each trial was terminated at different times. The defatted TKP was separated from solvent by a vacuum filter using Whatman filter paper No.1 and subsequently allowed to dry at room temperature. The final product was analyzed for the amount of fat by soxhlet extraction method (AOAC. 920.39) [8] and calculated for the fat removal percentage. The fat removal percentage (dry basis) was expressed in equation (1), where W_i and W_r are initial and residual fat (g/g TKP), respectively.

$$\mathit{Fat\ removal}\,(\%) = \frac{W_i - W_r}{W_i} \times 100 \qquad (1)$$

2.3 Scanning electron microscopy (SEM) analysis

The surface characteristic of the TKP samples were observed by scanning electron microscope (SEM) (JEOL, JSM-5410LV, Japan)

3. Results and Discussions

3.1 Effect of solvent type on fat removal

As shown in Fig. 1, the fat removal obtained from the extraction of small size TKP with variety of solvents. The results show that fat removal obtained from 99.9% IPA and 95% ethanol were around 97 and 41%, respectively. To find the explanation, 99.9% ethanol was also carried out in order to compare fat solubilization in solvent. The result shows similar trend to 99.9% IPA. This means that 5% water in ethanol solution plays an important role to the solubility of fat in solvent. The water content in ethanol reduces extraction efficiency because the fat can be solubilized lower in aqueous ethanol. This is in good agreement with previous works data published by Kwiatkowski et al. [4], Zhang et al. [5], Franco et al. [7], and Chatsettakul et al. [9] which showed a drastic loss of extraction efficiency for ethanol and isopropanol in case of water presence. Absolute ethanol and IPA were effective solvent but absolute ethanol was more harmful. Focusing on the price of solvent, absolute IPA is more expensive than 95% ethanol. Therefore, co-solvent between 95% ethanol and 99.9% IPA was carried out in order to reduce solvent costs.

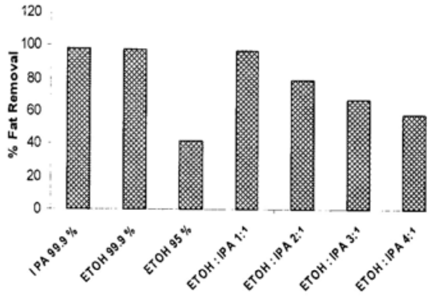

Fig.1 The effect of solvent type on fat removal at 35 ^{0}C, 400 rpm, 3 min of extraction time, and TKP:solvent ratio 1:3.

Moreover, Fig. 1 also shows the ratio of ETOH:IPA at 1:1 (V/V) gave higher % fat removal than 2:1, 3:1, and 4:1 due to the effect of water content in ethanol solution. % Fat removal yielded from ethanol:IPA at 1:1 was 96 which was 1% lower than that obtained from 99.9% IPA. Thus, the use of co-solvent for extraction is more preferable than single alcohol.

3.2 Effect of particle size and extraction time on fat removal

The effect of particle size and extraction time on fat removal was illustrated in figure 2. As seen in figure 2, the results show a decrease in percentage of fat removal when the particle size increase. The % fat removal from small size TKP gave the highest extraction rate and the highest yield (98%) within a few minutes of extraction time because it has higher specific area than the large one. In small particles, the cell wall was destroyed by grounding step resulting to exhibit more fat content on the particle surface. Therefore, the fat can be easily and rapidly extracted under no internal mass transfer limitation. Regarding to medium and large TKP, the maximum % fat removal (84% for medium size and 75% for large size) occurred at 90 min extraction. At 30 min of extraction, medium and large TKP % fat removal were about 74 and 66 %, respectively, which had 10% fat removal lower than 90 min. Considering on the mixing energy consumption, the extraction time at 30 min was more suitable. For the large particle, penetration of solvent to the inside of particle takes longer times. Thus, the internal mass transfer in large particle was limited and resulted in a decrease in the amount of fat extracted. These results are consistent with that reported by Amarasinghe et al. [10]. Although a small size TKP is more effective for extraction but it poses the problem of huge energy consumption in size reduction and difficulty to handle. Therefore, the larger particles were chosen to avoid above problems. At 30 min extraction, %fat removal from medium size was 8% higher than that obtained from large size. Thus, the use of medium size TKP for extraction is more preferable than large TKP.

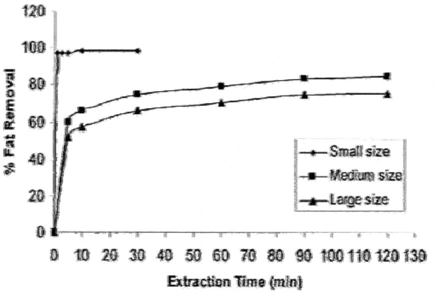

Fig.2 The effect of particle size and extraction time on fat removal at 35 ^{0}C, 400 rpm, and TKP:solvent ratio 1:3.

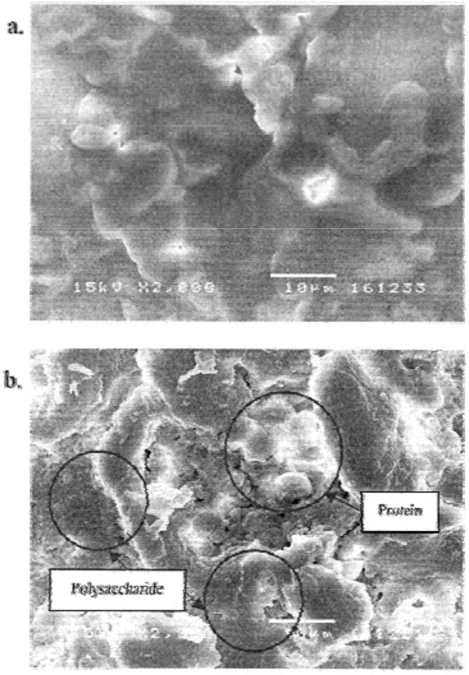

Fig. 3 Scanning electron microscopy images of large TKP particle. (a) Untreated. (b) At 30 min co-solvent extraction.

Table.1 shows the compositions, yield, and viscosity of raw material and product.

TKP	Composition (%)				Yield (%)	Viscosity (cp) at 2% wt/V
	Fat	Polysaccharide	Protein	Others		
Raw material	8.05	70.47	17.33	4.15	-	540
Small size product at 3 min	0.28	76.86	18.13	4.73	90.59	790
Medium size product at 30 min	2.05	74.02	18.97	4.96	92.85	685
Large size product at 30 min	2.76	73.35	18.56	5.33	93.56	654

Table.1 shows the compositions, yield, and viscosity of raw material and product. The product viscosity was higher than raw material due to the increasing of polysaccharide content as a viscosity enhancer. The fat in TKP sharply decreased from 8.05 to 0.28, 2.05, and 2.76% for small, medium, and large size, respectively, so the higher product quality is obtained. The high quality product showing low fat and high viscosity had a potential for using in food industry. The SEM images of the large TKP surface are illustrated in Fig. 3. There was a significant fat covering on the surface of TKP particle (Fig. 3(a)). At 30 min co-solvent extraction, most of the fat from TKP had been extracted (Fig. 3(b)) resulting in clearing surface.

4. Conclusion

It can be concluded that 99.9% IPA was more effective than 95% ethanol for fat removal. The water content reduction improved the extraction efficiency of co-solvent. Co-solvent ETOH:IPA at 1:1 V/V gave high extraction efficiency as well as absolute IPA. The extraction was more effective with small particle due to the increase in surface area. To avoid high energy consumption for size reduction and operation problems, the medium size TKP was suggested to use in fat removal process. The suitable condition for fat removal from TKP was at extraction time of 30 min, TKP:solvent ratio 1:3 g/ml, medium particle TKP (0.5 mm), and co-solvent 1:1 V/V. At this condition, the fat removal and product viscosity were 74% and 685 cp, respectively. High quality product as low fat and high viscosity had a potential for using in food industry as viscosity enhancer.

5. Acknowledgement

This work is supported by TRF-Master Research Grants (MRG-OSMEP505E016). The authors thank Mr.Chai Sukhochaiyakit, GM Ichihara (Thailand) Co., Ltd., for his support in Tamarind kernel powder as raw material.

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VITA

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