Diffusion Properties of Garcinia Fruit Acids (Garcinia atroviridis)

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ABSTRACT

The diffusion properties of garcinia fruit acids (Garcinia atroviridis) were investigated in a batch reactor. The influences of two variables were studied: material thickness and extraction temperature. Stirring was continuous to assure turbulent flow inside the vessel. Garcinia fruits were sliced into infinite slabs of two different thicknesses, 2.5 and 4.0 mm. Each group of a given thickness was then separated into three sub-groups. Each sub-group was then subjected to individual extraction temperatures of 50 °C, 60 °C and 70 °C. Distilled water was used as the extraction solvent. The experiment was carried out in well-agitated vessel with a ratio of water to fruit material of 30:1 (w/w). Each experiment took an operating extraction time of 3 h; unless it was under equilibrium conditions, in which case it was left for a further 24 h. Solid samples were taken at different periods and the amount of total acids determined. The total acid concentration was then calculated. Diffusion properties like solute diffusivities, times of cell plasmolysis, equilibrium distribution coefficients and volumetric mass transfer coefficients were determined to be in the range of 7.95×10⁻¹⁰ - 12.14×10^{-10} m²/s for diffusivities, 346.3 - 1158.4 s for times of cell plasmolysis, 1.06 - 1.07 for equilibrium distribution coefficients, and 3.85×10^{-4} - 9.40×10^{-4} s⁻¹ for volumetric mass transfer coefficients. These values are thus available for simulation purposes and optimization of the processing operation.

Keywords: Garcinia fruit, *Garcinia atroviridis*, diffusion properties, mass transfer characteristics, diffusivity, equilibrium distribution coefficient, time of cell plasmolysis, mass transfer coefficients

INTRODUCTION

Obesity is defined as an increase of adipose mass resulting from a chronic imbalance between energy intake and expenditure [1]. Potential risks of obesity include cardiovascular diseases, diabetes, cancer and hormonal imbalances (especially in women), leading to sterility [2]. It was reported that, according to the world health organization, there are over 300 million obese adults globally [3]. The reason is due to low levels of physical activity and sedentary lifestyles [4].

Using weight loss drugs that suppress appetite, reduce food intake and increase energy expenditure have been found to be effective but are unfortunately accompanied by adverse side effects [5]. Hence, supplementation with safe and natural products in addition to a healthy diet and exercise may be helpful [6].

Among a number of natural essences, (-)-hydroxycitric acid (HCA), the principal acid of garcinia fruit (*Garcinia atroviridis* or *G. cambogia*) has been widely investigated [1-9]. HCA is a potent metabolic regulator of obesity and lipid abnormalities in mammalian systems. The antiobesity potency of HCA has been clinically screened and confirmed [10,11]. It was shown to be a competitive inhibitor of adenosine 5'-triphosphate (ATP) citrate lyase, the enzyme that catalyzes the extramitochondrial cleavage of citrate to oxaloacetate and acetyl CoA. This action reduces the acetyl CoA pool, thus limiting the availability of two-carbon units required for the first step in the biosynthesis of fatty acids and cholesterol [1,8,11-13].

For the last 30 years, considerable research has been focused on using HCA from fruit extracts; surprisingly, no operation or any physical information such as diffusion properties have been reported. Thus, to fill up some engineering gaps, this paper aims to investigate the mass transfer properties of diffusion like solute (acid) diffusivities, mass transfer coefficients, equilibrium distribution coefficients, and times of cell plasmolysis. This information will be available for effective operation and simulation purposes, especially in industrial processes.

Garcinia fruits are native to tropical Asia. The first common strain, *Garcinia cambogia*, is generally found in Southern India; while the other strain, *Garcinia atroviridis*, is commonly found in Peninsular Malaya [14]. The plants are semi-growing wild and have been used as food for centuries [15]. The fruits contain 10 - 30 % HCA on a dry basis [16], while in some reports, 16 - 18 % HCA on a wet basis [7,10].

In this study, *G. atroviridis* were used as raw materials due to their ready availability for the experiment since the plants are widely cultivated especially in Southern Thailand and Northern Malaysia.

MATERIALS AND METHODS

Sample Selection and Preparation

Fresh unripe garcinia fruits (*G. atroviridis*), green in color, were purchased locally from fruit gardens in Kiriwong Sub-district, Nakhon Si Thammarat, Southern Thailand. The average weight of the fruit was selected to be 300 - 350 g. The unpeeled fruits were cut into flat slabs 2.5 and 4 mm in thickness, depending on the specific experiment plan. The diameter of the slab was in the range of 7 - 10 cm. The skin was not removed to minimize the moisture diffusion and evaporation in the radial dimension. The fruit slabs then kept at 4 °C with a relative humidity of 75 % RH until used for the experiment.

Quality of Raw Material: Some Physical and Chemical Properties of Garcinia Fruit

To obtain density and initial moisture content of garcinia, thirty fruit samples were used. The skin was removed manually with a knife and the fruits were cut into small pieces 2 - 4 mm in length. The density of garcinia was measured by a 25-ml picnometer and the moisture content was determined by vacuum drying using a vacuum oven. The color according to the Hunter system of a fresh fruit slice was examined by a colorflex instrument, model JP 7100 F. In addition, the hardness of unpeeled fruits was measured by a Texture analyzer model TA-TX 2i, Stable Microsystem, operating with a solid cylinder probe, diameter 2 mm, punching into the whole fruit at 10 different points around its circumference with a penetration distance of 5 mm and a velocity of 1.5 mm/s, as well as a compression force of 50 kg. Total acidity as citric acid was carried out according to the method of AOAC [17], while total soluble solid was determined by using a hand-held Refractometer.

The Diffusion Experiments

The diffusion experiments were done by batch extraction over a period of 3 h. The effect of extraction temperature and thickness of prepared fruits were investigated. Temperatures in the vessel (water bath) were varied from 50, 60 and 70 °C. Unpeeled sliced fruits were divided into two groups of two different thicknesses, 2.5 and 4 mm. Each experiment was performed in duplicate.

The ratio of water to raw material was 30:1 (w/w). Stirring was continuous to maintain a turbulent flow. Samples of the solid phase were taken at 2, 4, 6, 8, 10, 15, 20, 40, 70 and 180 min, respectively. Acidity as citric acid in the solid sample was determined by titration with 0.05 N NaOH. The moisture content of the solid phase at the end of each experiment was also determined. The purpose of each diffusion

experiment was to establish solute diffusivities, times of cell plasmolysis, solute equilibrium distribution coefficients and volumetric mass transfer coefficients.

Evaluation of Solute Diffusivities

The diffusivity was calculated using analytical solutions of Fick's second law of unsteady-state diffusion, developed by Siripatana. The solution is available for symmetrical solids and the mathematical expression is as follows [18]:

$$E = \frac{\overline{x} - x^*}{\overline{x}_0 - x^*} = \frac{\overline{y} - y^*}{y_0 - y^*} = \sum_{n=1}^{\infty} C_n \exp\left[-q_n^2 \tau\right]; \quad \tau = \frac{Dt}{a^2}$$
 (1)

where E is the remaining fraction of extractable solute.

 \overline{x} , \overline{x}_0 , x^* are the average concentration, initial concentration and equilibrium concentration of the solute in solid phase, respectively.

 \overline{y} , y_0 , y^* are the average concentration, initial concentration and equilibrium concentration of the solute in liquid phase, respectively.

 q_n is eigenvalues.

 C_n is a series coefficients obtained by solving the equation of eigenvalues.

au is Fick's number.

D is the solute diffusivity.

a is the characteristic length; and

t is the time of diffusion.

For an infinite slab, the equation for q_n [19] and C_n [18] is shown:

$$\tan q_n = \frac{q_n \alpha Bi}{\alpha q_n^2 - Bi} \tag{2}$$

$$C_{n} = \frac{2\nu\alpha(\alpha+1)}{\alpha^{2} \left[\left(\frac{q_{n}^{2}}{Bi} - \frac{\nu}{\alpha} \right)^{2} + q_{n}^{2} \left(1 - \frac{1}{Bi} \right) + \frac{\nu^{2}}{\alpha} \right]}$$
(3)

where α is the draft of extraction; $\alpha = \hat{E}m/\hat{R}$

 \hat{E} is the volume of extract phase.

 \hat{R} is the volume of solid phase.

m is the solute equilibrium distribution coefficient.

Bi is the Biot number of mass transfer; and

 ν is the shape factor of the solid phase calculated by the ellipsoid solid model [18]. The value is 1, 2 and 3 for an infinite slab, infinite long cylinder and sphere, respectively.

After a long period of time, where $\tau > 0.1$, the extraction process approaches essentially a pseudo-steady state condition where E decreases exponentially. This suggests a first order extraction kinetics and the process can be described by a simple model. Hence, Eq. (1) is thus simplified to

$$E = \frac{\overline{x} - x^*}{\overline{x}_0 - x^*} = \frac{\overline{y} - y^*}{y_0 - y^*} = C_1 \exp\left[-q_1^2 \tau\right]; \quad \tau = \frac{Dt}{a^2}$$

$$\tag{4}$$

Thus, solute diffusivity may be determined from a plot of ln(E) against time t, where the slope $(-q_1^2D/a^2)$ and the dimensionless concentration in the solid phase was selected for the calculation.

Evaluation of Time of Cell Plasmolysis

The time of cell plasmolysis (t_p) is the time lag in the diffusion process from/to solid particles which is not taken into account in the diffusion model. It is calculated using the following expression [18].

$$t_p = \frac{\ln C_1 - \ln j}{S} \tag{5}$$

where

j is the intercept on y-axis of the relationship between ln(E) and t according to Eq. (4). In the actual extraction where cell plasmolysis takes place, $j \neq C_1$.

S is the slope of the linear portion of the extraction curve according to Eq. (4).

 C_1 is the parameter defined in the case of no cell plasmolysis and expressed in Eq. (6) [18]:

$$C_{1} = \frac{2\alpha(\alpha+1)}{(\alpha+1)+(\alpha q_{1})^{2}}$$
 (6)

Evaluation of Solute Equilibrium Distribution Coefficient

To determine the solute equilibrium distribution coefficient, each batch system was left overnight to come to equilibrium and reach a steady-state process. The solute

concentration in the solid phase (x^*) is thus in equilibrium with that in the liquid phase (y^*) . Nevertheless, the two values need not be the same, depending on the solid phase composition and the liquid phase type. The relationship is expressed in Eq. (7)

$$y^* = mx^* \tag{7}$$

where m is the solute equilibrium distribution coefficient.

 x^* is obtained by determining the moisture content of the solid material at equilibrium. Then the corresponding equilibrium concentration can be calculated. However, the density of the inert solid must be measured and calculated.

 y^* is easily determined due to fact that there is no inert solid in liquid phase.

Evaluation of Volumetric Mass Transfer Coefficients

Volumetric mass transfer coefficient (K_x) is expressed as follows [18]:

$$K_{r} = K'A_{\rho} \tag{8}$$

where A_e is the surface area per unit volume of the solid phase; $A_e = A/V$

A is the area of the solid phase where mass transfer takes place.

V is the volume of the solid phase; and

K' is the mass transfer coefficient calculated by the equation

$$Sh = \frac{2K'a}{D} = \frac{2}{\nu} \left(\frac{\gamma}{\gamma + 1}\right) q_1^2 \tag{9}$$

here

Sh is a Sherwood number as defined by Spaninks [19].

 γ is α in case of batch extraction and $-\alpha$ in case of continuous extraction.

One difficulty in applying Eq. (4) and (9) to even simple geometries is that root q_1 depends on, at least, α , ν and Bi. Numerous tasks were carried out for this and finally a simple relationship, proposed by Siripatana was given for approximation of the Sherwood number at infinite Bi (Sh_{∞}) [18].

$$Sh_{\infty}/2 = 2.4391 - 3.7766 \times 10^{-1}/\gamma + 2.1857 \times 10^{-2}/\gamma^2$$
 (10)

The relation is only applicable for an infinite slab material, especially for $0.1 < \alpha < \infty$. In addition, the Biot number of mass transfer is given by a following equation:

$$Bi = \frac{K_x ma}{D} \tag{11}$$

RESULTS AND DISCUSSION

Quality of Raw Materials: Some Physical and Chemical Properties of Garcinia Fruit.

Garcinia fruits, less than 3 months old which were green in color, were selected for the investigation. Corresponding properties are shown in **Table 1** [15].

Table 1 Some physical and chemical properties of garcinia fruit (*Garcinia atroviridis*).

Composition	$\overline{\mathbf{X}} \pm \mathbf{S}\mathbf{D}$	_
Moisture content (% wet basis)	86.47±1.35	
Total acidity as citric acid (%w/w)*	5.54±0.13	
Soluble solid (%)	6.34±0.25	
Hardness (N)	85.14±1.89	
Density (kg/m ³)	$1,035.00\pm0.03$	
Color value:		
L (lightness)	66.30 ± 0.33	
A (light green attribute)	-1.42 ± 0.69	
B (yellow attribute)	27.57±1.08	

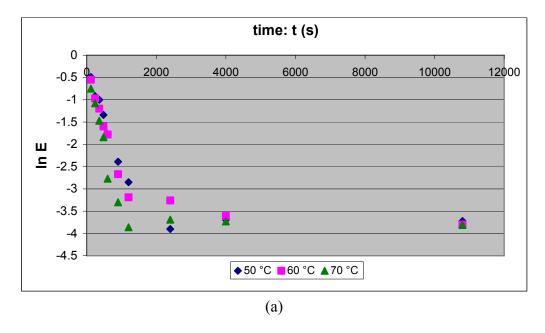
^{*} value calculated based on the basis of g of acid per 100 g of raw materials.

Source: W Rittirut and C Siripatana [15]

The Diffusion Experiments

Evaluation of Solute Diffusivities

The relationship between the remaining fraction of extractable solute " $\ln E$ " and the time of extraction "t" for different thicknesses of infinite slab materials at different extraction temperatures is shown in **Figures 1** and **2**. Slab material with a thickness of 2.5 mm, reaches a steady-state after ~2000 s (**Figure 1a**). Only the linear portion was selected for diffusivity calculations and the selected time range was 0 - 1200 s (**Figure 1b**).



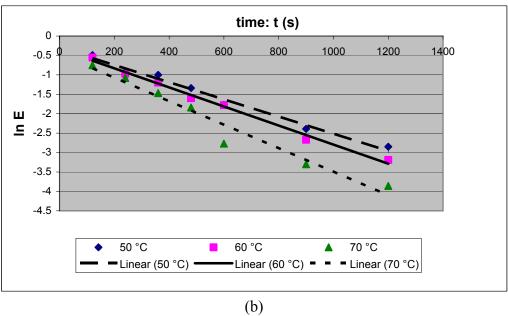


Figure 1 The relationship between $\ln E$ and t for garcinia slab materials with a thickness of 2.5 mm at different extraction temperatures according to (a) the overall extraction time; and (b) the selected time for diffusivity calculation.

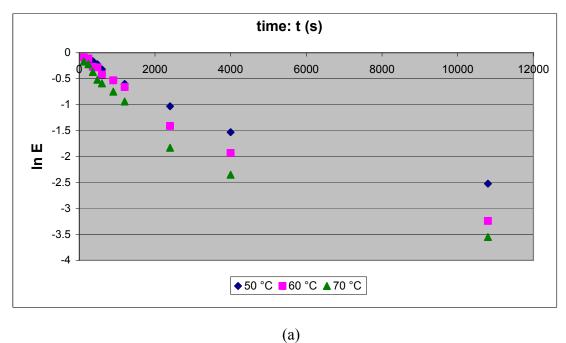
In the case of 4.0 mm-thick materials a steady-state condition was not reached (**Figure 2a**). The reason for this is not yet clear. However, we postulate that the interactions between the internal solid structure of the materials and the solute components result in the steady-state condition not being reached. Linear portions from 0 - 1200 s were selected for diffusivity calculations (**Figure 2b**).

The solute (acid) diffusivities are shown in **Table 2**. It was found that the higher the temperature, the higher the diffusivity obtained. In contrast, the thicker the material, the slightly lower the diffusivity observed. The explanation is that the thicker material slowed down the rate of heat transfer [20], especially at the early period of extraction as well as the plasmolysis. Another possible cause is the different extent of surface washing where the thinner the slab thickness, the greater the effect of surface damage due to cutting. In addition, the values of solute diffusivities here were established for the total acids. Although HCA in the fruit of *Garcinia cambogia* is found to be the major part of organic acids (the content is greater than 93.5 %), other acids are also presented, these are citric, malic and two other unidentified acids [10].

It was also found that the order of magnitude of acid diffusivities in garcinia fruit was the same as that of water, which is 10^{-9} - 10^{-11} m/s [20] (**Table 2**). For instance, Schwartzberg and Chao (1982), reported that the water diffusivity in apple and sugar beet slices at 75 °C was $11.8\pm0.9\times10^{-10}$ and $8.30\pm0.9\times10^{-10}$ m/s, respectively [21]. In addition, each component diffusivity not only depended on the temperature, but also their structure and composition of raw materials [20].

Table 2 Solute (acid) diffusivities in garcinia fruit of different slab thicknesses at different temperatures.

Thickness (mm)	Temperature (°C)	Solute diffusivity: D $(m^2/s) \times 10^{-10}$
2.5	50	8.86
2.5	60	8.87
2.5	70	12.14
4.0	50	7.95
4.0	60	8.42
4.0	70	11.26



(a)

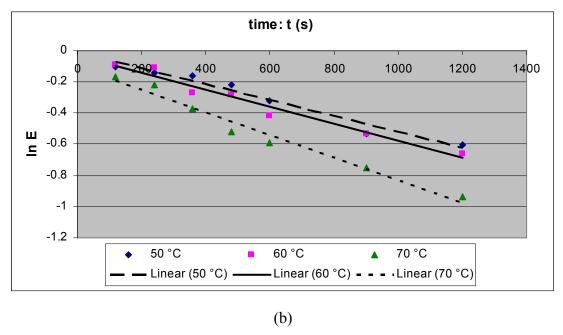


Figure 2 The relationship between $\ln E$ and t for garcinia slab materials with a thickness of 4.0 mm at different extraction temperatures according to (a) the overall extraction time; and (b) the selected time for diffusivity calculation.

Evaluation of Time of Cell Plasmolysis

Since the diffusion theory is firstly developed for chemical process industries [22], not for living cells, the theory thus assumes no time is required to denature the cell structure of biological material to reach its ultimate diffusivity. If the concentration distribution in the solid is uniform, the solute concentration will decrease rapidly at the beginning of the extraction process. Shortly after the rapid decrease, the remaining solute fraction will decrease exponentially. In practice, to extract the solute from a living cell effectively, heat or mechanical stress must be applied to disrupt or modify the structural barriers in order to obtain the maximum diffusion rate. Thus we define "time of cell plasmolysis", in other words, "time delay due to cell plasmolysis (t_p)" as a waiting time before the diffusion proceeds at its maximum potential.

For garcinia fruit, times of cell plasmolysis are summarized in **Table 3**. It was found that the higher the temperature, the lower the time was obtained. The reason is that the higher temperature causes higher in cell disruption. In contrast, the thicker material causes a longer time due to the slowing down of heat transfer leading to slower in cell disruption. Focusing on the temperature effect, our results give a similar trend to that found by Pairote and Sirichai [23]. They reported that times of cell plasmolysis for fresh papaya peel at temperatures of 50, 60 and 70 °C were 12 - 14, 11 - 19 and 5 - 14 min, respectively. In other work, it was claimed that between 50 - 70 °C, the time of cell plasmolysis could vary from a few minutes up to 30 min, depending on cell structure and composition [24]. The results obtained in the current study, 346.3 - 1158.4 s (~ 5.8 - 19.3 min), fall well within these limits. Nevertheless, at a room temperature, the time of cell plasmolysis might be a number of days, depending on parts and kinds of plant materials [18,24,25].

Table 3 Times of cell plasmolysis of garcinia fruit at different slab thicknesses and different temperatures.

Thickness (mm)	Temperature (°C)	Time of cell plasmolysis: t_p (s)
2.5	50	402.1
2.5	60	373.5
2.5	70	346.3
4.0	50	1158.4
4.0	60	1079.3
4.0	70	949.1

Evaluation of Solute Equilibrium Distribution Coefficient

Solute equilibrium distribution coefficients (m) are summarized in **Table 4**. This parameter does not depend on the extraction temperature, but on the structure and composition of the solid phase [24]. This fact corresponds well to what was observed in our work, where the average value of the coefficient is calculated to be 1.07. The value is essential for simulation purposes in diffusion phenomena, not only for batch but also for continuous operation.

Table 4 Solute equilibrium distribution coefficients at different temperature and thickness.

Thickness (mm)	Temperature (°C)	Solute equilibrium distribution coefficient: <i>m</i> (dimensionless)
2.5	50	1.07
2.5	60	1.07
2.5	70	1.07
4.0	50	1.06
4.0	60	1.07
4.0	70	1.07
	Average	1.07

From our experiments, all "m" values are slightly greater than 1.0 and seems to be constant. The concentration of solid phase (x^*) is calculated on the basis of three compositions, that is the solute, moisture, and inert solid. However, in general when the inert solid is not included, m must be less than 1.0.

In case of a system where the draft of extraction (α) can be regarded as approaching infinity ($\alpha >> 20$) [18], which is found in our study, where α is greater than 30 ($\alpha = m\hat{E}/\hat{R}$); the equilibrium distribution coefficient (m) can be simply calculated by using a number of 100 divided by the moisture content of the solid phase at equilibrium. The reason is because, presumably, only the moisture content and the inert solid appear in the solid phase and thus the liquid phase holds all the solute substances. Inert solid density (moisture-free basis) is calculated to be 1034±1.03 kg/m³.

Evaluation of Volumetric Mass Transfer Coefficients

Volumetric mass transfer coefficients, a parameter depending on agitation level of the extraction system, were calculated and shown in **Table 5**. The values are found in the range of approximately 4×10^{-4} - 10×10^{-4} s⁻¹. All values are determined based on infinite Biot number experimental system; It follows that there will be no surface

resistance of mass transfer (Bi >> 20) [18,24,25]. The internal diffusion thus controls the mass transfer rate. It was also observed that the values of volumetric mass transfer coefficients followed a similar trend to those of diffusivities. The higher the temperature, the higher the coefficient obtained, moreover, the thicker the material, the lower the coefficient was. Nevertheless, to correctly determine the volumetric mass transfer coefficient, especially for a system with weak agitation and low convection, where the Biot number of the system can not be considered as infinite, the calculation should be done according to a more appropriate formulation given elsewhere [18,26].

Table 5 Volumetric Mass	Transfer Coefficients at	t different temperature and thickne	ess.

Thickness (mm)	Temperature (°C)	Volumetric Mass Transfer Coefficient: $K_x \times 10^{-4}$ (s ⁻¹)
2.5	50	6.86
2.5	60	6.87
2.5	70	9.40
4.0	50	3.85
4.0	60	4.08
4.0	70	5.45

CONCLUSIONS

Some physical and chemical properties of garcinia fruit materials were characterized. Diffusion properties of total organic acids in the fruit were investigated. All experiments were carried out using a batch system. Two variables influenced the extraction process, the thickness of fruit slab material and the extraction temperature. Mass transfer characteristics (acids diffusion properties) like solute diffusivities, times of cell plasmolysis, equilibrium distribution coefficients and volumetric mass transfer coefficients were reported. It was found that, for total acids in garcinia fruit, those values were in the range of 7.95×10^{-10} - 12.14×10^{-10} m²/s for diffusivities, 346.3 - 1158.4 s for times of cell plasmolysis, 1.06 - 1.07 for equilibrium distribution coefficients, and 3.85×10^{-4} - 9.40×10^{-4} s⁻¹ for volumetric mass transfer coefficients.

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บทคัดย่อ

ไวกูณฐ์ ฤทธิรุฒม์ และ ชัยรัตน์ ศิริพัธนะ² คุณสมบัติในเชิงการแพร่ของกรดผลส้มแขก

งานวิจัยนี้เป็นการศึกษาคุณสมบัติในเชิงการแพร่ของกรดผลส้มแขก (Garcinia atroviridis) โดยดำเนินการ ทคลองในถังปฏิกรณ์แบบแบทซ์ ทั้งนี้ได้ศึกษาอิทธิพลของตัวแปรกระบวนการที่เกี่ยวข้องสองตัวแปรคือ ความหนา ของตัวอย่างวัตถุดิบและอุณหภูมิในการสกัด โดยทุกการทดลองกำหนดให้ระบบได้รับการกวนผสมเป็นอย่างดี เพื่อให้เกิดกระแสไหลปั่นป่วนภายในถังปฏิกรณ์และกำจัดความด้านทานการถ่ายโอนมวลที่ผิวหน้าของวัตถุดิบ จาก การศึกษาได้หั่นผลส้มแขกเป็นรูปทรงแผ่นราบโดยกำหนดความหนาสองระดับคือ 2.5 และ 4.0 มิลลิเมตร และใช้ อุณหภูมิในการสกัดสามระดับคือ 50, 60 และ 70 องศาเซลเซียส ทั้งนี้ตัวทำละลายที่ใช้สกัดคือน้ำกลั่น กำหนดให้ อัตราส่วนของน้ำกลั่นต่อวัตถุดิบเป็น 30:1 โดยน้ำหนัก แต่ละการทดลองใช้เวลาการสกัดนาน 3 ชั่วโมง อย่างไรก็ ตามเนื่องจากต้องการให้เกิดสภาวะสมดุลจึงเดินระบบทิ้งไว้เป็นเวลาอย่างน้อย 24 ชั่วโมง ตัวอย่างวัตถุดิบส่วนที่เป็น ของแข็งถูกตรวจวัดที่ระยะเวลาต่างๆของการสกัดแล้วคำนวณหากรดผลไม้รวมและความเข้มข้นคงเหลือในเฟส ของแข็ง ทั้งนี้จากผลการทดลองพบว่า ค่าสัมประสิทธิ์การแพร่ของกรดผลส้มแขกอยู่ระหว่าง 7.95×10⁻¹⁰ - 12.14×10⁻¹⁰ ตารางเมตรต่อวินาที เวลาล่าช้าเนื่องจากเซลล์พลาสโมไลซิสอยู่ระหว่าง 346.3 - 1158.4 วินาที สัมประสิทธิ์การกระจายสมดุลของตัวถูกละลายเป็น 1.06 - 1.07 และสัมประสิทธิ์การถ่ายโอนมวลเชิงปริมาตรมีค่า อยู่ในช่วง 3.85×10⁻⁴ - 9.40×10⁻⁴ วินาที⁻¹

่ สำนักวิชาเทคโนโลยีการเกษตร มหาวิทยาลัยวลัยลักษณ์ อำเภอท่าศาลา จังหวัดนครศรีธรรมราช 80161

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