

Enzymatic Destruction Kinetics of Oil Palm Fruits by Microwave Sterilization

Maya Sarah and Mohd. Rozainee Taib

Abstract—Microwave sterilization of oil palm fruit is carried out to deactivate lipase and soften the fruits. This study aims to determine enzymatic destruction kinetics from microwave sterilization of oil palm fruits such as decimal reduction time (D -value), temperature sensitivity (z -value), kinetic constant (k) and activation energy (E_a). Three power levels (medium, medium high and high) of the microwave oven were used and lipase assayed was conducted to determine the lipase activity. Microwave sterilization of oil palm fruits depends on the destruction kinetic parameters such as D -value, z -value and E_a . It required only 8.333 to 16.949 minutes to deactivate the lipase, and the process is not temperature sensitive which is indicated by z -value. The z -value indicated requirement to increase temperature up to 71.5, 77.0 and 83.0°C respectively from initial maximum temperature to reduce the D -value. Minimum energy required to start the destruction process of lipase was 13.927 to 14.049 kJ/mole obtained from microwave sterilization of 1 kg oil palm fruits at all power levels. Oil quality observed from free fatty acid (FFA) concentration that indicated FFA below 3.5%.

Index Terms—Destruction, lipase, microwave, sterilization, oil palm fruits.

I. INTRODUCTION

Sterilization refers to a process that eliminates the microorganism which carried out by utilizing heat, chemicals, irradiation, high pressure and filtration. Microwave irradiation had been proven to effectively eliminate and deactivate the microorganism and bio molecules such as enzyme [1]-[3]. It is mainly used for food preservation and medical disinfectant.

Milling sterilization of oil palm fresh fruits bunch (FFB) is important and influences oil quality. Sterilization purpose to deactivate lipase that produce free fatty acid (FFA), soften the fruit pulp and loosen the fruits from the bunch [4]. However this current technology is time consuming and utilizing high pressurized steam. Microwave irradiation is introduced to improve microwave sterilization of oil palm fruits. By using microwave irradiation, oil palm fruits are expected to be sterilized very fast with high stripping efficiency.

Some studies reported the utilization of microwave irradiation for oil palm fruits sterilization [5]-[8], include investigation on time-temperature profile, dielectric properties, quality of oil and strip ability of the fruit from the bunch.

Sterilization process is time and temperature dependency,

Manuscript received April 2, 2013; revised May 24, 2013.

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but there are lacks or no study available on the enzymatic destruction kinetics in sterilization of oil palm fruits, hence this study was carried out to investigate enzymatic destruction kinetics of oil palm fruits.

The decimal reduction time (D -value) and temperature sensitivity (z -value) were considered. The D -value for oil palm sterilization is the time to reduce lipase activity by a factor of 10 [9]-[10]. The z -value was determined so as to obtain the temperature sensitivity of the sterilization process, which is effective in eliminating the lipase activity.

II. MATERIALS AND METHODS

A. Materials

The materials used for the study include; FFB (Tenera variety) obtained from UTM Plantation Skudai Johor Malaysia, chemicals (acetone, ethanol, NaOH and phenolphthalein produced by Merck) microwave oven (Sharp Model: R-958A), data logger (Pico Temperature Data Logger, PT 104), and hydraulic presser (fabricated).

B. Methods

The experimental rigs for microwave sterilization are illustrated in Fig. 1. The microwave oven was connected with data logger and a computer to monitor and record the temperature of oil palm FFB during sterilization process.

1) *Sample Preparation*: The sample preparation was cutting fresh oil palm fruit bunch using chain saw (Tokai 3600) into smaller desired parts and each weighted by 0.5, 1.0 and 1.5 kg respectively. Sample was placed in a dry environment before sterilization.

2) *Microwave Sterilization Process*: Oil palm FFB's sample was placed in the centre of the microwave. The oil palm FFB was exposed to microwave irradiation at high, medium high and medium power level and the temperature changes were monitored, measured and recorded for every 4, 7, 10, 13 and 16 minute intervals using thermocouple type J which was punched into the oil palm fruit at three points. The sterilization was carried out in triplicates. After the sterilization, the fruits were pressed using hydraulic presser to squeeze the oil and the lipase assayed was conducted immediately.

3) *Lipase Assayed*: It was conducted according to the method proposed in [11]-[13] using a mixture of acetone and ethanol (50:50 v/v). The palm oil (0.1 g) was dissolved stirred for about 45 minutes and the temperature was adjusted at 37.0°C. Mixture of acetone and ethanol (50:50 v/v) of approximately 10 ml was added to inactivate the lipase at the end of reaction time. It was immediately transferred into a titration flask and titrated with 0.1N sodium hydroxide solution using phenolphthalein as indicator until the first

permanent pink colour appears. Each test was conducted in duplicates. The blank test was also conducted.

4) *FFA Test*: It was conducted according to Malaysia Palm Oil Board (MPOB) test method. It used neutralized

2-propanol in the amount of 50 ml to neutralize 0.5-2.0 g of palm oil sample. After regulating the temperature to 40.0°C, the sample was titrated against standard NaOH (0.1M) using phenolphthalein 1% to the first permanent pink [14]-[15].

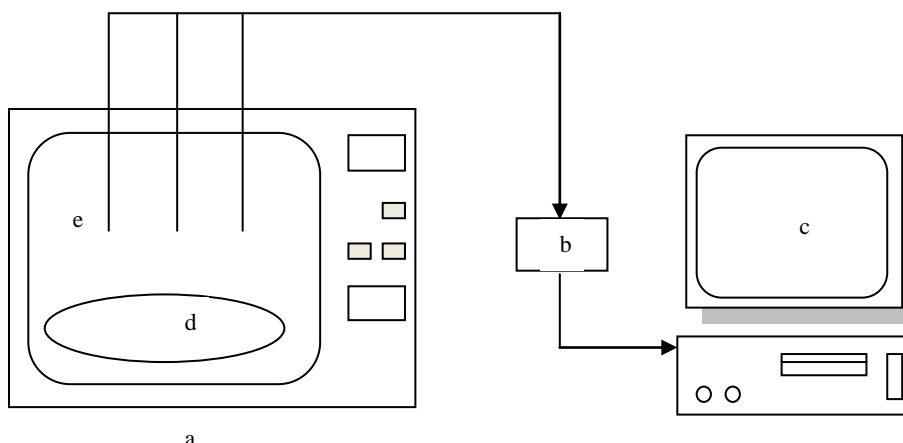


Fig. 1. Experimental rigs for microwave sterilization: (a) microwave oven, (b) data logger, (c) computer, (d) sample's tray, (e) thermocouple type

III. RESULTS AND DISCUSSION

A. Destruction Rate Kinetics

Destruction rate of lipase from microwave sterilization of oil palm fruits is shown in Table I. The destruction rate of lipase predicted obeyed first order kinetics [10]-[11] as a function of lipase activity [10], [16] and [17], and can be expressed as:

$$\frac{dA}{dt} = -kc \quad (1)$$

Destruction rate was influenced by power density and microwave intensity. Power density is the number of power apply to the oil palm fruits sample per kg, while microwave intensity is the time of exposure. The fastest destruction rate generally obtained by sterilization with high power density. This can be achieved using small sample and high power level.

Destruction rates in this study were observed between 0.041 to 0.153 U/ml min. Minimum destruction rate observed

from microwave sterilization of 1.5 kg oil palm fruits sample using medium and high power level or power density of 239.51 and 399.88 W/kg respectively. Maximum destruction rate observed from sterilization of smaller sample using high and medium high power level. However utilization of very high power density (1,199.63 W/kg) resulted very dry sample (moisture loss was over 35%) which made the fruits difficult to press and no palm oil obtained.

The lipase activity measured in this experiment was actually between 0.3 to 0.8 U/ml respectively. Other worked reported lipase activity in palm oil of approximately 0.5 U/ml [13].

B. Decimal Reduction Time

D-value from the microwave sterilization of oil palm fruits is shown in Table I. Integration of the Eq. (1) for the limits, A_1 at $t_1=0$, and A at time t results Eq. (2).

$$\log A = \log A_1 - \frac{kt}{2.302} \quad (2)$$

TABLE I: THE *D*-VALUE AND KINETIC CONSTANT FROM MICROWAVE STERILIZATION OF OIL PALM FRUITS

Oil Palm Fruits (kg)	Power Level	Power Density (W/kg)	Microwave Intensity (min)	Destruction Rate (U/ml min)	Kinetic Constant, <i>k</i> (min ⁻¹)	<i>D</i> -value	
						(min)	R ²
0.5	Medium	718.53	16	0.111	0.182	12.658	0.931
	Medium High	854.58	16	0.103	0.237	9.708	0.920
	High	1,199.63	13	0.146	0.276	8.333	0.983
1.0	Medium	359.27	16	0.105	0.161	14.286	0.980
	Medium High	427.29	16	0.153	0.179	12.821	0.973
	High	599.82	16	0.105	0.186	12.346	0.963
1.5	Medium	239.51	16	0.041	0.136	16.949	0.858
	Medium High	284.86	16	0.112	0.161	14.286	0.987
	High	399.88	16	0.091	0.164	14.085	0.959

Graphical expression of Eq. (2) is a semi-logarithmic graph of lipase activity as a function of time at constant

temperature which shown in Fig. 2. The kinetic constant, *k* of lipase destruction rate is summarized in Table I. *D*-value was

evaluated between 8.333 and 16.949 minutes. The microwave sterilization with those certain irradiation times able to reduce or inactivate 90% of initial lipase activity. It is faster than common industrial sterilization practice which carried out for about 1.5 hours using pressurized steam of 40 lb/in² [4]. Even for laboratory scale, we obtained *D*-value more than 2 hours using steam with *t* pressure of 0.10 to 0.45 kg/cm².

Observation toward temperatures during microwave sterilization process indicated low temperature requirement to obtained *D*-value as shown in Table II. The temperature was observed between 70 to 82°C using power density of 239.51 to 1,199.63 W/kg. Those temperatures were lower as to compare to laboratory scale of steam batch sterilization which observed temperature of 105°C to obtain *D*-value of 2 hours. Even milling process on oil palm fruits sterilization utilizes steam of 3 kg/cm² in pressure to maintain temperature of 140°C [18].

Sterilization of oil palm fruits by microwave irradiation with power density of 1,199.63W/kg for about 8.333 minutes was better as to compare to others. It required 49.89 Joule to maintain temperature of 82.00°C The other options is sterilization using power density of 854.88 W/kg due to power requirement for such sterilization is 58.13 Joule to maintain the temperature of 80.46°C. Others microwave sterilization process needed longer time than 10 minutes to proceed and required higher energy to be completed.

C. Resistance Coefficient

The *z*-value, also named as the resistance coefficient, represents the range of temperature increase to reduce the *D*-value by a factor of 10 [9]. It can be determined by thermal death time (TDT) method using an equation:

$$\log \left(\frac{D}{D_R} \right) = \frac{T_R - T}{z} \quad (3)$$

The TDT is an empirical model for temperature dependence. Usually *T_R* of common sterilization process can be referenced as 121.1°C or 250.0°F, but in this case, no information available regarding *D_R* for oil palm sterilization, so Eq. (3) can be solved by plotting log *D*-value against *T*, thus having -1/*z* as slope, and *z*-value can be calculated. The *z*-value of this study are listed in Table II.

The *z*-value from microwave sterilization of 0.5 and 1.5 kg oil palm fruits samples were 71.5 and 77.0°C respectively, while *z*-value from the sterilization of 1 kg oil palm fruits sample was 83.0°C. The *z*-value indicated requirement to increase temperature up to 71.5, 77.0, and 83.0°C respectively from the initial maximum temperature to reduce the *D*-value. Normally increasing the power for sterilization process will increase the temperature. However since further heating have been proven to promote high moisture loss, temperature increase from initial maximum temperature was inappropriate and the enzymatic destruction rate is relatively not temperature sensitive.

Other critical point is quality of the palm oil which indicates by low FFA (usually below 3.5%) [19]. The FFA concentration from microwave sterilization in this

experiment are shown in Table III. They were measured approximately from 0.3 to 1.39%.

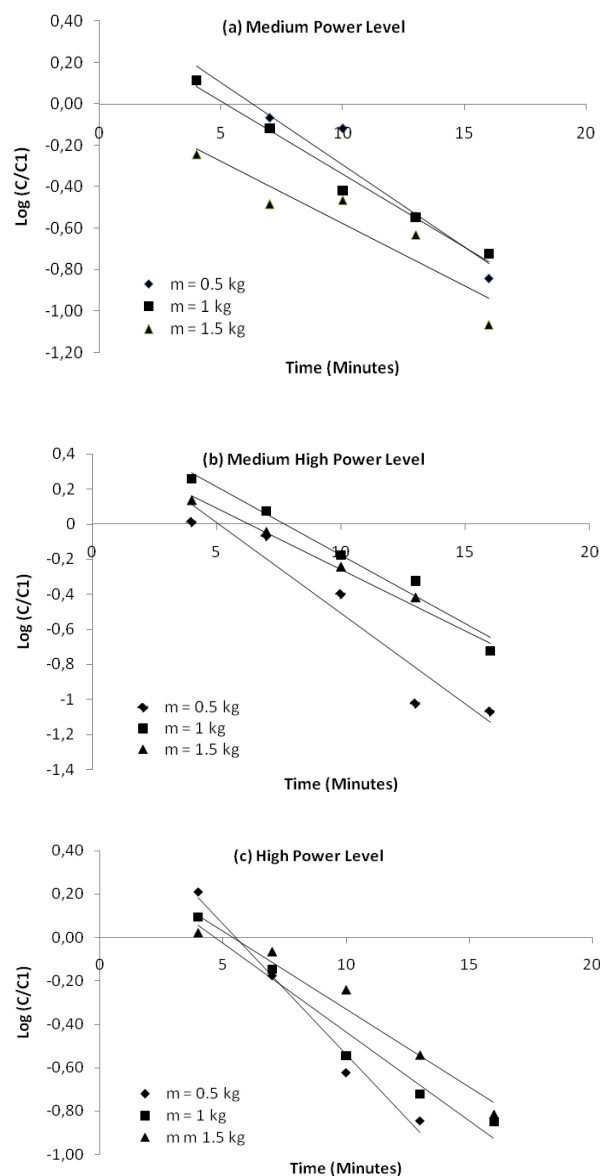


Fig. 2. First-order destruction of lipase from the microwave sterilization of oil palm fruits

TABLE II: THE *Z*-VALUE AND ACTIVATION ENERGY FROM MICROWAVE STERILIZATION OF OIL PALM FRUITS

Power Density (W/kg)	<i>T</i> (°C)	<i>z</i> -value (°C)		<i>E_a</i> (kJ/mole)
		<i>z</i>	<i>R</i> ²	
718.53	70.50	71.429	0.940	0.522
854.58	80.46			0.518
1,199.63	82.00			0.485
359.27	75.00	83.333	0.975	0.591
427.29	78.00			0.582
599.82	80.00			0.585
239.51	70.00	76.923	0.976	0.601
284.86	75.00			0.591
399.88	76.50			0.599

D. Activation Energy

Terminology of activation energy (*E_a*) in the microwave sterilization is the minimum energy required to start destruction process. The Arrhenius equation provide a

quantitative basis of the relationship between the E_a and the rate at which lipase destruction proceeds. Arrhenius equation [10] is expressed as:

$$k = s \exp \frac{-E_a}{RT} \quad (4)$$

The frequency factors can be evaluated by letting the reaction rate constant be k_1 at temperature T_1 . Then,

$$s = k_1 \exp \frac{-E_a}{RT_2} \quad (5)$$

Substitution of Eq.(5) into Eq.(4) and taking the logarithm yields

$$\log \frac{k}{k_1} = \frac{-E_a}{2.303R} \left[\frac{T_1 - T}{T_1 T} \right] \quad (6)$$

Thus E_a can be calculated from the Eq.(6). The E_a from the microwave sterilization of oil palm was observed between 0.485 to 0.601 kJ/mole and they are given in Table III, while relationship between mean E_a and z -value shown in Fig. 3.

TABLE III: THE FFA CONCENTRATION AS KEY PARAMETER FROM MICROWAVE STERILIZATION OF OIL PALM FRUITS

Oil Palm Fruits (kg)	Power De (W/kg)	1.5 kg	FFA Concentration (%)	1 kg
0.5	718.53		1.09	
	854.58		0.44	
	1,199.63		0.48	
1.0	359.27		0.60	
	427.29		0.70	
	599	0.5 kg	0.36	
1.5	239.34		1.01	
	284.86		1.39	
	399.88		0.30	

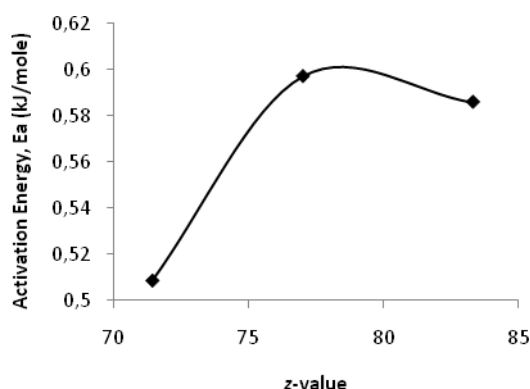


Fig. 3. Relationship between E_a and z value from the microwave sterilization of oil palm fruits

Based on the relationship between E_a and z -value, a more energy is required in order to sterilize 1.5 kg compared to others samples. However, it is more easy and safe to sterilize 1.0 kg sample because the z was higher which indicates the process is not temperature sensitive compare to 0.5 kg sample.

IV. CONCLUSION

Microwave sterilization of oil palm fruits depends on

the destruction kinetic parameters such as D -value, z -value and E_a . It required only 8.333 to 16.949 minutes to deactivate the lipase, and the process is not temperature sensitive which is indicated by z -value. The z -value indicated requirement to increase temperature up to 71.5, 77.0 and 83.0°C respectively from initial maximum temperature to reduce the D -value. Reliable minimum energy required to start the destruction process of lipase was 0.485 to 0.601 kJ/mole obtained from microwave sterilization of 1.0 kg oil palm fruits at all power level. Oil quality observed from FFA concentration that indicated FFA below 3.5%.

NOMENCLATURE

T	Temperature	°C
t	Time	s
A	Final lipase activity	U/ml
k	First-order reaction rate constant	min ⁻¹
s	A constant, the frequency factor	min ⁻¹
E_a	Activation energy	cal/mol
R	Gas constant (1.987 cal/K mol)	
D	D -value from the experiment	min

SUBSCRIPTS

1	Initial
R	Reference (at reference temperature)

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He got many experiences in incineration, fluidization process and application of microwave in drying, heating and sterilization process. He published more than 50 technical papers in international and local refereed journals as well as in international and local conference proceedings.

Prof. Mohd. Rozainee Taib received award from Institution of Chemical Engineering, UK, for the Best Paper Award at IChemE Research Event in 1997. His patent title "Process for Production of high Purity, Amorphous, Carbon Free Silica" has filed to Intellectual Property Corporation of Malaysia in 2005. Prof. Mohd. Rozainee Taib is a member of Malaysian Institute of Chemical Engineers and Institution of Chemical Engineers, UK