PERFORMANCE OF MICROWAVE ASSISTED AQUEOUS ENZYMATIC TECHNIQUE FOR *ELATERIOSPERMUM TAPOS* SEED OIL EXTRACTION

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To my beloved husband, family and friends

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ABSTRACT

Green, sustainable and effective extraction method is currently in demand for extraction of valuable compounds from natural resources. In this study, microwave assisted aqueous enzymatic extraction (MAAEE) which uses an electromagnetic wave as heating medium incorporated with non-toxic enzymatic aqueous solution as solvent was applied to extract omega-3 (ω-3) rich oil from *Elateriospermum tapos* seed. The effect of parameters in the MAAEE process including optimization, oil characterization, and mass transfer models relationship were investigated. In this study, experiments were carried out based on the design of experiment by central composite design using the design expert software. The optimization of extraction was analyzed by the response surface methodology and mass transfer models which were correlated with the models of the modified Fick's law, Patricelli's model and mass balance based on broken and intact cells. The results revealed that low microwave power (110 W) and small particle size (0.5 mm) gave significant effect on the extraction yield whereas increasing concentration of enzyme cocktail from 1 to 3% significantly increased the concentration of extracted ω-3 fatty acid. The optimum conditions were determined to be 110 W microwave power, 30 second extraction time, 1% enzyme cocktail concentration and 0.5 mm particle size, resulted in 46.12 ± 1.48% recovery of extraction. Meanwhile, the optimum ω-3 fatty acid concentration was achieved at microwave power of 550 W, extraction time of 75 second, enzyme cocktail concentration of 3% and particle size of 0.5 mm resulted into 348.96 ± 24.88 mg ω-3/g extracted oil compared to 106.57 ± 4.32 mg ω-3/g of oil from Soxhlet extraction. The modeling study indicated mass balance model based on broken and intact cells as the best fitted model which gave the highest value of R^2 (0.9932) and lower value of absolute average relative deviation (3.7983) at 550 W of microwave power. Thus, it was proven that MAAEE was able to accelerate the extraction process and provided high quality of ω-3 rich oil extract at the same time.

ABSTRAK

Teknologi pengekstrakan hijau yang lestari dan berkesan kini menjadi pilihan untuk mengekstrak sebatian berharga daripada sumber alam semula jadi. Dalam kajian ini, pengekstrakan akueus enzimatik berbantukan gelombang mikro (MAAEE) menggunakan gelombang elektromagnet sebagai media pemanasan yang mana digabungkan dengan larutan enzim yang merupakan pelarut bukan toksik telah digunakan untuk mengekstrak minyak yang kaya dengan omega-3 (ω-3) daripada biji benih *Elateriospermum tapos*. Kesan parameter terhadap proses MAAEE; iaitu pengoptimuman, pencirian minyak, dan hubungan model pemindahan jisim telah dikaji. Penyelidikan telah dijalankan berdasarkan reka bentuk eksperimen menggunakan reka bentuk komposit pusat oleh perisian design expert. Pengoptimuman pengekstrakan dianalisis oleh kaedah gerak balas permukaan dan model pemindahan jisim yang telah dikolerasikan dengan model-model daripada hukum Fick terubah suai; model Patricelli dan imbangan jisim berdasarkan sel pecah dan sel tak terusik. Hasil kajian menunjukkan bahawa gelombang mikro yang berkuasa rendah (110 W) dan saiz zarah yang kecil (0.5 mm) memberikan kesan yang ketara ke atas hasil pengeluaran minyak sebaliknya peningkatan kepekatan enzim koktel dari 1 hingga 3% memberi kesan yang ketara kepada peningkatan jumlah kepekatan ω-3. Keadaan optimum telah ditentukan pada kuasa gelombang mikro 110 W, masa pengekstrakan 30 saat, kepekatan koktel enzim 1% dan saiz zarah 0.5 mm dengan memberi 46.12 ± 1.48% daripada jumlah hasil pengekstrakan. Kepekatan optimum ω-3 diperoleh pada keadaan kuasa gelombang mikro 550 W, masa pengekstrakan 75 saat, kepekatan enzim koktel 3% dan saiz zarah 0.5 mm dengan memberikan 348.96 ± 24.88 mg ω-3/g minyak yang dikeluarkan berbanding dengan pengekstrakan soxhlet 106.57 ± 4.32 mg ω-3/g minyak. Untuk kajian pemodelan, model imbangan jisim berdasarkan sel pecah and sel tak terusik merupakan model paling sepadan yang memberi nilai R^2 (0.9932) yang lebih tinggi dan nilai sisihan bandingan purata mutlak (3.7988) yang lebih rendah pada kuasa gelombang mikro 550 W. Oleh itu, MAAEE terbukti mampu mempercepatkan proses pengekstrakan dan pada masa yang sama dapat menghasilkan minyak yang kaya ω-3 yang berkualiti tinggi.

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LIST OF ABBREVIATIONS

AARD	-	Absolute Average Relative Deviation
AEE	-	Aqueous Enzymatic Extraction
AHA	-	American Health Association
ALA	-	Alpha Linolenic Acid
ANOVA	-	Analysis of The Variance
ATR	-	Attenuated Total Reflectance
BIC	-	Broken and Intact Cells
BFRs	-	Brominated Flame Retardants
CCD	-	Central Composite Design
CH_3	-	Methyl
CHD	-	Coronary Heart Disease
COOH	-	Carboxyl Group
CV	-	Cardiovascular
CVD	-	Cardiovascular Disease
DHA	-	Docosapentaenoic Acid
DOE	-	Design of Experiment
DTGS	-	Deuterated Triglycine Sulphate
DW	-	Dry Weight
EPA	-	Eicosapentanoic Acid
ET	-	*Elateriospermum Tapos*
ETS	-	*Elateriospermum Tapos* Seed
FAO	-	Food And Agriculture Organization
FAMEs	-	Fatty Acids Methyl Ester
FDA	-	Food and Drug Administration
FTIR	-	Fourier Transform Infrared
GAE	-	Gallic Acid Equivalents

GC	-	Gas Chromatography
GHz	-	Gigahertz
GRAS	-	Generally Recognized As Save
HCN	-	Hydrogen Cyanide
HDL-C:LDL-C	-	High Density Lipoprotein Cholesterol to Low Density Lipoprotein Cholesterol
HIPS	-	High-Impact Polystyrene
HPLC	-	High Performance Liquid Chromatogram
ISM	-	Industrial, Scientific And Medical
L	-	Linoleic
La	-	Lauric
LA	-	Linoleic Acid
LDL	-	Low Density Lipoprotein
LLL	-	Linoleic-Linoleic-Linoleic
LLLn	-	Linoleic-Linoleic-Linolenic
Ln	-	Linolenic
LnOO	-	Linolenic-Oleic-Oleic
M	-	Myristic
MAE	-	Microwave Assisted Extraction
MAAEE	-	Microwave Assisted Aqueous Enzymatic Extraction
MUFA	-	Monounsaturated Fatty Acids
NA	-	Not Applicable
ND	-	Non-Detectable
O	-	Oleic
OLL	-	Oleic-Linoleic-Linoleic
OLLn	-	Oleic-Linoleic-Linolenic
OOL	-	Oleic-Oleic-Linoleic
OOO	-	Oleic-Oleic-Oleic
P	-	Palmatic
PLL	-	Palmatic-Linoleic-Linoleic
PLL-MOL	-	Palmatic-Linoleic-Linoleic-Myristic-Oleic-Linoleic
Po	-	Palmitoleic
POL	-	Palmatic-Oleic-Linoleic

POO	-	Palmatic-Oleic-Oleic
PPL	-	Palmatic-Palmatic-Oleic
PoPoPo	-	Palmitoleic-Palmitoleic-Palmitoleic
PCB	-	Polychlorinated Biphenyl
PUFAs	-	Polyunsaturated Fatty Acids
PSE	-	Pressurized Solvent Extraction
RSM	-	Response Surface Methodology
S	-	Steric
SAFA	-	Saturated Fatty Acids
SE	-	Soxhlet Extraction
SEM	-	Scanning Electron Microscope
SFE	-	Supercritical Fluid Extraction
SSE	-	Sum of Squares Regression
SSR	-	Sum of Squares Error
SST	-	Sum of Squares Total
TAG	-	Triglycerol
TAGs	-	Triglycerides
TC	-	Serum Cholesterol
TPC	-	Total Phenolic Content
UAE	-	Ultrasonic-Assisted Extraction
USA	-	United States of America
UV	-	Ultraviolet
VOCs	-	Volatile Organic Compounds
WHO	-	World Health Organization

LIST OF SYMBOLS

3D	-	three dimensional
A	-	microwave power parameter in RSM
A	-	cellulase parameter in crossed mixture design
b	-	coefficients for extraction kinetics in washing step
B	-	extraction time parameter in RSM
B	-	pectinase parameter in crossed mixture design
C	-	concentration solute in the solid matrix
C	-	enzyme cocktail concentration
C	-	proteinase parameter in crossed mixture design
C	-	concentration of omega 3 fatty acid or amygdalin obtained from calibration curve
cm	-	centimeter
CV	-	coefficient of variation
d	-	Particle Size
df	-	degree of freedom
D	-	diffusion coefficient of the solute
D	-	particle size parameter in RSM
D	-	temperature parameter in crossed mixture design
E	-	field strength
E	-	incubation time parameter in crossed mixture design
f	-	frequency
g/d	-	gram per day
H	-	extraction bed length
J	-	flux of solute
k	-	constant
k	-	coefficients for diffusion step

kV	-	kilo voltage
m	-	meter
$\dot{m}$	-	solvent flow rate
mg	-	milligram
mg/g	-	milligram per gram
mg/kg	-	milligram per kilogram
mg/L	-	microgram per liter
mg/mL	-	microgram per milliliter
min	-	minute
mL	-	milliliter
mL/min	-	milliliter per minute
mL/g	-	milliliter per gram
mm	-	millimeter
$M(t)$	-	mass of the extract
$M(\infty)$	-	maximum value for the extracted omega-3
N	-	mass flux of solute
n	-	integer number
nm	-	nanometer
pH	-	potential of Hydrogen
P	-	power
r	-	radius of the particle
R	-	distance
rpm	-	revolution per minute
s	-	second
S/F	-	solvent to solid (feed)
t	-	extraction time
T	-	temperature
u	-	superficial fluid velocity
V	-	volume
v/v	-	volume per volume
W	-	watt
W/W	-	weight per weight
x	-	distance inside the porous part of the solid matrix

x	-	mass fraction in solid phase
Y	-	mass fraction in fluid phase
y	-	output peak area of the absorbance
z	-	axial co-ordinate
ε	-	bed void fraction
µL	-	microliter
µm	-	micrometer
%	-	percentage
>	-	greater than
<	-	lower than
°C	-	degree Celsius
tan δ	-	loss tangent
ω-3	-	omega-3
R^2	-	correlation coefficient
Y^*	-	equilibrium fluid phase mass fraction
ε'	-	dielectric constant
ε''	-	dielectric loss
C_0	-	initial concentration of solute in sample particle
C_{amyg}	-	concentration of the extracted amygdalin
Ci	-	concentration of solute at the interface of sample particle
C_t	-	concentration of solute extracted as function of time
C_1	-	amount of solute equilibrium yield at washing step
C_2	-	amount of solute equilibrium yield at diffusion step
C_∞	-	concentration of solute in the extraction solvent after infinite time
$C_{\omega-3}$	-	concentration of the extracted omega-3
C_{exp}	-	experimental yield of omega-3 concentration
C_{model}	-	predicted yield of omega-3 concentration
k_1	-	mass transfer coefficient during washing step
k_2	-	mass transfer coefficient during diffusion step
k_f	-	fluid phase mass transfer coefficient
k_s	-	solid phase mass transfer coefficient
Ms	-	mean square

P_{diss}	-	microwave power dissipation per unit volume
x_0	-	initial mass fraction of solute in solid phase
x_k	-	easily accessible solute in solid phase
W_{oil}	-	weight of the extracted oil
X_i and X_j	-	independent parameters level
Y_1	-	total oil yields
Y_2	-	concentration of omega-3
β_0	-	constant
β_i	-	Linear coefficient
β_{ii}	-	quadratic coefficient
β_{ij}	-	interactive coefficient
ρ_a	-	apparent density
ρ_f	-	solvent density
ρ_s	-	solid density
ρ_s	-	real density

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CHAPTER 1

INTRODUCTION

1.1 Background

Nowadays, researches on therapeutical compounds from natural resources gain major interest from academic researchers. Omega-3 fatty acid is believed to be one of the therapeutical compounds that are currently attracting a great deal of attention. There are increasing amount of evidences citing omega-3 fatty acid ability to be used in the treatment and prevention of chronic diseases especially cardiovascular disease (CVD) known as the leading cause of death among people around the world. Omega-3 fatty acid is an essential fatty acid that need to be consumed through supplements or food products due to the incapability of human body to generate the compound naturally by itself (Poudyal *et al.*, 2011; Kapoor and Patil, 2011). Omega-3 fatty acid is frequently found in marine animals and plants. However, due to several safety concerns, marine animals are no longer a remarkable source of omega-3 fatty acid as most of the researchers nowadays focus on expanding the discovery of omega-3 from plant as an alternative source. One underutilized local plant seed which is known as Perah or scientifically known as *Elateriospermum tapos* seed (ETS) had recently been reported to be rich of omega-3 fatty acid. A research by Yong and Salimon (2006) claimed that 17.4% of alpha linolenic acid (ALA), an omega-3 fatty acid is contained in the ETS. However, there is no further literature for the quantification of the specific amount of omega-3 fatty acid concentration in the ETS.

The primary method in extracting valuable active compounds from plants is the Soxhlet extraction (SE). This method has been comprehensively used as a standard reference to other methods of extraction due to its >99% extraction recovery (Pradhan *et al*., 2010). The microwave assisted aqueous enzymatic extraction (MAAEE) method is a promising new extraction method that is green, fast, efficient and energy saving. This method eliminates the disadvantages of conventional solvent extraction method which is the undesirable effect on oil quality due to the organic solvent usage. Water and aqueous based solvent system offer an increasingly crucial choice for the replacement of conventional organic solvent (Gai *et al*., 2013). MAAEE had been applied for oil extraction from seed crops such as pumpkin seed (Jiao *et al*., 2014), *Isatis indigotica* seed (Gai *et al*., 2013), yellow horn (Li *et al*., 2013), and *Forsythia suspense* seed (Gai *et al*., 2013). Microwave uses electromagnetic wave which penetrates into certain materials to provide volumetric heating through ionic conduction and dipole rotation (Chan *et al*., 2014). Treatment of ETS with enzymes enhance the extraction of oil yield due to its hydrolyzed structural polysaccharide of the cell walls and proteins associated with the lipid bodies (Jiao *et al*., 2014). Hence, a novel combination of MAAEE and enzymatic treatment will create effective synergy in enhancing the oil extraction process. To our knowledge, the combined use of enzymes and microwave assisted extraction of omega-3 fatty acid from ETS has not been previously reported.

The performance and efficiency of MAAEE depend upon many factors including microwave power, temperature, extraction time, solid to solvent ratio, and particle size. Some of these factors should be considered for the optimization of extraction condition. According to Baş *et al*. (2007), the optimization process can be carried out effectively using Response Surface Methodology (RSM) in which has become a powerful tool to determine the effect of the factors and their interaction. This method is the preferred experimental design technique for fitting polynomial model to analyze the response of multi-factor combination.

Mathematical modeling is useful in improving, optimizing, stimulating and scaling up a process design of the extraction process. It must be considered as a fundamental step during the operation of industrial process (Franco-vega *et al*., 2016;

Xavier *et al*., 2011). Mass transfer model of MAAEE is developed for the purpose of explaining the physical mechanism of extraction process based on mass transfers fundamentals.

Therefore, the aims of this research work are to study the interaction of different operating parameters on the extraction of ETS in order to attain the maximum yield of oil and omega-3 fatty acid concentration from ETS as well as the optimization purposes. Meanwhile, performances of the MAAEE are further characterized by physicochemical properties of ETS oil and compared with microwave assisted extraction (MAE) and SE methods. Furthermore, with the intention of evaluating the kinetic behavior (mass transfer coefficient) of data from the experiments; a mathematical model was also developed to study the relationship and correlate the best fit with three different mass transfer models.

1.2 Problem Statement

In Malaysia, *Elateriospermum tapos* seed (ETS) is a local seed that is in abundance but underutilized, which had been found to contain high valuable omega-3 fatty acid. Up to date, non-specific amounts of omega-3 fatty acid concentrations had been found in the seed according to previous studies. In addition, ETS also contains an antinutritional compounds known as amygdalin which is one of the cyanogenic glycoside compound that causes dizziness when consume in a large quantity. Thus, it is risky for consumers to consume omega-3 fatty acid directly from perah seed oil due to the presence of amygdalin in the seed. Hence, it is necessary to remove the amygdalin compound in order to obtain high quality omega-3 fatty acid. Amygdalin can be removed in sufficient amount using conventional methods such as fermentation, roasting and boiling. A study by Ngamriabsakul and Kommen (2009) reported that the amount of amygdalin was reduced from 660 mg/L to 100 mg/L and 25 mg/L after the seeds had been cooked and fermented, respectively. However, these methods are time consuming and the bioactive compounds present in ETS oil might be thermally oxidized. Thus, green and effective methods are desired in order to extract and separate both omega-3 fatty acid and amygdalin simultaneously so that pure and high quality ETS oil can be attained.

There are various techniques of extraction that can be employed for the recovery of therapeutic compounds from plants. Recently, the MAAEE technique had been successfully applied in the extraction of oil from various seeds due to its special heating mechanism, fast extraction time and non-tolerable with organic solvent. However, this technique is considered as a new combination extraction technique which deals with enzymes and microwave assisted extraction. In MAAEE, it is crucial to study useful data for optimization such as ratio of the enzyme cocktail and operating parameters such as microwave power, extraction time, enzymes concentration, and particle size. These data need to be further studied as the extraction conditions available in the literature are only applicable for specific microwave system where different instrumental setup of microwave system would results to different performance if applied with the same operating conditions. In other words, the optimum extraction conditions reported in the literature are valid only as guides and references for new extraction microwave system. Therefore, optimization and response of interactions of the operating parameters need to be determined in order to maximize the yield of omega-3 fatty acid.

In addition, scarce availability of mass transfer data for ETS oil extracted by MAAEE reported also contributes to the problem of this study. The data is essential for further use as reference, for scaling up the production and for predicting the extraction behavior. Hence, mass transfer modeling of MAAEE needs to be in correlation with appropriated models.

1.3 Objectives of the Study

The main objective of this study is to investigate the potential of MAAEE as extraction method of seed oil by determining the most optimum parameters of ETS oil extraction and by modeling the process efficiency via MAAEE technique. Thus, the specific objectives of this research are:

i) To identify and quantify the omega-3 fatty acid and amygdalin compounds in *Elateriospermum tapos* seed (ETS) oil.

ii) To investigate the effect of MAAEE operating parameters and to optimize the extraction condition on the ETS oil yield and omega-3 fatty acid concentration using Responses Surface Methodology (RSM).

iii) To characterize and compare the morphology behavior, chemical structure, fatty acids profile and triglycerides (TAGs) composition among different methods namely MAAEE, microwave assisted extraction (MAE) and Soxhlet extraction (SE).

iv) To investigate the relationship and best fit model representing mass transfer of extracted oil.

1.4 Scope of the Study

In order to achieve the objectives, the scopes of the study are stated as follows. Identification and determination of omega-3 fatty acid compound were carried out using gas chromatography (GC). This analysis was carried out in order to confirm the presence of omega-3 fatty acid and also to quantify the exact amount of omega-3 concentration in ETS oil. High performance liquid chromatography (HPLC) was used to detect and quantify the exact amount of amygdalin compounds, which is one of cyanogenic compound that is risky for direct consume by human.

In order to minimize the number of experiments and parameters involved for process optimization, some important parameters of MAAEE were set as constant parameters which are solvent to solid ratio, ratio of enzyme cocktail and solvent pH. Solvent to solid ratio needs to be determined in order to know the solvent required to extract the maximum amount of extraction oil. Ratio of the enzyme cocktail concentration is essential for determination in order to know the ratio of each enzyme used for the cell wall hydrolysis. Solvent pH is believed to cause denaturation or aggregation of protein which probably can be utilized for separating oil in extraction process.

In order to determine the optimum condition of MAAEE parameters on extraction yield of ETS oil and omega-3 fatty acid using Response Surface Methodology (RSM), the experiment was carried out at selected conditions at power, P (110-1100 W), extraction time, t (30-120 s), enzyme cocktail concentration, C (1-5%), and particle size, d (0.5-1.5 mm) according to the central composite design (CCD) of experiment using the Design Expert software. The microwave power range was chosen in accordance to low, medium and high level microwave power while the selection of extraction time is not exceeding 120 s or the extraction will no longer be significant. For enzyme cocktail concentration, the amount was selected to be not too high due to the expensive cost of enzymes while the selection of particle size is based on preliminary experiment.

The next scope is to continue the research with the investigation of the effects (P, t, c and d) and their interaction on extracted ETS oil yield and omega-3 fatty acid from Responses Surface Methodology (RSM) and analysis of variance (ANOVA). Low to medium microwave power is expected to favor the extraction efficiency as high power would rapidly increase the temperature. Meanwhile, the extraction yield would increase as extraction time increased until certain level. Increasing amount of enzyme cocktail concentration might speed up the extraction rate while smaller sample particle size would increase the extraction efficiency.

In order to investigate the performance of MAAEE, characterization of extracted ETS oil is compared with other methods (MAE and SE) including extraction yield, the amount of omega-3 concentration, the reduction amount of amygdalin, the properties of the oil including morphology image before and after extraction of ETS using SEM, chemical structure using FTIR, fatty acids analysis by GC and TAGs profile by HPLC.

The last scope of this research covers the investigation of the mass transfer models relationship and best fit model using modified Fick's Law model, Patricelli's model and mass balance model by broken and intact cells.

1.5 Significance of Study

This research looks into the abundant amounts of local seed that were under-utilized as a new source of omega-3 fatty acid. Usually, rich omega-3 fatty acid products from plant sources such as flaxseed, linseed, canola, and walnut (Simopoulos, 2002) are imported from other countries which require high production and exportation costs. Thus, this new local source of omega-3 fatty acid from ETS provides huge benefit in term of production cost and less expensive omega-3 fatty acid oil could be produced. Moreover, only few people consume *Elateriospermum tapos* seed (ETS), thus it is not in competition with other food sources as compared to marine life. Hence, a sufficient supply of ETS in Malaysia could contribute to the low cost alternative of omega-3 fatty acid production.

Furthermore, from the aspect of academic contribution, the novel part of this research is the manipulation of microwave assisted extraction condition to remove the undesired amygdalin from ETS in order to produce green, safe and high quality oil. The capability of the MAAEE technique to extract desired concentration of target compounds with fast and low cost extraction setup could be established. Besides, the optimization, influences of operating parameters and mass transfer modeling data are significant to industry as a guideline and references in order to upgrade to industrial scale. A side from that, the quantification method of omega-3 fatty acid concentration using GC conducted in this study could provide consumers with the information of sufficient intake of omega-3 fatty acid. Thus, the extracted ETS oil has huge potential to be further developed either in the food, pharmaceutical or oleo chemical industries.

1.6 Thesis Outline

This thesis consists of five chapters and the content of each chapter are describe as follows:

Chapter 1 introduces the background and objectives of this research. It also discusses problem statement, scope of the study, novelty contribution and thesis outline.

Chapter 2 reviews the properties of omega-3 fatty acid, amygdalin compounds and botanical information of ETS. This chapter also discusses the fundamental and effect of parameters of the microwave assisted extraction (MAE) technique as well association of hydrolytic enzymes in MAAEE which was employed in most plant extracts. The optimization method using response surface methodology (RSM) is also described in this chapter. This chapter also covers the reviews on several relevant mass transfers modeling.

Chapter 3 presents the research methodology for optimization and modeling of MAAEE. It also describes the analysis methods for ETS oil characterization.

Chapter 4 encompasses the results and discussion on the optimization of the ETS oil yield and omega-3 fatty acid concentrations as well as the influences of the extraction parameters. The comparison of MAAEE with the conventional SE as well as MAE is also presented in this chapter which also includes the quantitative amount of omega-3 fatty acid and amygdalin and also the characterizations of the oil. Moreover, the relationship and best fitting of mass transfer models is also evaluated in this chapter.

Chapter 5 concludes all the findings of this research and proposes recommendations for future work.

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