

Multi-stage Microwave-Assisted Transesterification of Soybean Oil—An Economic Design for High Yield Biodiesel Production

Chien-Hsiu Hung, Ying Ming Chew, Yi-Chung Fu, and Chin-Shuh Chen

Abstract—A multi-stage microwave-assisted transesterification apparatus for biodiesel production is designed by connecting a microwave to a low-pressure reactor to form a flow-through microwave's circulation reaction system. After each stage of reaction, the mixture is centrifuged to expel glycerol phase and transferred to the reactor for subsequent reaction stages, namely Stage II to V. Refilling tests of different reactants are operated with four conditions (Condition A – D). Among these conditions, Condition B, C and D can produce high FAME yield (100%) since Stage II. With persistent refilling of reactants, as Condition C, the reactions can be performed for at least five stages with a steady SBO amounts (approximately 0.1 mole) and low surplus rates of oil (5 to 8%). The reaction time can be shortened to 6 minutes, with 3 minutes' intermittent microwave irradiation. This study has demonstrated that multi-stage microwave-assisted transesterification is effective, feasible, and affordable for scale-up biodiesel production.

Index Terms—Microwave-assisted transesterification, multi-stage, circulation system, fatty acid methyl ester, soybean oil.

I. INTRODUCTION

The limited amount of fossil fuel has brought about concerns of world-wide energy crisis. In recent years, studies on biodiesel, or called fatty acid methyl ester (FAME), as renewable energy with attempts to replace fossil fuel have received increased attention. Nonetheless, the current yield of biodiesel can hardly cover the huge demand of energy with the increasing world population [1].

To improve yield of biodiesel, microwave technology has been proposed in many reports. Some of these reports have been discussed in the review articles by Motasemi and Ani [2], Gude *et al.* [3], Sajjadi *et al.* [4] Such technology as an electromagnetic wave can significantly improve the conversion rate of vegetable oil during transesterification reactions mainly due to its dielectric heating properties on dipole molecules [5]. Microwave-assisted transesterification reaction (MATR) is said to be more suitable than the conventional methods also because it provides a cleaner and more comfortable way of working with chemical reactions [6]. Depends on the type of reactants, applying microwave energy as heating medium has been proved to increase most

organic reaction rate to at least twice as fast as the conventional conductive heating methods can achieve [7].

Despite the intensive research of MATR on FAME production, these studies are focusing mostly on the batch type reaction [8]. Batch reaction is normally less preferable for industrial MATR because several concerns should be considered when the size of microwave reaction vessels is enlarged. Depth of microwave penetration can be the major concern for current research. Depends on the dielectric constant of materials, microwave energy can normally penetrate a few centimeters into the reactants during MATR process [3], [9], [10]. Transesterification rate can be affected if microwave energy does not reach the center of reaction vessel. In addition, an enlarged batch-MATR process can also lead to safety concerns [11]. Some of these concerns include the stability of excess pressure caused by highly intensive microwave radiation emitted in the closed system [12], the undetected leakage of microwave radiation that brings about health issues to the operators [13], and the thermal runaway of superheating generated by microwave and dielectric molecule's interactions [12]. In practical, the higher capital cost of investment and maintenance required for novel technologies can be another key barrier to the well-equipped industrial MATR system [10].

Methanol is an alcohol commonly used as a reactant to produce FAME [14], [15]. Due to reversibility of the reaction, excess methanol (or alcohol) is usually added to react with triglycerides (or oil) so that forward reaction rate can be enhanced to achieve high FAME yield [16]. This may lead to high amount of residual methanol dissolved in FAME and glycerol phase, which should be separated and recovered in the process followed by transesterification. Methanol residue is normally wasted in the washing process in conventional transesterification reaction [17] which causes environmental pollution. In recent years, several reports have proposed to recover methanol by distillation from FAME and glycerol [17], yet few reports are associated with the effect of recycled methanol refilling in the MATR process.

In this study, an apparatus for multi-stage MATR of methanol and soybean oil is proposed. With the consideration of industrial feasibility and practicality, the apparatus is designed by connecting a modified household microwave with a low-pressure reaction vessel so that a flow-through microwave heating and circulation system is constructed. With the purpose to achieve high FAME yield, investigation on the effects of persistently refilling recycled methanol and raw reactants after each stage of MATR

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process is also an important objective of this study.

II. MATERIAL AND METHODS

A. Materials

Commercial food grade soybean oil (SBO) and HPLC grade methanol (99.9% purity) are used as the main reactants for MATR. Sodium methoxide (NaOCH_3 , 98% purity) as catalyst and methyl pentadecanoate ($\text{C}_{15:0}$) as internal standard are purchased from Sigma-Aldrich Pty Ltd. Hexane as diluent and anhydrous sodium sulfate as water eliminator are purchased from Merck and Union Chemical Works Ltd, respectively.

B. Flow-through MATR Circulation System

The apparatus consists of a 900 W household microwave (Sharp R-588D, Japan) connected to a 300 ml low pressure reactor (Parr 5100, USA) with 500 cm silicon tube (i.d. = 5 mm; o.d. = 9 mm) installed on a peristaltic pump, forming a flow-through MATR circulation system (Fig. 1). Of the 500 cm silicon tube, 350 cm is designed to pass through the microwave field.

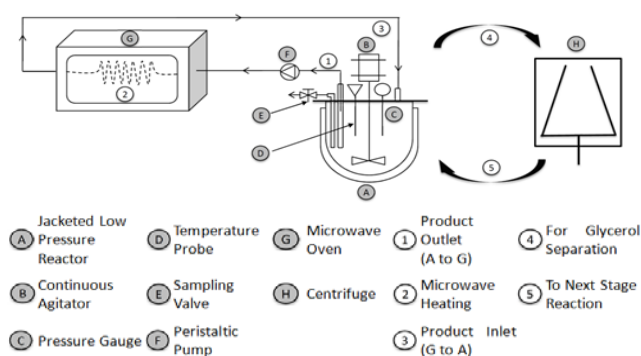


Fig. 1. Schematic diagram of the multi-stage flow-through MATR circulation system.

Each stage of the MATR process begins with turning on both microwave and peristaltic pump to deliver the reactants from Parr 5100 to the microwave (A to G) and flow back to Parr 5100 after passing through the microwave field (G to A). During the process, Parr 5100 is agitated at 1000 rpm while circulation rate of reactants is controlled at 540 ml/min. The circulation is maintained for a desired time period followed by transferring the reactants from Parr 5100 to a centrifuge (A to H). FAME and glycerol phase are separated at 12000 g centrifugation for 5 min. While the glycerol phase consisting of glycerol and methanol is expelled, FAME phase containing FAME, unreacted SBO, methanol, and catalyst is collected and transferred to the reactor (H to A) for the next stage of reaction.

In the subsequent stages, certain amount of raw reactants (raw SBO, raw methanol, and catalyst) and/or recycled methanol is refilled and mixed with FAME phase. Through distillation (below 55 kPa at 55°C) of the glycerol phase, recycled methanol can be obtained.

The reaction time is determined based on total time of microwave heating. In each stage, microwave heating is controlled at 5 minutes. One ml of sample is taken per minute of reaction for FAME yield determination. Samples are stored within the ice bath immediately to stop reaction.

The overall process is carried out in a closed system with temperature recorded.

C. Multi-stage MATR with Different Operation Conditions

Based on the above-mentioned flow-through MATR circulation system, multi-stage MATR of SBO and methanol can be carried out. In this study, five stages (Stage I to V) MATR is demonstrated. To determine the effects of four different reactant refilling conditions, namely Condition A – D, are investigated after each stage of MATR process (Fig. 2). In each condition, Stage I remains unchanged, while reactant refilling procedures in Stage II is repeated in Stage III, IV, and V. Reaction details for Stage I and Condition A - D shown in Fig. 2 are described below.

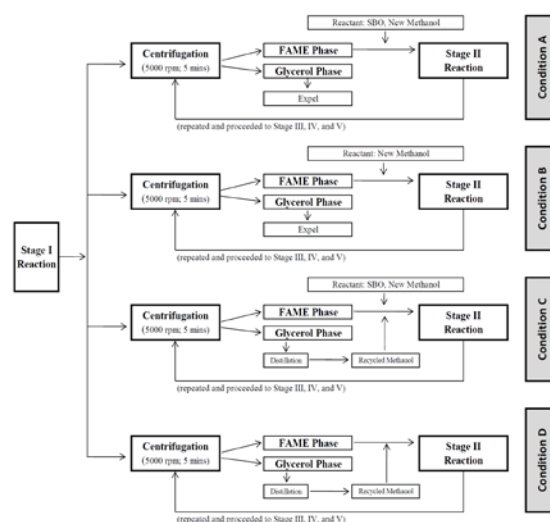


Fig. 2. Flow diagram of the multi-stage MATR with four different conditions.

1) [Stage I]: Initial reaction

NaOCH_3 -methanol solution (SMS) is prepared by dissolving 1 % (wt.) NaOCH_3 (relative to oil) in methanol (45.90 g), while SBO (139.04 g) is filled into Parr 5100 in advance. To initiate the reaction, SMS is added into Parr 5100 and mixed with SBO followed by turning on the microwave, peristaltic pump, and agitator. Molar ratio of methanol and SBO (average MW=874 g/mol) [18] is adjusted to 9:1.

2) [Condition A]: Raw reactant refilling

Raw reactants (SBO and SMS) are refilled and mixed with FAME phase separated from the former stage. The quantity of reactants refilled is adjusted to 50% (wt.) of the total reactants added in Stage I.

3) [Condition B]: Raw methanol refilling

Similar refilling process to Condition A, SMS is added and mixed with FAME phase. The amount of SMS added is adjusted to a quantity equivalent to 3:1 methanol/SBO molar ratio, the theoretical alcohol/oil molar ratio to produce FAME.

4) [Condition C]: Raw reactants and recycled methanol refilling

While refilling procedure of raw reactants is the same as Condition A, methanol recycled from the former stage is also added in Condition C. The quantities of recycled methanol that can be obtained by distillation in Stage II – V are respectively 54, 87, 120, and 152 % (wt.) of the

methanol added in Stage I.

5) [Condition D]: Recycled methanol refilling

Same refilling procedure of recycled methanol as that mentioned in Condition C. The quantities of recycled methanol that can be obtained in Stage II – V are respectively 54, 50, 49, and 48 % (wt.) approximately of the methanol added in Stage I.

D. Stage Effect of MATR

Multi-stage MATR is carried out according to the procedures stated in Condition D. However, only three-stage MATR was performed with reaction time reduced to 3 minutes per stage. The FAME yields obtained is then compared with two-stage MATR with reaction time up to 5 minutes per stage. In other words, total reaction time for three-stage MATR is 9 minutes as compared with 10 minutes in total for two-stage MATR.

E. Analysis

Samples were centrifuged at 10,000 rpm for 10 minutes so that ester layer can be separated. Ester layer (or upper layer) was transferred to a volumetric bottle (5 ml) and diluted to 5 ml with hexane. Aliquot of 1 ml methyl pentadecanoate (10 mg/ml) was then added followed by addition of anhydrous sodium sulfate. The samples were analyzed by FID gas chromatograph (Trace GC Ultra, Thermo electron, USA) equipped with HP88 column (100 m × 0.251 mm × 0.20 μm) using helium (99.999%) as carrier gas. Sample of 1 μl was injected and analyzed with the following heating program: $T_{\text{initial}} = 50$ °C, heating rate of 5 °C/min till 70 °C, holding 3 minutes, then 20 °C/min till 245 °C, and finally 5 min at 240 °C before cooling. The flow rate of the carrier gas was 1 mL/min with the split ratio of 100: 1. The injection port and detector temperatures were set at 260 °C and 260 °C separately.

At least triplicate was carried out for analysis of each sample, while the data shown are result of the average values. Calculation of the total FAME yield is in accordance with the equation below:

$$\% \text{ESTER}_{\text{C}_{16:0}} = \frac{\text{area}_{\text{C}_{16:0}} / \text{area}_{\text{C}_{15:0}} / \text{CTRF}}{\times W_{\text{C}_{15:0}} / W_{\text{sample}} \times f_{\text{TGC}_{15:0}}} \times 100\%$$

where % $\text{ESTER}_{\text{C}_{16:0}}$ indicates the methyl palmitate yield (%) of the sample; $\text{area}_{\text{C}_{16:0}}$ is the peak area of palmitic acid methyl ester; $\text{area}_{\text{C}_{15:0}}$ is the peak area of methyl pentadecanoate (internal standard); CTRF (calibrated theoretical relative response factor) is the respective areas of the peaks corresponding to fatty acid methyl esters and internal standard; $W_{\text{C}_{15:0}}$ is the mass (g) of methyl pentadecanoate; W_{sample} is sample mass (g); $f_{\text{TGC}_{15:0}}$ is 0.9947, according to AOAC [19] 996.06E. It represents the equivalent conversion of internal standard (C15:0 fatty acid methyl ester) to triglyceride. The data was analyzed using SAS analysis system followed by mean separation using Duncan's multiple range test.

F. Calculation of Oil Surplus Rate

Oil surplus rate is calculated to determine the degree of SBO conversion to FAME in the MATR process. In this study, oil surplus rate is also an indicator to show the efficiency of the forward reaction. The value of oil surplus

rate is calculated by the following equation:

$$\text{Oil Surplus Rate (wt.\%)} = [\text{SBO mole} \times \text{MW} \times (100\% - \text{FAME yield\%})] / W_{\text{SBO}} \times 100\%$$

Where SBO mole is the total moles of raw and residual SBO; FAME yield is determined according to the above-mentioned method; MW is the average molecular weight of SBO (874 g); and W_{SBO} is the total weight of SBO.

III. RESULTS AND DISCUSSION

Preliminary studies on the flow-through MATR circulation system were carried out by using SBO and methanol as raw materials. The reactions catalyzed by 1 wt% sodium methoxide were then optimized at 9:1 methanol/SBO molar ratio with the flow rate 540 ml/min. and agitated at 1000 rpm for 5 minutes.

A. Feasibility of the Flow-through MATR Circulation System

The flow-through MATR circulation system has been demonstrated by performing four different operation conditions, namely Condition A-D. Among the four conditions, Condition A and B performed by adding raw reactants (either SBO and methanol or solely methanol) in the subsequent stages were respectively compared with Condition C and D to determine the effect of recycled methanol refilling. While Condition B and D are designed to show the possibility of replacing raw methanol with recycled methanol in the multi-stage MATR system, Condition A and C were to demonstrate the feasibility of industrial biodiesel production by using the system proposed in this study. For example, in Condition C, the accumulated amount of methanol recycled from Stage II to V (54 to 152 wt% of the initial amount added) also indicates that amount of raw methanol refilled in the later stages (i.e. Stage VI, VII, VIII...etc.) can be reduced (Fig. 2).

B. Effect of Multi-stage MATR with Different Operation Condition

Effect of multi-stage MATR is determined based on the results of FAME yield, oil surplus rate, methanol/oil molar ratio, and amount of SBO from Stage I to V. In this study, it is aimed to decrease oil surplus rate and increase FAME yield in order to achieve high efficiency of MATR process.

According to the FAME yield and oil surplus rate of multi-stage MATR with different conditions shown in Fig. 3, the result under Condition A indicated that oil surplus rate increased but FAME yield decreased if raw reactants were refilled at the same ratio. Under Condition B, the result showed that the forward reaction can be driven by refilling additional methanol in the reaction mixture, leading to decreasing oil surplus rate but increasing FAME yield. Similar result had been found in the effect of refilling recycle methanol as in Condition D. MATR process under both Condition B and D can achieved 100% FAME yield with almost 0% oil surplus rate after Stage V reaction. Condition C appeared to be the combination of Condition A and D, which showed in the result that FAME yield and oil surplus rate maintained respectively at above 90% and 10%, while maintaining a steady SBO amount at approximately 0.1 mole and also finding an increase of methanol/oil molar

ratio, 16:1 to 31:1 (Fig. 4). This implies that the MATR process can be further proceeded to subsequent stages (eg. Stage VI, VII, VIII...etc.) with a reduced amount of methanol refilling.

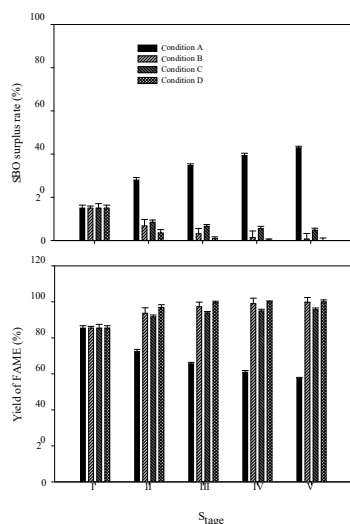


Fig. 3. Yield of FAME and SBO surplus rate of multi-stage MATR with different operation conditions. Conditions A-D are shown schematically in Fig. 2. Oil Surplus Rate (wt.%) = $[\text{SBO mole} \times \text{MW} \times (100\% - \text{FAME yield} \%)] / W_{\text{SBO}} \times 100\%$.

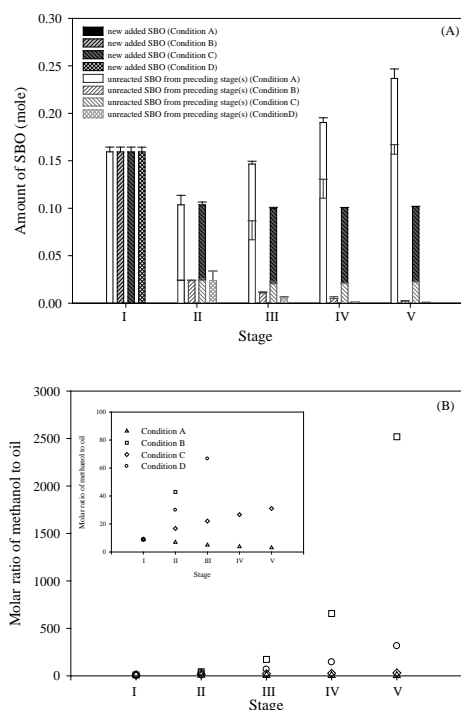


Fig. 4. The initial SBO amount (A) and molar ratio of methanol to oil (B) for each stage in the multi-stage MATR with four different operation conditions. The figure inside figure 4B shows those molar ratios of methanol to oil that are lower than 100.

It can also be observed in Fig. 4 for the initial SBO amount and methanol/oil molar ratio of multi-stage MATR with different condition that refilling raw reactants at the same ratio (Condition A) can lead the MATR process to increasing SBO amount but decreasing methanol/oil ratio, which resulted in gradual reduction of FAME yield. In contrast to the result under Condition A, however, refilling raw and recycled methanol as in Condition B and D caused

a larger increase in methanol/oil ratio that led to a more aggressive forward reaction and eventually achieved 100% FAME yield.

One reason that causes decrease in FAME yield in Condition A is due to removal of methanol dissolved in glycerol phase expelled at the end of each stage, which results in lower methanol/oil ratio. The other reason can be reversibility of the reaction caused by the accumulated FAME. The FAME yield can be enhanced by refilling methanol, as in Condition C, to remain a more stable (or higher) methanol/oil ratio. Addition of raw or recycled methanol increased collision rates between the reactants and thus accelerated the forward reaction. Hence, at higher molar ratio of methanol/oil as in Condition B and D (approximately 10 to 80 times of that under Condition C), the data shows a more complete reaction of oil to produce FAME. This result is consistent with the studies reported by Vyas *et al.* [20] and Gabriel *et al.* [21] in terms of the association between methanol/oil molar ratio and FAME yield. Both of these studies have agreed that forward reaction can be driven by increasing methanol/oil ratio to achieve higher yield of FAME. Fig. 5 shows the accumulated FAME amount of multi-stage MATR. It can be seen from the data shown in Condition B and D that little amount of FAME was produced after Stage II reaction, indicating that glycerol expelling and methanol refilling can provide the effect of a more complete reaction. In Condition A and C, it was observed that FAME amount was accumulated steadily after each stage. Especially in Condition C, not only larger FAME amount than that in Condition A had been produced, but the FAME yield can also be maintained at above 90% from Stage II-V with a stable oil surplus rate and SBO amount (Fig. 3 and 4). This also indicates a steady performance of the overall multi-stage MATR process under this condition.

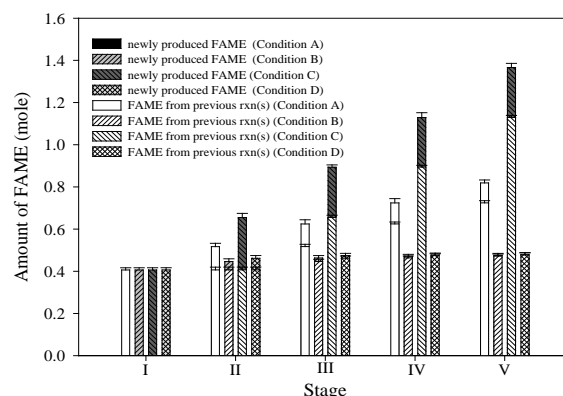


Fig. 5. The accumulated amount of FAME for each stage in the multi-stage MATR. The amount of FAME obtained in stages after Stage I comprised of two portions, newly produced and those from previous stage(s) under different operation conditions.

C. Stage Effect of MATR on FAME Yield

The stage effect of multi-stage MATR is determined by comparing the FAME yields obtained from two- and three-stage MATR. Two-stage MATR was performed for 10 minutes with 5 minutes microwave heating per stage. As for three-stage MATR, total time of microwave heating was shortened to 9 minutes with only 3 minutes per stage. Both

two- and three-stage MATRs were carried out under Condition D.

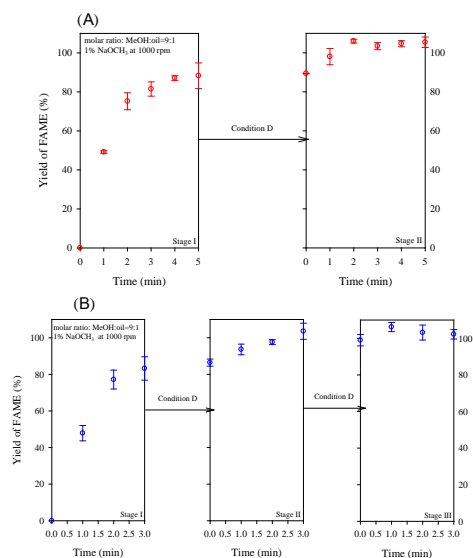


Fig. 6. Stage effect of MATR on FAME yield. The two-stage MATR was performed for 10 minutes with 5 minutes microwave heating per stage. (b) The three-stage MATR, total time of microwave heating was 9 minutes with 3 minutes per stage. All processes were carried out under Condition D shown in Fig. 2.

According to the data for stage effect of MATR on FAME yield shown in Fig. 6, both two- and three stage MATR can achieve 100% FAME yield at Stage II with respectively 82% (two-stage) and 80% (three-stage) FAME yield in Stage I. For three-stage MATR, the 100% FAME yield remained at Stage III also indicated the reaction had completed within two stages. In other words, the FAME yields for two- and three-stage MATR appeared to be similar despite the shortened heating time in the three-stage reaction. The overall result also indicates that heating time for multi-stage MATR can be shortened to 3 minutes per stage with a total of only 6 minutes heating time to achieve 100% FAME yield.

IV. CONCLUSION

The MATR system designed in this study has been proved to be effective, economic, and practical for the scale-up production of biodiesel. The FAME yield results under Condition D show a significant effect on improving the forward reaction. While comparing the results with Condition B, it shows that refilling of both recycled and fresh methanol can provide high FAME yield accompanied with low oil surplus rate, which symbolizes a complete reaction. The comparison between results of Condition A and C also shows that persistent refilling of raw reactants accompanied with recycled methanol refilling can also show a stable SBO content in each stage of reaction, indicating that more stages of reactions can be performed after Stage V. As for the results for stage effect of MATR, in addition, it shows similar effect in terms of FAME yield and oil surplus rate with either 3 and/or 5 minutes microwave irradiation. By using the MATR system designed, in conclusion, FAME yield can achieve 100% at Stage II with only 6 minutes of microwave heating time (3 minutes per stage). Meanwhile, the multi-stage transesterification with persistent reactant

refilling and glycerol removal has been proved to be not only effective in generating high yield of FAME, but also practical in providing a reference for scale-up production of biodiesel.

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Chien-Hsiu Hung was born in Chang Hua county of Taiwan in 1983. Presently, she is pursuing her doctorate of science degree in the field of food science at National Chung Hsing University, Taiwan. During 2012 to 2017, she has published 1 book and 2 SCI research papers in the field of food and chemical engineering.

As a professional food technician who holds licenses issued by Taiwan Government Sector in different field of specializations, which include technician for chemistry, food inspection/analysis, food baking, and dietitian, Ms. Hung is currently the manager of food safety department at Tzong Hsin Co., Ltd.



Ying Ming Chew was born in Malaysia in 1980, holds a BS. Degree and a MBA degree from National Chung Hsing University (Taiwan) and University of Southern Queensland (Australia), respectively. He is currently working on his doctoral program at Department of Food Science and Biotechnology with National Chung Hsing University.

With up to 10 years work experience in food industry, Mr. Chew has approximately 5-year experience as a Senior Food Technologist with Riken Vitamin Group and 5-year experience in flavoring industry.



Yi-Chung Fu (1960-2015) was an associate professor with National Chung Hsing University, Taiwan. He spent approximate 20 years of his life on academic research in food industry.

Being a professional scientist in the field of food engineering, microwave heating technology, bioseparation technology, minimal bioresource processing, and artificial intelligent (AI) expert system, Professor Fu had contributed at least 20 research papers to SCI journals, 5 books, and 1 patent in the food engineering lab at Department of Food Science and Biotechnology.



Chin-Shuh Chen has been a professor with Food Science and Biotechnology Department of Taiwan National Chung Hsing University since 1988. In the past 30 years, Professor Chen has been working aggressively on food technology, applied microbiology, and enzyme production and application research, while numerous talents (eg. professors, officers in government sectors, or the owners of several well-known food manufacturers)

have been educated and nurtured in his lab.

As a veteran and expert of food science, microbiology and enzyme engineering, Professor Chen has more than 30 publications contributed to the field, including SCI and non-SCI journals, industrial patents, and academic books and handouts.