CHAPTER III
RESEARCH PROCEDURES

3.1 Reference Data Source of the Plant

3.1.1 Introduction to the Biodiesel Plant Process in Thailand

A flowchart of the biodiesel production process is shown in Figure 3.1.

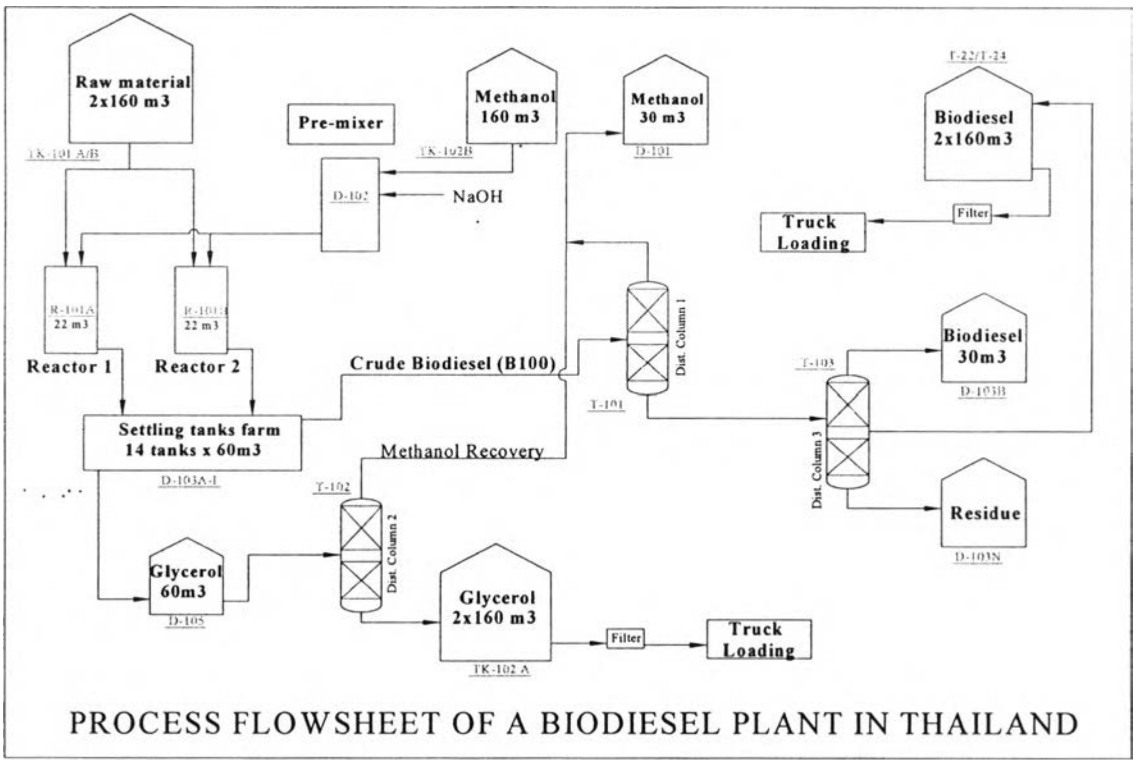

Figure 3.1 Biodiesel process of Veerasunwan Company (Thailand).

TK-101 A/B:	Raw Material storage tanks	T-101, 102, 103:	Vacuum distillation column
TK-102B:	Methanol tank	FH 2-1, 2-2:	Heater
D-102:	Pre-mixer drum	T22/T24:	Product tanks (Biodiesel)
R-101A/B:	Reactor	TK-102C:	B100 tank
D-103A-L:	Setting tanks	TK-102A:	Glycerol tank

In this process, refined palm stearine oil (Grade A) is used as the raw material, so pretreatment is not needed. Since the cost of the raw material is rather high, it is difficult to reduce the cost of the finished products. Palm oil is pumped from tanks into two reactors (R-101A and R-101B) and combined with a mixture of methanol and NaOH by a mixer. These reactors are jacket reactors; heat supplied from the hot oil keeps the temperature of the reactors at about 65-68°C. (The hot oil is heated up by furnace). The reactors are batch-operated and have a capacity of 22,000 liters/batch, so the maximum capacity of the plant is 44,000 liters/batch. The mixing and heating time for the mixture is about 90 minutes. After the reaction is completed, the mixture is transferred to the settling tank farm, which consists of 14 tanks with capacity of 60 m^3 each. The settling time is about 90 minutes. Centrifuge equipment is not used in the process, so many settling tanks (14 tanks) need to be used. This problem poses a challenge in building bigger plants.

After separation into two phases in the settling tanks, crude biodiesel from the top of the settling tanks goes to vacuum distillation column 1 (T-101) (packing type). In this stage, the common method of washing by water is not used; vacuum distillation is used to separate methanol and biodiesel from the mixture, and the biodiesel product (B100), which will be collected at the bottom, will be sent to vacuum distillation column 3 (T-103) to separate out the glycerides. Glycerides are esters formed from glycerol and fatty acids. Glycerol has three hydroxyl functional groups that can be esterified with one, two, or three fatty acids to form monoglycerides, diglycerides, and triglycerides—and biodiesel. Glycerides are collected at the bottom as soaps and can be used as fuel. Pure biodiesel (B100 – commercial product) is collected from the top and the side-stream of the distillation column.

Methanol at the top will be recovered at methanol tank and transferred to methanol plants to be refreshed, because the required methanol used in the transesterification reaction must be very pure. The streams from the bottom of settling tanks containing methanol, glycerin, and NaOH will enter distillation column 2 (T-102) to separate methanol at the top, and glycerol with NaOH at the bottom will be removed from the bottom product.

3.1.2 <u>Main Components in the Production Process</u>

The main operational parameters of each unit in the process are listed below:

I. Reactor: R-101 A/B

- 2 reactors with a capacity of 22,000 liters each
- Stainless steel
- Stir tanks with motor on the top
- Jacket–tank type with hot stream (Oil 300) running outside of the tank
- Heating oil supply at 120°C
- Temperature: kept stable at 65-68°C
- Reaction time: 90 min
- Reaction yield: minimum 95%

II. Purification unit or distillation column

Vacuum Distillation Column 1 (T-101)

- Packing column
- Vacuum distillation
- Add heat at the bottom by hot oil
- Operation parameters:
  - Inlet stream at 117°C
  - Top temperature: 89°C
  - Bottom temperature: 140°C
  - Pressure: 0.21 atm

III. Vacuum distillation column 3 (T-103)

- Packing column
- Vacuum distillation
- No added heat at the bottom
- Operation parameters:
  - Inlet stream at 274°C
  - Top temperature: 231°C
  - Bottom temperature: 210°C
  - Side draw: 245°C
  - Pressure: 0.11 atm

IV. Vacuum distillation column 2 (T-102)

- Packing column
- Vacuum distillation
- Add heat at the bottom
- Operation parameters:
  - Inlet stream at 110°C
  - Top temperature: 89°C
  - Bottom temperature: 120°C
  - Pressure: 0.21 atm

V. Pre-mixer: Catalyst mixing drum (D-102)

Purpose: to dissolve NaOH in methanol before mixing with palm oil in the reactors.

- Stir tank
- Stainless steel tank
- Volume: 8,000 liters

VI. Settling tanks: D – 103A-L

Settling tanks (separation units) consist of 14 tanks with a volume of 60 m^3 each to separate the biodiesel that exits at the top and glycerol at the bottom.

VII. Raw material storage tanks: TK-101 A/B

Consist of two tanks a the volume of 160 m^3 each, loaded by tank truck. Because palm stearine oil very easily to solidifies, it is necessary to add heat to each tank in order to keep the temperature at about 50-60°C.

VIII. Vacuum pump

Purpose: to produce a vacuum pressure in the three distillation columns: T-101, T-102, and T-103.

3.1.3 Products Obtained from the Production Process

Table 3.1 Certificate of Analysis

Product: Biodiesel (B100)

TEST ITEM	TEST METHOD	LIMIT	RESULT
1. Appearance	Visual	Good	Good
2. Density @ 15°C, kg/m^3	ASTM D 4052-96	860-900	871.9
3. Kinematic Viscosity @ 40°C, mm^2/s	ASTM D 445-04	3.5-5.0	4.427
4. Sulphated Ash, % wt.	ASTM D 874-00	Max 0.02	0.001
5. Sulphur Content, ppm by wt.	ASTM D 2622-03	Max 10	2.17
6. Water and Sediment, %vol.	ASTM D 2709-96	Max 0.2	<0.005
7. Flash Point, (P.M.), °C	ASTM D 93-02a	Min 120	>120
8. Copper Strip Corrosion (3hrs. @ 50 °C)	ASTM D 130-94	Max No.1	Ia
9. Total Acid Number, mg KOH/g	ASTM D 664-01	Max 0.50	0.37
10. Water Content, %wt.	EN 12937	Report	0.04
11. Micro Carbon Residue, % wt.	ASTM D 4530-93	Report	0.01
12. Oxidation Stability at 110°C, hours.	EN 14112	Min 6	3.4
13. Iodine Value, g I_2/100g	EN 14111	Max 120	27

Product: Biodiesel (B100)

Item	Property	Unit	Test Method	Result
1	Monoglyceride	%wt	EN 14105	<0.250
2	Diglyceride	%wt	EN 14105	<0.050
3	Triglyceride	%wt	EN 14105	<0.050
4	Free glycerin	%wt	EN 14105	<0.005
5	Total glycerin	%wt	EN 14105	0.031*
6	Methyl ester	%wt	EN 14105	100.0
7	Linoleic acid methyl ester	%wt	EN 14105	Nil
8	Methanol content	%wt	Inhouse method	0.01
9	Oxidation stability at 110°C	Hr.	EN 14112	4.0
10	Sodium	mg/kg	ASTM D5185	Nil
11	Potassium	mg/kg	ASTM D5185	2.4
12	Calcium	mg/kg	ASTM D5185	Nil
13	Magnesium	mg/kg	ASTM D5185	Nil
14	Phosphorous	%wt	ASTM D4951	Nil

Remarks: *calculated from extrapolated calibration curve

3.2 Software Programs

3.2.1 Software Introduction

3.2.1.1 ICAS Software

ICAS was developed by CAPEC – Technical University of Denmark. (CAPEC software is closely related to the CAPEC research projects and it is not commercial software). ICAS is an Integrated Computer Aided System with "toolboxes" that help to solve a multitude of problems with high efficiency. ICAS software combines computer-aided tools for modeling, simulation (including property prediction), synthesis/design, control, and analysis into a single integrated system. These tools are present in ICAS as toolboxes. The user may move from one toolbox to another to solve problems requiring more than one tool while working with the software. We can, therefore, use the analysis and utility toolbox, together with the synthesis toolbox, design toolbox, or analysis toolbox at the same time. We can start from any toolbox; it is possible to invoke the simulation engine to perform a steady state and/or dynamic simulation for batch and/or continuous process operations. With the synthesis toolbox, we can invoke the solvent design tool (in the design toolbox) if a solvent is needed for a specific separation task. There is also a utility toolbox, which determines properties, phase diagrams, etc., which can be used by the other toolboxes or by the user to analyze the behavior of the specified system. (Source from: http://www.capec.kt.dtu.dk/Software/CAPEC-Software)

3.2.1.2 PRO/II Software

PRO/II is a Process Engineering Suite product (PES) developed by SimSci-Esscor. This is a commercial product and is very powerful and popular in the oil refinery industry. The PRO/II steady-state simulator "performs rigorous mass and energy balances for a wide range of processes. From oil and gas separation to reactive distillation, PRO/II combines the data resources of a large chemical component library and extensive thermodynamic property prediction methods with the most advanced and flexible unit operations techniques. The users benefit from computational facilities to perform all mass and energy balance

calculations needed to model most steady-state processes within the chemical, petroleum, natural gas, solids processing, and polymer industries". PRO/II can be run in a Windows®-based GUI environment and can be applied in designing new processes, valuating alternate plant configurations, modernizing and revamping existing plants, determining environmental impact, troubleshooting and debottlenecking plant processes, and monitoring, optimizing, and improving plant yields and profitability.
(Source: www.simsci-esscor.com/us/eng/products/productlist/proII/default.htm)

3.2.2 Combining ICAS and PRO/II to Achieve the Objective

ICAS is used for analysis, development of models, and selected optimization problems for existing bio-diesel plants; PRO/II will be used for simulation of the operation of the plant.

3.3 Methodology (Systematic Method)

3.3.1 Why Design and Optimization of Biodiesel Plants are Important

According to the above information, we can see that although biodiesel is a product of great interest for the many benefits it brings in, we still must deal with a lot of challenges in terms of economy and technology.

Design and optimization is a way to help investors to maximize its benefit and reduce disadvantages such as high cost. There are a number of software programs available to assist the design and optimization phase.

3.3.2 Systematic Method

The aim of this project to improve the efficiency of a biodiesel from palm oil production plant in Thailand. Several factors, such as technical, social, and economic aspects, are taken into consideration.

A methodology has been created in order to assist companies in the selection of appropriate criteria for the implementation of biodiesel production. The methodology is divided into four parts, as listed in the Figure 3.2 below:

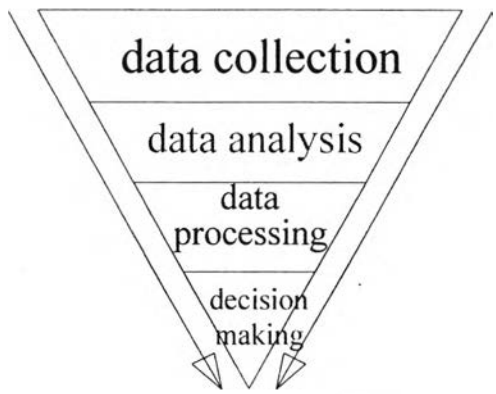

Figure 3.2 Methodology steps.

3.3.2.1 Data Collection

In this step, various data, such as raw materials, products, market, consumption, future market, techniques, and other information, are collected from various biodiesel companies in Thailand.

3.3.2.2 Data Analysis

The collected data will be analyzed by ICAS or PRO/II to discover sensitive points. This is the most important step of the project. It includes five main steps:

1- Build the structures of five components not in the database of PRO/II and then the property predictions of component can be found and are filled in the database of PRO/II.
2- Build the biodiesel plant in PRO/II and set-up mass balance for process using data collected from the real plant.
3- Build the energy balance for process.
4- Find the cost associated with each operation.
5- Find the largest operating costs in term of percentage of total cost, to be analyzed.

3.3.2.3 Data Processing

Depending on the results from data analysis step, the analyzed data will be used as input data to run a simulation by PRO/II. The purpose of this step is to evaluate:

- Economic effect
- Environmental impacts

The four main steps of this section are shown below:

1- Maximize the recovery of B100 by rigorous design for distillation column T-103 and also minimize the energy supply for this column.

2- Perform rigorous design and optimize for distillation column T-101.

3- Do heat integration: to find the heat exchanger network that minimizes utility consumption.

4- Evaluate environmental impact: to find the benefit of this modified process to the environment.

3.3.2.4 Decision–Making

Based on the processed data, feedback and final decisions can be derived, and the investors can make use of them to improve the economics of the plant.