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## Characterizing Particle Board Made of Oil Palm Empty Fruit Bunch Using Central Composite Design

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### Cover Page Footnote

We would like to express our deep gratitude to Dr. Darmadi for his valuable assistance with the optimization software. Our thanks are also extended to the staffs and technicians of the laboratory of the Chemical Engineering Department of Sumatera Utara University for their guidances in using the equipment in their laboratory.

## Characterizing Particle Board Made of Oil Palm Empty Fruit Bunch Using Central Composite Design

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### Abstract

Particle board was made using processed oil palm empty fruit bunch and casein glue. This research used two factor variables and eight response variables to determine the best matrix/filler ratio and compression pressure for particle board production. The factor variables, (matrix/filler ratio and compression pressure), determined the response variables: modulus of rupture, water content, density, tensile strength, and both endothermic-and-exothermic temperatures and heats. This research aimed to optimize the mechanical and physical property of particle boards by using the bunch/glue ratio. The processing of data was undertaken by Response Surface Method (RSM) with Central Composite Design (CCD). The optimal conditions under which particle board created were at matrix/filler ratio of 50:50 and compression pressure of 3.53 kg/cm<sup>2</sup>; resulting in a modulus of rupture of 325.32 kg/cm<sup>2</sup>, water content of 0.009%, density 0.826 g/cm<sup>3</sup>, and tensile strength of 2.573 kg/cm<sup>2</sup>. This research also indicates that casein glue results in particle board with optimum parameters of endothermic temperature of 247.74 °C, an endothermic heat of -4.122 mW, exothermic temperature of 431.08 °C, and exothermic heat of 7.526 mW. Based on physical testing, most particle board properties obtained fulfilled the standards of water content and density as required by the Indonesian National Standard/SNI 03-2105-2006.

### Abstrak

**Karakterisasi Papan Partikel dari Tandan Kosong Kelapa Sawit dengan Menggunakan Desain Central Composite.** Papan partikel dibuat dengan menggunakan tandan kosong kelapa sawit yang diolah dengan perekat kasein. Penelitian ini menggunakan dua variabel faktor dan delapan variabel respons untuk menentukan rasio matriks/filler dan tekanan kompresi terbaik untuk produksi papan partikel. Variabel-variabel faktor tersebut, (rasio matriks/filler dan tekanan kompresi), menentukan variabel respons: keteguhan patah, kandungan air, densitas, kekuatan tarik, temperatur dan panas eksotermis dan endotermis. Penelitian ini bertujuan untuk mengoptimasi sifat mekanik dan fisika papan partikel tersebut dengan menggunakan rasio tandan terhadap perekat. Pengolahan data dilakukan dengan menggunakan *Response Surface Methodology* (RSM) dengan *Central Composite Design* (CCD). Kondisi optimum yang menghasilkan papan partikel adalah pada rasio matriks terhadap filler sebesar 50:50 dan tekanan kompresi sebesar 3,53 kg/cm<sup>2</sup>, yang menghasilkan kekuatan patah 325,32 kg/cm<sup>2</sup>, kandungan air 0,009%, densitas 0,826 g/cm<sup>3</sup>, dan kekuatan tarik 2,573 kg/cm<sup>2</sup>. Penelitian ini juga menunjukkan bahwa perekat kasein menghasilkan papan partikel dengan parameter optimum temperatur endotermis sebesar 247,74 °C, panas endotermis -4,122 mW, temperatur eksotermis 431,08 °C, dan panas eksotermis 7,526 mW. Berdasarkan hasil pengujian fisik dan mekanik, papan partikel yang diperoleh sebagian besar telah memenuhi standar kandungan air dan densitas yang disyaratkan oleh *Indonesian National Standard/SNI 03-2105-2006*.

**Keywords:** casein, central composite, oil palm empty fruit bunch, particle board

### Introduction

Many oil palm plantations are present in Indonesia, owned by the government and privately. The oil palm area in Indonesia increases by around 11,444,808 hectare/year with a production of 30,948,931 tons/year [1], and it

would be needed 0.88–1.05 Mega hectare of oil palms to meet the annual increase in demand [2]. The increase of oil palm production increases solid waste production of substances such as oil palm empty fruit bunch (OPEFB), pressed fiber of fruit, midriff, and oil palm fiber. It is assumed that 23–25% OPEFB is obtained from the total

mass of fresh fruit bunch, giving a potential OPEFB production in Indonesia of 2,632,306–2,861,202 tons/year. OPEFB is 72.67% fiber and it has a particle size that matches the fabrication needs of particle board. OPEFB use for particle-board fabrication has a positive impact by increasing the added value of oil palm and reduces waste that pollutes the environment.

The principal problem using OPEFB is the high amount of extractive matter present that reduces adhesive properties, and ability to be used as a thermoplastic adhesive, cement, or a thermosetting adhesive. Oil palm waste needs to undergo treatment to cope with this problem and reduce the extractive matter composition. This is carried out by first, the cutting the raw material using a chipped machine [3] so that the particle board resembles wood shavings [4]. Casein, found in mammalian milk, was used as an adhesive by craftsmen in ancient European times. Casein provides an adhesive content of 6–14% and the best treatment is achieved with an adhesive content of 14% because generally this fulfills the JIS A 5908-2003 standard [5]. Because of this, this research aims to investigate the adhesive percentage variation at the percentile to determine the quality of particle board formed.

Research on the use of oil palm waste to produce a more valuable product has been conducted but there are currently few commercial uses for it [6]. OPEFB board produced by [7] has fulfilled FAO requirements of density and modulus of rupture. Density is one of physical properties that show comparison between the amount of matter mass and its volume unit. Density testing is useful to know particle board density resulted. However, previous research has not investigated endothermic or exothermic temperature and heat. This research therefore investigates the ability of casein glue to produce particle board. The OPEFB and glue ratio are varied. Differential Scanning Calorimetry (DSC) is used to determine the temperature and heat.

## Methods

In order to know the tensile strength, modulus of rupture, and particle board properties, a tensile testing machine and compressive test equipment were used. Modulus of rupture is a mechanical property of particle board related to board ability to hold loads. Modulus of rupture is an external force that is applied to a maximum and tends to change the material form [8]. Modulus of rupture can be calculated from the load deflection curve [9] to determine compliance with the specifications of materials [10]. This research result is further optimized statistically by *Response Surface Methodology* (RSM) using Design-Expert 6.0.6 software through Central Composite design (CCD). Factorial designs allow researchers to look at how multiple factors affect a dependent variable, both independently and simultaneously. Factorial design studies

are named for the number of levels of the factors. CCD is useful to adjust experimental plan, where the design of the formula is by combination  $2^k$  factorial dependent variables with a simple design. The design is typically very efficient to find the number of experiments conducted. CCD combines two-level-full-factorial design with at least one dependent variable in the middle of the research variables being investigated [11], which allow the estimation of the second-order polynomial equation [12]. It enables mathematical modelling such as the linear and quadratic models to describe the correlation among independent variables monitored. The CCD is an alternative to three-level-full-factorial design because it demands fewer experiments to provide comparable results. CCD has three different points: edge points as two-level design, star points that take quadratic effects, and centre points. It has three variants namely circumscribed, inscribed, and face centered.

In engineering, determining which factors are significant and their response is often difficult. In such cases, full factorial design is useful to find all possible combinations. The selection of full factorial design is appropriate when one needs accurate measurement results with various operating conditions and when the responses change unpredictably. This approach often needs a large number of experiments because the number of experiments increases geometrically with factors tested. Furthermore, interpretation of data is difficult and unnecessary, because engineering applications focus on trends and how appropriate factors that affect a response. Therefore, the response surface method with CCD is an efficient way to experimentally explore the relationship of investigating factors and response. In this research, the evaluation of the effect of matrix/filler ratio and compression ratio uses CCD combined with RSM. The purpose is to get and to confirm experimentally, the optimum operating conditions that meet maximum modulus of rupture, density, and exothermic temperature. The method of analysis here is useful for investigations of new types of particle boards.

## Materials and Methods

**Source of oil palm empty fruit bunch.** Oil palm empty bunch taken was from a community's plantation in Desa Lhok Gayo, Kecamatan Babah Rot, Aceh Barat Daya. This solid waste was from cutting machine. This waste contained large amount of potassium oxide, had a brown color and unpleasant odor. The bunch taken was put into a plastic bag and cooled in refrigerator at a temperature of 4 °C. The bunch characteristics are given in Table 1.

**Material** Materials used in this research were casein, which was used as a glue, and oil palm empty fruit bunch (OPEFB) used as filler. The glue or casein was an organic polymer (matrix) and with maleic anhydride (MAH) 2% added by sample mass [13] to increase strength and

**Table 1. Analysis of Oil Palm Empty Fruit Bunch Composition**

Parameter	Composition (%)
Nitrogen	0.8
Phosphor	0.2
Potassium Oxide	2.9
Magnesium Oxide	0.2
Calcium Oxide	0.3
Chlor	0.4

impact properties [14]. The OPEFB was cut by cutting machine into small shapes of 5 cm. This OPEFB piece still contained impurities needed decocting at a temperature of 40 °C for two hours. The *coupling agent* used was maleic anhydride (MAH). Whereas, starch and threethanolamine were useful as materials to add to the casein adhesive power.

**Preparation of particle board.** This research uses a glass reactor (15 cm × 10 cm × 1 cm), equipped with magnetic stirrer on a hot plate. The chemicals used was casein adhesive, which had extreme heat stability [15], made by modifying its adhesive composition [13, 16, 17]. This was achieved by mixing 33.1% casein, 26.5% starch, 36.2% cold distilled water, and 4% threethanolamine. The agitator motor on the *hot plate* was then turned on. 43% of sample mass of distilled water was then added to the mixture in the reactor and this was heated at a temperature of 60 °C. Furthermore, MAH was added at about 2% of available sample mass. After boiling, the next stage was drying the OPEFB in sunlight for one day to reduce the content of water still contained in OPEFB fiber. Following this, OPEFB was dried in an oven at 100 °C for 10 minutes, ground in a spice-grinding machine, and screened with a 80–100 screening mesh. The OPEFB particle was entered into a reactor that consists of the mixture of casein, starch, cold water, threethanolamine, and MAH. The blending of the mixture was carried out with a particular ratio of matrix until homogenous which took about 15 minutes. The homogeneous mixture was put outside the reactor and into sample container in a cast for casting with a heat press. Compression took place at 130 °C for 15 minutes as shown by CCD. The particle board obtained was left for 24 hours before analysis.

**Experimental design.** By using RSM, the relationship between independent process variables and the result could be determined. In order to find the relationship, independent variables for the process were the ratio of filler/matrix (matrix percentage, or A) and compression pressure (B). The real values of process variables for the process are presented in Table 2. Optimization by Design-Expert 6.0.6 software with CCD was carried out on this random experimental method of particle board fabrication from OPEFB. The relationship selection of

matrix/filler ratio of A (15:85; 25:75; 35:65; 45:55; 55:45) and compressive pressure of B (0.8; 1.6; 2.4; 3.2; 4.0 kg/cm<sup>2</sup>) uses the CCD that resulted in 13 treatment times (Table 3). Response variables in this process are the modulus of rupture, water content, density, tensile strength, endothermic temperature, endothermic heat, exothermic temperature, and exothermic heat (Tables of 4 and 5).

**Characterization of particle board.** A mechanical test was also carried in the Polymer laboratory. Modulus of Rupture (MOR) determination refers to SNI 03-2105-2006 for particle board by using *Servo Control System Universal Testing Machine A1-7000M* [18]. In order to get the optimum conditions, the best modulus of rupture measurement was based on all variables. The optimum modulus of rupture could indicate the effective process and the most efficient energy use.

Tensile strength test took place by cutting the board in a dog bone shape. The load during the test used continuously at bearing motion of testing machine was 4 mm/minute. Self tightening grips were used to transmit the load to the board. If either grip cause a failure within 12 mm, the value was disregarded. The next analysis was

**Table 2. Code Factor and Input Variable of Response Surface Methodology with Central Composite Design**

Code Factor	A	B
Input Variable	Ratio of filler/matrix	Compression pressure (kg/cm <sup>2</sup> )
-1.41	15:75 (written as 15)	0.8
-1.00	25:75 (written as 25)	1.6
0.00	35:65 (written as 35)	2.4
1.00	45:55 (written as 45)	3.2
1.41	55:45 (written as 55)	4.0

**Table 3. Central Composite Design**

Run	Standard	Ratio of filler/matrix	Compression Pressure (kg/cm <sup>2</sup> )
1	8	35:65	4.0
2	10	35:65	2.4
3	7	35:65	0.8
4	13	35:65	2.4
5	2	45:55	1.6
6	1	25:75	1.6
7	11	35:65	2.4
8	5	15:85	2.4
9	6	55:45	2.4
10	3	25:75	3.2
11	12	35:65	2.4
12	4	45:55	3.2
13	9	35:65	2.4

**Table 4. Actual and Predicted Values of Mechanical and Physical Properties using Central Composite Design**

Run	Ratio of matrix/filler	Compression Pressure (kg/cm <sup>2</sup> )	Modulus of Rupture (kg/cm <sup>2</sup> )		Water Content (%)		Density (g/cm <sup>3</sup> )		Tensile Strength (kg/cm <sup>2</sup> )	
			Act.	Pred.	Act.	Pred.	Act.	Pred.	Act.	Pred.
1	35:65	4.0	260.28	258.78	0.45	1.23	0.70	0.70	1.55	1.58
2	35:65	2.4	198.91	204.62	0.94	0.67	0.64	0.65	1.31	1.34
<b>3</b>	<b>35:65</b>	<b>0.8</b>	160.08	150.45	<b>7.72</b>	<b>6.28</b>	0.57	0.59	1.08	1.10
4	35:65	2.4	198.91	204.62	0.94	0.67	0.64	0.65	1.31	1.34
5	45:55	1.6	242.31	231.96	0.39	2.30	0.75	0.72	1.85	1.77
6	25:75	1.6	122.59	123.11	1.23	2.88	0.54	0.52	0.77	0.75
7	35:65	2.4	198.91	204.62	0.94	0.67	0.64	0.65	1.31	1.34
<b>8</b>	<b>15:85</b>	<b>2.4</b>	<b>102.18</b>	<b>95.77</b>	0.83	0.63	<b>0.47</b>	<b>0.45</b>	<b>0.39</b>	<b>0.39</b>
<b>9</b>	<b>55:45</b>	<b>2.4</b>	288.57	313.47	0.26	0.21	<b>0.82</b>	<b>0.84</b>	<b>2.58</b>	<b>2.64</b>
10	25:75	3.2	164.45	177.28	0.76	0.19	0.56	0.57	0.92	0.89
11	35:65	2.4	198.91	204.62	0.94	0.67	0.64	0.65	1.31	1.34
<b>12</b>	<b>45:55</b>	<b>3.2</b>	<b>325.04</b>	<b>286.13</b>	<b>0.24</b>	<b>0.07</b>	0.79	0.77	2.21	2.12
13	35:65	2.4	198.91	204.62	0.94	0.67	0.64	0.65	1.31	1.34

**Table 5. Actual and Predicted Values of Endothermic and Exothermic Heats using Central Composite Design**

Run	Ratio of matrix/filler	Compression Pressure (kg/cm <sup>2</sup> )	Endothermic Temperature (°C)		Endothermic Heat (mW)		Exothermic Temperature (mW)		Exothermic Heat (mW)	
			Actual	Predict	Actual	Predict	Actual	Predict	Actual	Predict
1	35:65	4.0	254.28	252.68	-5.96	-5.86	405.08	406.19	10.71	10.42
2	35:65	2.4	251.81	252.44	-6.42	-6.41	402.26	404.20	11.40	11.31
<b>3</b>	<b>35:65</b>	<b>0.8</b>	249.34	249.35	-6.87	-6.95	<b>399.44</b>	<b>403.18</b>	12.09	12.15
4	35:65	2.4	251.81	252.44	-6.42	-6.41	402.26	404.20	11.40	11.31
5	45:55	1.6	253.42	249.11	-5.39	-5.33	410.86	405.24	9.756	9.43
6	25:75	1.6	257.83	255.53	-8.37	-8.29	421.42	414.73	14.51	14.95
7	35:65	2.4	251.81	252.44	-6.42	-6.41	402.26	404.20	11.40	11.31
<b>8</b>	<b>15:85</b>	<b>2.4</b>	<b>259.93</b>	<b>258.86</b>	-9.84	-9.84	426.52	429.48	19.14	18.64
<b>9</b>	<b>55:45</b>	<b>2.4</b>	245.04	246.02	<b>-4.00</b>	<b>-3.98</b>	<b>428.35</b>	<b>430.24</b>	<b>7.311</b>	<b>7.58</b>
10	25:75	3.2	253.58	249.11	-7.62	-7.71	410.43	406.36	13.30	14.09
11	35:65	2.4	251.81	252.44	-6.42	-6.41	402.26	404.20	11.40	11.31
12	45:55	3.2	249.23	249.35	-4.69	-4.81	419.61	416.61	8.535	8.55
13	35:65	2.4	251.81	252.44	-6.42	-6.41	402.26	404.20	11.40	11.31

the water content as conducted by [19, 20] and density as in JIS A 5908:2003 [21]. A dry sample of 10 cm × 10 cm × 1 cm is weighed to measure its density.

A thermal test took place by using *Differential Scanning Calorimeter* (DSC) in the Laboratory of Catalyst and Catalysis, Chemical Engineering, Universitas Syiah Kuala. For the thermal test, the board was cut by saw so that its thickness and length the same as the sample container and its mass was 4 mg (ASTM D3418-12e1). The first thermal cycle took place by heating sample at rate of 5 °C/minute in the range of 70–100 °C. The temperature was held constant for 10 minutes. A sample was heated to a maximum temperature of 380 °C at a heating rate of 10 °C/minute [22, 23]. Then, sample cooling took place to 212 °C by using rate of 10 °C/minute and its cooling curve was recorded.

## Results and Discussion

### Response analysis using central composite design.

Particle board made by OPEFB (filler) uses cellulose and halocellulose contained in OPEFB. The board is light brown with a relatively smooth and compact surface. The particle board manufacture also uses five different ratios of the matrix and filler. Processing of OPEFB from cutting machine uses process variables such as the matrix/filler ratio (15:75–55:45) and compression pressure (0.8–4.0 kg/cm<sup>2</sup>). Both process variables become the main factor run in the CCD. Based on these factors, investigation focuses on knowing the effect of process variables on modulus of rupture, water content, density, tensile strength, endothermic and exothermic temperatures, endothermic heat, and exothermic heat. The codes of design matrix entered are matrix/filler ratio and compression pressure (kg/cm<sup>2</sup>).

The results obtained are: water content, density, tensile strength, modulus of rupture, exothermic temperature and heat, exothermic temperature and heat. Results are summarized in Table 4 and Table 5.

Based on Table 4, the twelfth run shows the highest value of the modulus of rupture with filler/matrix ratio of 45:55 and compression pressure of 3.2 kg/cm<sup>2</sup>. In such conditions, the modulus of rupture obtained is 325.04 kg/cm<sup>2</sup>. On the other hand, the lowest modulus of rupture is 102.18 kg/cm<sup>2</sup> obtained during the eighth run at filler/matrix ratio of 55:45 and compression pressure of 2.4 kg/cm<sup>2</sup>. The relationship between process variable and modulus of rupture at response obtained provides a mathematical equation to predict the response. The Design Expert 6.0.6 software results in Equation 1–Equation 8 for each response.

$$\begin{aligned} \text{MOR} &= -67.11 + 5.44 A + 33.85 B & (1) \\ \text{Water Content} &= 11.56 + 0.036 A - 7.71 B - 1.152 \times 10^{-3} A^2 + 1.2 B^2 + 9.938 \times 10^{-3} AB & (2) \\ \text{Density} &= 0.23 + 9.583 \times 10^{-3} A + 0.032 B & (3) \\ \text{Tensile Strength} &= 0.094 + 0.01 A - 0.083 B + 4.389 \times 10^{-4} A^2 + 1.977 \times 10^{-3} B^2 + 6.438 \times 10^{-3} AB & (4) \\ \text{Endothermic Temperature} &= 263.32 - 0.32 A + 0.15 B & (5) \\ \text{Endothermic Heat} &= -14.04 + 0.24 A + 0.4 B - 1.253 \times 10^{-3} A^2 + 6.277 \times 10^{-4} B^2 - 1.781 \times 10^{-3} AB & (6) \\ \text{Exothermic Temperature} &= 532.76 - 5.95 A - 21.56 B + 0.064 A^2 + 0.19 B^2 + 0.62 AB & (7) \\ \text{Exothermic Heat} &= 27.71 - 0.59 A - 0.48 B + 4.504 A^2 - 9.008 \times 10^{-3} B^2 - 4.687 AB & (8) \end{aligned}$$

Equations 2–8 show that variables of the matrix/filler ratio (A) and compression pressure (B) are proportional to each response indicated by a constant value that has positive sign. The higher the ratio of the matrix (A) and compression pressure (B) provided, the higher the density, tensile strength, endothermic and exothermic temperatures, and exothermic obtained. Table 4 shows the result where the highest value of water content appears in the third run with a ratio of matrix/filler of 35:65 and a pressure of 0.8 kg/cm<sup>2</sup>. In such conditions, the water content obtained is 7.72%. Whereas the lowest

water content obtained was in the twelfth run at the matrix/filler ratio of 45:55 and compression pressure 3.2 kg/cm<sup>2</sup> namely 0.24%. This means that the water content is inversely proportional to compression pressure.

Analysis of variance (ANOVA) is useful as a parametric test to distinguish the average value of more than two data groups by comparing their variances and deciding on responses among their variables [24] using a single test [25]. Table 6 summarizes ANOVA for the process of OPEFB to decide the significance of the interaction between factor variable and response variable. Table 6 predicts that values of modulus of rupture, density, and tensile strength approach the model by more than 90%. This R<sup>2</sup> value indicates that the research result obtained appears true and significant to a linear model. Table 6 also shows the effect of the inter-variable interaction. Interaction of a significant or insignificant variable is decided based on a probability value (p-value) > F that equals 0.1. A value greater than 0.1 indicates the model terms are not significant. The usage of the matrix/filler ratio indicates that significant variables in this research are mostly dominated by A and A<sup>2</sup>. Significant variables have an important effect on the modulus of rupture, density, endothermic and exothermic temperatures, and endothermic and exothermic heat. For density, compared to compression pressure whose p-value is 0.0007, variable of matrix/filler ratio is more significant with p-value < 0.0001. These different p-values show that variable of matrix/filler ratio have larger interaction effect in determining density of particle board.

By using research result data, constraint could be written mathematically:

$$\begin{aligned} 15:85 &\leq \text{Matrix/filler ratio} \leq 55:45 \\ 0.8 \text{ kg/cm}^2 &\leq \text{Compressive pressure} \leq 4 \text{ kg/cm}^2 \\ 102.18 \text{ kg/cm}^2 &\leq \text{Modulus of rupture} \leq 325.04 \text{ kg/cm}^2 \\ 0.237\% &\leq \text{Water content} \leq 7.724\% \\ 0.466 \text{ g/cm}^3 &\leq \text{Density} \leq 0.824 \text{ g/cm}^3 \\ 0.392 \text{ kg/cm}^2 &\leq \text{Tensile strength} \leq 2.576 \text{ kg/cm}^2 \\ 245.04 \text{ }^\circ\text{C} &\leq \text{Endothermic temperature} \leq 259.93 \text{ }^\circ\text{C} \\ -9.8372 \text{ mW} &\leq \text{Endothermic heat} \leq -4 \text{ mW} \\ 399.44 \text{ }^\circ\text{C} &\leq \text{Exothermic temperature} \leq 428.35 \text{ }^\circ\text{C} \\ 7.3113 \text{ mW} &\leq \text{Exothermic heat} \leq 19.1441 \text{ mW} \end{aligned}$$

**Table 6. ANOVA for the Probality Value Probability of Mechanical Properties of Processed of Oil Palm Empty Fruit Bunch**

Source	Modulus of Rupture		Water Content		Density		Tensile Strength	
	P-value	Remark	P-value	Remark	P-value	Remark	P-value	Remark
Model	< 0.0001	significant	0.0283	significant	< 0.0001	significant	< 0.0001	significant
A	< <b>0.0001</b>		0.8916		< <b>0.0001</b>		0.4486	
B	0.0002		0.0497		<b>0.0007</b>		0.6222	
A <sup>2</sup>			0.6594				0.0092	
B <sup>2</sup>			0.0178				0.9211	
AB			0.8982				0.1242	
R <sup>2</sup>	<b>94.25%</b>		78.22%		<b>97.24%</b>		<b>99.40%</b>	
Adj R <sup>2</sup>	93.10%		62.66%		96.69%		98.97%	

**Table 7 ANOVA Results for the Probability of Endothermic and Exothermic Heats of Processed Oil Palm Empty Fruit Bunch**

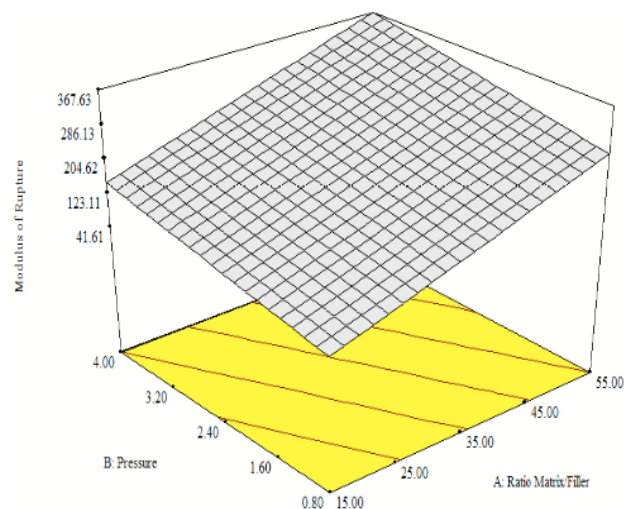
Source	Endothermic Temperature		Endothermic Heat		Exothermic Temperature		Exothermic Heat	
	P-value	Remark	P-value	Remark	P-value	Remark	P-value	Remark
Model	0.0012	significant	<0.0001	significant	0.0037	significant	<0.0001	significant
A	<b>0.0003</b>		<b>&lt;0.0001</b>		<b>0.0005</b>		<b>0.0004</b>	
B	0.8460		0.1259		0.1281		0.7029	
A <sup>2</sup>			<b>0.0002</b>		<b>0.0003</b>		<b>0.0019</b>	
B <sup>2</sup>			0.9825		0.9033		0.9523	
AB			0.7461		0.0692		0.9870	
R <sup>2</sup>	74.02%		99.82%		88.31%		98.62%	
Adj R <sup>2</sup>	68.82%		99.68%		79.95%		97.63%	

**The effect of matrix and filler ratio on modulus of rupture.** The results of testing the modulus of rupture of particle board are presented in Figure 1. The lowest value of modulus of rupture is 102.18 kg/cm<sup>2</sup>, which is obtained at particle board with the matrix/filler ratio of 15:85 and compressive pressure of 0.8 kg/cm<sup>2</sup>. The highest value of modulus of rupture is at a matrix/filler ratio of 55:45 and compressive pressure of 4.0 kg/cm<sup>2</sup>. This result indicates that the value of modulus of rupture increased with the increase of the matrix/filler ratio, and was influenced by the adhesive power. Besides the content and type of casein material used (phosphoprotein), adhesive power, and fiber length also influences the value of modulus of rupture.

While the ratio of matrix/filler increased from 15:85–55:45, the modulus of rupture in this research increased in the range of 102.18–325.04 kg/cm<sup>2</sup>. According to SNI 03-2105-2006, the value of modulus of rupture determined was a minimum of 82 kg/cm<sup>2</sup>. Thereby, particle board at all matrix/filler ratios investigated fulfilled the standard. This ratio indicated that at the same relative density, particle board strength tended to increase with the decrease in the wood constituent element. The high value of modulus of rupture was probably because of the fact that the OPEFB was the main component forming the particle board. The bunch had a relatively narrow glue-surface area so that adhesive distribution occurs evenly. Finally, during the modulus of rupture test the particle board was able to support testing load.

Modulus of rupture value relates to the closeness or estrangement of the particles in board that uses particle fiber size of length  $\pm 5$  cm. Modulus of rupture values for particle board will be higher with the increase in adhesive content used. This is similar to a bamboo particle board with 6% added urea formaldehyde (UF) adhesive with modulus of rupture of 56.86 kg/cm<sup>2</sup> and an increase to 187.56 kg/cm<sup>2</sup> at 12% UF [26].

The increase of the matrix/filler ratio (A) has a positive effect on modulus of rupture as shown in Figure 1. In addition, the higher compression pressure enhances the



**Figure 1. The Effect of Matrix/filler Ratio and Compression Pressure on Modulus of Rupture**

modulus of rupture. This trend could be of use when considering large scale production of particle board from OPEFB. Optimization with CCD indicates that the optimum modulus of rupture of particle board is 325.32 kg/cm<sup>2</sup> (Table 8) with an adhesive ratio of 50:50 and compressive pressure of 3.53 kg/cm<sup>2</sup>. Therefore, for quality and technical ease reasons, it is better that the matrix/filler ratio is 50:50 and compressive pressure is 3.53 kg/cm<sup>2</sup> because SNI standard is fulfilled.

**The effect of matrix/filler ratio on water content.** Matrix/filler ratio is an important variable in the processing of OPEFB. By increasing matrix/filler ratio, the organic compound in OPEFB processed will be less. Another important research variable is compression pressure. The compression pressure could influence the stability of the mixture in the particle board. The setting of the compression pressure could give the same impact. The water content of particle board is in the range of 0.237–7.724% with the highest content increase in the matrix/filler ratio of 35:65 and pressure of 0.8 kg/cm<sup>2</sup>. This is possible because at the ratio of 35:65 and pressure of 0.8 kg/cm<sup>2</sup>, contact between particles increases,



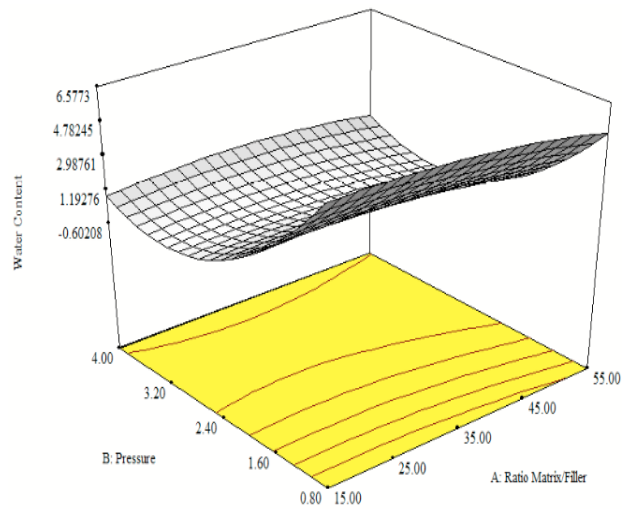
**Table 8. The Optimal Condition Values that are Appropriate for Variables Investigated**

No	Ratio of matrix/filler	Compress. Pressure (kg/cm <sup>2</sup> )	MOR (kg/cm <sup>2</sup> )	Water Content (%)	Density (g/cm <sup>3</sup> )	Tensile Strength (kg/cm <sup>2</sup> )	Endo. Temp. (°C)	Endo. Heat (mW)	Exo. Temp. (°C)	Exo. Heat (mW)	Des
1	<b>50:50</b>	<b>3.53</b>	325.32	<b>0.009</b>	0.826	2.573	247.74	-4.122	431.08	7.526	1
2	50:50	3.55	325.23	0.037	0.826	2.568	247.78	-4.130	430.93	7.534	1
3	50:50	3.64	326.02	0.194	0.825	2.560	247.93	-4.145	430.84	7.539	1
4	50:50	3.66	326.68	0.225	0.825	2.563	247.94	-4.142	430.95	7.533	1
5	50:50	3.66	327.32	0.210	0.826	2.573	247.90	-4.127	431.29	7.514	1

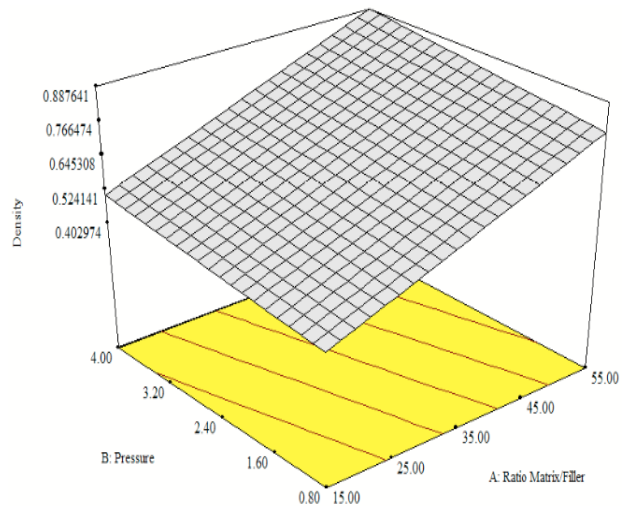
they are more densely packed, so that it is hard for water to enter between particles. Water content decreases down to a ratio of 55:45 and pressure of 2.4 kg/cm<sup>2</sup>. This research result indicates that almost all particle boards fulfill SNI 03-2105-2006, namely that they contain less than 14% water (Figure 2). All matrix/filler ratios result in water content less than 14%.

The contour plot in Figure 2 indicates the effect of the matrix/filler ratio on water content. The increase of the matrix/filler ratio in the range of 15:85–55:45 leads to water content in the range of 0.07–6.28%. Figure 2 indicates that the increase of matrix/filler ratio has a positive impact on water density. However, the increase in compression pressure indicates the water content is slightly decreased initially, and then increases again. This shows that as the compression pressure increases, less water is contained up to a certain point because the pressure causes water contained in particle board to evaporate. It is a requirement that only a small amount of water is contained within the particle board. Based on CCD, it could be said that optimum water content is 0.009% (Table 8).

**The effect of matrix and filler ratio on density.** In previous works, the common variable examined when making particle board is density. Board density is influenced by density of raw material used. Density testing results that relate to the correlation between particle board density and compression pressure are given in Figure 3. The lowest density value of particle board is at a matrix/filler ratio of 15:85 and pressure of 2.4 kg/cm<sup>2</sup> namely 0.47 g/cm<sup>3</sup> (Table 4). Based on Figure 3, the higher compression pressure gave the higher density value. In Figure 3, up to compression pressure 4 kg/cm<sup>2</sup>, density was slightly up from 0.45–0.84 g/cm<sup>3</sup>. Increasing compression pressure resulted in the near density value. In this case, the value determines the compression pressure. The highest density of particle board is at a matrix/filler ratio of 55:45 and compressive pressure of 4 kg/cm<sup>2</sup>, namely 0.82 g/cm<sup>3</sup>. A higher density value is possibly caused by the lower porosity of particle board at compression pressure of 4 kg/cm<sup>2</sup>. High porosity could occur due to air trapping while agitating and casting took place. According to SNI 03-2105-2006, the density standard is 0.4–0.9 g/cm<sup>3</sup>, thus all compression pressures fulfill the standard.



**Figure 2. The Correlation between Water Content and Matrix/filler Ratio (A) and Compressive Pressure (B)**



**Figure 3. The Correlation between Matrix/filler and Particle Board Density at Various Pressures**

Figure 3 is a surface graph for the density. It shows that the more compact particle board, the higher the density and vice versa. When the other variable, the matrix/filler ratio, is higher, this indicates that there will be large molecules that are bound so that color of particle board becomes lighter. Particle board thickness could be one of

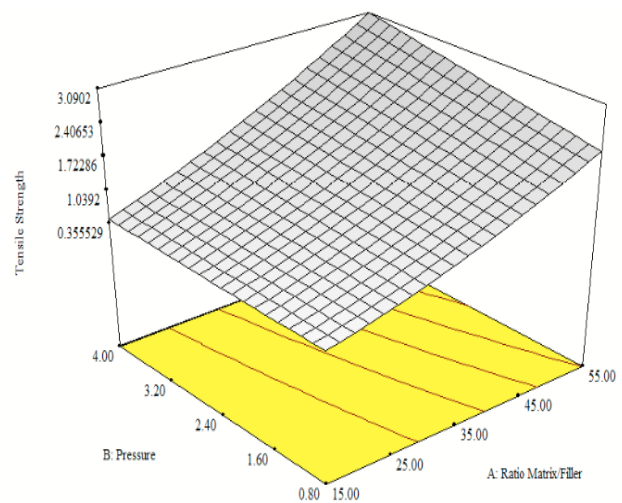
the quality indicators of particle board. It shows that the thicker particle board has less content of casein so that particle board contains more filler. Based on such content, it is expected that density of particle board obtained is high but it is still in the range of particle board's density namely  $0.4\text{--}0.9\text{ g/cm}^3$ .

The surface graph for density is different from that for water content, but the optimum point for density is similar to that for modulus of rupture, at a matrix/filler ratio 50:50 and compression pressure  $3.53\text{ kg/cm}^2$ , and the density is  $0.826\text{ g/cm}^3$ . This optimization (Table 8) aims to obtain a matrix/filler ratio and compressive pressure that could give good quality of particle board with low operating cost, reachable standard and simple technique.

Particle board quality depends on the minimum water content and the maximum density. In order to reduce operating costs, the matrix/filler ratio and compressive pressure of particle board must be kept to a minimum. The higher matrix/filler ratio used, the more casein, maleic anhydride, and threethanolamine needed, thus more such mixture that must be provided. Other mixture constituents are not too problematic because they can be obtained easily; however, needing more casein will add cost. The glue used in this research is casein which is relatively costly. For compressive pressure, the less the pressure, the lower the energy requirement and operational costs despite the long usage duration of the technique.

Therefore, for conditional optimization of board fabrication, density should be obtained in the range of  $0.45\text{--}0.84\text{ g/cm}^3$  as based on these findings, the lowest density obtained is  $0.45$  and the highest is  $0.84\text{ g/cm}^3$  (Table 4). Even though high density is an important variable in the high ratio of matrix/filler, tensile strength is also important in finding the optimum process and conditions for this process.

**The effect of matrix/filler ratio on tensile strength of particle board.** This section is an overall view of a matrix/filler ratio and compression pressure on tensile strength of particle board. Both variables have different optimum levels in the processing of OPEFB. In making particle board, the higher ratio of the matrix/filler mostly results in the higher tensile strength (Figure 4). Testing results indicate an increase in tensile strength from the matrix/filler ratio of 15:85 up to the matrix/filler ratio of 55:45. The tensile strengths are summarized in Table 4. The lowest tensile strength obtained for particle board is at the matrix/filler ratio of 15:85 and pressure of  $2.4\text{ kg/cm}^2$  namely  $0.39\text{ kg/cm}^2$ . Whereas the highest tensile strength of particle board obtained is at the matrix/filler ratio of 55:45 and pressure of  $2.4\text{ kg/cm}^2$  namely  $2.64\text{ kg/cm}^2$ .



**Figure 4. The Correlation between Tensile Strength and Matrix/filler Ratio and Compressive Pressure**

Tensile strength for both variables is also obtained for the optimum condition. This optimum condition is a result of using the response surface method with CCD that refers to theoretical and experimental data. The higher desirability (equal to 1) determines the optimum condition. The desirability or Derringer function is the most important and most used multi-criteria methodology in the optimization of analytical procedures nowadays [24]. This method is initially based on the construction of a desirability function for each response. The goal is to set the matrix/filler ratio in the range of 15:85–55:45 and compression pressure in the range of  $0.8\text{--}2.4\text{ kg/cm}^2$ .

These constraints will be useful in the RSM with CCD. By entering the constraint into the software of Design-Expert 6.0.6 thus it will give one optimal solution that agrees with the expected criteria. This solution appears in Table 8. One solution in Table 8 was selected as the optimum conditions for particle board fabrication with constraints and standards determined. Based on the solution, the selection of the actual conditions closest to the optimum considers aspects of lower compressive pressure and water content. The optimum condition by using the matrix/filler ratio of 50:50 and compression pressure of  $3.53\text{ kg/cm}^2$  results in tensile strength of  $2.573\text{ kg/cm}^2$  (Table 8).

Based on aspects of MOR, water content, density and tensile strength, the condition is not that different from the real compression pressure of  $3.2\text{ kg/cm}^2$ , thus this pressure could be considered as the best condition. The tensile strength obtained in this research will not be comparable to the available standard. It is in accordance with SNI 03-2105-2006 and the American National Standard Institute for general-use particle boards (ANSI A208.1-1999), where the parallel surface tensile strength is not a required measurement for general particle board.

**Differential scanning calorimetry.** The effect of the matrix/filler ratio and compression pressure on endothermic and exothermic temperatures are also investigated. The research parameters were also optimized by observing the effect of interactions among variables on endothermic and exothermic temperatures by using a RSM with CCD. Figure 5 is a graph that shows how endothermic temperature relates to the matrix/filler ratio and compression pressure. As the compression pressure increases, endothermic temperature also improves. The most influential variable is the compression pressure as is explained by the CCD equation. The optimum condition is obtained by using a matrix/filler ratio of 50:50 and compression ratio of 3.53 kg/cm<sup>2</sup> with endothermic temperature of 247.745 °C and endothermic heat of -4.12145 mW (Figure 6). At thermogram of all particle boards in the temperature range of 70–380 °C shows an endothermic peak that is probably caused by water evaporation process (Figure 7). Based on Figure 7, the DSC curve of particle board indicates the lowest endothermic peak wave in the range of 245.03–259.95 °C and this probably emerges because of the evaporation of water molecules.

The second peak around 360 °C is a new peak that indicates that bonding between OPEFB and casein has possibly occurred and causes an increase in melting point of the board. A DSC thermogram of board sample indicates that the melting temperature of board increases when bunch cellulose modification occurs. This change relates to cross linkage that occurs among cellulose granules. Based on the curve, it appears that OPEFB decomposition is complete at temperature of 380 °C. OPEFB constituents such as cellulose melts at around 260–280 °C. The higher temperature indicates that the OPEFB constituent is crystalline cellulose. At temperatures of 200–400 °C decomposition process of hemicellulose occurs, whereas at temperatures above 400 °C lignin decomposition and ash formation occurs (Lacerda *et al.*, 2009). Figure 7 indicates that the highest heat required to melt particle board is at a matrix/filler ratio of 55:45. Whereas the lowest heat needed to melt particle board is at the matrix/filler ratio of 15:85. This means that at the ratio of 55:45, matrix and filler mixing is good and results in stronger particle board so that higher heat is required to melt it. All peaks in Figure 7 indicate the endothermic temperature of particle board, which is in the range of 245.03–259.95 °C, with insignificant range of heat namely -10.564– -3.333 mW.

Based on Table 5, the highest exothermic temperature of particle board appears in run 9 with the matrix/filler ratio of 55:45 and compression pressure of 2.4 kg/cm<sup>2</sup>. Under such conditions, the temperature obtained is 428.35 °C. However, the lowest exothermic temperature occurs in run 3 at the matrix/filler ratio of 35:65 and compression pressure of 0.8 kg/cm<sup>2</sup>. By using CCD, the optimum exothermic temperature is obtained at a matrix/

filler ratio of 50:50 and compression pressure of 3.53 kg/cm<sup>2</sup> (Table 8) with exothermic temperature of 431.08 °C (Figure 8). At a lower matrix/filler ratio, the change of exothermic temperature is also relatively low.

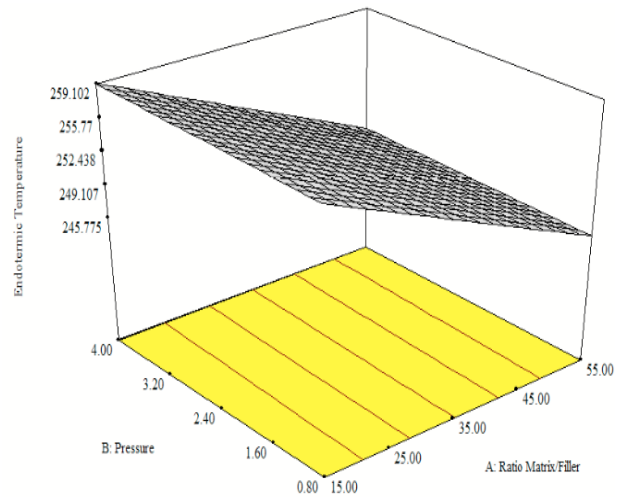


Figure 5. The Correlation of Matrix/filler Ratio and Endothermic Temperature at Various Pressures

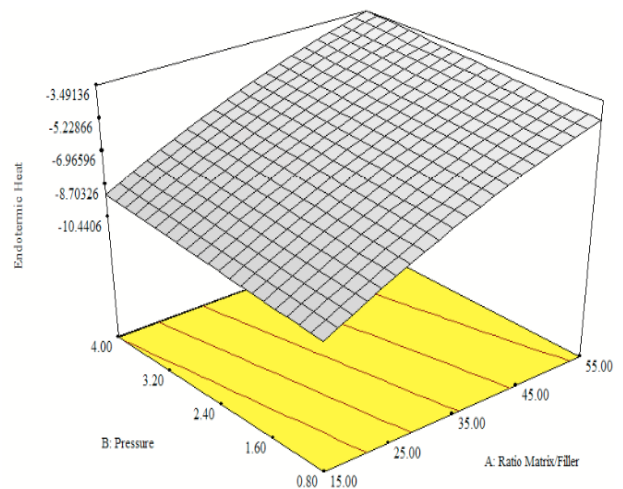


Figure 6. The Effect of Matrix/filler Ratio and Compression Pressure on Endothermic Heat

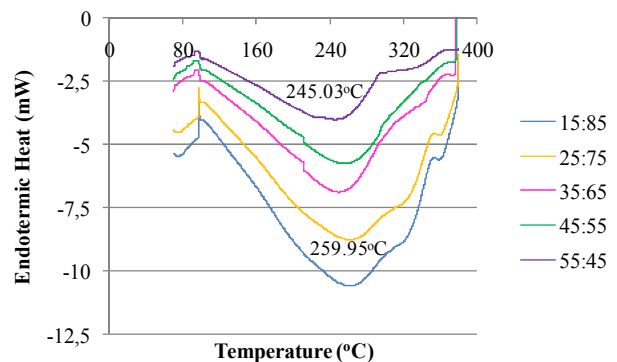
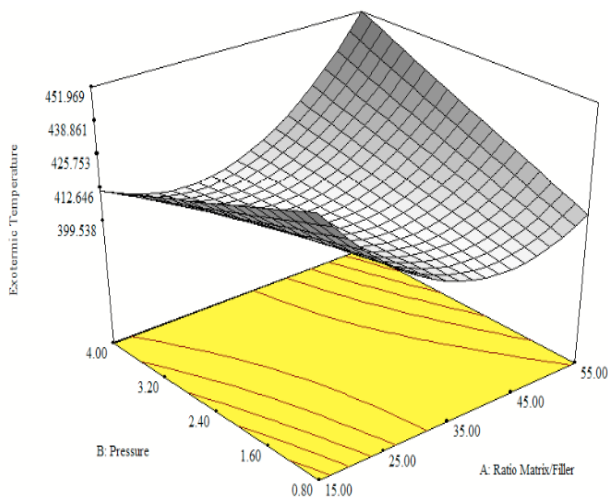


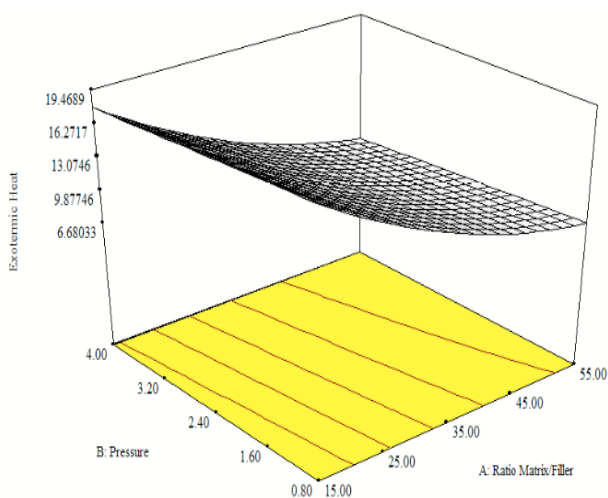
Figure 7. Correlation between Temperature and Endothermic Heat at Various Matrix/filler Ratio

At a higher ratio, there is an addition of exothermic temperature that could be influenced by compression pressure. The pressure could influence exothermic heat in the particle board to be lower than that at the lower matrix/filler ratio (Figure 9). In this research, the most influential independent variable does not exist because there are only two variables used and both are considered influential. It is the reason the research uses CCD, not Box-Behnken design that requires three variables.

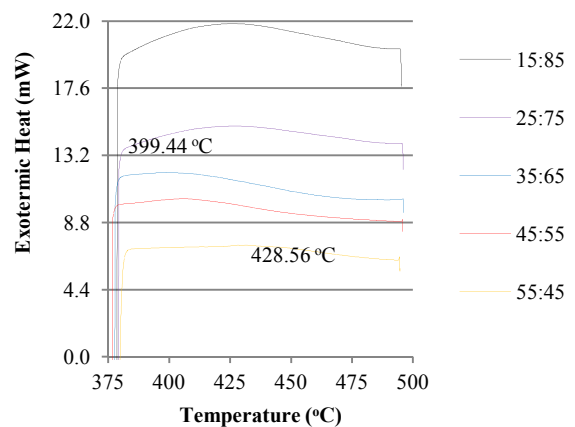
Figure 8 indicates that at compressive pressure of 0.8 kg/cm<sup>2</sup> the highest heat required for crystallization of particle board is at matrix/filler ratio of 15:85. Whereas the lowest heat needed for crystallization of particle board is at matrix/filler ratio of 55:45. The crystallization is not very different to that which occurs in the range of 5.036–21.832 mW and temperature range of 399.44–428.55 °C. The curve in Figure 8 indicates an exothermic



**Figure 8. The Effect of Matrix/filler Ratio and Compression Pressure of Exothermic Temperature**



**Figure 9. The Effect of Matrix/filler Ratio and Compression Ratio on Exothermic Heat**



**Figure 10. Correlation between Temperature and Exothermic Heat at Various Matrix/filler Ratio**

peak at the temperature where oxidized cellulose becomes levoglucosan, water, carbon monoxide, and carbon dioxide.

After mixing the board and glue together, DSC showed many changes through both exothermic and endothermic reactions. An exothermic reaction that released heat occurred because OPEFB was hydrophilic and had a cellulose structure of –OH groups that was easily degraded. Based on data, the melting peaks obtained at temperatures around 245, 359, and 360 °C was endothermic and resulted from the glue degradation. Furthermore, the board experienced an exothermic glass transition phase at a range of 380–428 °C with exothermic peak around 426 °C (ratio 15:85). This reaction ended with decomposition that occurred at temperatures of 470–495 °C. Based on these data, using casein glue would influence the thermal effects of OPEFB because the entry of casein glue could increase specimen solidity so that it is more difficult to decompose. The use of MAH as coupling agent probably could be also increase thermal stability of OPEFB, so that it helps casein in handling thermal effect provided on OPEFB.

### Conclusions

Based on research conducted, the optimum conditions for producing particle board from OPEFB are with a matrix/filler ratio of 50:50 and compressive pressure of 3.53 kg/cm<sup>2</sup>. All particle board ratios fulfill the mechanical and physical testing standards. All these particle board ratios fulfill the water content requirements of SNI 03-2105-2006, that it is less than 14%. This research result indicates that all particle board ratios fulfill SNI 03-2105-2006 for densities of 0.4–0.9 g/cm<sup>3</sup>. Tensile strength testing indicates results in the range of 0.39–2.58 kg/cm<sup>2</sup>. Therefore, based on this research, the optimal operating condition that results in maximum modulus of

rupture, density, and exothermic temperature is at ratio of 50:50 and compressive pressure of 3.53 kg/cm<sup>2</sup>. This conclusion was reached by observing aspects of ratio and minimum compressive pressure. The conditions give modulus of rupture of 325.32 kg/cm<sup>2</sup>, water content of 0.009%, density of 0.826 g/cm<sup>3</sup>, tensile strength of 2.573 kg/cm<sup>2</sup>, endothermic temperature of 247.74 °C, and endothermic heat of -4.122 mW. Such conditions also provide an exothermic temperature of 431.08 °C and exothermic heat of 7.526 mW.

Optimization needs further improvement by referring to modified casein glue. The CCD implemented in this research could be improved by using another optimization method such as Box-Behnken. OPEFB particle board could be used in decorative interiors and also for buliding exteriors if the thermal stability and modulus of rupture can be increased. Furthermore, research on impregnation with a resin is also necessary in the future to improve particle board quality from type 8 to be type 13 (particle board with medium flexural standard). In such a way, it could fulfill standards required in SNI 03-2105-2006.

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