

**CHAPTER VIII**  
**EFFECT OF METAL TYPE ON PARTIAL HYDROGENATION OF**  
**RAPESEED OIL-DERIVED FAMES**

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**8.1 Abstract**

Supported SiO<sub>2</sub> catalysts were studied for the partial hydrogenation of rapeseed oil-derived fatty acid methyl esters (FAMES) for improving its oxidative stability. The effect of metal type: Pt, Pd, and Ni, on catalytic activity and *cis-trans* selectivity was investigated. Hydrogenation activity was studied in terms of turn over frequency (TOF) of C18:3, C18:2, C18:1, and C18:0 FAMES. The highest TOF of C18:3, C18:2, and C18:1 was found for Pd catalyst. However, C18:0 TOF of Pt is higher than that of the Pd catalyst. The higher in C18:0 TOF can explain the low selectivity towards *trans*-monounsaturated FAMES of the Pt catalyst, which is due to the subsequent hydrogenation of the intermediate *trans*-monounsaturated to saturated FAMES. On the other hand, Ni showed the lowest TOFs when compared with the Pt and Pd catalysts.

**Keywords:** partial hydrogenation, supported SiO<sub>2</sub> catalyst, Pt, Pd, Ni, biodiesel, oxidative stability

## 8.2 Introduction

Oxidative stability of biodiesel fuel (BDF) depends on the degree of saturation of fatty acid in the starting vegetable oil. Biodiesel produced from vegetable oil, which contains large amounts of polyunsaturated fatty acids such as rapeseed and soybean oil, exhibits a low oxidative stability [1,2]. One approach to solve this problem is the hydrogenation of polyunsaturated fatty acid methyl esters (FAMES) in biodiesel. However, complete hydrogenation of polyunsaturated FAMES to saturated FAMES dramatically results the worse cold flow properties of biodiesel [1]. Therefore, partial hydrogenation of polyunsaturated FAMES to monounsaturated FAMES is a promising solution to improve oxidative stability, with minimal effect to cold flow properties, particularly when blending with petroleum diesel.

Several studies have been done on the hydrogenation of vegetable oil to increase its shelf life by improving its oxidative and thermal stability [3]. The catalysts used in the hydrogenation of vegetable oil are supported catalysts based on Ni, Cu, and noble metals. The different metals exhibit different catalytic activity as well as *cis-trans* isomerization selectivity [3,4]. Consequently, the type of metal will has considerable influence on the hydrogenation of vegetable oil derived-FAMES. However, the effect of metal type on hydrogenation of FAMES has not been widely studied. Thus, this work aimed to study the effect of metal type on partial hydrogenation of rapeseed BDF for the improvement of oxidative stability. In our previous work, Pd supported on SiO<sub>2</sub> support with an average pore diameter of 45.3 nm, shows good performance for partial hydrogenation of rapeseed oil derived-FAMES [5,6]. Therefore, SiO<sub>2</sub> was selected as a support in this study. The catalytic activity of different metal supported SiO<sub>2</sub> catalysts was investigated in terms of turn over frequency (TOF). In addition, the effect of metal type on *cis-trans* selectivity was also studied. Finally, the oxidative stability and cold flow properties of feed and hydrogenated products were investigated

## 8.3 Materials and Methods

### 8.3.1 Materials

A commercial silica (SiO<sub>2</sub>-Q30) with an average pore diameter of 45.3 nm and particle size of 75–500 μm, obtained from Fuji Silysia Chemical Co. Ltd., was used as a support. The Pt(NH<sub>3</sub>)<sub>4</sub>Cl<sub>2</sub>.xH<sub>2</sub>O and Pd(NH<sub>3</sub>)<sub>4</sub>Cl<sub>2</sub>.xH<sub>2</sub>O purchased from N.E. Chemcat Corp., JAPAN, were used as a Pt and Pd precursor, respectively. On the other hand, Ni(NO<sub>3</sub>)<sub>2</sub>.6H<sub>2</sub>O purchased from Wako Chemicals Co. Ltd., was used as a Ni precursor. In addition, tetrahydrofuran (stabilizer free, Wako Chemicals Co. Ltd.) was used as a solvent for gas chromatography (GC).

### 8.3.2 Catalyst Preparation

The Pt, Pd, and Ni on SiO<sub>2</sub> catalysts were prepared by the incipient wetness impregnation technique using Pt(NH<sub>3</sub>)<sub>4</sub>Cl<sub>2</sub>.xH<sub>2</sub>O, Pd(NH<sub>3</sub>)<sub>4</sub>Cl<sub>2</sub>.xH<sub>2</sub>O, and Ni(NO<sub>3</sub>)<sub>2</sub>.6H<sub>2</sub>O precursors, respectively. An exact amount of the metal precursor was dissolved in distilled water. Then, this solution was impregnated into the dried support and kept for 1 day. After that, the catalyst was dried, calcined at 300°C (Pt and Pd) or 450°C (Ni) for 3 h under the oxygen flow of 1 l/min, and reduced at 300°C (Pt and Pd) or 400°C (Ni) for 2 h under the hydrogen flow of 100 ml/min before use in the partial hydrogenation reaction. In order to compare the activity and selectivity of the catalysts, equimolar amount of Pt and Pd were prepared ( $9.49 \times 10^{-4}$  mole or 1.82 wt.% Pt and 1 wt.% Pd). On the other hand, 10 wt.% Ni on SiO<sub>2</sub> was prepared and the amount of Ni catalyst used was ten times higher than that of Pd due to its low catalytic activity in comparison to the Pd and Pt catalysts.

### 8.3.3 Catalyst Characterization

The specific surface area, pore volume, and pore size distribution of the support were determined by N<sub>2</sub> physisorption using a Quantachrome Autosorb-1 MP surface area analyzer. Before analyzing, the sample was heated in a vacuum atmosphere at 250°C overnight to eliminate volatile species that had adsorbed on the surface.

Metal dispersion of the prepared catalysts was determined by pulse chemisorption of 10.1% CO/He at 50°C, using Temperature-Programmed Desorption/Oxidation/Reduction (TPD/R/O) Ohkura R6015. The metal dispersion was calculated by assuming a stoichiometry of CO:metal = 1:1. Before analysis, the calcined sample was pretreated with hydrogen at the temperature same as reduction temperature for 1 h and followed by purging with helium at the same temperature for 10 min.

#### 8.3.4 Transesterification of Rapeseed Oil

Fatty acid methyl esters (FAMES) of rapeseed oil were prepared by transesterification reaction catalyzed by potassium hydroxide (KOH). The reaction took place in a 2 L three-necked round-bottomed flask, equipped with a stirrer and a condenser. The amount of catalyst used was 1 wt.% compared to the starting rapeseed oil with 9:1 methanol to oil molar ratio. KOH and methanol were added into rapeseed oil and stirred at 60°C for 2 h and cooled down. After that, the lower glycerine phase was removed. The upper phase was washed with 60°C distilled water several times to remove remaining KOH, methanol, and possible soap. Finally, it was dried by a rotary evaporator at 60°C, to remove remaining washed water. The FAME composition of the rapeseed BDF was determined by GC.

#### 8.3.5 Partial Hydrogenation of Polyunsaturated FAMES

The partial hydrogenation reaction was carried out in a 388 ml stainless steel semi-batch reactor at a temperature and hydrogen partial pressure of 80°C and 0.3 MPa, respectively. The stirring rate was maintained at 1000 rpm and the flow rate of hydrogen gas was 200 ml/min. First, the supported SiO<sub>2</sub> catalyst was added into 180 ml of rapeseed biodiesel under argon atmosphere to prevent oxidizing of the reduced catalyst and placed into the reactor. Second, the reactor was connected to the reaction line and the system was purged with hydrogen three times to remove any remaining air. Third, the reaction was started by increasing the temperature and pressure to the desired point. Finally, the liquid products were collected every 30 minutes. The total reaction time was 3 h.

### 8.3.6 Biodiesel Analysis

FAME composition in the biodiesel before and after the partial hydrogenation reaction was determined by using a Hewlett Packard gas chromatograph 6890N equipped with a flame ionization detector (GC-FID). An HP-88 (100 m x 250  $\mu\text{m}$  x 0.2  $\mu\text{m}$ ) capillary column was used. Samples of 1  $\mu\text{l}$  were auto-injected under the following conditions: the carrier gas was helium with a flow rate of 2.4 ml/min, an injector temperature was 200°C with a split ratio of 75:1, and a detector temperature was 230°C. The sample was injected at an oven temperature of 155°C. After an isothermal period of 20 min, the GC oven was heated to 230°C at a rate of 2°C/min and held for 2.5 min with a total running time of 60 min. The FAME composition was identified by reference to the retention time.

To identify the oxidative stability of feed and hydrogenated BDF, the Rancimat test according to EN 14112 was performed in a Metrohm 743 Rancimat. The sample was aged at 110°C under a constant air stream with flow rate of 10 L/h. The oxidative stability is defined based on the period of time before FAMEs are degraded to such an extent that the formation of volatile acids can be recorded through an increase in conductivity.

Cold flow properties (cloud point and pour point) of biodiesel before and after partial hydrogenation were measured by a Tanaka mini pour/cloud point tester Series MCP-102, which was developed according to ASTM D6749.

## 8.4 Results and Discussion

### 8.4.1 Support and Catalyst Characterization

The surface area and average pore diameter of  $\text{SiO}_2$  support determined by BET-surface area analyzer is 112.1  $\text{m}^2/\text{g}$  and 45.3 nm, respectively, which can be classified as a mesoporous material. Metal dispersion of different metal supported on  $\text{SiO}_2$  catalysts, determined by CO-chemisorption technique, is displayed in Table 8.1. The metal dispersion is an important characteristic that tells how well metals disperse on the surface of the catalyst, and consequently tells how much is the catalytic activity. It showed that different catalysts with different metal

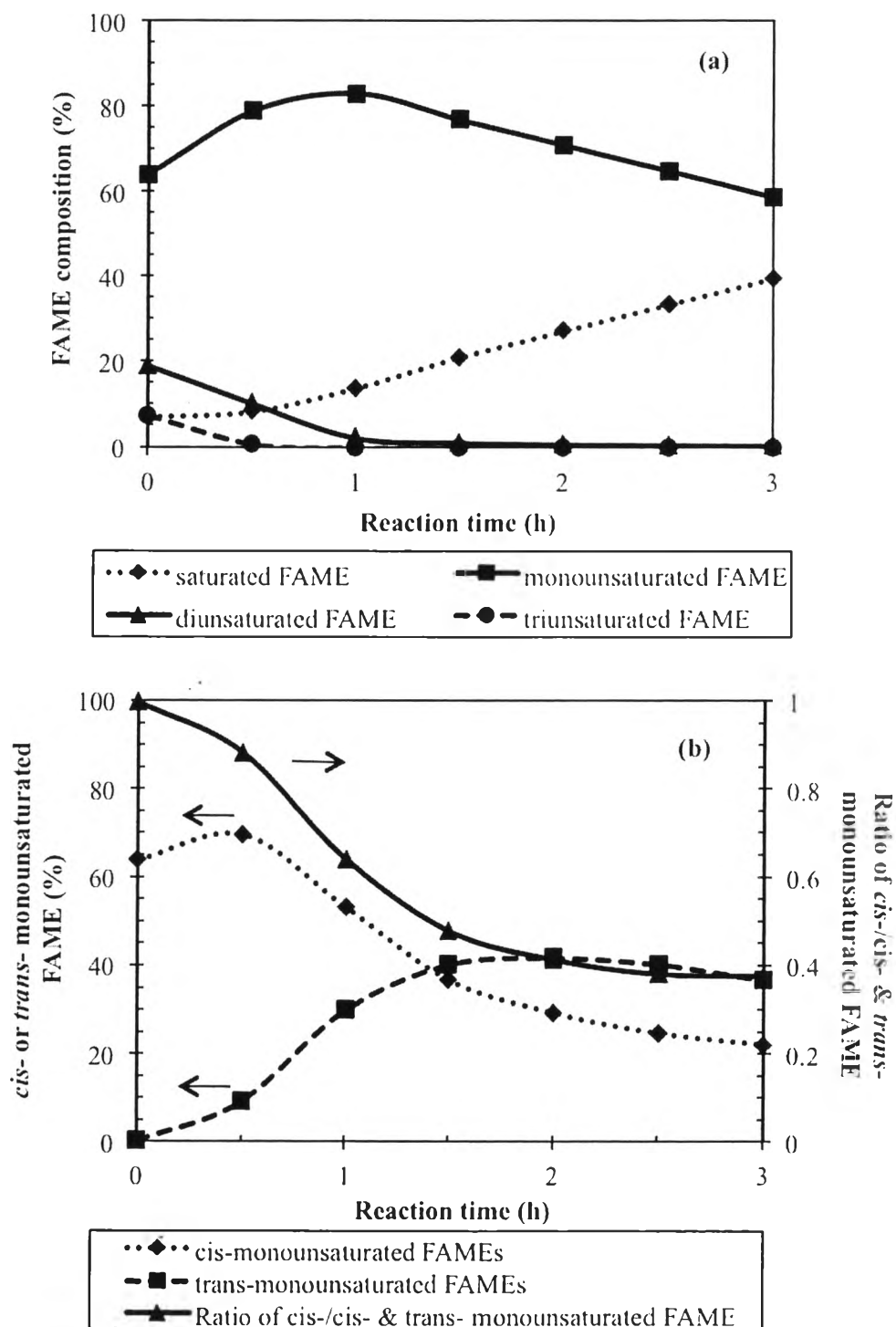
loadings exhibit different metal dispersions. However, in this study, the activity of each catalyst was determined and compared based on the turnover frequency (TOF), not directly from metal dispersion.

**Table 8.1** Amount of metal loading and metal dispersion of Pt, Pd, and Ni supported SiO<sub>2</sub> catalysts.

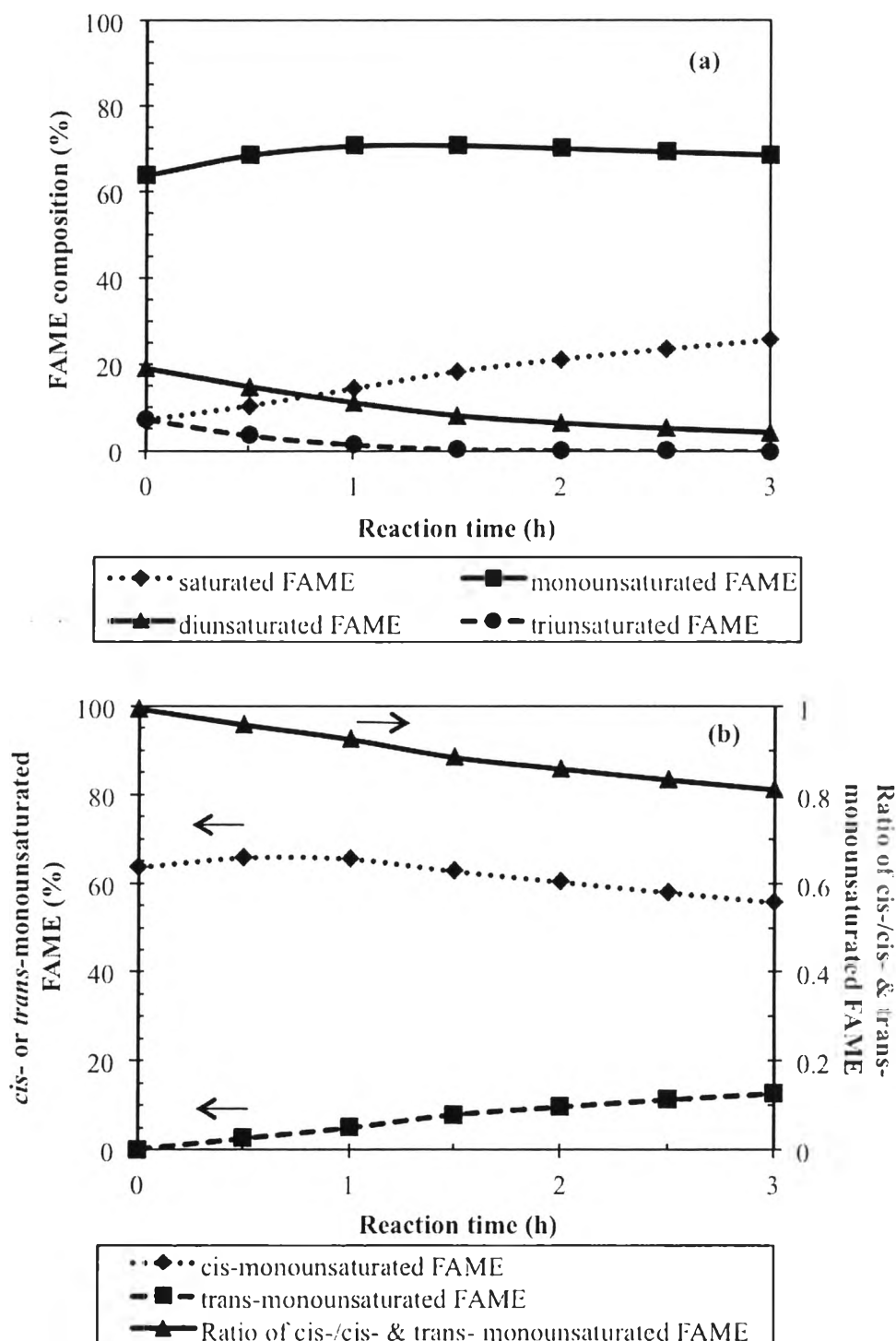
Catalyst	Metal loading (wt.%)	Metal dispersion (%)
Pt	1.82	8.77
Pd	1.00	6.55
Ni	10.00	6.16

#### 8.4.2 Partial Hydrogenation of Polyunsaturated FAMES

Figure 8.1(a) and (b) present the composition of FAME as a function of reaction time using Pd/SiO<sub>2</sub> catalyst under the reaction condition of 80°C, 0.3 MPa, 200 ml/min hydrogen flow rate, and 1000 rpm stirring rate. As shown in Figure 8.1(a), gradually hydrogenation of triunsaturated and diunsaturated FAMES with a gradual increase of monounsaturated and saturated FAMES were observed, at the beginning of reaction. After that, at 0.6 h of reaction; triunsaturated FAMES are fully hydrogenated, followed with a gradually decrease of monounsaturated and a linearly increase of saturated FAMES. This suggests that hydrogenation at this condition using Pd/SiO<sub>2</sub> catalyst meets the purpose in the partial hydrogenation point of view, which shows the behavior of consecutive reactions from tri-, di-, and monounsaturated FAMES to saturated FAMES. This reaction condition was used to study with other metal supported SiO<sub>2</sub> catalysts further. The FAME profile during hydrogenation time using Pt/SiO<sub>2</sub> and Ni/SiO<sub>2</sub> catalysts are presented in Figure 8.2 and Figure 8.3, respectively. It evidently shows that Pt and Ni catalysts exhibited lower hydrogenation activity than the Pd catalyst, where a slowly decrease of polyunsaturated FAMES could be observed in both catalysts.

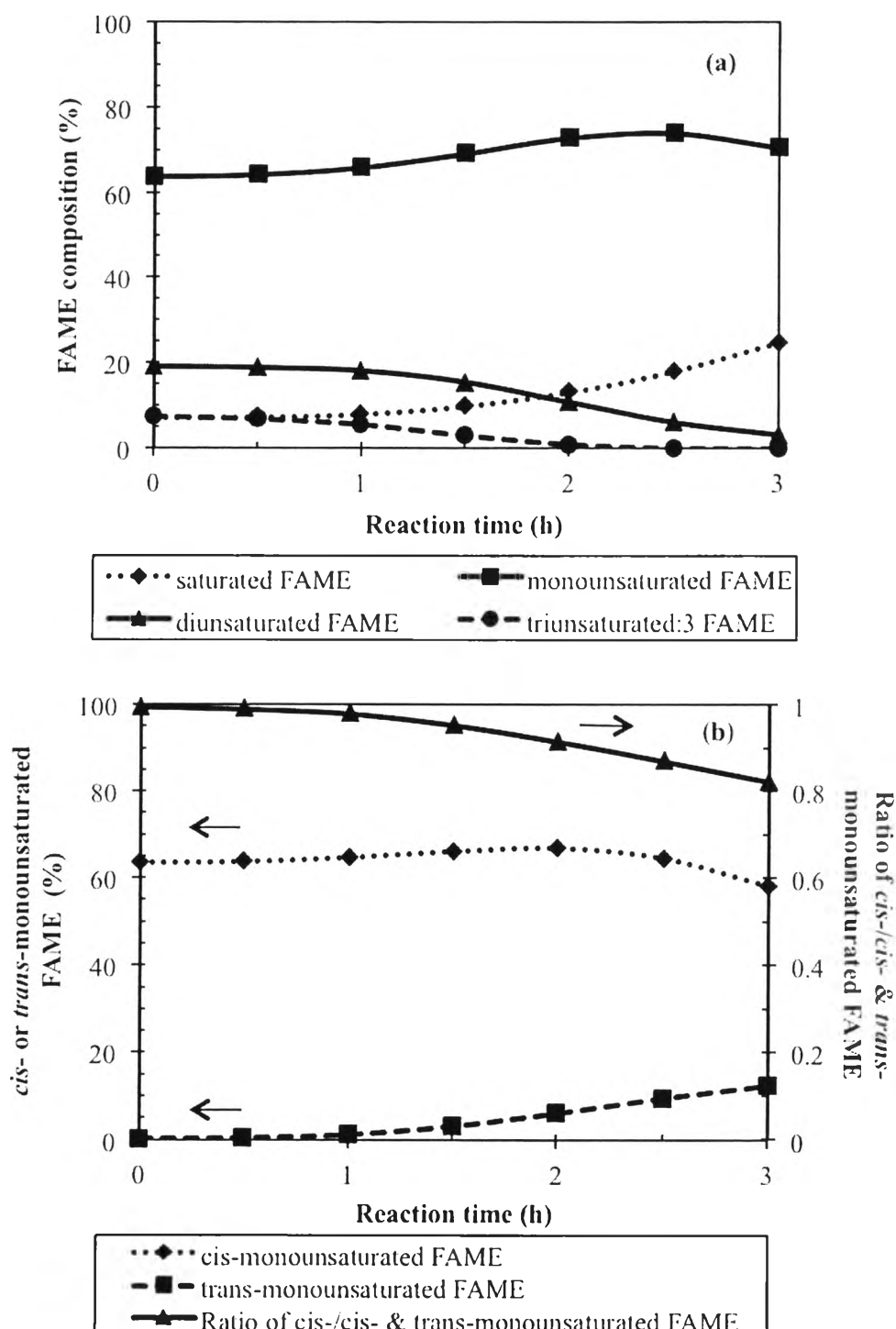


**Figure 8.1** Overall FAME composition, (a) and *cis*- or *trans*-monounsaturated FAME composition, (b) as a function of reaction time using Pd/SiO<sub>2</sub> (Reaction conditions: 80°C, 0.3 MPa, 200 ml/min hydrogen flow rate, and 1000 rpm stirring rate).



**Figure 8.2** Overall FAME composition, (a) and *cis*- or *trans*-monounsaturated FAME composition, (b) as a function of reaction time using Pt/SiO<sub>2</sub> (Reaction conditions: 80°C, 0.3 MPa, 200 ml/min hydrogen flow rate, and 1000 rpm stirring rate).





**Figure 8.3** Overall FAME composition, (a) and *cis*- or *trans*-monounsaturated FAME composition, (b) as a function of reaction time using Ni/SiO<sub>2</sub> (Reaction conditions: 80°C, 0.3 MPa, 200 ml/min hydrogen flow rate, and 1000 rpm stirring rate).

Table 8.2 presents the FAMES composition and some fuel properties of rapeseed BDF and hydrogenated BDF after 1 h of reaction using Pt, Pd, and Ni supported SiO<sub>2</sub> catalysts. After hydrogenation, a decrease in polyunsaturated FAMES (C18:3 and C18:2) along with an increase of monounsaturated (C18:1) and saturated FAMES (C18:0) was detected for all catalysts. Here, C18:2 and C18:3 were considered as a reactant and the conversion was calculated by combining the composition of C18:2 and C18:3. As can be seen in Table 8.2, for the Pt catalyst after 1 h of reaction; the C18:2 and C18:3 conversion was 52.3%, it provided BDF product with 9.09% of saturated C18:0 and 69.20% of monounsaturated C18:1. For Pd catalyst, after 1 h of reaction; it exhibited higher C18:2 and C18:3 conversion (91.6%) when compared with the Pt catalyst. However, the Pd catalyst provided BDF product with smaller amount of saturated C18:0 (8.51%) and higher amount of monounsaturated C18:1 (81.35%). These data show that Pd catalyst is superior in maximizing the amount of monounsaturated C18:1 and minimizing the deep hydrogenation into saturated C18:0. On the other hand, Ni catalyst shows very poor activity, which provided only 12.7% of C18:3 and C18:2 conversion.

Moreover, it apparently shows that partial hydrogenation can improve the oxidative stability of biodiesel even without the addition of any antioxidants. However, the hydrogenated biodiesel exhibited a poor pour point and cloud point, which exposes a drawback of hydrogenation of biodiesel. This problem can be solved by the optimization of hydrogenation degree to compromise between the oxidative stability and cold flow properties. In addition to the saturation of unsaturated FAMES during the hydrogenation reaction, transformation of some of the natural *cis*-monounsaturated FAMES to *trans*-configurations also takes place [3,4,7] as can be seen in Figure 8.1(b), 8.2(b), and 8.3(b). This is another disadvantage commonly observed during the hydrogenation of vegetable oil or FAMES due to the lower cold flow properties of the *trans*-isomers when compared with *cis*-isomers [8].

**Table 8.2** FAMES composition and some fuel properties of rapeseed BDF and hydrogenated BDF after 1 h of reaction using Pt, Pd, and Ni supported SiO<sub>2</sub> catalysts.

	Rapeseed BDF feed	Hydrogenated BDF after 1 h		
		Pt	Pd	Ni
<b>FAMES composition (%)</b>				
Saturated FAME	7.14	14.59	13.89	8.20
C12:0	0.01	0.03	0.01	0.02
C14:0	0.04	0.04	0.05	0.04
C16:0	4.05	4.09	4.06	4.08
C17:0	0.05	0.05	0.04	0.04
C18:0	1.82	9.09	8.51	2.78
C20:0	0.62	0.75	0.71	0.68
C22:0	0.34	0.36	0.35	0.35
C24:0	0.15	0.17	0.17	0.16
Monounsaturated FAME	64.11	70.86	83.02	66.14
<i>trans</i> -Monounsaturated FAME	0.13	5.16	29.93	1.26
t-C16:1	0.03	0.04	0.16	0.04
t-C18:1	0.10	5.11	29.30	1.23
t-C20:1	0.00	0.00	0.42	0.00
t-C22:1	0.00	0.00	0.00	0.00
t-C24:1	0.00	0.01	0.04	0.00
<i>cis</i> -Monounsaturated FAME	63.97	65.70	53.09	64.88
c-C16:1	0.21	0.20	0.10	0.22
c-C18:1	62.32	64.09	52.05	63.15
c-C20:1	1.26	1.24	0.83	1.31
c-C22:1	0.03	0.03	0.02	0.03
c-C24:1	0.16	0.15	0.10	0.16
Diunsaturated FAME	19.55	11.32	2.30	18.22
C18:2	19.55	11.32	2.30	18.22
Triunsaturated FAME	7.77	1.70	0.00	5.64
C18:3	7.77	1.70	0.00	5.64
<b>Fuel properties</b>				
oxidative stability (h)	1.89	4.71	38.98	3.50
pour point (°C)	-11	4	5	-10
cloud point (°C)	-3	6	11	-1

#### 8.4.2 Effect of Metal Type on Hydrogenation Activity

The hydrogenation activity of different metal supported on SiO<sub>2</sub> catalysts was evaluated in terms of turnover frequency (TOF) of each C18 FAMES: C18:3, C18:2, C18:1, and C18:0 at a hydrogenation time of 0.5 h. The TOF is the amount of reactant reacted per surface area of active site per time and can be calculated as follows:

$$\text{TOF} = \frac{\text{mole of FAMES converted (C18:3 and C18:2) or formed (C18:1 and C18:0) per time}}{\text{mole of Pd metal exposed on the surface}} \quad (8.1)$$

As shown in Table 8.3, the order of hydrogenation activity based on TOF compared on the same mole basis is: Pd > Pt >> Ni. This result is consistent with many works done on the hydrogenation of vegetable oil [9–14]. They reported that Pd is the most active catalyst, while Pt also exhibits good activity, but slightly lower than Pd. On the other hand, Ni shows the lowest activity. The TOF of C18:3, C18:2, and C18:1 for the Pd catalyst is around 1–2 times higher than those of the Pt catalyst. However, the C18:0 TOF of Pt is almost 4 times higher than that of Pd. This implies that the Pt catalyst exhibits deeper hydrogenation activity than the Pd catalyst, while Ni shows the lowest hydrogenation activity, which exhibits TOFs of 1000 times less than those of the Pt and Pd catalysts. These results are in agreement with the composition of FAMES and fuel properties of BDF products after 1 h of hydrogenation time using Pt, Pd, and Ni catalysts, presented in Table 8.2. Hydrogenated rapeseed BDF using the Pd catalyst presents the lowest amount of polyunsaturated FAMES, whereas the Ni catalyst shows the highest amount of polyunsaturated FAMES. As expected, the oxidative stability of partial hydrogenated BDF was improved from 1.89 h to 4.71 h using Pt, 38.98 h using Pd, and 3.50 h using Ni catalyst.

**Table 8.3** C18:3, C18:2, C18:1, and C18:0 TOFs of Pt, Pd, and Ni supported SiO<sub>2</sub> catalysts at 0.5 h of hydrogenation time

Catalyst	TOFs ( $\times 10^3 \text{ h}^{-1}$ )			
	C18:3	C18:2	C18:1	C18:0
Pt	32.58	36.39	41.01	27.71
Pd	38.79	50.76	83.79	7.14
Ni	0.041	0.018	0.041	0.012

#### 8.4.3 Effect of Metal Type on Selectivity

As above described, the isomerization of original *cis*-monounsaturated FAMES to *trans*-monounsaturated FAMES commonly takes place during hydrogenation. The *cis*-monounsaturated FAME is preferable in terms of cold flow properties, since its melting point is lower than that of *trans*-isomers, for example;  $-20.2^\circ\text{C}$  for *cis*-C18:1 FAME, but  $9.9^\circ\text{C}$  for *trans*-C18:1 FAME [8]. Hence, it is noteworthy to prepare a catalyst that gives low selectivity towards *trans*-isomers. Table 8.4 shows that the selectivity towards *trans*-monounsaturated FAMES is in following order: Pd  $\gg$  Ni  $>$  Pt. This order of *trans*-selectivity displays the same trend as other studies on hydrogenation of vegetable oil [9–14]. However, it apparently showed that the low selectivity towards *trans*-monounsaturated FAMES of the Pt catalysts was accompanied with a high formation of saturated FAMES, which is consistent with its highest C18:0 TOF. This implies an interesting result that the low *trans*-selectivity of both Pt and Ni catalysts is due to the subsequent hydrogenation of the intermediate *trans*-monounsaturated FAMES to saturated FAMES.

**Table 8.4** Comparison of *cis*-monounsaturated, *trans*-monounsaturated, and saturated FAMES composition at the same conversion of C18:2 and C18:3 (55% conversion)

Catalyst	<i>cis</i> -monounsaturated FAME (%)	<i>trans</i> -monounsaturated FAME (%)	Saturated FAMES (%)
Pt	65.03	5.83	15.54
Pd	69.27	8.85	8.36
Ni	66.98	6.06	13.30

According to the main purpose of partial hydrogenation, which is to minimize polyunsaturated FAMES and maximize monounsaturated FAMES, in order to optimize the oxidative stability and cold flow properties. Therefore, Pd is the best catalyst in terms of partial hydrogenation, which results in a lower saturated FAMES composition when compared with Pt and Ni catalysts at the same conversion. In addition, Pd shows the highest hydrogenation activity, which provides an advantage in terms of mild operating conditions. Moreover, it could be suggested that changing the reaction conditions for hydrogenation using a Pd catalyst is an alternative way to reduce the *trans*-isomers formation in the hydrogenation of vegetable oil derived-FAMES.

## 8.5 Conclusions

Partial hydrogenation of polyunsaturated FAMES of rapeseed biodiesel has been investigated on different types of metal supported on SiO<sub>2</sub> catalysts. It was found that the type of metal used had a significant effect on the catalytic activity and *cis-trans* selectivity for the partial hydrogenation of polyunsaturated FAMES. The catalytic activity in terms of C18:3, C18:2, and C18:1 TOFs was in this order: Pd > Pt >> Ni. On the other hand, selectivity towards *trans*-monounsaturated FAME was in the following order: Pd >> Ni > Pt. It was revealed that the low *trans*-selectivity of both Pt and Ni catalysts is due to the subsequent hydrogenation of the intermediate

*trans*-monounsaturated to saturated FAMES. Therefore, Pd is the best catalyst in terms of partial hydrogenation, which generates biodiesel product with a good oxidative stability and cold flow properties.

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