

## CHAPTER IV

### RESULTS AND DISCUSSION

As described in chapter II, this research was intended to improve the quality of used oil by means of two main methods. First, the used oil was treated with the physical and chemical treatment method, and the second by the catalytic hydrogenation method. Because of the physical and chemical treatment method gave high waste byproduct and disposal problem. Then this research was studied closely to solve or to overcome this undesirable problem by treating the used oil directly with the catalytic hydrogenation method. This step had an objective to compare the catalytic activity of those three catalysts (Raney nickel,  $\text{NiO}/\text{MoO}_3/\text{Al}_2\text{O}_3$ , and  $\text{NiO}/\text{WO}_3/\text{Al}_2\text{O}_3$ ). Among these catalysts, Raney nickel catalyst had shown its proper characteristic that not only in good activity in hydrogenation but also gave reasonable catalyst cost in production. Besides, the other objective of this research is to study the optimum condition in hydrogenation of used lubricating oil using Raney nickel catalyst.

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Physical and Chemical Treatment Method

The physical and chemical properties of used oil and the treated oil that obtained from this method were shown in the following Table:

Table 4.1 Physical and chemical properties of acid-clay treated oil compared with an original used oil

Physical and Chemical Properties	Used Oil	Acid/Clay Treated Oil
API Gravity @ 60 °F	28.6	30.8
Color, ASTM	1.65	1.20
Pour Point, °C	-12	-8
Viscosity, Kin @ 40 °C, cSt	54.74	43.52
Viscosity, Kin @ 100 °C, cSt	7.05	6.85
Viscosity Index	81	113
Sulfur Content, % wt	0.87	0.30
Oxidation Point, °C	366.5	365.3
Oxidative Compound, % wt	18.7	15.9
Carbon Content, %	76.67	72.91
Cp	21.61	26.42
Cn	1.72	0.67
Ca	-	60-70
Yield, %	-	-

In comparison between used oil and treated oil, all properties of acid-clay treated oil were improved. API gravity of treated oil is higher than untreated oil, that means the oil treated with acid-clay process had more saturated portion, or had more paraffinic hydrocarbon contents.

Besides, the other properties such as color, viscosity index, sulfur content, etc., were improved by this treatment. From this process, some contaminants, unsaturated, unstable compounds were removed to make oil to be clean and more stable.

From the Table 4.1, some aromatic carbons were reduced, the oil had better in most of the properties especially in color, VI, sulfur removal. This means that some oxidative and some undesirable compounds were removed by this process and gave oil yield for about 60-70%.

#### **Effect of Catalyst Types on Hydrogenation of Acid-Clay Treated Oil**

This section studied the catalytic hydrogenation by using three catalysts under the same condition. These catalysts were Raney nickel, nickel oxide/molybdenum oxide/alumina, and nickel oxide/tungsten oxide/alumina. The results obtained from this method were in the following Table.

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Table 4.2 Physical and chemical properties of oil obtained from hydrogenation of acid-clay treated oil

Physical and Chemical Properties	R-Ni	Ni/Mo	Ni/W
API Gravity @ 60 °F	32.0	32.0	32.7
Color, ASTM	L 0.5	L 1.0	L 0.5
Pour Point, °C	-7.5	-6.5	-7.5
Viscosity, Kin @ 40 °C, cSt	37.99	36.88	36.88
Viscosity, Kin @ 100 °C, cSt	6.30	6.13	6.13
Viscosity Index	115	118	122
Sulfur Content, % wt	0.06	0.06	0.02
Oxidation Point, °C	348.0	344.7	340.0
Oxidative Compound, % wt	21.8	21.6	20.9
Carbon Content, %			
Cp	77.38	76.99	77.13
Cn	21.94	22.32	21.98
Ca	0.68	0.69	0.90
Yield, %	93	90	92

Among these three catalysts used in hydrogenation of acid-clay treated oil, hydrotreating catalyst such as NiO/MoO<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> and NiO/WO<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub>, gave oils which had more desirable properties, especially in terms of viscosity index, color, paraffinic carbon content and naphthenic carbon content, and sulfur removal. It could be considered that in some properties of treated oil such as VI values, by using hydrotreating catalysts, the data show better VI, when used NiO/MoO<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> and NiO/WO<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub>, the VI values were 118 and 122, respectively, whereas Raney nickel catalyst gave VI value of 115. In sulfur removal, NiO/WO<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> was the best catalyst of all.

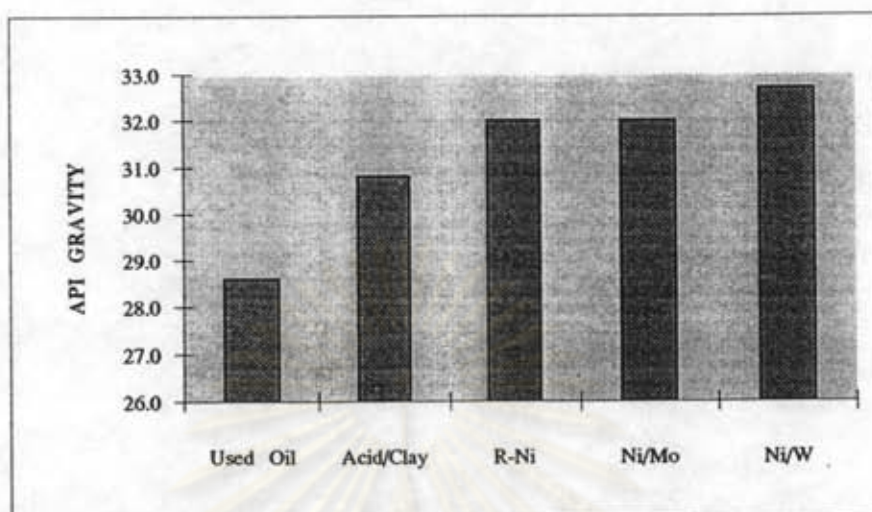


Figure 4.1 Effect of catalyst types on API gravity in hydrogenation of acid-clay treated oil compared with acid-clay treated oil and original used oil

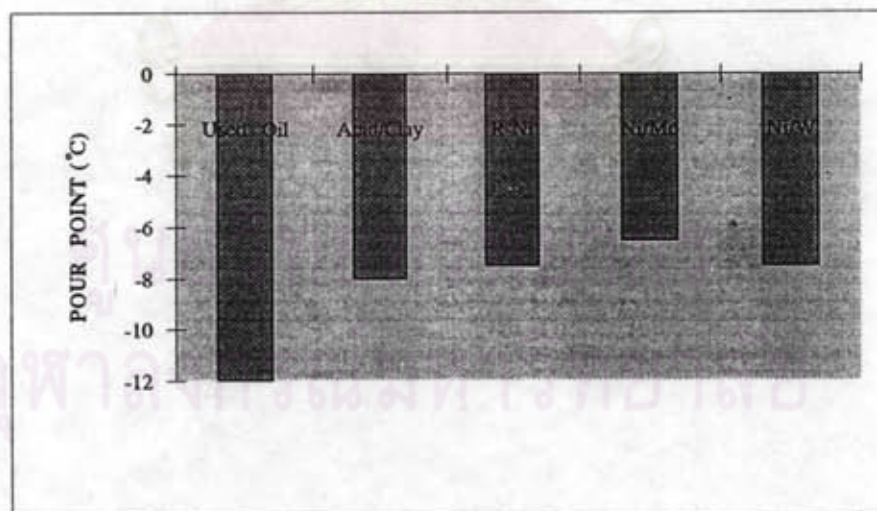


Figure 4.2 Effect of catalyst types on pour point in hydrogenation of acid-clay treated oil compared with acid-clay treated oil and original used oil

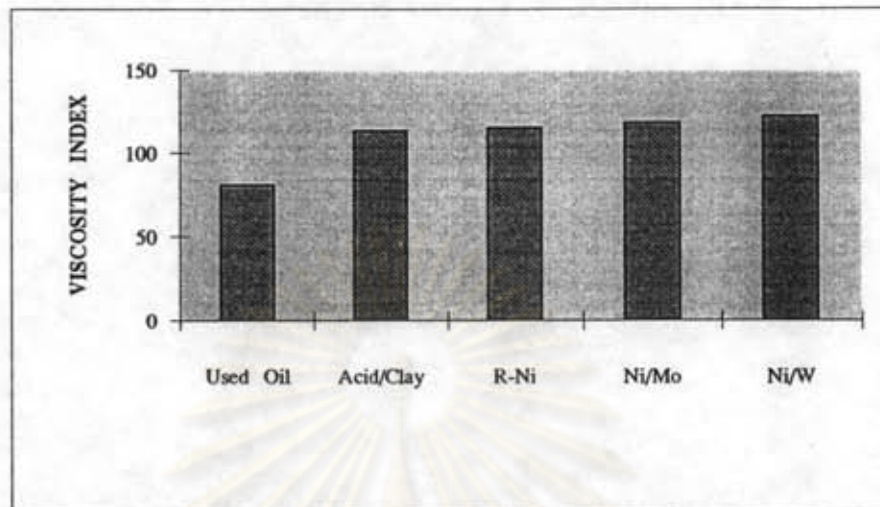


Figure 4.3 Effect of catalyst types on viscosity index in hydrogenation of acid-clay treated oil compared with acid-clay treated oil and original used oil

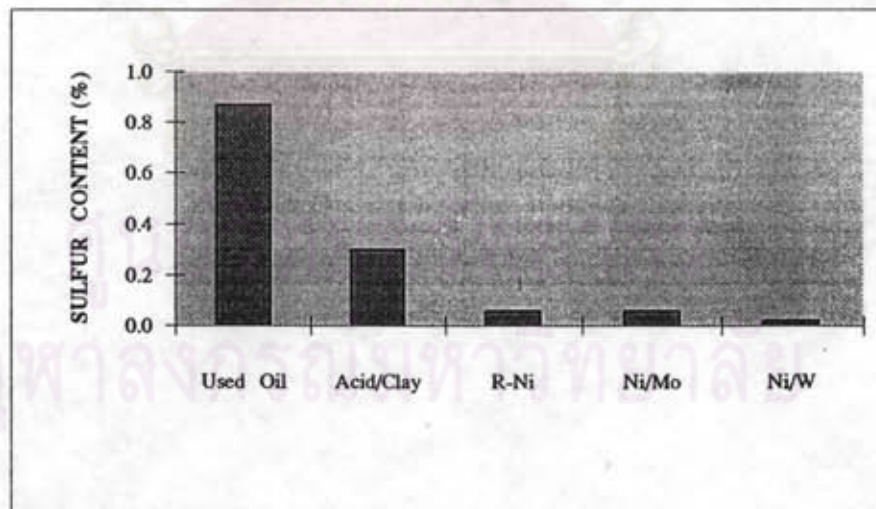


Figure 4.4 Effect of catalyst types on viscosity index in hydrogenation of acid-clay treated oil compared with acid-clay treated oil and original used oil

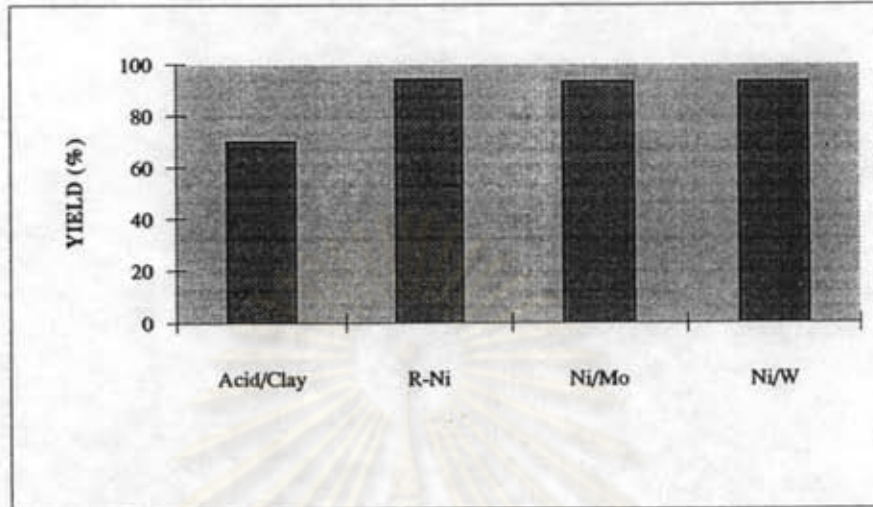


Figure 4.5 Effect of different treatments on oil yield

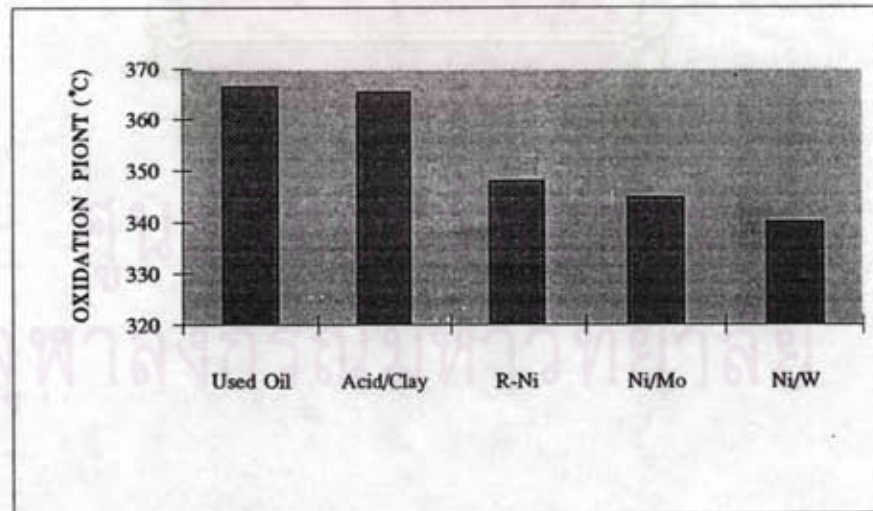


Figure 4.6 Effect of catalyst types on oxidation point in hydrogenation of acid-clay treated oil compared with acid-clay treated oil and original used oil

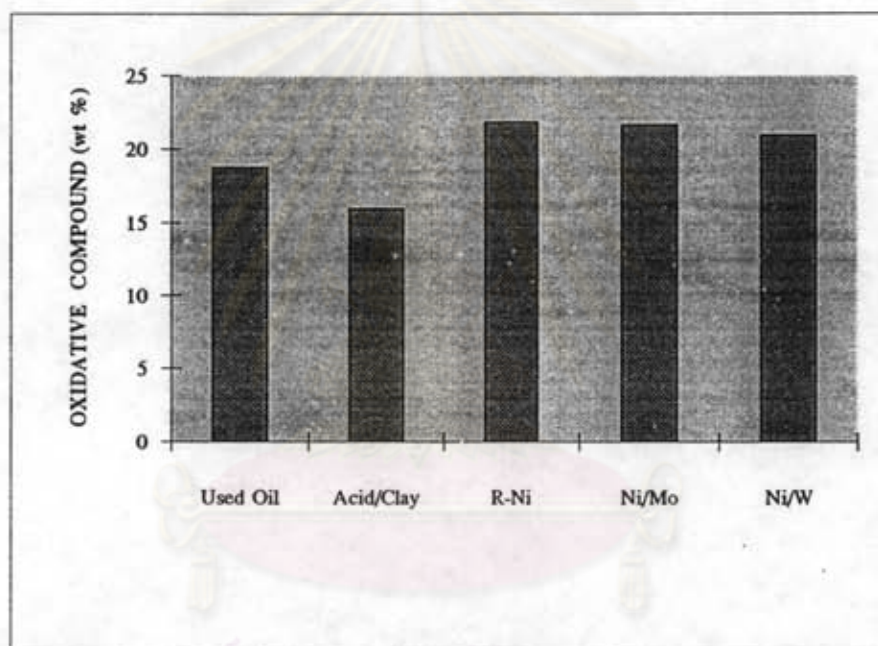


Figure 4.7 Effect of catalyst types on oxidative compound in hydrogenation of acid-clay treated oil compared with acid-clay treated oil and original used oil



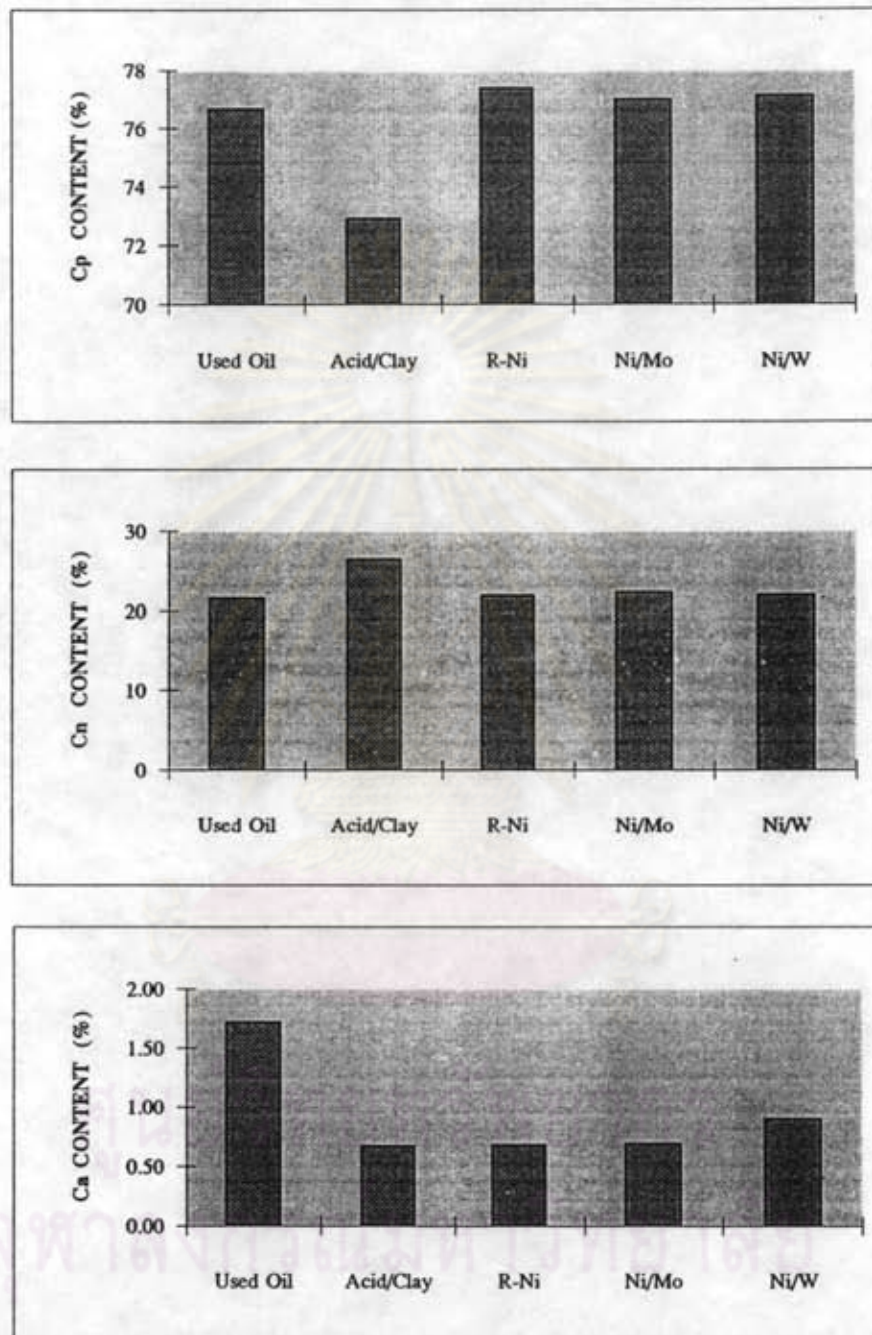


Figure 4.8 Effect of catalyst types on various types of carbon content in hydrogenation of acid-clay treated oil compared with acid-clay treated oil and original used oil

Because of the previous acid-clay treatment, the oil used in this catalytic hydrogenation had somewhat been cleaned which was good to react with hydrogen in the presence of catalysts. However, from the Table above, these three catalysts still gave the appreciated oil properties that were not significant different.

### **Effect of Catalyst Types on Hydrogenation of Used Lubricating Oil**

In this section, used oil was treated with the same three type catalysts as the previous section. The condition for hydrogenation was still same. The objective of this experiment is to study the catalytic activity among these catalysts in hydrogenation of used lubricating oil.

The oil obtained from this experiment were shown in the following Table.



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Table 4.3 Physical and chemical properties of oils obtained from hydrogenation of used oil by using various catalysts compared with original used oil

Physical and Chemical Properties	Used Oil	R-Ni	Ni/Mo	Ni/W
API Gravity @ 60 °F	28.6	29.7	30.2	30.2
Color, ASTM	L 6.5	L 2.0	L 1.5	L 1.5
Pour Point, °C	-12.0	-7.5	-7.5	-6.5
Viscosity, Kin @ 40 °C, cSt	54.74	41.46	38.46	38.75
Viscosity, Kin @ 100 °C, cSt	7.05	6.47	6.27	6.19
Viscosity Index	81	106	111	106
Sulfur Content, % wt	0.87	0.50	0.24	0.23
Oxidation Point, °C	366.5	354.5	346.5	369.7
Oxidative Compound, % wt	18.7	23.9	22.0	15.6
Carbon Content, %				
Cp	76.67	73.93	79.59	79.08
Cn	21.61	24.58	19.46	19.82
Ca	1.72	1.49	0.95	1.10
Yield, %	-	94	92	92

#### Effect of Concentrations of Raney Nickel Catalyst on Hydrogenation of Used Lubricating oil

This experiment had the objective to compare the resulting oil properties when using Raney nickel catalyst in the different concentrations (4%, 6%, and 8% by weight of used oil), while the other condition parameters were identical. The data obtained from this was shown in the following Table:

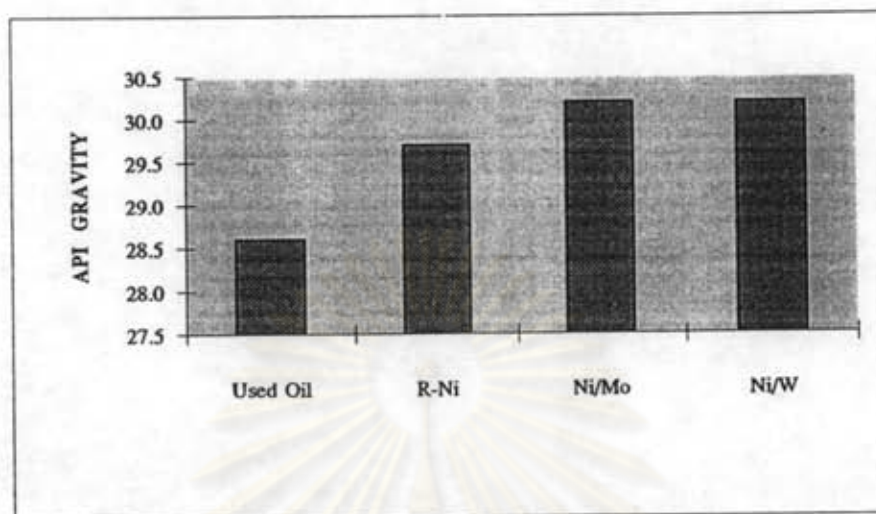


Figure 4.9 Effect of catalyst types on API gravity in hydrogenation of used oil compared with an original used oil

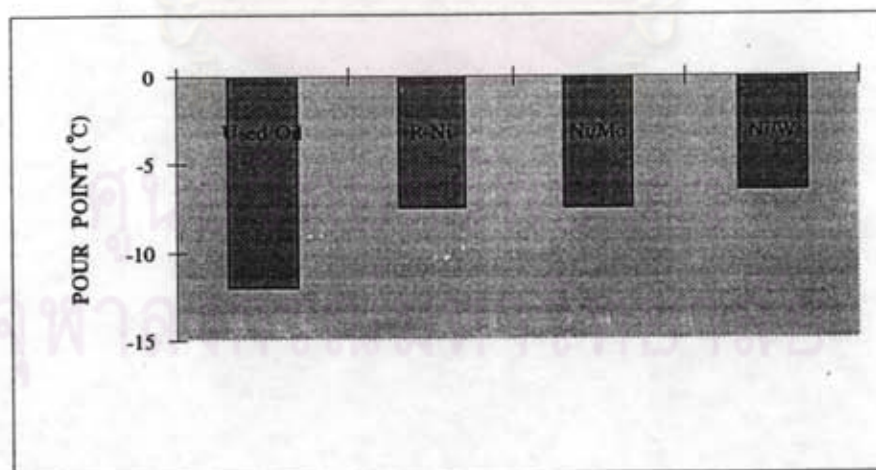


Figure 4.10 Effect of catalyst types on pour point in hydrogenation of used oil compared with an original used oil

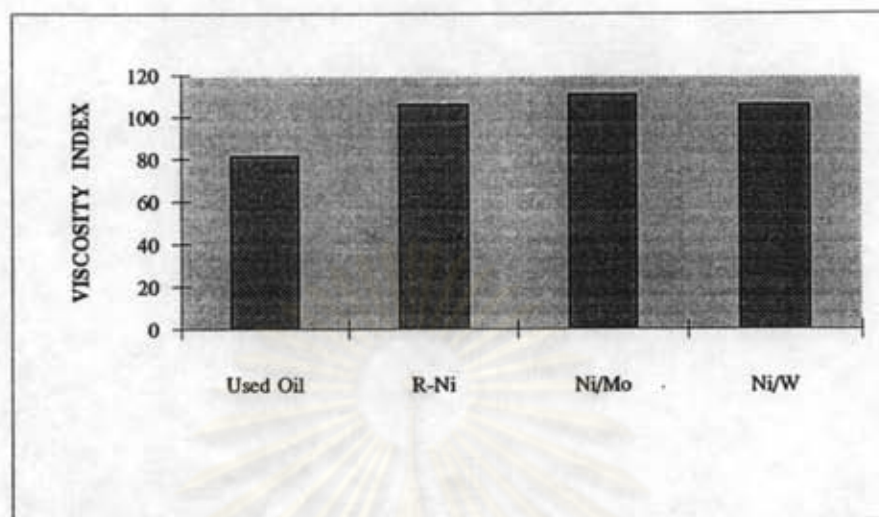


Figure 4.11 Effect of catalyst types on viscosity index in hydrogenation of used oil compared with an original used oil

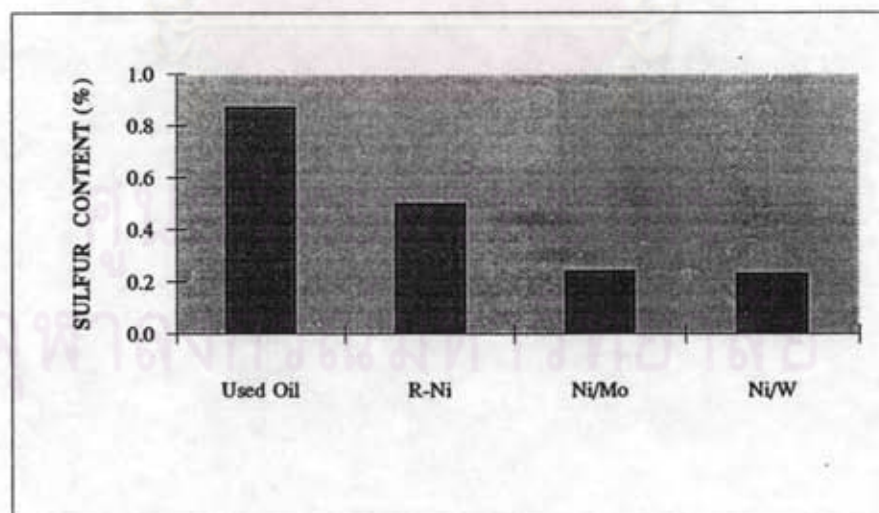


Figure 4.12 Effect of catalyst types on sulfur content in hydrogenation of used oil compared with an original used oil

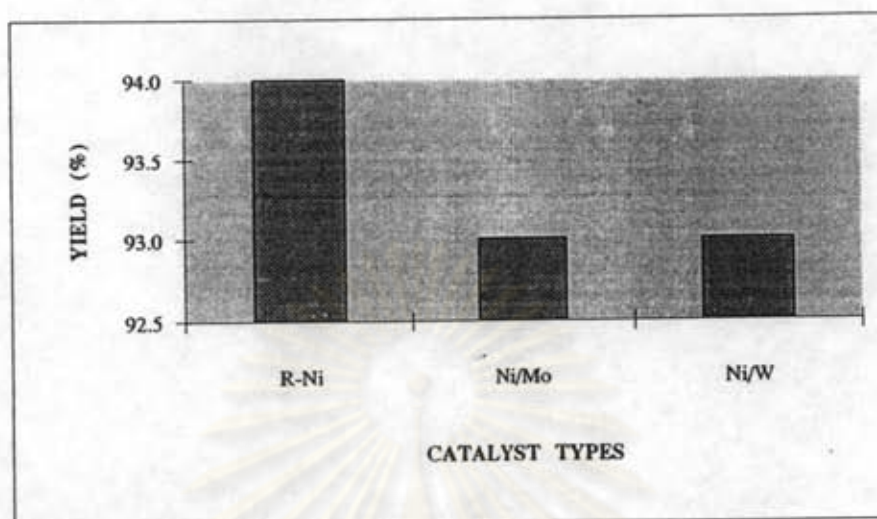


Figure 4.13 Effect of catalyst types on % yield in hydrogenation of used oil

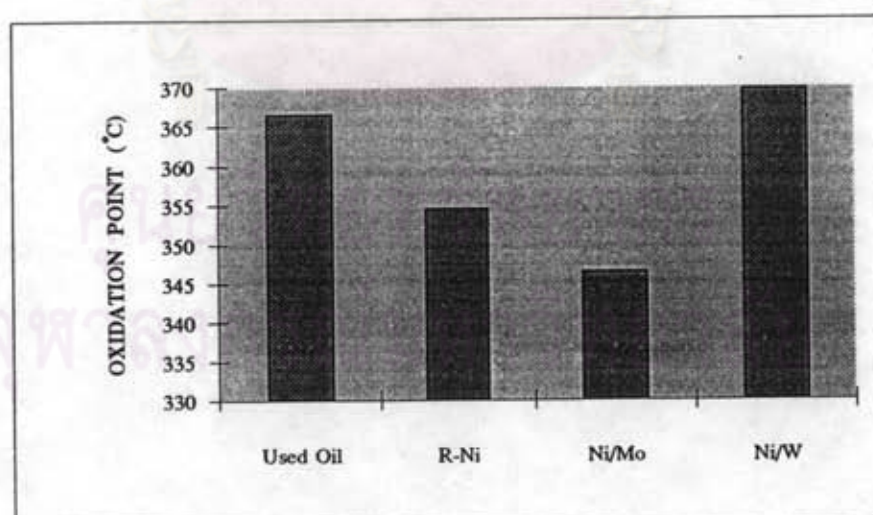


Figure 4.14 Effect of catalyst types on oxidation point in hydrogenation of used oil compared with an original used oil

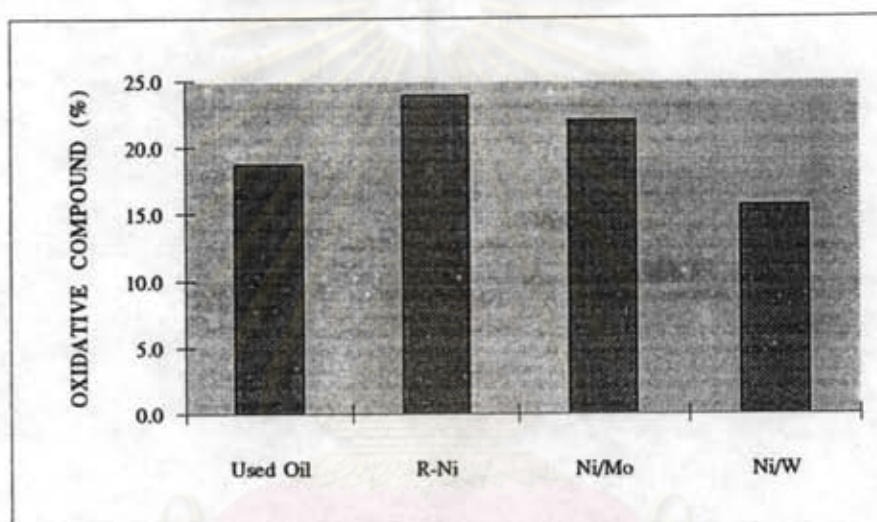


Figure 4.15 Effect of catalyst types on oxidative compound in hydrogenation of used oil compared with an original used oil

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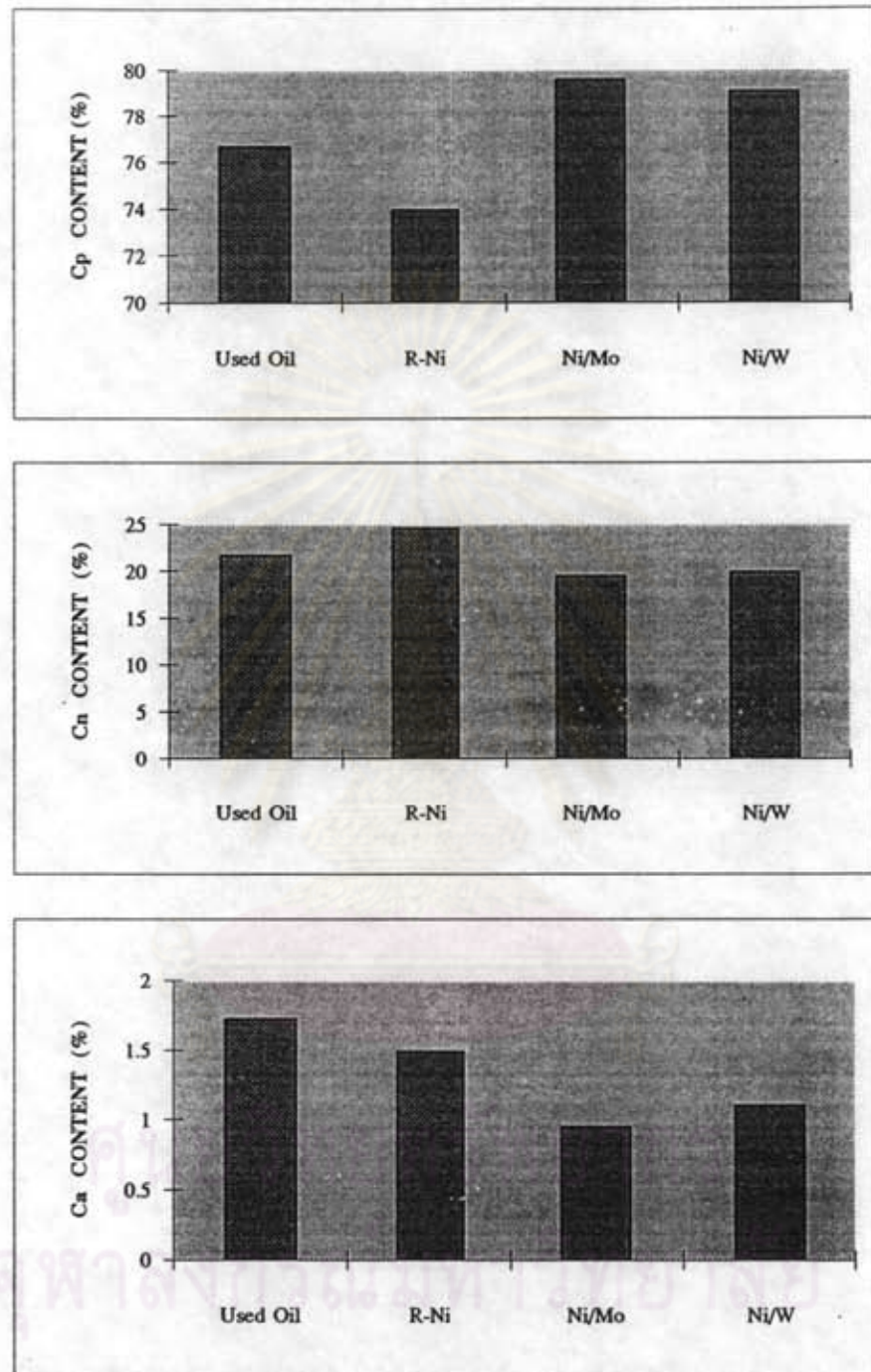


Figure 4.16 Effect of catalyst types on various types of carbon content in hydrogenation of used oil compared with an original used oil



Table 4.4 Physical and chemical properties of oils obtained from hydrogenation of used oil by using Raney nickel catalyst in different concentrations

Physical and Chemical Properties	Concentration of catalyst, %		
	4	6	8
API Gravity @ 60 °F	29.7	30.1	30.0
Color, ASTM	L 2.0	L 1.5	L 1.5
Pour Point, °C	-7.5	-8.0	-9.0
Viscosity, Kin @ 40 °C, cSt	41.46	39.80	38.24
Viscosity, Kin @ 100 °C, cSt	6.47	6.18	6.18
Viscosity Index	106	101	108
Sulfur Content, % wt	0.50	0.33	0.36
Oxidation Point, °C	354.5	371.6	351.0
Oxidative Compound, % wt	23.9	15.5	21.3
Carbon Content, %			
Cp	73.93	80.15	77.48
Cn	24.58	18.85	21.50
Ca	1.49	1.00	1.03
Yield, %	93	93	93

From the above data, Raney nickel catalyst in higher concentration had more activity in hydrogenation of used oil. It gave better results in both physical and chemical properties especially in color, pour point depression, oxidative stability. It should be considered that when used this catalyst in concentration of 6 %, the VI value was reduced to 101, if considered other properties such as carbon content in oil composition, the more naphthene content in the oil, the more value of VI. In this case, when Raney nickel catalyst in concentrations of 4% and 8% was used, the quantity of Cn were 24.58% and 21.50% respectively, and when used Raney nickel catalyst in the

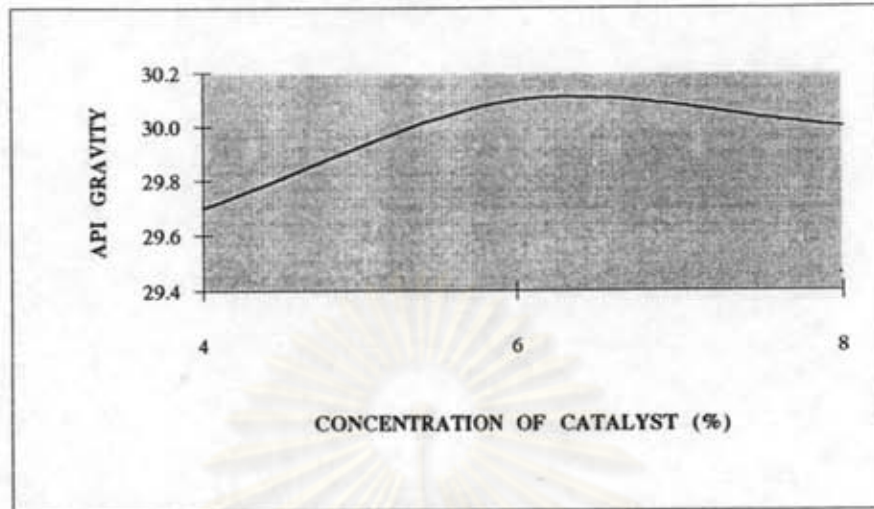


Figure 4.17 Effect of concentration of Raney nickel catalyst on API gravity in hydrogenation of used oil

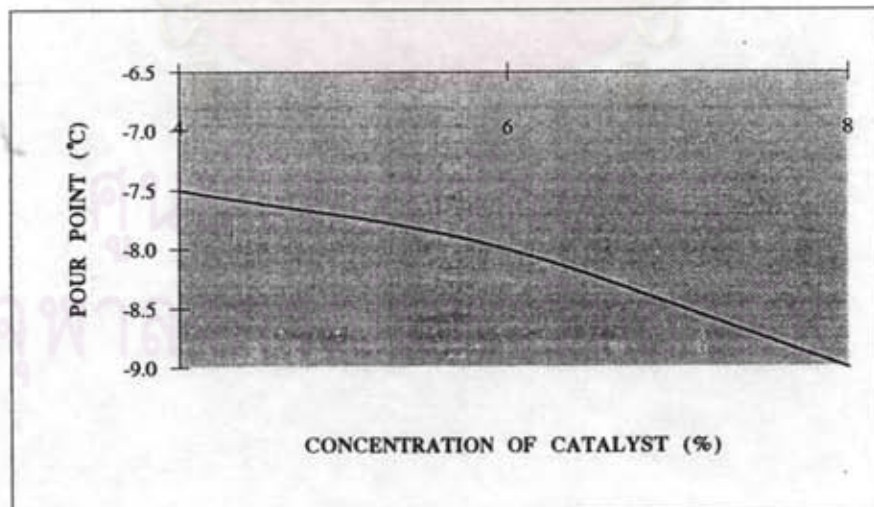


Figure 4.18 Effect of concentration of Raney nickel catalyst on pour point in hydrogenation of used oil

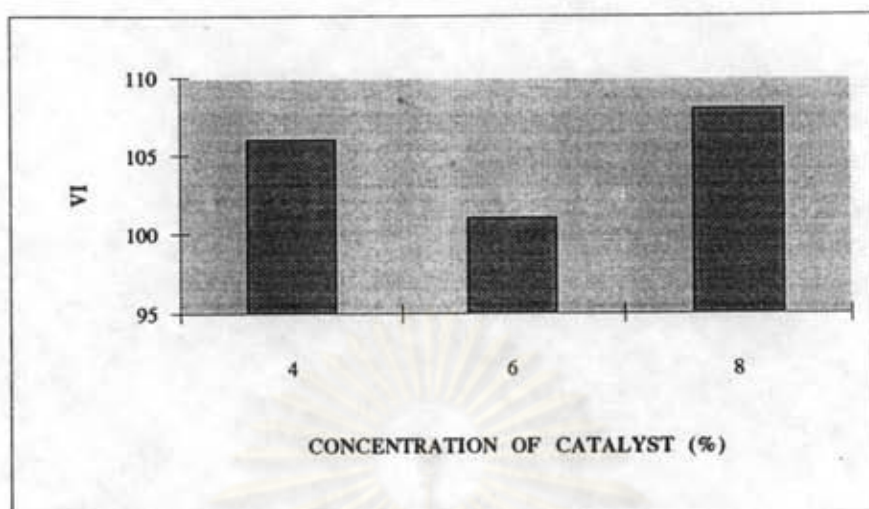


Figure 4.19 Effect of concentration of Raney nickel catalyst on viscosity index in hydrogenation of used oil

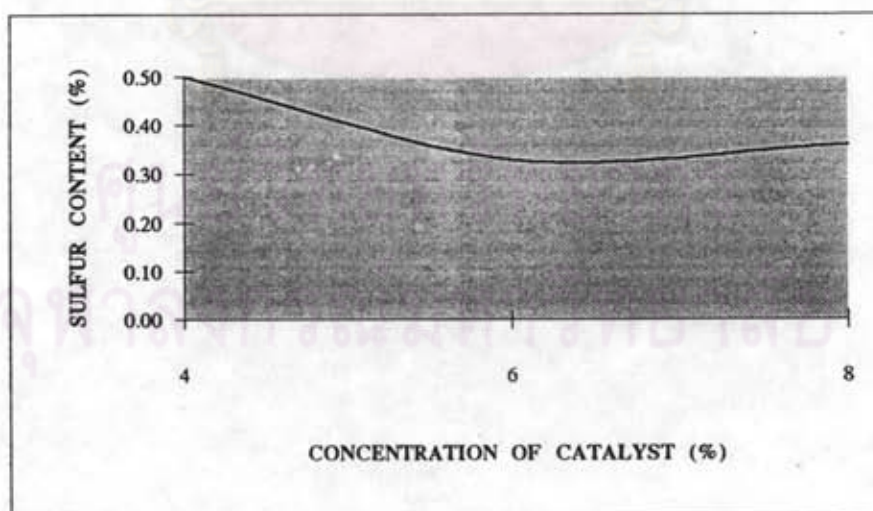


Figure 4.20 Effect of concentration of Raney nickel catalyst on sulfur content in hydrogenation of used oil

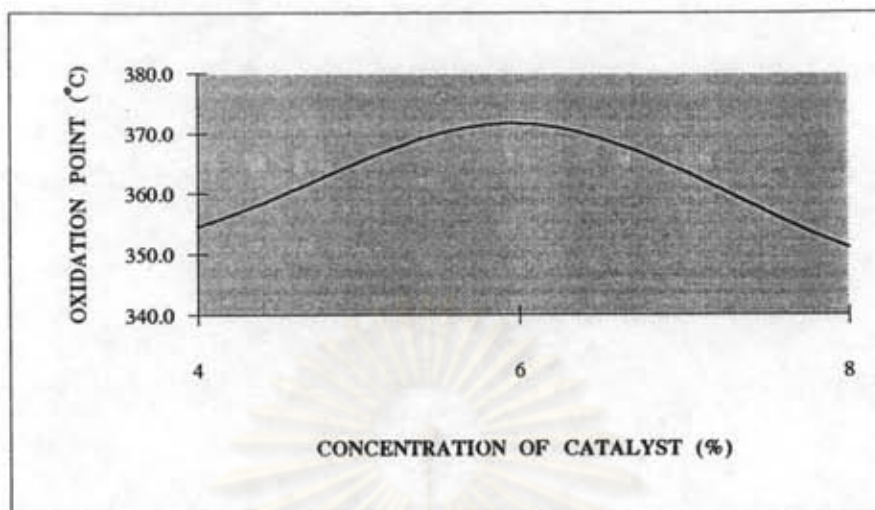


Figure 4.21 Effect of concentration of Raney nickel catalyst on oxidation point in hydrogenation of used oil

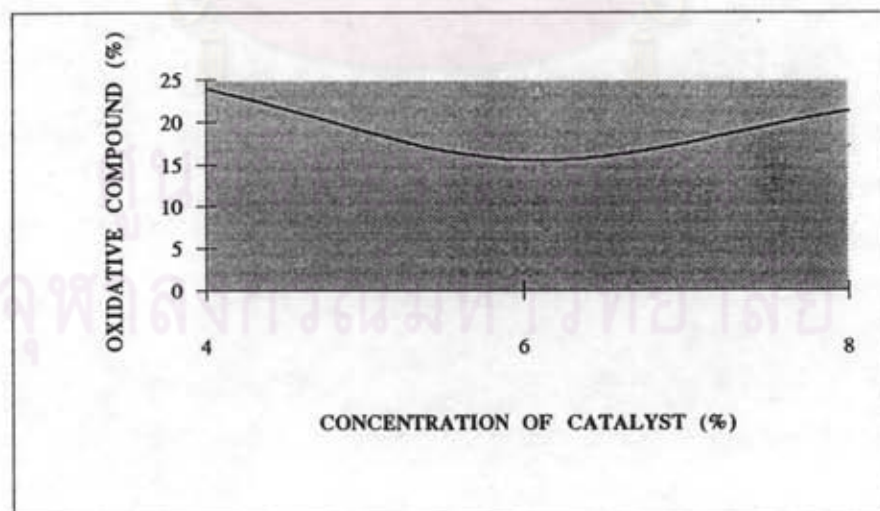


Figure 4.22 Effect of concentration of Raney nickel catalyst on oxidative compound in hydrogenation of used oil

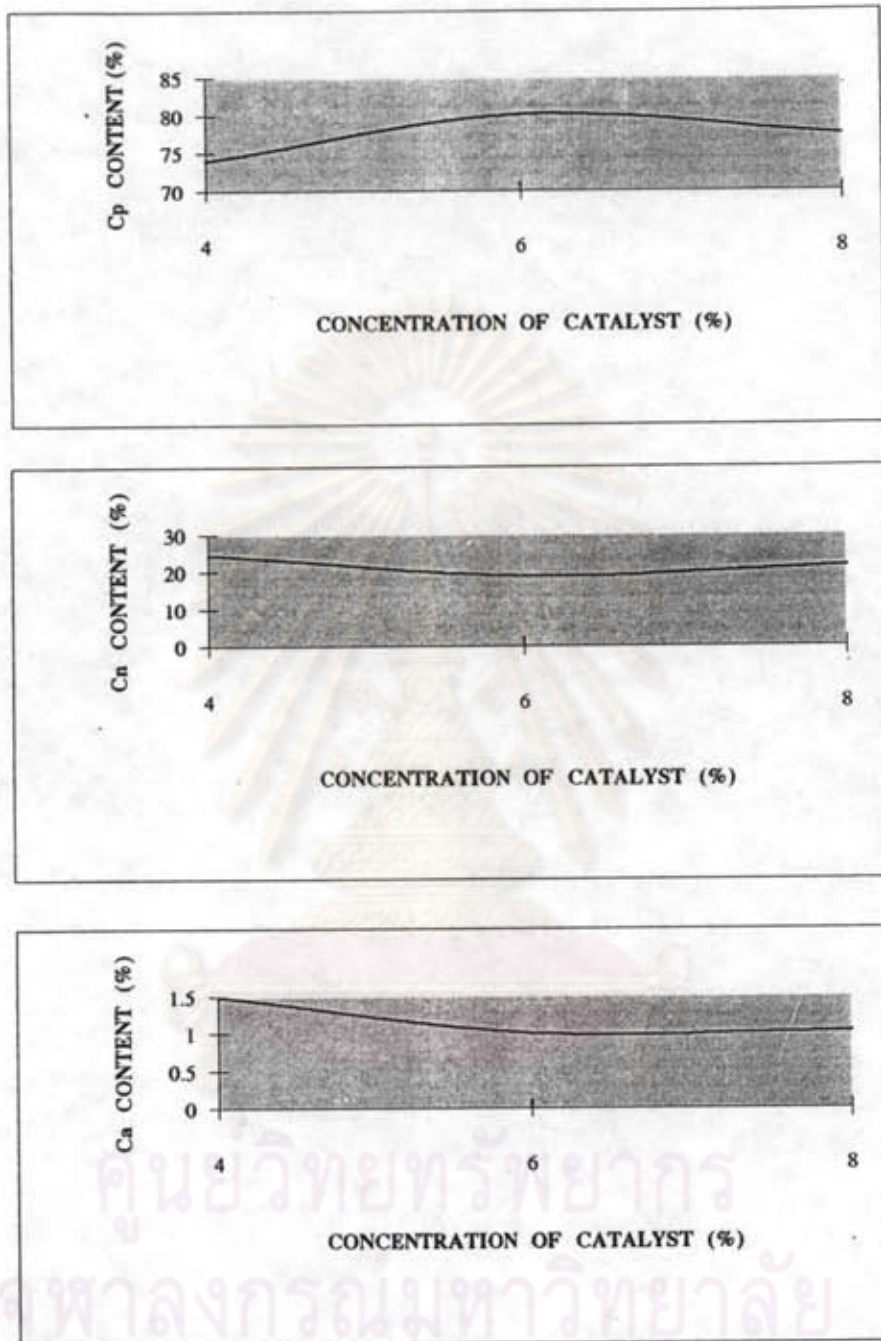


Figure 4.23 Effect of concentration of Raney nickel catalyst on various types of carbon content in hydrogenation of used oil

concentration of 6%, it gave lower VI value of 101. In comparison, if used catalyst in higher concentration, the data showed that the oil had good oxidation stability that gave lower in oxidative compound.

In some cases, the used oil had properties in better value than treated oil such as pour point, oxidation point for examples. This can explained that in used oil may had some available additives to maintain the good results of oil. In this experiment, data shows that by using Raney nikel catalyst it had high oil yield because of its ease of oil separation.

Ultimately, the catalyst in various concentrations used in this hydrogenation process were still gave the good quality of treated oil but they were not in significant differences.

#### **Effect of Reaction Times on Hydrogenation of Used Lubricating Oil**

This section studied hydrogenation of used lubricating oil by using the same amount of Raney nickel catalyst but varied the reation times. Reaction times for this experiment were 4, 6, and 8 hours respectively whereas other conditions were the same. By this way, all data obtained were shown in the following Table:

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Table 4.5 Physical and chemical properties of oils obtained from hydrogenation of used oil by using Raney nickel catalyst in the same concentrations but in different reaction times

Physical and Chemical Properties	Reaction Time, hours		
	4	6	8
API Gravity @ 60 °F	29.7	30.0	30.1
Color, ASTM	L 2.0	L 2.5	L 2.5
Pour Point, °C	-7.5	-8.0	-8.0
Viscosity, Kin @ 40 °C, cSt	41.46	38.99	38.89
Viscosity, Kin @ 100 °C, cSt	6.47	6.34	6.31
Viscosity Index	106	111	111
Sulfur Content, % wt	0.50	0.39	0.37
Oxidation Point, °C	354.5	365.2	361.0
Oxidative Compound, % wt	23.9	18.2	18.9
Carbon Content, %			
Cp	73.93	77.10	74.72
Cn	24.58	21.87	24.47
Ca	1.49	0.88	0.81
Yield, %	93	94	93

From Table 4.5, the more reaction times in hydrogenation, the more desirable properties of treated oils were obtained. Resulting data were rather closed values, excepted in VI values, and aromatic carbon contents. By using 6 and 8 hours for reaction times, the values of VI were the same of 111 whereas using 4 hours for reaction time, the value of VI was 106. In considered Ca contents, 6 and 8 hours could reduced the aromatic carbon contents to 0.88% and 0.81% respectively whereas 4 hours gave higher aromatic carbon content of 1.49%.

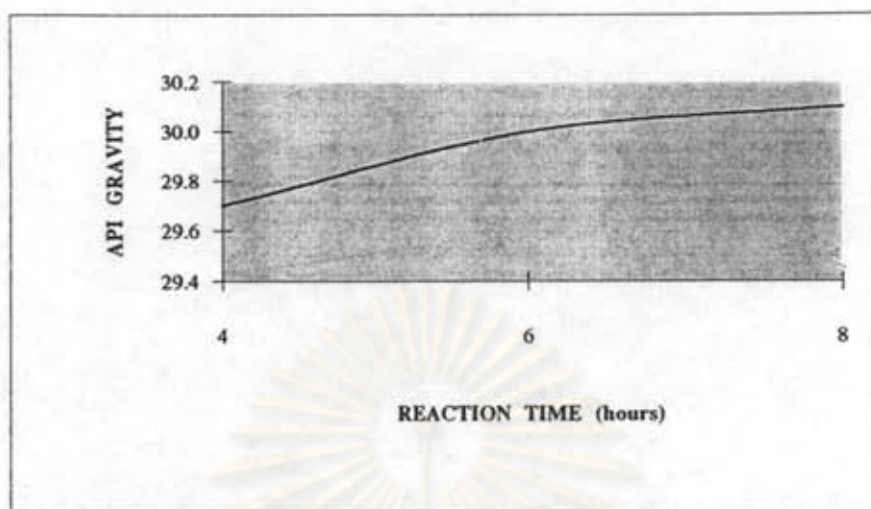


Figure 4.24 Effect of reaction time on API gravity in hydrogenation of used oil

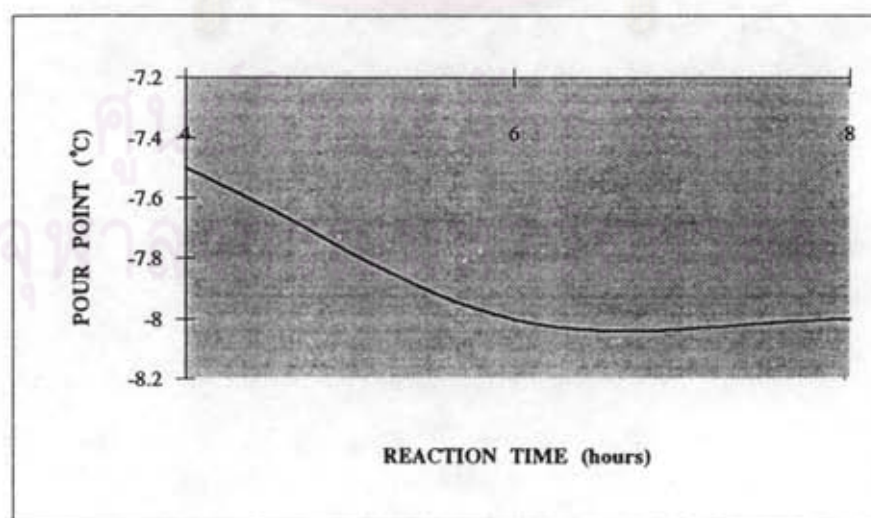


Figure 4.25 Effect of reaction time on pour point in hydrogenation of used oil



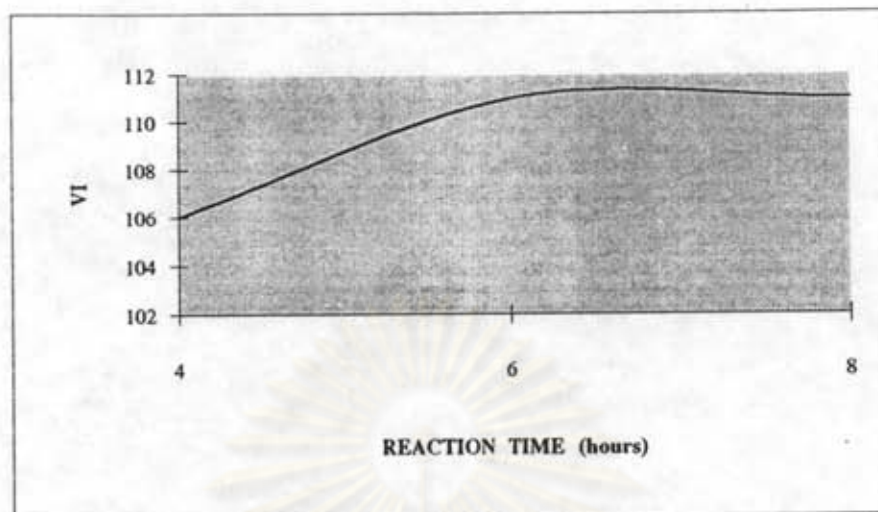


Figure 4.26 Effect of reaction time on viscosity index in hydrogenation of used oil

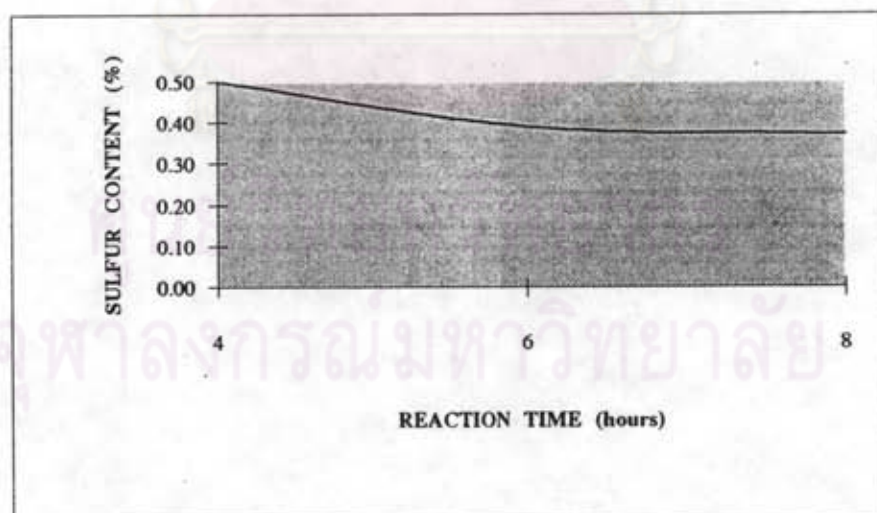


Figure 4.27 Effect of reaction time on sulfur content in hydrogenation of used oil

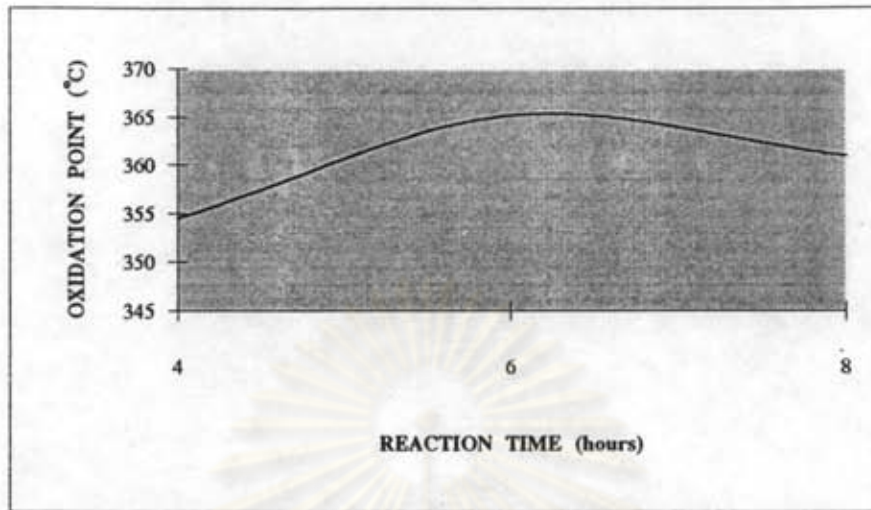


Figure 4.28 Effect of reaction time on oxidation point in hydrogenation of used oil

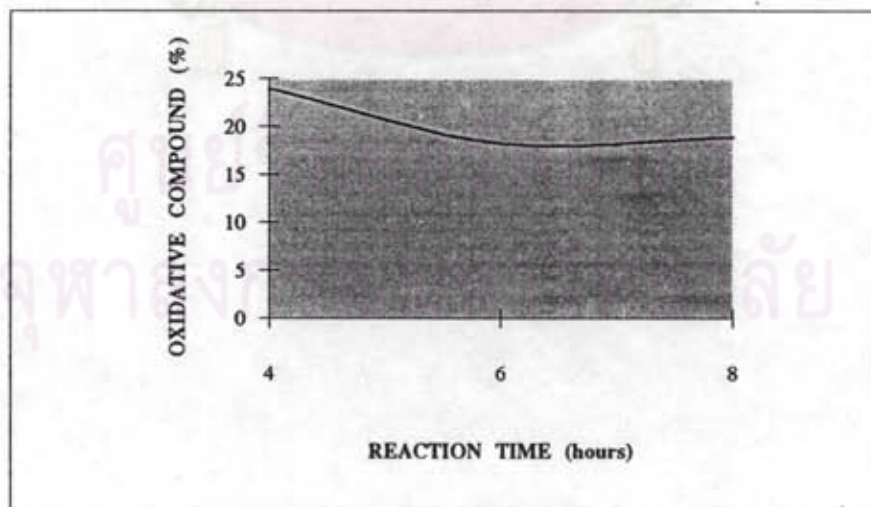


Figure 4.29 Effect of reaction time on oxidative compound in hydrogenation of used oil

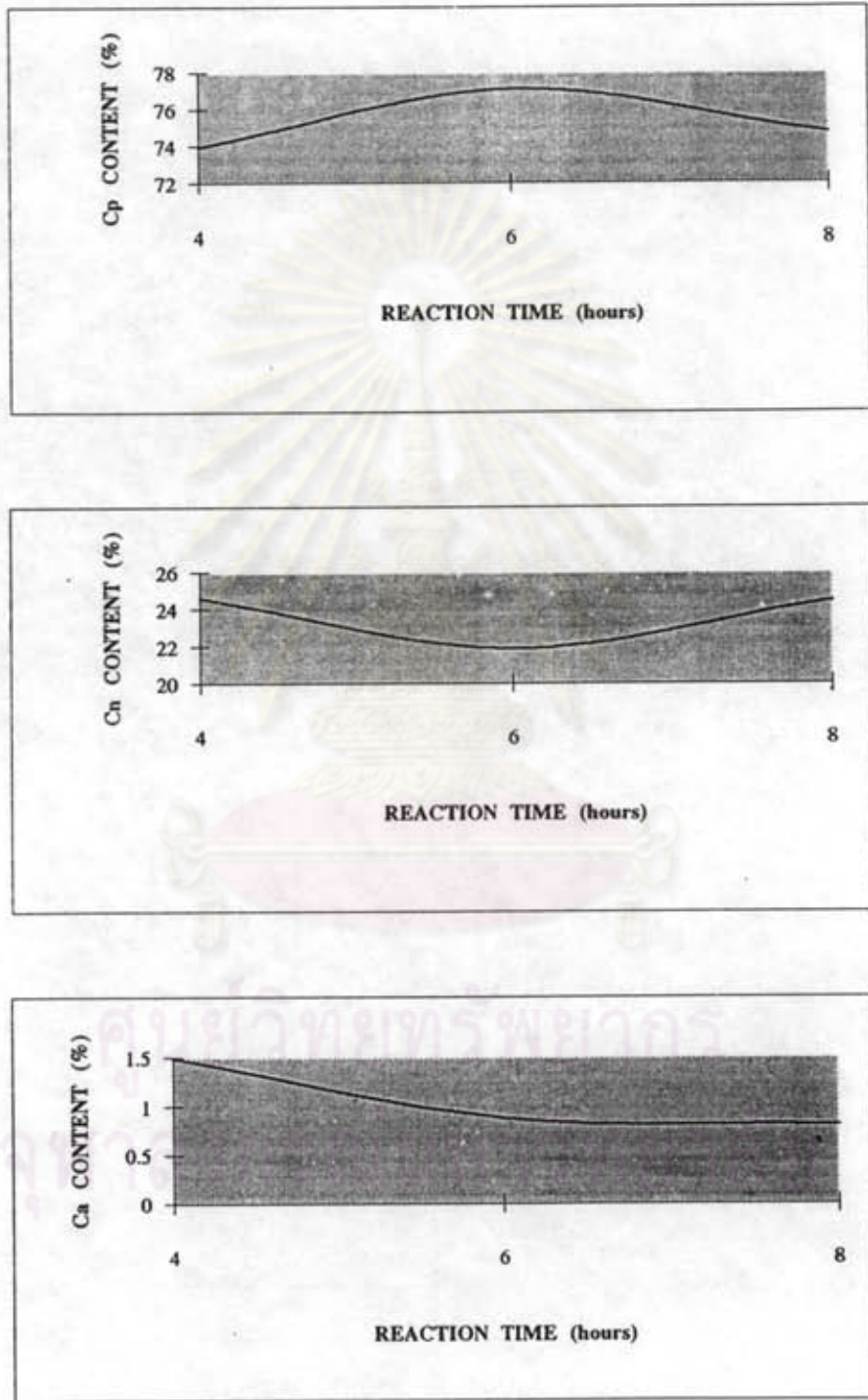


Figure 4.30 Effect of reaction time on various types of carbon content in hydrogenation of used oil

By changing reaction times, other oil properties such as color, API gravity, pour point, oxidative stability, Cp, and Cn were not significant different. But longer times let more sulfur removal. The used oil obtained by the reaction times of 6 and 8 hours had sulfur content of 0.39% and 0.37% respectively.



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