



Research Article

# Preparation, Characterization, and Activation of Co-Mo/Y Zeolite Catalyst for Coal Tar Conversion to Liquid Fuel

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## Abstract

One of many efforts to convert coal tar into alternative liquid fuel is by hydrocracking. This research aims to determine the impregnation of Co-Mo/Y zeolite, its characteristics, the effect of impregnation temperature and time, and also the best Co-Mo/Y zeolite impregnation condition for the conversion of coal tar. This research was conducted in several steps, impregnating Co from  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  and Mo from  $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$  into Zeolite Y in liquid media, drying at 100 °C for 24 hours, and calcination at 550 °C for 3 hours. Coal tar was then reacted with hydrogen gas (as a reactant), and Co-Mo/Zeolite Y (as a catalyst) was conducted at 350 °C. Characteristic analysis showed that Co and Mo had impregnated into the Y zeolite, as well as it made no change of catalyst's structure and increased the total acidity. The higher of impregnation temperature was increased the catalyst crystallinity, total acidity, and yield of gasoline. The longer impregnation time was reduced crystallinity value, but total acidity and yield were increased. GC analysis showed that products included into the gasoline product (C<sub>8</sub>, C<sub>9</sub>, and C<sub>10</sub>). Copyright © 2017 BCREC Group. All rights reserved

**Keywords:** Catalyst Activation; Catalyst Characterization; Coal tar; Co-Mo/Y zeolite; Catalyst Preparation

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## 1. Introduction

The use of petroleum as a fuel in Indonesia is increasing every year, while the oil production constantly decreased. To overcome this matter, the government officially issued Regulation No. 5 Year 2006 on National Energy Policy where in it is mentioned to reduce petroleum consumption and increase the consumption of other energies such as coal [1].

Coal should not be directly burned, but would be more meaningful and efficient if con-

verted into synthetic fuel or other high value petrochemical materials. One method is by a gasification process. Coal gasification is a process to convert solid coal into flammable coal gas. This process produces by-products such as quite large coal tar. Coal tar has black or dark brown color and categorized as a high viscosity liquid. Coal tar has very complex chemical components, includes monocyclic aromatic compounds, polycyclic aromatic, and heterocyclic so it has potential to be processed into synthetic fuel. Coal tar is mostly composed of C, H, and O, and also lower concentration of S and N [2]. The aromatics and heteroatoms (S and N) contained in coal tar must be changed and the molecular

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weight and viscosity should be lowered. To lower the molecular weight, viscosity, and aromatics content, the coal tar must be processed by hydrogenation and cracking or hydrocracking. The cracking process requires a catalyst that has dual functions, i.e. metal component as the hydrogenation catalyst and acid component as the cracking catalyst [3]. Ones of the transition metals that most commonly used in the hydrocracking process are Co and Mo. Meanwhile, the most common catalyst used in catalytic cracking and hydrocracking is zeolite [4-10].

Wang *et al.* [11] has done a research using zeolite catalyst, that reaction initially took place at high temperature (without a catalyst), but when using a catalyst, the reaction could take place at lower temperature. Research by Emelik [12] and Tsitsishvili [13] about the effect of Ni and Mo metals showed that catalyst with Ni and Mo required higher specific surface area to produce greater distribution of Mo. Anggoro *et al.* [14] and Zeno [15] studied the effect of Co and Mo metals addition on Y zeolite and found that at higher concentration of the metal, the catalyst acidity was higher if supported by equitable distribution. Therefore, in this research, the Co and Mo impregnated into Y zeolite was applied to convert coal tar into liquid fuels.

The effect of impregnation temperature and time need to be considered to determine the best preparation of Co-Mo/Y zeolite condition which can be used as a hydrocracking catalyst of coal tar. This has encouraged research to determine the effect of impregnation condition of Co and Mo into Y zeolite in coal tar hydrocracking into liquid fuel. Therefore, the objectives of this research are to determine the effect of temperature and length time of impregnation on the preparation of the Co-Mo/Y zeolite catalyst, to characterize the Co-Mo/Y zeolite catalyst, and to test the catalyst on the conversion of coal tar to liquid fuel.

## 2. Materials and Method

### 2.1. Materials

The materials used were a coal tar from PT. Sango Ceramic Indonesia, Y-zeolite (99.9 %) and ZSM-5 (99.9 %) from Zeolyst International,  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  (99.0 %) from Merck,  $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$  (99.3 %) from Merck, ammonia from Merck, and hydrogen gas (99.99 %) from PT. Aneka Gas.

### 2.2. Instrumentation

The functional group detection of the catalysts was analyzed by Fourier Transform Infra Red (FTIR) Shimadzu IR Prestige-21. The crystallinity of catalyst and its structure were done by X-Ray Diffraction (XRD) Shimadzu XRD-7000, and the total acidity determination was conducted using method of ammonia adsorption gravimetrically. The liquid fuel product was analyzed using Gas Chromatography.

### 2.3. Preparation and characterization of catalyst

The Co-Mo/Y zeolite catalyst was prepared using impregnation method. Five grams of Y zeolite, cobalt metal obtained from  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  0.985 grams, and molybdenum metal from  $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$  4.34 grams were dissolved in 50 mL of distilled water. The solution was then heated to temperature as stated in independent variables and stirred for the length time as stated in the variables (Table 1). Thus, the samples were filtered using a vacuum pump until no more water was dripping. Further, the samples were dried in an oven at temperature of 100 °C for 24 hours. The dried samples were then calcined at temperature of 550 °C for 3 hours. The functional group of the catalysts was analyzed by FT-IR method. The stability of catalyst structure was analyzed by X-Ray Diffraction. The total acidity of catalysts was determined using the ammonia gravimetrically method [16].

### 2.4. Catalyst testing and analysis of product

Hydrocracking process was performed in a 1000 mL three-neck flask size as a place of coal tar (100 mL), evaporated using a hot plate (300

**Table 1.** Matrix of experimental design

Run	Temperature (°C)	Time (minutes)
1	35	10
2	35	30
3	55	10
4	55	30
5	31	20
6	59	20
7	45	6
8	45	34
9	45	20
10	45	20

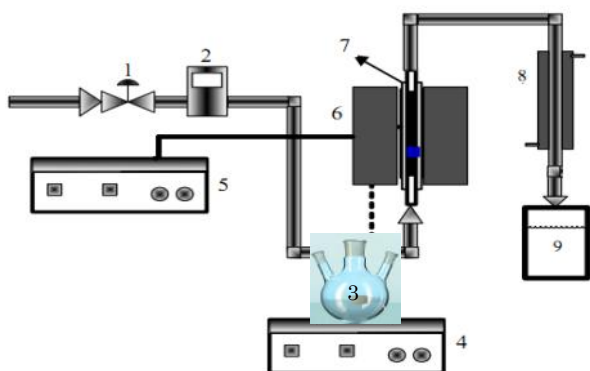
°C, and completed with a magnetic stirrer. Hydrogen gas used as the reactant gas of the hydrocracking. The reactor used was a stainless steel tube reactor completed with ceramic and glass wool in order to support position of the catalyst as shown Figure 1. There were a temperature indicator and a heater in the reactor, so the temperature reaction was maintained at 350 °C. Seven grams of catalyst, consisted of 5 grams catalyst which had been prepared (Co-Mo/Y zeolite) and 2 grams ZSM-5 catalyst, was put in the reactor tube. The cooling system of the reactor used a condenser and ethylene glycol (as coolant). Length time of the hydrocracking reaction was 1 hour.

### 3. Results and Discussion

#### 3.1. Catalyst characterization using FTIR

Catalyst characterization was intended to determine whether Co and Mo had impregnated successfully or not into the Y zeolite. Figure 2 explained FTIR spectra to show the characteristic of the Y zeolite and the Co-Mo/Y zeolite. From the Figure 2, it is indicated that impregnation of Co and Mo at temperature and length time as independent variables did not cause changes structure of the catalyst. Preparation of catalyst using impregnation method aims to elicits metal which can expand the surface active site catalyst.

In comparison to standard FT-IR spectrum of Y zeolite, the Co-Mo/Y zeolite catalyst showed that the ten samples had frequency or wave number shifted at 1199.87  $\text{cm}^{-1}$  and 1067.88  $\text{cm}^{-1}$ , which indicates the range of O-Si-O and O-Al-O [17]. Presumably, this shift is caused by a reduction of Al atoms in the framework to become Al non-framework due to dealumination during catalyst calcination [17].



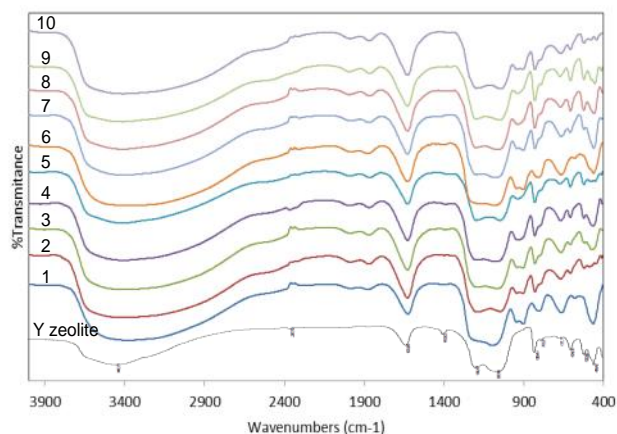
**Figure 1.** Experimental setup: (1) valve, (2) flowmeter, (3) glassware, (4) hot plate + magnetic stirrer, (5) temperature controller, (6) split tube furnace, (7) reactor, (8) condenser, (9) glass bottle

At temperature of 200 °C, protons in zeolite have high mobility, while at 550 °C are separated as water to form the Lewis site as described in Figure 3.

The presence of water vapor constantly strengthens the structure stability of Lewis site structure, and the result is called "actual Lewis sites" which can be seen in Figure 4. The peak in the standard spectrum of Y zeolite at 1400.62  $\text{cm}^{-1}$  is present, but not detected in ten samples. According to Coates [17], the peak indicates -OH. There is also a peak at 829.49  $\text{cm}^{-1}$  on spectrum of Y zeolite standard which indicates C-H [17]. Meanwhile, the spectrums of Co-Mo/Y zeolite have a shift at that wave numbers thought caused by impregnation of the Co and Mo. Also, there was appearance of two peaks near wave numbers of 902  $\text{cm}^{-1}$  and 945  $\text{cm}^{-1}$ , while there was not detected in spectra standard of Y zeolite before. The peak area indicates asymmetrical strain O-Si-O and O-Al-O [17]. The appearance peak strengthens the presumption of Co and Mo had impregnated into catalyst accordance impregnation temperature and time variables.

#### 3.2. Effect of impregnation temperature and time to the Co-Mo/Y zeolite crystallinity

It can be observed in Figure 5 that the intensity of the ten Co-Mo/Y zeolite catalysts diffractogram tend to decrease. The catalyst crystallinity is calculated by comparing the intensity of the eight strongest peaks of Co-Mo/Y zeolite with that of Y zeolite (Table 2). The catalyst crystallinity decreased when Co and Mo were impregnated on the zeolite. This is probably due to the presence of Co and Mo into Y zeolite. However, the ten catalysts have similar pattern with the Y zeolite, so it can be said



**Figure 2.** FTIR spectra of Y zeolite and Co-Mo/Y zeolite

that the crystallinity of Y zeolite was not damaged after impregnating Co and Mo [14-15]. This is consistent with the results of FTIR analysis where the graph shows that the structure does not change after impregnating Co and Mo.

Based on Figure 6 (a), at the same impregnation time, it can be seen that higher the temperature, higher the degree of catalyst crystallinity. According to Krichko [18], the increasing of impregnation temperature may increase the mobility of the chains bond in the catalyst, which may occur in the area of the crystal structure. The high mobility leads to the composition of the chain becomes tight and thus expand the areas of crystal structure. However, there are deviations in the chart, which is a decrease of crystallinity as the temperature rises at impregnation time of 20 minutes and temperature of 60 °C.

Figure 6 (b) indicates that at the same impregnation temperature, there is an increasing of crystallinity due to the longer impregnation time. The decline trend occurred may be caused by the metal deposits on the catalyst samples that cover the surface of the pores which in turn changes the characteristics of the zeolite crystal and causes a decrease in the intensity of the curve [19]. The longer impregnation time causes the high amount of Co and Mo are impregnated, and the catalyst crystallinity has decreased.

### 3.3. Effect of impregnation temperature and time to total acidity

According to Figure 7, it can be seen that increasing of total acidity occurs after impregnating Co and Mo into Y zeolite. The measured

acidity of catalyst is calculated as the sum of total Brönsted acid and Lewis acid. In the catalysts impregnated with metal, the metals donate the amount of the acidic Lewis site. This is consistent with the analysis results of increasing trend in the acidity of the catalyst with the addition of Co and Mo into Y zeolite [14,15].

At the same impregnation time, it can be seen in Figure 8 (a) that the raising impregnation temperature increases the total acidity at 10 minutes and decreases at 30 minutes. Meanwhile, at 20 minutes impregnation length time decreases followed increases. To find out the cause of this phenomenon is expected for further Co-Mo/Y zeolite catalyst characterization.

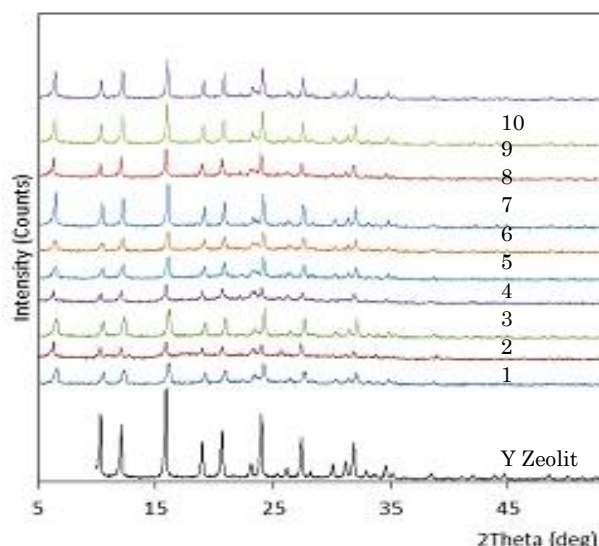


Figure 5. XRD diffractogram of Y zeolite and Co-Mo/Y zeolite

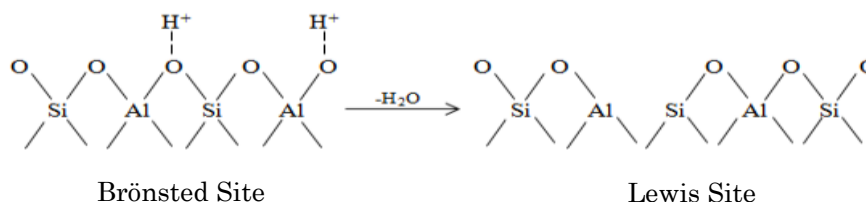


Figure 3. The process of release of a proton from Brönsted site into Lewis site

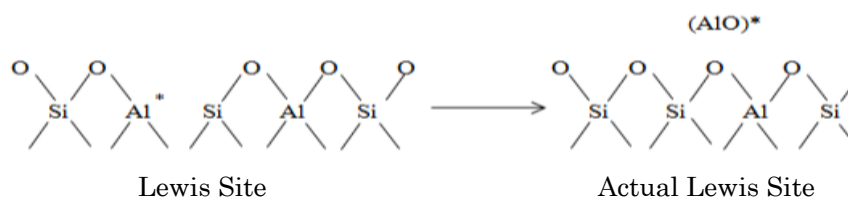


Figure 4. The process of structural stability from Lewis site into actual Lewis site

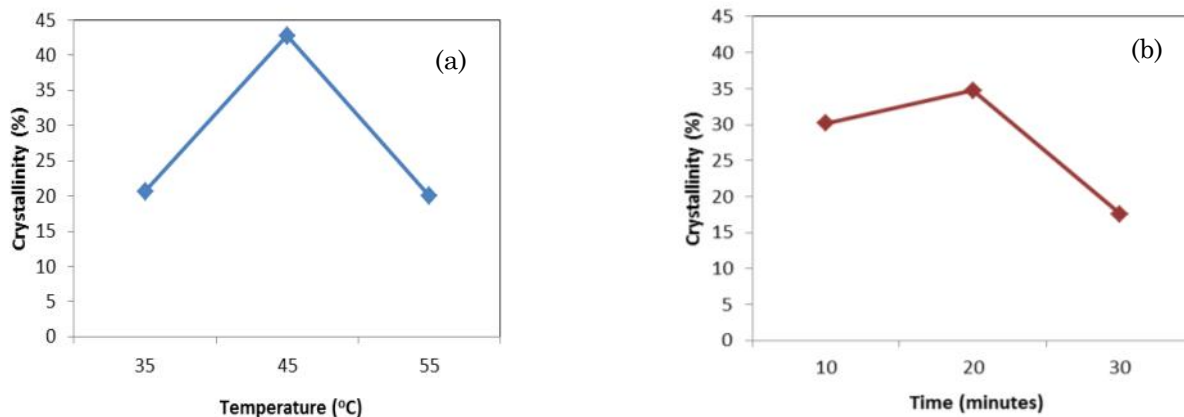
Based on Figure 8 (b), at same impregnation temperature, it can be seen that at the longer impregnation time, the total acidity of the catalyst is increased. This is likely due to that at the longer time, the more ammonia is absorbed by the Co-Mo/Y zeolite catalyst. However, a deviation occurs at 45 °C and 20 minutes caused by incompletely distribution of ammonia adsorption.

### 3.4. Effect impregnation temperature and time to liquid fuel product

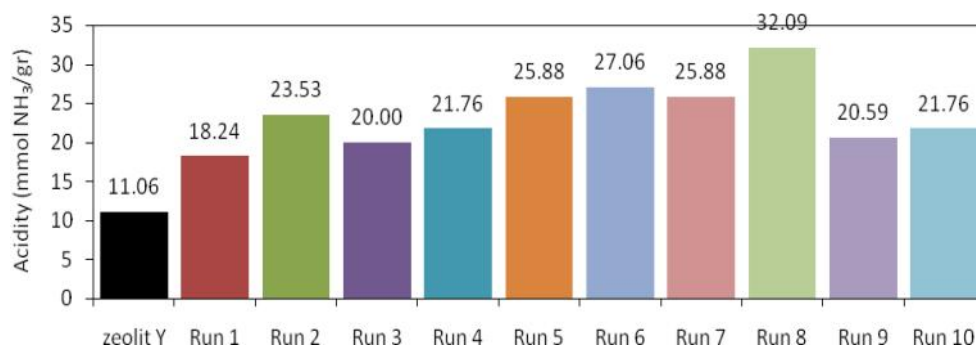
According to Figure 9 (a), at the same impregnation temperature, increasing in time causes increasing in product yield. This may be due to roles of acidity and crystallinity of the catalyst. This is consistent with the previous discussion in which the increase in impregnation length time leads to increased acidity. Therefore, the high acidity of catalyst enhances the catalyst performance during cracking process. In Figure 9 (b), increasing in yield occurs as the temperature rises at the same time. This is influenced by increasing in crystallinity as temperature rises. The high percentage of crystallinity indicates that the catalyst structure did not change significantly and tend to be more stable, so the ability of the catalyst can increase and accelerate the formation of reaction product. Data of catalyst crystallinity and the concentration of the product gas in the GC product analysis results were shown in Figure 10. The crystallinity of the zeolite can be calculated from X-ray diffractogram [20]. The crystallinity of the zeolite is expressed as the percentage of crystallinity using the intensity of

**Table 2.** Co-Mo/Y zeolite crystallinity

Run	Crystallinity (%)
1	23.84
2	17.39
3	36.49
4	17.73
5	25.35
6	23.31
7	49.84
8	30.86
9	46.53
10	43.92



**Figure 6.** Relationship of impregnation (a) temperature and (b) length time with crystallinity



**Figure 7.** Value of total acidity of Y zeolite and Co-Mo/Y zeolite

the main peak at  $2\theta = 22.5-24.4^\circ$  in the XRD pattern.

From Figure 10, it is obtained the relationship between the Co-Mo/Y zeolite crystallinity at impregnation temperature and impregnation length time with the concentration of the gas contained in coal tar products of the hydro-cracking process. The figure indicated that the impregnation temperature and the impregnation length time caused the differences of crystallinity. However, the concentration of gasoline (%) increases with increasing both variables. According to Figure 10, it can be seen that at higher percentage of the Co-Mo/Y zeolite crystallinity, the greater the concentration of the fuel produced, except for crystallinity at 17.73 and 23.31 %. The crystallinity is a very important factor that influencing the nature of the catalyst. The high crystallinity indicates that the catalysts, which are free from impurities and physical properties (catalytic properties of high, stable at high temperatures, and extensive porosity), are not disturbed [21-23].

As a result, performance can be run optimally, so that produce more product.

### 3.5. Analysis of liquid fuel

GC-MS analysis showed peak gasoline in the range of 1.3-19.9 minutes explaining ten different products. The gasoline concentration on the ten products can be determined by calculating the concentration of GC peak results which are in the range of gasoline. As comparison, a commercial gasoline sample is obtained from gas station where GC-MS analysis indicates that the components contained in the produced gasoline have a number of chains of carbon atoms ranging from  $C_2-C_{10}$ .

Consideration of the suitability using hydrocarbons for gasoline is based on the volatility and octane number. In general, the gasoline has a component of  $C_5-C_8$ , but some products are up to  $C_9$  or more [19]. In the range of  $C_5-C_8$ , it contained aromatic isomers with branched-chain or cyclic good in the use of gasoline as a motor fuel. This is consistent with the results

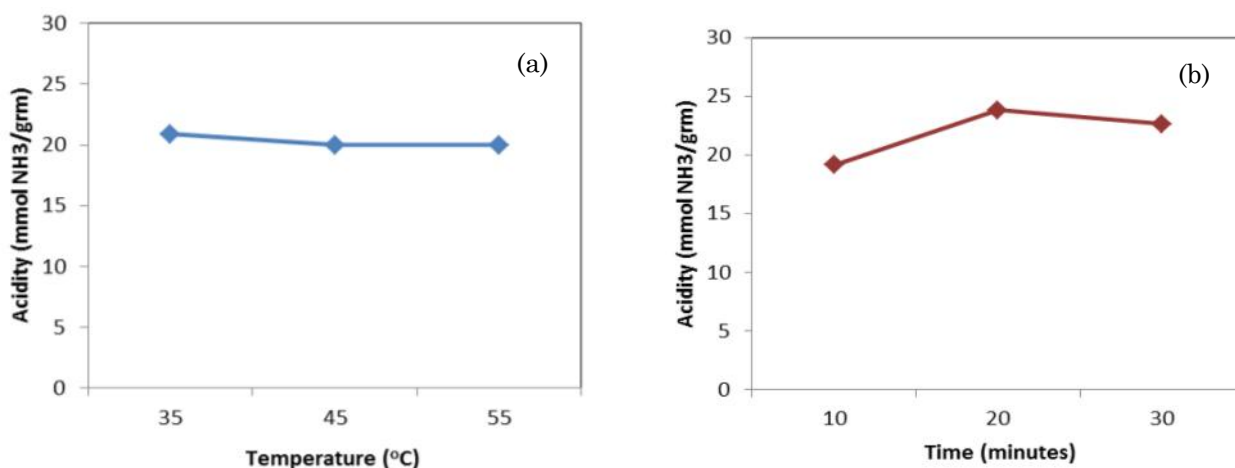


Figure 8. Relationship of impregnation (a) temperature and (b) time with acidity

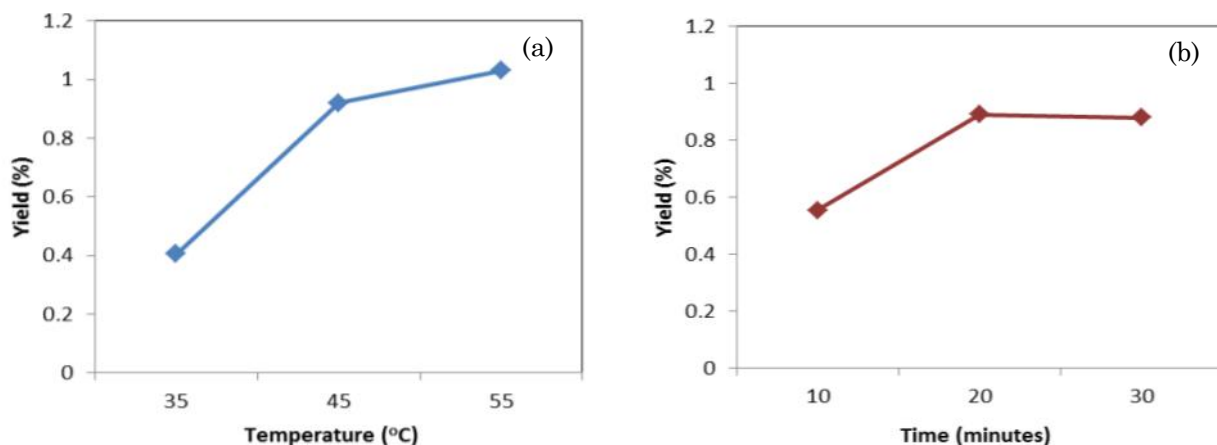


Figure 9. Relationship of impregnation (a) time and (b) temperature with yield

of GC analysis of the ten products in Table 3 that most of products contains C<sub>8</sub>, C<sub>9</sub>, and C<sub>10</sub>. Table 3 shows that the best impregnation condition to get the best yield is by impregnation method at temperature of 55 °C and 30 minutes. Results of Table 3 and Figure 7 showed that the yield of gasoline depends on the acidity of catalyst. The yield of gasoline increases with increasing of the catalyst acidity.

#### 4. Conclusions

In conclusion, the catalyst performance of Zeolite Y and Co-Mo/Zeolite Y was significantly affected by physicochemical properties of the catalyst. Impregnation of Co and Mo on the zeolite at temperature and length time of impregnation did not affect structure of the zeolite Y catalyst, and even increased the total acidity of the zeolite Y. Increasing the impregnation temperature led to improved catalyst crystallinity and acidity as well as the yield of gasoline. Meanwhile, the longer the impregna-

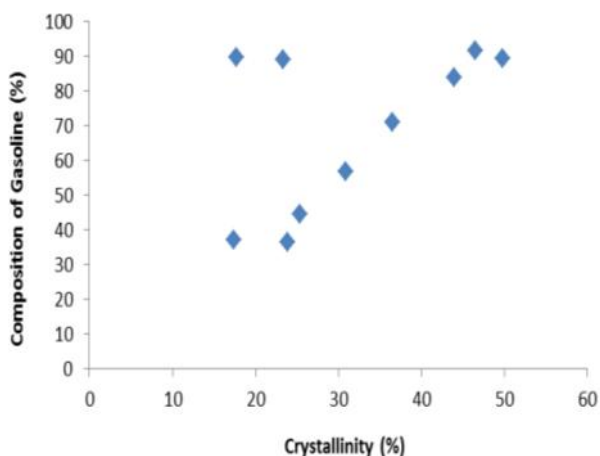


Figure 10. Relationship between crystallinity and concentration of gasoline

tion length time, the more reduced the catalyst crystallinity, but total acidity the catalyst and yield increased. From GC analysis showed that the liquid fuel products in the range of gasoline comprising C<sub>8</sub>, C<sub>9</sub>, and C<sub>10</sub>. Crystallinity of the Co-Mo/zeolite Y catalyst was the highest when impregnated at a temperature range of 40-50 °C and length time of 5-20 minutes. In order to get the highest liquid fuel yield, the catalyst should be impregnated at temperature of 55 °C and length time of 30 minutes.

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Table 3. Yield and composition of C<sub>8</sub>, C<sub>9</sub>, and C<sub>10</sub> in gasoline

Run	Yield of Gasoline (%)	Composition (%)		
		C <sub>8</sub>	C <sub>9</sub>	C <sub>10</sub>
1	0.40	0.00	90.31	9.69
2	0.41	0.00	0.00	100.00
3	0.71	9.13	9.11	81.77
4	1.35	2.78	1.53	95.69
5	0.54	0.00	56.30	43.70
6	1.16	0.00	95.86	4.14
7	0.80	11.60	7.73	80.67
8	1.02	0.00	0.00	100.00
9	1.10	2.18	57.96	39.86
10	0.76	2.92	0.00	97.08

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