

# FISCHER-TROPSCH SYNTHESIS: EFFECT OF TEMPERATURE AND IRON-COBALT RATIO IN Fe-Co/meso-HZSM-5 CATALYST ON LIQUID PRODUCT DISTRIBUTION

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## Abstract

The Fischer-Tropsch synthesis converted hydrogen and carbon monoxide into linear hydrocarbons as liquid fuel. Iron and cobalt were used as polymerization catalyst, that impregnated on HZSM-5. The Fe-Co/HZSM-5 could be applied as bifunction catalyst which combined polymerizing synthesis gas and long chain hydrocarbon cracking for making biofuel. The objective of this study is observing the effect of temperature and composition of iron and cobalt combination, supported by HZSM-5 (Fe-Co/HZSM-5) catalyst on fuel product composition. The results obtained from this study would be used to find optimum condition for various iron and cobalt ratio in the catalyst. The mesoHZSM-5 was prepared from ammonium ZSM-5 over calcination, desilication, and dispersion. The mixed solution consisted of  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  and  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  were used as precursor for incipient wetness impregnation on HZSM-5. The catalyst performance was observed in a continuous fixed bed reactor using Fe-Co/meso-HZSM-5 catalyst with synthesis gas at various composition iron and cobalt ratio (10–40 % wt. Fe in Co), various temperature (225–275 °C) at 20 bars. All catalysts were reduced in situ in the reactor. The 10Fe-90Co/mesoHZSM-5 catalyst was more suitable for FTS at 250 °C with alkane (20.49 %) as the main product and alcohol as the by-product (79.51 %). The others catalysts composition of 20–40 % Fe (by weight) in Fe-Co were more suitable for FTS at 225–250°C because under these conditions, alkanes as the main product were obtained in relatively higher compositions compared to other compounds. The mechanism of paraffins, olefins, aldehydes and alcohols formation in this FTS reaction followed the hydrogen assisted CO dissociation with CO-insertion mechanism.

**Keywords:** synthesis gas, catalyst, zeolite, HZSM-5, iron-cobalt, biofuel, temperature, fixed-bed reactor, Fischer-Tropsch.

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## 1. Introduction

The HZSM-5 was one of zeolite that could be applied in catalytic process (i.e. cracking in Fischer-Tropsch). Microporous HZSM-5 had limitation in catalyzing the large molecules [1, 2]. Desilication formed mesopores over decreasing of silicon content in zeolite. This process created the larger pore size, improved reaction efficiency and minimized channel closure [3–6]. Commonly, the alkali was applied as desilication agent. The potassium hydroxide (KOH) induced greater mesoporous volumes than sodium hydroxide (NaOH), but KOH desilication showed zeolite crystal fracture. The NaOH could increase mesopore surface area and volume of HZSM-5. The Fe-Co alloys were observed with less oxide of iron ( $\text{Fe}_2\text{O}_3$ ) and cobalt ( $\text{Co}_3\text{O}_4$ ) in the produced catalysts [7].

The Fischer-Tropsch synthesis (FTS) converted hydrogen ( $\text{H}_2$ ) and carbon monoxide (CO) into linear hydrocarbons as liquid fuel in various products (i.e. olefins, paraffins and oxygenated

compounds). Water Gas Shift (WGS) activity, that produced more water, occurred significantly over iron (Fe) catalyst, but it could be ignored in cobalt (Co) catalyst. Iron and cobalt were used as polymerization catalyst, that impregnated on HZSM-5. The Fe-Co/HZSM-5 could be applied as bifunction catalyst which combined polymerizing synthesis gas and long chain hydrocarbon cracking for making biofuel [4, 8–11]. The cobalt gave some advantages, instead of iron catalyst. Iron was added in less amount to escalate the cobalt catalyst performance (improved olefin selectivity, reduce methane selectivity, reduce temperature and pressure sensitivity to product distribution and lower the catalyst price) [12, 13].

The Fe catalyst was applied in High Temperature Fischer-Tropsch (HTFT) process for high conversion, more olefins, high WGS activity and low resistance to deactivation due to coking, carbon deposition, iron carbide formation. The HTFT operated at 300–350 °C and 20 bars [14]. The Fe/HZSM-5 catalyst (at 290 °C and 17 bars) could produce 24.2 % of C<sub>5–19</sub> hydrocarbon as biofuel fraction [9]. The Cobalt was the optimal choice in low temperature FTS with high activity, high selectivity to linear paraffin, high resistance to deactivation, low WGS activity. Co-based catalysts were used in Low Temperature Fischer Tropsch (LTFT) which operated at 200–240 °C and 27 bars [16]. The Co/HZSM-5 catalyst (at 240 °C and 15 bars) produced 48 % of C<sub>5–11</sub> [4]. The combination of Fe and Co (bimetallic Fe-Co) could increase catalytic activity. The Fe-Co catalyst on conventional supports like TiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> had been investigated. They could increase the biofuel reaction conversion and fraction compared to the use of Fe and Co catalysts separately [15–18].

The objective of this study is observing the effect of temperature and composition of iron and cobalt combination, supported by HZSM-5 (Fe-Co/HZSM-5) catalyst on fuel product composition. The results obtained from this study would be used to find optimum condition for various iron and cobalt ratio in the catalyst. The combination of polymerizing and cracking catalyst, should be subtracting the reactor in the industrial application.

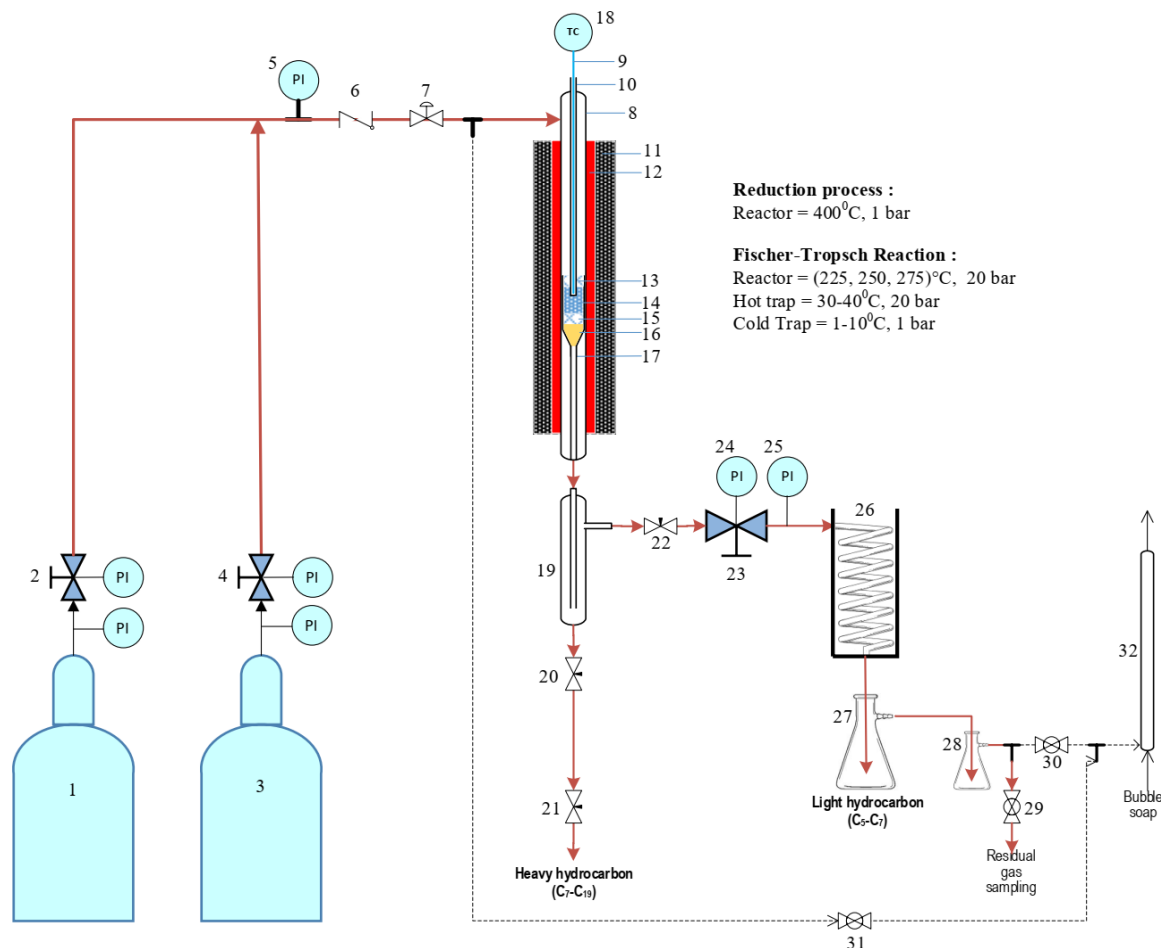
## 2. Materials and Methods

The equipment used were syringe, digital scales, hotplate magnetic stirrer, vacuum filter, tubular furnace, quartz furnace crucible, air compressor, hot air oven, and standard glassware for catalyst synthesis. The continuous fixed-bed Fischer-Tropsch reactor (**Fig. 1**), hot trap, cold trap, bubble soap meter, vacutainer (vacuum container) were used for reaction.

The Fe-Co/meso-HZSM-5 catalyst was prepared from ammonium ZSM-5 (CBV 8014, Amberlyst International) over calcination, desilication with NaOH (Merck), dispersion, nitric acid (Merck) treatment [7]. The mixed solution consisted of Co(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O (Merck) and Fe(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O (Merck) were used as precursor for incipient wetness impregnation (IWI) process on meso-HZSM-5. Loading iron-cobalt catalyst on meso-HZSM-5 was 10 % (w/w). The catalyst consisted of 10–40 % Fe (by weight) in Fe-Co catalyst. The impregnated HZSM-5 was kept in desiccator at room temperature for 24 hours, dried in oven at 120 °C for 12 hours and then calcined at 550 °C for 2 hours.

The catalyst performance was observed in a continuous fixed bed reactor (**Fig. 1**) using 1 gram Fe-Co/meso-HZSM-5 catalyst, 25 mL/min synthesis gas (15 % CO, 30 % H<sub>2</sub>, 55 % N<sub>2</sub>) at various composition iron and cobalt ratio (10Fe-90Co/meso-HZSM-5, 20Fe-80Co/meso-HZSM-5, 30Fe-70Co/meso-HZSM-5, 40Fe-60Co/meso-HZSM-5), various temperature (225 °C, 250 °C, 275 °C) at 20 bars. The catalyst was mixed with 2 mm glass beads (Marienfeld) as much as 6 times of the catalyst weight, inserted into the catalyst bowl which was held with glass wool at the bottom and top of the catalyst to prevent the spilling out of the catalyst during process [19, 20]. All catalysts were reduced in situ in the reactor with 25 mL/min flowing hydrogen (PT. Samator Gas Industry, Surabaya, Indonesia), 1 bar, 400 °C for 10 hours. The reactor was cooled to reaction temperature and the hydrogen gas flow was stopped, then it was replaced by the synthesis gas flow (PT. Samator Gas Industry, Surabaya, Indonesia). The modified pressure regulator was installed after the reactor to maintain 20 bars pressure. The flow rate was calibrated using bubble soap meter at 25 mL/min at the operating pressure and temperature. Some hydrocarbon fractions were condensed through hot trap at 30–40 °C and 20 bars. The lower hydrocarbon would be condensed in a cold trap (1–10 °C, 1 bar).

The reaction was carried out for 120 hours with gas sampling every 24 hours and liquid sampling at the end of the whole reaction. Gas samples were taken using a 10 mL syringe and injected into 10 mL vacutainer.



**Caption :**

- |   |  |
|---|--|
| 1. Hydrogen gas (H <sub>2</sub> )                                   | 17. Catalyst bowl                        |
| 2. Pressure regulator for H <sub>2</sub>                            | 18. Temperature panel controller         |
| 3. Synthesis gas (15% CO, 30% H <sub>2</sub> , 55% N <sub>2</sub> ) | 19. Hot trap                             |
| 4. Pressure regulator for synthesis gas                             | 20. Needle valve I (Swagelok)            |
| 5. Pressure gauge   | 21. Needle valve II (Swagelok)           |
| 6. Check valve  | 22. Ball valve I (Swagelok)              |
| 7. Micro metering valve   | 23. Pressure Regulator (modified)        |
| 8. Fischer-Tropsch reactor  | 24. Pressure gauge for reactor outlet    |
| 9. Temperature probe sensor (type K)                                | 25. Pressure gauge for condenser         |
| 10. Thermo well   | 26. Condenser (cold trap)                |
| 11. Heat Isolator (rockwool)  | 27. Separator for liquid and gas product |
| 12. Electric Heater   | 28. Bubble gas indicator                 |
| 13. Glass beads   | 29. Ball valve II (Sankyo)               |
| 14. 10Fe-90Co/mesoHZSM-5 catalyst                                   | 30. Ball valve III (Sankyo)              |
| 15. Glass beads   | 31. Needle valve III (Swagelok)          |
| 16. Glass wool  | 32. Bubble soap meter                    |

**Fig. 1.** The reactor unit for continuous Fischer-Tropsch synthesis for Fe-Co/meso-HZSM-5 catalyst using synthesis gas (15 % CO, 30 % H<sub>2</sub>, 55 % N<sub>2</sub>), 25 mL/min, 225–275 °C, 20 bars, 120 hours on various catalyst composition (10–40 % Fe in Fe-Co)

Brunauer-Emmett-Teller (BET), X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) with Energy Dispersive X-ray spectroscopy (EDX) analysis were used for catalyst characterization. Gas Chromatography Mass Spectrum (GCMS) measured hydrocarbon composition

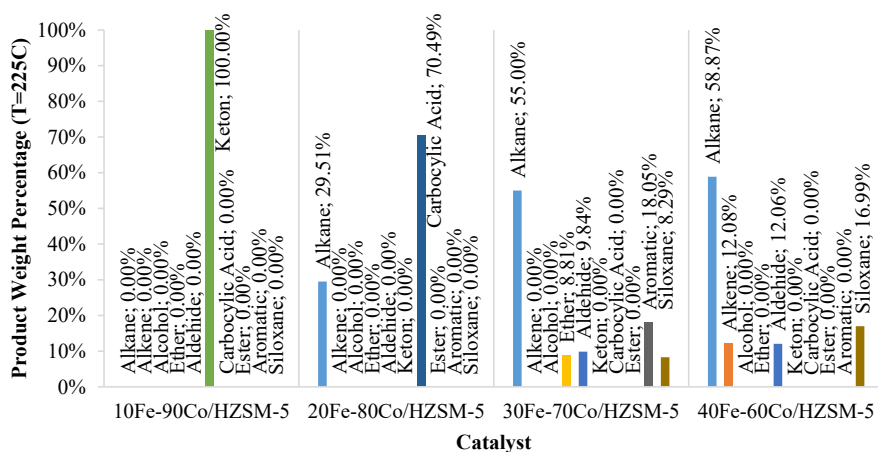
of liquid product. Gas Chromatography with Thermal Conductivity Detector (GC-TCD) measured the exit gas composition.

### 3. Results and discussion

Ammonium ZSM-5 (Si/Al=40) was converted to microporous HZSM-5 through calcination process in the tube furnace reactor. The formation of mesoporous structures was obtained through the desilication process in which silicon was partly-removed from zeolite framework using NaOH as desilication agent. The desilicated HZSM-5 properties obtained from BET analysis were 6.1–29.9 nm mesoporous diameter, 0.3496 cc/g average mesoporous volume, 526.035 cc/g pore surface area, and from EDX analysis gave 22.1059 Si/Al ratio.

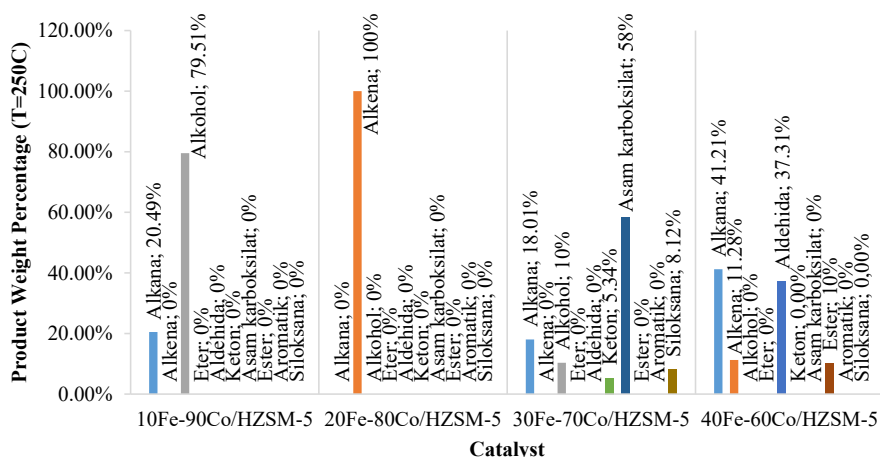
The previous studies [15, 17, 21] applied Fe-Co in FTS which produced the best products at temperatures of 220–270 °C. The cobalt weight was greater than Fe, so it could be estimated that the reaction should run well between LTFT (200–240 °C) and HTFT (300–350 °C) optimal temperature range. Because of the iron composition was varied from 10–40 % (w/w), the temperature was set slightly above LTFT (225–275 °C).

The liquid product was analyzed using GCMS to measure the hydrocarbon compounds produced. The effect of catalyst and temperature variations on the weight percentage of the liquid product was presented in a bars chart for each functional group. **Fig. 2** showed the liquid product distribution from Fischer-Tropsch reaction at 225 °C. The 10Fe-90Co/meso-HZSM-5 catalyst gave ketone products only. The alkane compounds (29.5 %) were formed on the 20Fe-80Co/meso-HZSM-5 catalyst, 30Fe-70Co/meso-HZSM-5 (55.0 %), 40Fe-60Co/meso-HZSM-5 (58.8 %). The more iron in the catalyst formed the more alkane compounds in the liquid product. Alkane were the main product desired in this biofuel production. The 20Fe-80Co/meso-HZSM-5 catalyst produced a carboxylic acid as by-product, while the others produced more varied by-product with a total amount below 50 %. The 30Fe-70Co/meso-HZSM-5 catalyst gave ether, aldehyde, aromatic compounds and siloxane as by-products. The 40Fe-60Co/meso-HZSM-5 catalyst gave alkenes and aldehydes as by-products. At 225 °C, the more Fe in the catalyst (40Fe-60Co/meso-HZSM-5) produced more alkanes (58.8 %) and alkenes (12.08 %) than other catalysts.



**Fig. 2.** The effect of iron and cobalt composition (10Fe-90Co, 20Fe-80Co, 30Fe-70Co, 40Fe-60Co) supported by HZSM-5 mesopore (1 gram catalyst weight) in FTS reaction (225 °C, 20 bars, synthetic gas flow rate 25 mL/min, 120 hours) to mass percentage of product (%w/w)

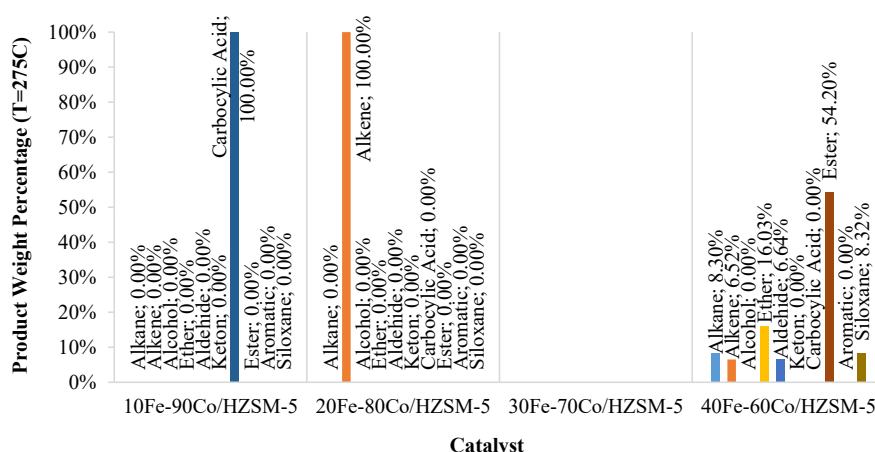
The liquid product composition from the Fischer-Tropsch reaction carried out at 250 °C can be seen in **Fig. 3**. The 10Fe-90Co/mesoHZSM-5 catalyst produced alkanes only (20.4 %) as the main reaction product and alcohol (79.51 %) as a by-product. Alkanes are also formed on the 30Fe-70Co/mesoHZSM-5 catalyst (18.01 %), 40Fe-60Co/mesoHZSM-5 catalyst (41.2 %). In this condition, the catalyst containing the most iron (40Fe-60Co/mesoHZSM-5) produced the best performance which was indicated by the highest number of alkane products compared to other catalysts.



**Fig. 3.** The effect of iron and cobalt composition (10Fe-90Co, 20Fe-80Co, 30Fe-70Co, 40Fe-60Co) supported by HZSM-5 mesopore (1 gram catalyst weight) in FTS reaction (250 °C, 20 bars, synthetic gas flow rate 25 mL/min, 120 hours) to mass percentage of product (%w/w)

The liquid product composition from the Fischer-Tropsch reaction carried out at 275 °C was showed on **Fig. 4**. Product composition data for the catalyst 30Fe-70Co/mesoHZSM-5 were not available because the sample was damaged during shipping. Under these conditions, only very small amounts of alkanes are formed in the Fischer-Tropsch reaction using the 40Fe-60Co/mesoHZSM-5 catalyst. Esters, the cracking reaction products had the highest composition compared to other cracking products such as ethers, aldehydes and siloxane. This catalyst gave better performance than other catalysts. The 10Fe-90Co/mesoHZSM-5 catalyst only produced carboxylic acid which was a cracking product. They indicated that the entire compound resulting from the Fischer-Tropsch reaction was cracked into carboxylic acids. The 20Fe-80Co/mesoHZSM-5 catalyst only produced alkenes which were one of the main Fischer-Tropsch reactions product. The best catalyst in this condition was 40Fe-60Co/mesoHZSM-5 with 8.30 % alkane and 6.52 % alkene product. The paraffins (alkanes) and olefins (alkenes) formation was the main reaction that occurs in the Fischer-Tropsch reaction. Long chain hydrocarbon compounds (wax) were not found after 120 hours of reaction. This indicated that the entire wax produced during the polymerization process was successfully cracked into short chain hydrocarbon compounds. The main FTS reactions produced the main liquid products of alkanes and alkenes, while the side reactions produced alcohols. The oxygenated compounds such as ethers, ketones, carboxylic acids, esters and siloxane were found as other by-product compounds produced from the cracking process. The Fischer-Tropsch reaction carried out at a temperature of 225 °C produced the most alkane products compared to the other temperature variations. The temperature increasing in the Fischer-Tropsch reaction would increase the hydrogenated compounds formation, thereby increasing the side products formation such as ketones and aromatics. The Co catalyst as hydrogenation catalyst was more active and running optimally at low temperatures (LTFT). The synthesis gas composition affected the selectivity of the product. The use of synthesis gas with 15 % mole CO reduced the surface coverage by CH<sub>2</sub> species, thereby reducing the chain growth possibility and higher desorption of CH<sub>2</sub> species. They were one of the reasons for the relatively low availability of alkanes as the main product in almost all catalysts. Each catalyst has a different character at increasing temperature.

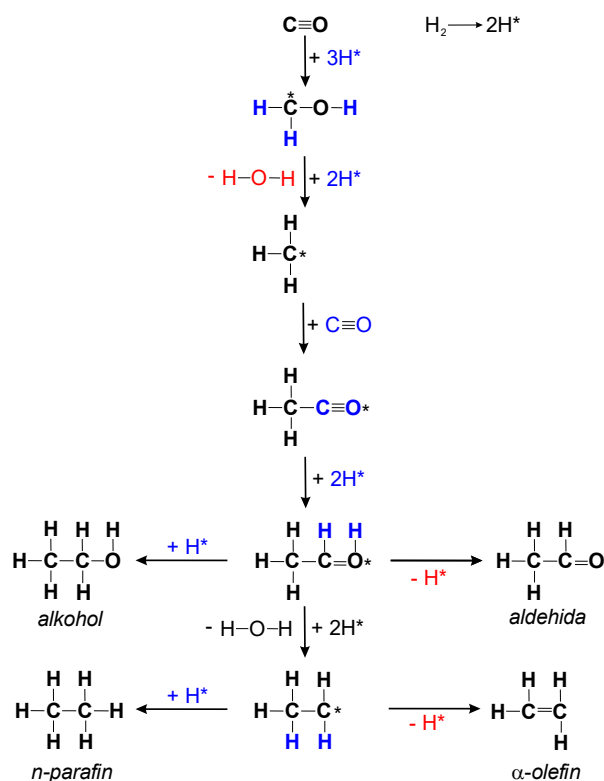
The 10Fe-90Co/mesoHZSM-5 catalyst was more suitable for FTS at 250°C with alkane (20.49 %) as the main product and alcohol as the by-product (79.51 %). Alcohol had high economic value so this reaction could be used as an alternative route for alcohol production. The catalysts of 20Fe-80Co/mesoHZSM-5, 30Fe-70Co/mesoHZSM-5, 40Fe-60Co/mesoHZSM-5 were more suitable for FTS at 225–250 °C because under these conditions, alkanes as the main product were obtained in relatively higher compositions compared to other compounds. The higher cobalt catalyst content in all catalysts could operate better at low temperatures. The tendency of Co activity to form olefins could be suppressed by the combination of Fe in a smaller composition. The olefins were almost not found in every variation at 225–250 °C.



**Fig. 4.** The effect of iron and cobalt composition (10Fe-90Co, 20Fe-80Co, 30Fe-70Co, 40Fe-60Co) supported by HZSM-5 mesopore (1 gram catalyst weight) in FTS reaction (275 °C, 20 bars, synthetic gas flow rate 25 mL/min, 120 hours) to mass percentage of product (%w/w)

The use of Fe-Co/HZSM-5 lead to the growth of long hydrocarbon chains (wax) which were cracked by HZSM-5 when desorbed out of the catalyst. If the reaction occurred in the pore, the wax would be trapped in the pore and could only come out when it had been cracked into smaller molecules.

Based on the resulting product variation, the FTS reaction followed the dissociation of CO with the hydrogen assisted and tend to lead to a CO-insertion mechanism. The mechanism of paraffins, olefins, aldehydes and alcohols formation had been showed at **Fig. 5**.



**Fig. 5.** The mechanism of paraffins, olefins, aldehydes and alcohols formation in FTS reaction (225–275 °C, 20 bars, synthetic gas flow rate 25 mL/min, 120 hours) followed the hydrogen assisted CO dissociation with CO-insertion mechanism

These results could be reached with procedure, operating conditions (temperature, pressure, time), materials and catalyst specification, equipments specification and that were explained in the materials and methods above. To improve the results obtained, further research is needed with the target of increasing the yield of short chain alkanes which are the desired biofuels.

The combination of a metal catalyst Fe-Co (polymerization) and mesoporous HZSM-5 zeolite (cracking) in one reactor could be applied to the biofuel industry which used the Fischer-Tropsch process. The currently Fischer-Tropsch plants still use two separated reactors.

#### 4. Conclusions

Fischer-Tropsch synthesis on synthesis gas (15 % CO, 30 % H<sub>2</sub>, 55 % N<sub>2</sub>) with various catalysts (10Fe-90Co, 20Fe-80Co, 30Fe-70Co, 40Fe-60Co)/mesoHZSM-5 and reaction temperature (225 °C, 250 °C, 275 °C) at 20 bars, synthetic gas flow rate 25 mL/min, 120 hours produced liquid fractions of alkanes, alkenes, alcohols, ethers, aldehydes, ketones, carboxylic acids, esters, aromatics and siloxane. The 10Fe-90Co/mesoHZSM-5 catalyst was more suitable for FTS at 250 °C with alkane (20.49 %) as the main product and alcohol as the by-product (79.51 %). Alcohol had high economic value so this reaction could be used as an alternative route for alcohol production. The catalysts of 20Fe-80Co/mesoHZSM-5, 30Fe-70Co/mesoHZSM-5, 40Fe-60Co/mesoHZSM-5 were more suitable for FTS at 225–250 °C because under these conditions, alkanes as the main product were obtained in relatively higher compositions compared to other compounds. The mechanism of paraffins, olefins, aldehydes and alcohols formation in this FTS reaction followed the hydrogen assisted CO dissociation with CO-insertion mechanism.

#### Conflict of interest

The authors declare that they have no conflict of interest in relation to this research, whether financial, personal, authorship or otherwise, that could affect the research and its results presented in this paper.

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#### Data availability

Data will be made available on reasonable request.

#### Use of artificial intelligence

The authors confirm that they did not use artificial intelligence technologies when creating the current work.

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