



Heterogeneous Catalyst HZSM5 in Biodiesel Production from Rapeseed Oil in Batch Process

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ABSTRACT

In this paper, HZSM5 zeolite was synthesized through reflux method on support material CaO (25, 35 and 45 wt%) in two specific methods: microwave and impregnation at high temperature. The zeolite catalyst was modified with impregnation of NaOH (2, 4, 8, 12 wt%) at room temperature. The modified zeolite was used in transesterification of rapeseed oil with methanol in a batch catalytic process. In transesterification of rapeseed oil, the catalytic activities of HZSM5, NaZSM5, KZSM5 were considered. The prepared catalysts were characterized by several techniques such as X-ray diffraction (XRD), Brunauer Emmett Teller (BET) surface area and also the surface image was scanned by scanning electron microscopy (SEM). The parameters affecting on biodiesel yield at optimum reaction conditions were investigated. The maximum yield was achieved with 8wt% of NaOH loaded on HZSM5 at reaction temperature of 65°C, reaction time of 12 hours and catalyst/oil mass ratio of 9. Also the yield of CaO loaded with impregnation at high temperature was more desired than CaO loaded with microwave. Meanwhile the catalytic activity of HZSM5, NaZSM5 and KZSM5 was nearly zero; and the catalytic activity of modified zeolite was HZSM5>NaZSM5>KZSM5 subsequently.

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INTRODUCTION

Fossil fuels reduction caused human concern and high attention of them to replace another source of energy instead of fossil fuels in the last decades [1, 2]. Some source of energy such as wind, solar, water and biomass can use instead of fossil fuels. Biodiesel is one of the best substitution of fossil fuel that many of scientists are studying on it. Some of biodiesel traits such as: renewability, non-toxic, oxygenated and environmental friendly [3], biodegradability [4], higher lubricity [5], stability [6], lower emissions of greenhouse gases [7, 8], higher combustion efficiency, similar physiochemical properties to fossil fuels make it good replacement for fossil fuels [6, 9].

Biodiesel is mono-alkyl ester derived from waste cooking oils, vegetable oils or animal fats in transesterification process of alcohols with lower molecular weight in presence of catalysts [6]. Figure 1 shows a simple reaction of transesterification that biodiesel product is on it.

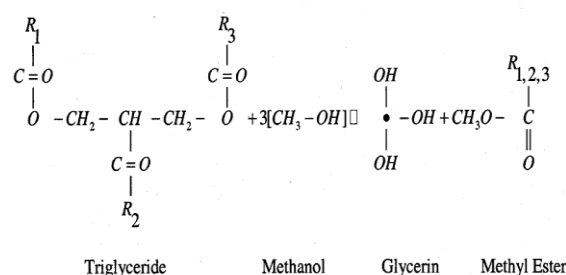


Figure 1. Transesterification process diagram

As you see in the shape three mole of alcohol react with one mole of triglyceride. The productions of reaction are glycerine and methyl ester. Methanol, ethanol, propanol and butanol can be used for alcohol source of reaction. But normally methanol has been used in industrial process because of lower cost and easy access. Catalysts used in this process are classified

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to heterogeneous (acid and base) and homogeneous (acid and base). High reaction rate, mild pressure and temperature condition, cheap and widely available are advantages of homogeneous base catalysts. NaOH and KOH are two examples of this kind of catalysts [10, 11]. Being sensitive to free fatty acid content of oil, product soap formed and high costs of separation and purification of it are disadvantages of these kind of catalysts [12]. Being insensitive to free fatty acid and water content of the oil, mild temperature and pressure reaction condition are advantages of homogeneous acid catalysts. On the other hand very slow rate of reaction, causing corrosion on reactor and pipeline and product separation problems from catalysts are disadvantages of these family of catalysts. [13, 14] Despite generally homogeneous catalysts have used in commercial activity up to now, but the problems that mentioned above caused researchers trying to find another catalysts to replace in order to omit these problems and also reuse catalysts [15].

Heterogeneous acid catalysts are kind of catalyst that scientists consider different parameters to replace of these catalysts instead of homogeneous catalysts. The advantages of these catalysts are: being insensitive to free fatty acid content of oil [12], omit the washing step of product [16], easy separation of catalyst from product, easy regenerating and recycling of catalyst, reduce corrosion problems [13]. Another group of catalysts that have developed for transesterification reaction is heterogeneous base catalysts. Higher rate in compare with acid-catalyzed transesterification, mild temperature and pressure reaction condition, easy separation and purification of catalyst from product and high possibility to reuse and regenerate of the catalyst are advantages of this group of catalysts [12]. Also low solubility in methanol and high basic strength make the quality of them higher [17, 18].

Zirconium oxide² [19], Titanium Oxide³ [20], Tin Oxide⁴ [21], Zeolites and etc are classified in heterogeneous catalysts that showed mentionable yield in transesterification reaction [12]. Zeolites are microporous crystalline solids that silicon (Si) and alumina (Al) constituted basic part of zeolite structure [22]. Shape selectivity, having characteristics of acidic sites, vary in pore structure, accommodating a wide variety of cations, having high surface area and specific surface; in which some of traits are reported [1].

NaX, NaY, ZSM5, ZSM11, Mordenite, fauja site, Beta etc are couple of zeolites that used in different reactions. Different kind of zeolites have used in transesterification of variety oils as catalyst [23]. Xie et

al. [24] used NaX zeolite with 23 ratio of Si/Al in transesterification of soybean oil after modifying it by 10% KOH loading on it and reported 85.6% conversion. Ramos et al. [25] considered zeolites (mordenite, Beta and X) catalytic performance on transesterification of sunflower oil and reported 93.5 - 95.1% yield at 60 °C reaction temperature. Haitang wu et al. [26] used zeolites (NaY, KL and NaZSM5) as a catalyst in transesterification of soybean oil after modifying them with variety amount of Calcium Oxide⁵ and reported 95% yield in presence of NaY with 30% loading of CaO on it. M Kim et al. [27] considered the effect of sodium on catalytic activity of ZnO-Al₂O₃/ZSM5 and SnO-Al₂O₃/ZSM5 for transesterification of vegetable oil with methanol and reported desired results.

In this paper HZSM5 with 15 Si/Al ratios synthesized with the method that A. Robert [28] patented it on 1972, then modified with different amount of CaO loading with couple of methods (impregnation and microwave irradiation) and modified with variety amount of NaOH with impregnation method and used as catalyst in transesterification of rapeseed oil in presence of methanol.

MATERIAL AND METHODS

Materials

Edible rapeseed oil acquired from Ladan company. The composition of rapeseed fatty acid consist of palmitic acid 4.7, stearic acid 1.9, oleic acid 69.8, linoleic acid 17.4% according to the manufacturer information. Tetraethylorthosilicate⁶ of Aldrich company as a silica source of HZSM5, Tetrapropylammoniumhydroxide⁷ of merck company as template of zeolite, aluminium isopropoxide⁸ as alumina source of HZSM5, Calcium carbonate⁹ and Sodium hydroxide¹⁰, Sodium chloride and Potassium chloride of merck company in order to modifying of catalyst, and methanol 99.99% purity for alcohol source of reaction.

Catalyst preparation

HZSM5 zeolite was synthesized with a suitable blend of water, TEOS, TPAOH, Al-ISO hydrothermally [28]. The molar composition of the gel mixture was found to be 29.16SiO₂:1Al₂O₃:11.436TPAOH:480.84H₂O. Also the ratio of Si/Al found to be 14.58. In order to modification of the zeolite three methods was used. Before modification of the zeolite CaCO₃ was calcined at 900 °C for 3hr in order to achieve CaO.

² ZrO₂

³ TiO₂

⁴ SnO₂

⁵ CaO

⁶ TEOS

⁷ TPAOH

⁸ Al-ISO

⁹ CaCO₃

¹⁰ NaOH

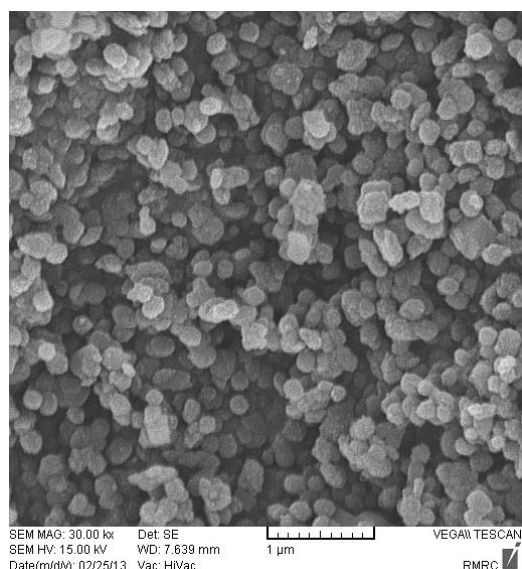


Figure 2. SEM analysis of HZSM5

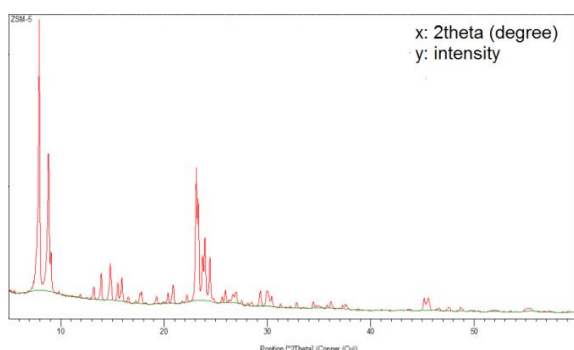


Figure 3. XRD pattern of synthesized HZSM5

Modification that pointed above was consisted of:

- I) Different amount of CaO (25, 35, 45, 55 wt%) was grinded for 15min in a mortar and titrated with certain amount of water, then an obtained mixture was irradiated by microwave for 30min in the microwave oven (the power of microwave oven was 900w and in this work 50% of power was used.). The solid of the mixture was separated with centrifuge and keep the catalyst at 120 °C over night subsequently. The CaO/HZSM5 obtained sample was calcined at 550 °C for 3 hr in order to activate the catalyst and using it in transesterification of rapeseed oil.
- II) Different amount of CaO (25, 35, 45, 55 wt%) proportion with zeolite added to distillate water and impregnated for 24hr at 60-70 °C in reflux method. The solid of the mixture was separated with centrifuge and keep the catalyst at 120 °C over night subsequently. The CaO/HZSM5 obtained sample was calcined at 550 °C for 3 hr in order to activate the catalyst and using it in transesterification of rapeseed oil.

- III) Different amount of NaOH (2, 4, 8, 12 wt%) proportion with zeolite added to distillate water and impregnated for 24hr at room temperature. The obtained solid was separated with filter paper and then keep at 120 °C over night subsequently. The NaOH/HZSM5 obtained sample was calcined at 550 °C for 3 hr in order to activate the catalyst and using it in transesterification of rapeseed oil.
- IV) HZSM5 was impregnated for 24hours at room temperature with different concentration of NaCl (0.5, 1, 2mol/liter) and KCl (0.5, 1, 2 mol/liter) in order to exchange H with Na and K and compare catalytic activity of HZSM5, KZSM5 and NaZSM5 with each other. The obtained solid was dried and calcined as same as last sections.

All of the modified catalysts should be kept in desimLators before using them in the reaction.

Catalyst Characterization

In order to characterize the composition and structure of any catalyst with the best result, several techniques were used. Powder X-ray diffraction (XRD) was chosen for phase composition identification by using Cu K α radiation. Scanning electron microscopy (SEM) was used to characterize the microstructure of the catalyst. The Brunauer-Emmett-Teller (BET) was used to measure pore size and surface area by multipoint N $_2$ adsorption-desorption method at -196 °C (liquid nitrogen temperature).

Transesterification procedure and methods of product analysis

In this paper all experiments were performed in a 250 mL Erlenmeyer flask. In any step 20 gram of rapeseed oil, variety of methanol/oil ratio and different amount of catalyst were used and the reaction was done in Erlenmeyer flask as batch reactor. Magnetic stirrer was used to agitating the mixture and a water cooled condenser was used in order to get the vaporized methanol back to the reactor. In all experiments the magnet velocity was constant at 800 rpm.

In any step, the oil kept half an hour in the bass water at the reaction temperature until the oil temperature was equaled to reaction temperature and catalyst and methanol added to reaction sphere together subsequently. Also all reaction was done at atmospheric pressure. After the reaction time was finished, the mixture was centrifuged to separate the solid catalyst. The upper layer of centrifuged product was kept at 84 °C for half an hour in order to evaporate the methanol of production. The yield of reaction was obtained by using GC gas chromatograph amLording to follow equation [29]:

$$C = \frac{\sum A - A_{IS}}{A_{IS}} \times \frac{C_{IS} \times V_{IS}}{m} \times 100\% \quad (1)$$

where:

ΣA = total peak area $C_{14:0} - C_{24:1}$

A_{IS} = internal standard (methyl pentadecanoate) peak area

C_{IS} =concentration of the internal standard solution, in mg/mL

V_{IS} = volume of internal standard solution used, mL

M = mass of the sample, in mg.

RESULTS AND DISCUSSION

Effect of different zeolite on biodiesel yield

HZSM5 impregnated with different concentration of NaCl (0.5, 1, 2 mol/liter) and KCl (0.5, 1, 2 mol/liter) for 24h at room temperature in order to exchange H with Na and K and any of catalysts (HZSM5, KZSM5, NaZSM5) used as catalyst in transesterification of rapeseed, subsequently. The parameters affecting on biodiesel yield have done with methanol/oil ratio of 12 for the reaction time of 24 hours at reaction temperature of 65°C, 75 °C and 90 °C and catalyst/oil mass ratio of 3. But the yield of reaction was zero for all processes.

Effect of CaO loading with microwave irradiation method on biodiesel yield

Variety of CaO loading (25, 35, 45, 55 wt%) proportion with HZSM5 was supported on it as instruction (I) mentioned above. The parameters affecting on biodiesel yield have investigated to optimize the reaction conditions. The maximum yield was achieved with 35wt% of CaO loaded on HZSM5, with methanol/oil ratio of 12 for the reaction time of 8 hours at reaction temperature of 65°C and catalyst/oil mass ratio of 10. Figures 3-6 show results of microwave irradiation method.3.3. Effect of CaO loading with impregnation method on biodiesel yield.

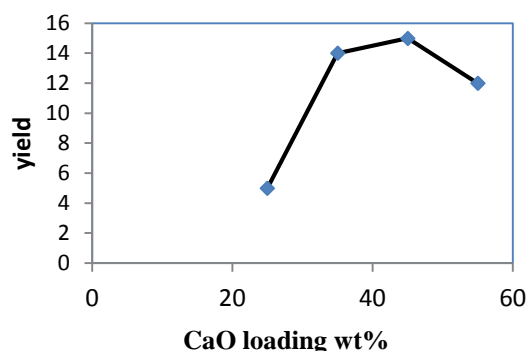


Figure 2. Effect of CaO loading on the yield of process. Reaction condition: methanol/oil molar ratio:12:1, catalyst amount:3wt%, reaction temperature: 65°C, reaction time: 5h.

Different amount of CaO impregnated with HZSM5 as the method number (II) mentioned earlier.

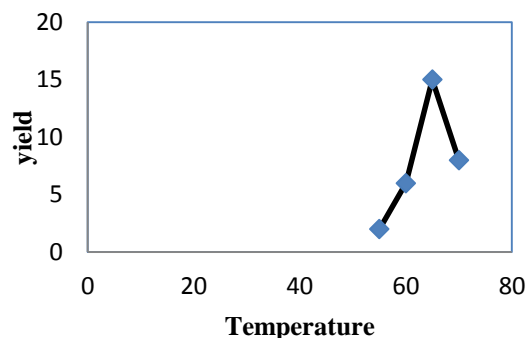


Figure 4. Effect of temperature on the yield of process. Reaction condition: time:5h, catalyst amount: 3wt%, methanol/oil molar ratio: 12:1, CaO loading: 35wt%.

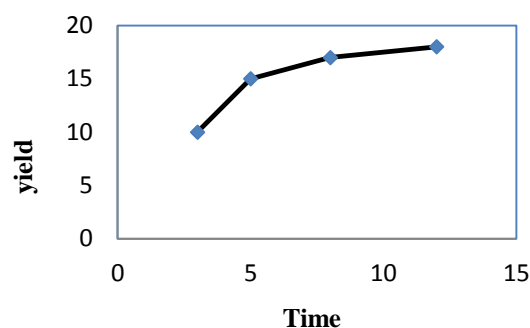


Figure 5. Effect of time on the yield of process. Reaction condition: temperature: 65°C, catalyst amount: 3wt%, methanol/oil molar ratio: 12:1, CaO loading: 35wt%.

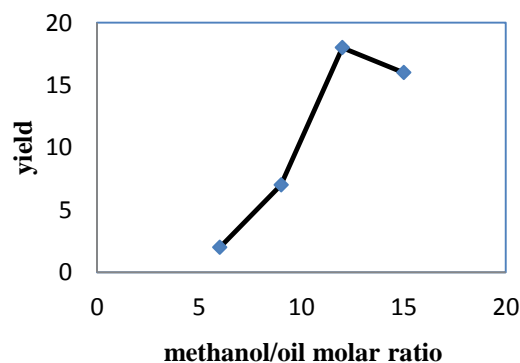


Figure 6. Effect of methanol/oil molar ratio on the yield of process. Reaction condition: time: 8h, catalyst amount: 3wt%, CaO loading: 35wt%, temperature: 65°C.

The experiments of this section was done according to optimized parameters of section 1.3. Also for comparing of HZSM5, NaZSM5 and KZSM5 catalytic activity, before doing CaO impregnation, HZSM5 was impregnated with different concentration of KCL and NaCL according to method (IV) and then impregnated with CaO as the same way of method number (II). Despite it was predicted NaZSM5/CaO has the highest catalytic activity, but the sequence of catalytic activity is:

HZSM5>NaZSM5>KZSM5. Also the best yield of KZSM5 and it is predictable if NaZSM5 is synthesized with NaOH basically, the yield of process will be better. All reaction of this section done at reaction condition of: time:8h, CaO loading: 35wt%, temperature: 65 °C, molar ratio of methanol/oil: 12:1, catalyst amount 10wt%. So the achievement yield in presence of 35wt% CaO/HZSM5, 35wt% CaO/NaZSM5 and 35wt% CaOKZSM5 was 57, 25 and 8% subsequently.

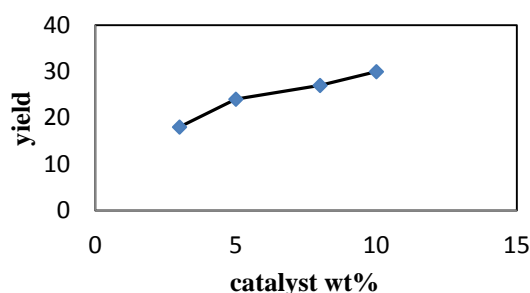


Figure 7. Effect of amount of catalyst on the yield of process. Reaction condition: time:8h, temperature 65°C, CaO loading: 35wt%, methanol/oil molar ratio: 12:1.

Effect of NaOH loading with impregnation method on biodiesel yield

Different amount of NaOH (2, 4, 8, 12 wt%) proportion to HZSM5 was impregnated with zeolite according to method number (III) and optimized different parameters affected on the reaction. The temperature was constant at 65 °C for all experiments of this section. The maximum yield was achieved with 8wt% NaOH loaded on HZSM5, with methanol/oil ratio of 9 for the reaction time of 12 hours at reaction temperature of 65°C and catalyst/oil mass ratio of 10.

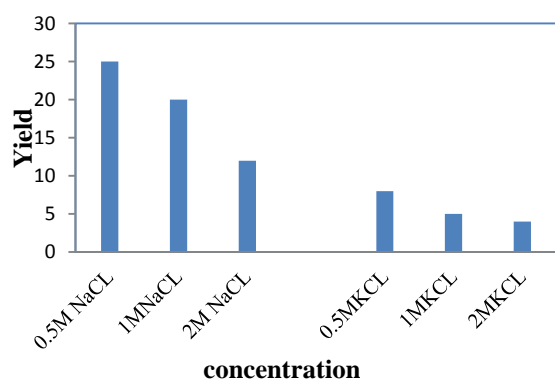
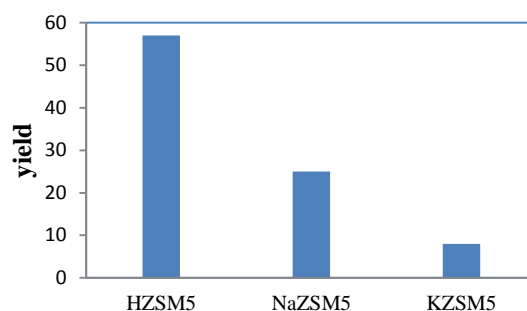


Figure 8. Effect of ion exchange concentration on the process yield. Reaction condition: time:8h, temperature 65°C, CaO loading: 35wt%, methanol/oil molar ratio: 12:1, catalyst mass ratio: 10wt%.

Effect of solvent on biodiesel yield

Whereas oil does not solve in methanol, kind of solvent such as n-hexane can increase the yield of process. In order to raising the yield of process and considering the effect of solvent on the yield of process 2mL of methanol proportion of 5mL methanol used in presence of 8% wt NaOH/HZSM5 in reaction condition of: time:12h, molar ratio of methanol/oil:9, mass ratio of catalyst/oil: 10wt%, temperature: 65 °C. The achievement yield was 70.2%.



35wt% CaO loading on zeolite

Figure 9. Comparing different element activity. Reaction condition: time:8h, temperature 65°C, CaO loading: 35wt%, methanol/oil molar ratio: 12:1, catalyst mass ratio: 10wt%.

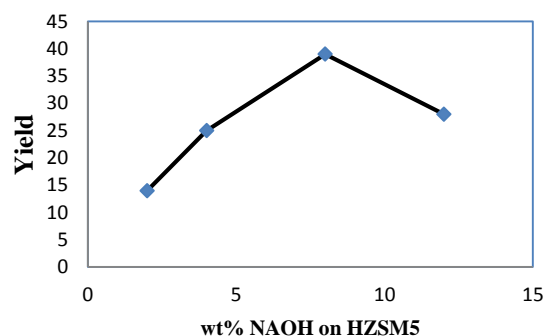


Figure 10. NaOH loading effect on the process yield. Reaction condition: time:8h, temperature 65°C, 35wt%, methanol/oil molar ratio: 12:1, catalyst mass ratio: 10wt%.

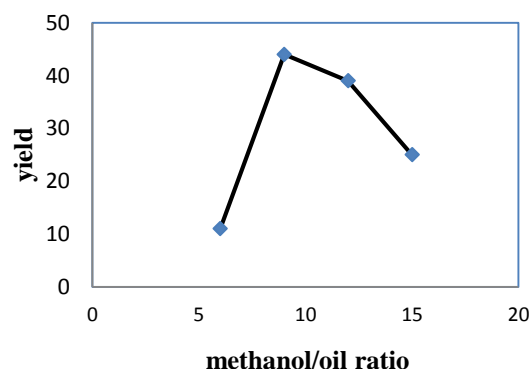


Figure 11. Methanol to oil molar ratio effect on the process yield. Reaction condition: time:8h, temperature 65°C, 8wt% NaOH loading on zeolite, catalyst mass ratio: 10wt%.

Reusability of catalyst

In order to considering reusability of catalyst after regenerating the NaOH/HZSM5 the catalyst catalytic activity was decreased sharply.

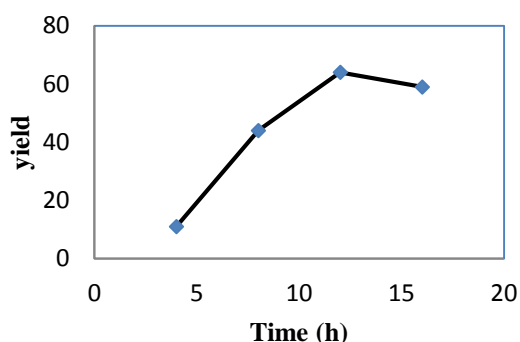


Figure 12. Time effect on the process yield. Reaction condition: methanol to oil molar ratio:9, temperature 65°C, 8wt%NaOH loading on zeolite,, catalyst mass ratio: 10wt%.

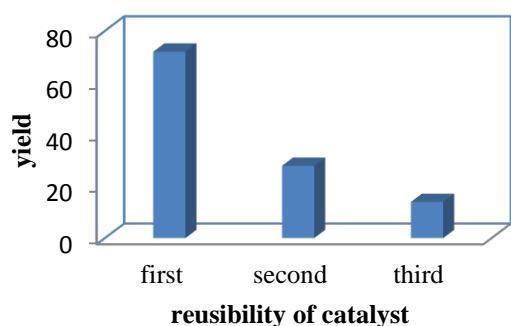


Figure 13. Considering catalyst reusability. Reaction condition: methanol to oil molar ratio:9, temperature 65°C, 8wt%NaOH loading on zeolite,, catalyst mass ratio: 10wt%, time:12h.

CONCLUSION

Among all variation 8%wt NaOH loading on HZSM5 has the best performance at temperature of 65 °C, methanol/oil molar ratio of 9, 12h reaction time, 10wt% catalyst mass ratio to oil. Also the HZSM5 modified with CaO at high temperature has higher yield from the modified zeolite with microwave. The highest yield achievement from CaO loading on HZSM5 was 57% that obtained at the reaction condition of: CaO loading:35wt% on HZSM5, time:8h, temperature:65 °C, methanol to oil molar ratio: 12 and catalyst mass ratio to oil: 10wt%. Replacing H with another element has negative effect on the yield of reaction. Yield reduction can be because of pore volume or surface activity

reduction in the middle of impregnation. But it is predictable if ZSM5 is being synthesized with NaOH basically the yield of reaction will be higher. The reason of that can be issue of more reaction activity of NA in compare with H. The observation show pure zeolite dose not have any effect on process yield and the yield of reaction in presence of HZSM5, NaZSM5 and KZSM5 was zero. Meanwhile it is predictable with raising catalyst amount in reaction the yield of reaction can be higher, but it is not suitable way do because the cost of process is not economic.

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چکیده

در این مقاله زئولیت HZSM5 با روش رفلکس بر روی پایه کلسیم اکسید (با درصد وزنی ۲۵، ۳۵ و ۴۵) با استفاده از دو روش مایکروویو و اشباع سازی در دمای بالا تولید شد. کاتالیست زئولیت با استفاده از اشباع سازی به وسیله سدیم هیدروکسید (با درصد وزنی ۲، ۴، ۸ و ۱۲) در دمای اتاق اصلاح شد. کاتالیست زئولیت اصلاح شده در واکنش تبادل استری روغن کلزا با متانول در یک فرایند ناپیوسته کاتالیستی مورد استفاده قرار گرفت. در تبادل استری روغن کلزا فعالیت کاتالیستی HZSM5، NaZSM5 و KZSM5 مورد بررسی قرار گرفت. ساختار کاتالیست تهیه شده با تکنیک های مختلفی نظیر XRD، BET و SEM سنجیده شد. پارامترهای تاثیرگذار در بازده تولید بیودیزل در شرایط بهینه واکنش بررسی شد. بیشترین بازده با HZSM5 اشباع شده با سدیم هیدروکسید ۸٪ وزنی، دمای واکنش ۶۵ درجه سانتیگراد، زمان واکنش ۱۲ ساعت و نسبت کاتالیست به جرم روغن ۹ به دست آمد. همچنین بازده تولید به وسیله کلسیم اکسید اشباع شده در دمای بالا در مقایسه با کلسیم اکسید اشباع شده به کمک مایکروویو افزایش یافت. در ضمن فعالیت کاتالیستی HZSM5، NaZSM5 و KZSM5 تقریباً برابر با صفر بود در حالی که فعالیت کاتالیستی زئولیت اصلاح شده $\text{HZSM5} > \text{NaZSM5} > \text{KZSM5}$ بود.
