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Bio-diesel fuel (BDF) synthesis by using solid catalyst based on ultrasonic irradiation

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Abstract. So far, a method of employing ultrasound irradiation for BDF synthesis has been suggested. However, since a large quantity of water is need to wash the BDF produced, it is very important to develop a new method based on the solid catalysis, by which water-washing is not necessary, At current paper, at first, several zeolite were prepared to increase their basic characteristics. Then the basic characteristics was characterized by using SEM and X-ray diffractometer. Beside, BDF synthesis experiment was carried out by using the selected zeolite based on ultrasonic irradiation. It was found that BDF yield ratio was higher when zeolite A5 was used.

1. Introduction

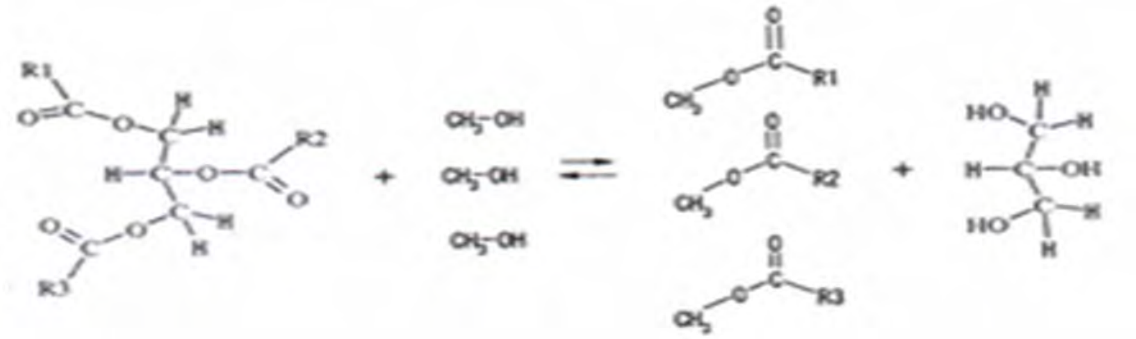
It is extremely important to promote using alternative fuel such as alcohol or BDF for automobile in the era when mankind is facing the crisis of global warming while at the same time crude oil price is soaring so high. Recently many literatures[1-6] have being published focusing on the methods of synthesizing biomass fuels and their applications.

BDF is a kind of energy that is clean and sustainable and can be used to automobiles of diesel engines. In EU and USA, with government's support and promotion, BDF used as B5 or B10 are officially recognized and gradually BDF's application has become wider and wider. However in Japan, only 100% BDF is permitted to be used on diesel automobiles. Also, since the production cost of BDF is higher than that of diesel fuel, reducing BDF's production cost is an urgent problem. In order to collect fundamental data for BDF standard establishment and make wide use of BDF in Japan, it is necessary to perform some basic studies on producing and analyzing and using of BDF.

So far, a method of employing ultrasound irradiation for BDF synthesis has been suggested. However, since a large quantity of water is need to wash the BDF produced, it is very important to develop a new method based on the solid catalysis, by which water-washing is not necessary, At current paper, at first, several zeolite were prepared to increase their basic characteristics. Then the basic characteristics was characterized by using SEM and X-ray diffractometer. Beside, BDF synthesis experiment was carried out by using the selected zeolite based on ultrasonic irradiation. It was found that BDF yield ratio was higher when zeolite A5 was used. Further, s numerical simulation based on Solidworks is carried out for flow-type ultrasonic reactor to investigate the influence of the temperature on the velocity distribution.

2. BDF synthesis principle

The zeolite-catalyzed transesterification equation for producing BDF is shown in Figure 1. When vegetable oil(triglyceride) is mixed with alcohol(methanol) under the condition of ultrasonic irradiation and zeolite after some time, BDF and the by-product of glycerin are yielded.



Vegetableoil (triglyceride) Alcohol(methanol) BDF Glycerin

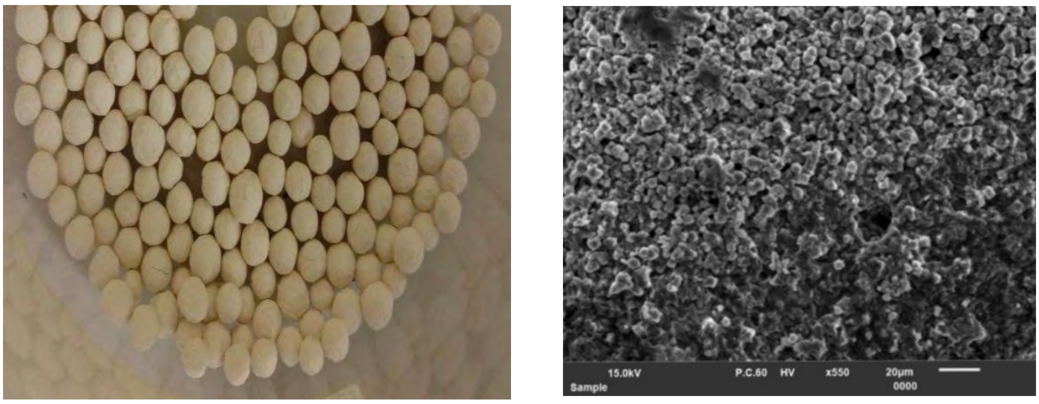
Figure 1. BDF synthesis principle

3. BDF synthesis experiment

3.1 Catalyst *preparation and evaluation*

The solid catalysts used for BDF synthesis in this paper are 5 kinds of zeolites (A3,A4,A5and F9) produced by Wako of Japan.

Figure 1 shows the size and shape of zeolite A5 and its SEM analysis result. A5 has the round shape whose average external diameter of A5 is around 5-7 mm. According to the SEM analysis result, the surface area of A5 is very large which could absorb large amount of liquid.



(a) Shape of zeolite A5 (b) SEM analysis result Figure 1. Shape of zeolite A5 and its SEM analysis.

Since base strengthen could properly influence the BDF yield ratio, before BDF synthesis, zeolite was prepared as follows:

Step 1: Soak samples A3,A4,5 and F9 with different NaOH concentration(3, 6 and 9 mol) for 24 hours Step 2: Wash the samples by using methanol and then dry the samples

Step 3: Sinter the samples by an electric furnace at the temperature of 400 ℃ for 2 hours

After zeolites weree prepared, an X-ray diffractometer is used to character their base strength characteristics.

3.2 BDF synthesis
During batch type BDF synthesis(Figure 2), firstly, the mixture of the reactants of vegetable oil and methanol is prepared inside a glass tube. Secondly, the power of the ultrasonic reactor of which water is switched on at frequency of 28 kHz. In order to decease BDF synthesis time, hot water is used too. Condition of BDF synthesis is listed in Table.1. The zeolites of A3,A4,A5 and F9 as catalyst were used respectively.

Table.1 BDF synthesis condition

Volume ratio of vegetable oil to methanol	5:1
Methanol purity	99.5%
Zeolite	A3,A4,A5 and F9
Quantity of Zeolite	0.33wt%

BDF components are detected by using a GC system. The sample of the BDF product is taken out every 20 min. The total BDF yield ratio is calculated based on the GC detection results.

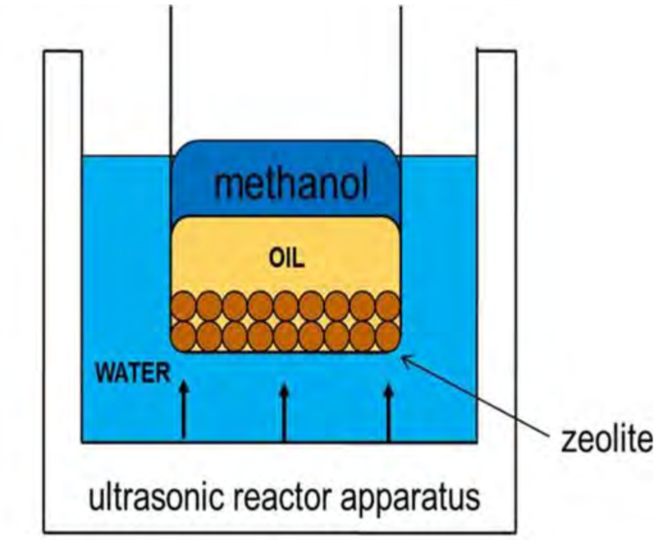


Figure 2. BDF synthesis by using batch method

3.2 Numerical simulations
Though a batch type BDF synthesis is used, according to our analysis, the flow type ultrasonic reactor will mostly increase the BDF yield ratio, at current paper, we suggest a flow U-type reactor inside which the zeolite is filled. The numerical model was made and analyzed by using Solidwrks. The main simulation conditions are listed in Table 2.

Table 2 Simulation conditions

Diameter of U type tuube	U type pipe Internal volume	Inlet Volume Flow	Reynolds number	Catalyst porosity	Liquids
20 mm	75 ml	5×10-5m^3/s	2326	0.7	oil

4. Results and discussions

4.1. Catalyst evaluation

Na^+ content change after zeolite preparation is shown in Figure 3 based on SEM analysis. As for surface investigation, the Na+ content increased if the high NaOH concentration is used. In case of A4, nd F9, the Na+ content are over 20%. In regard of inner investigation, the Na^+ content is lower than that of the surface because less NaOH is infiltrated in the inner of the zeolite.

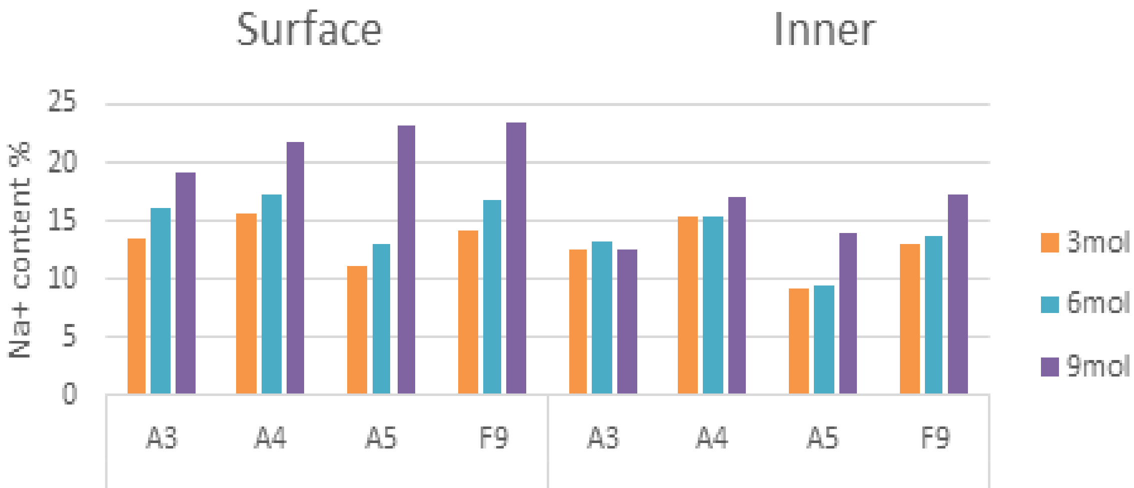


Figure 3. Na^+ content change after zeolite preparation

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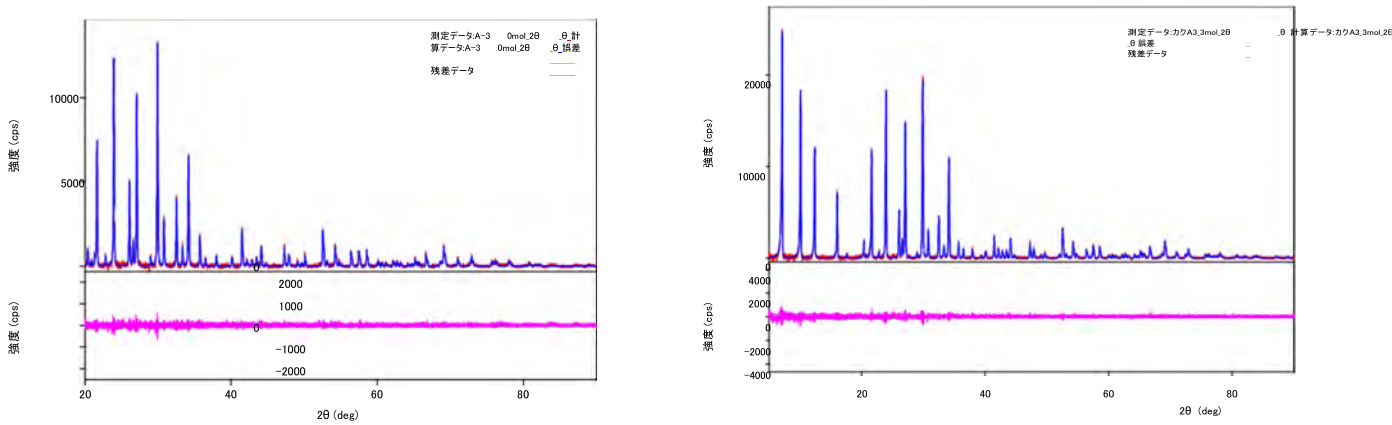
Analysis result based on X-ray diffractomete are shown in Figures 4-7 for zeolite A3, A4, A5 and F9.

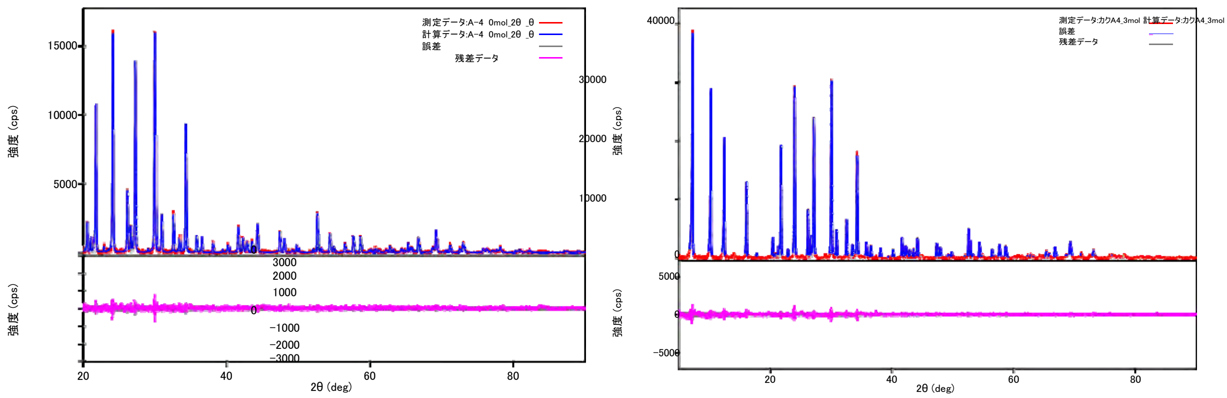


(a) Before preparation

(b) After preparation for 3mol

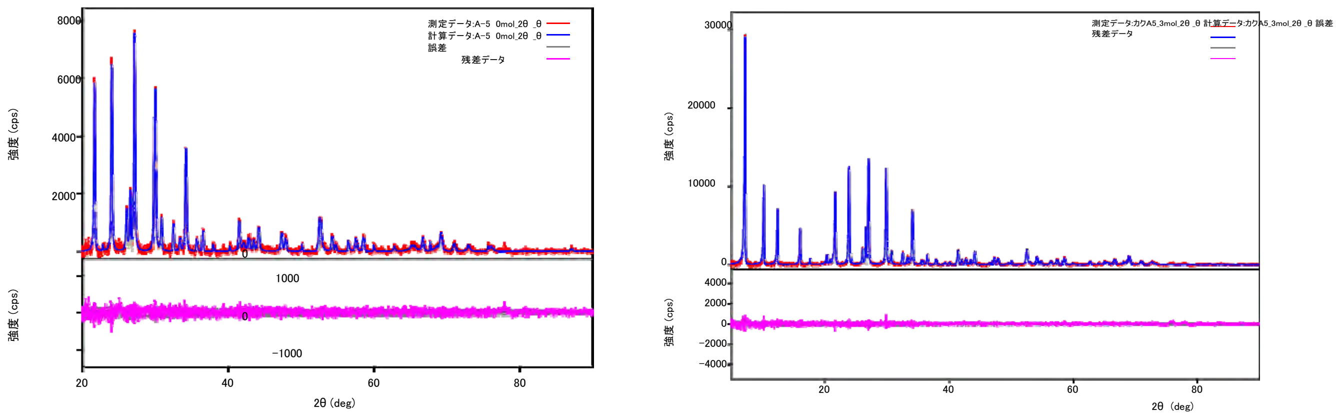
Figure 4. X-ray diffractomete result for A3

Before preparation, the X-ray diffractomete result is different because each zeolite has different structure. After preparation, the X-ray diffractomete result changed too because the soaked Na^+ content changed its structure to some extent., which could properly effect the BDF synthesis efficiency



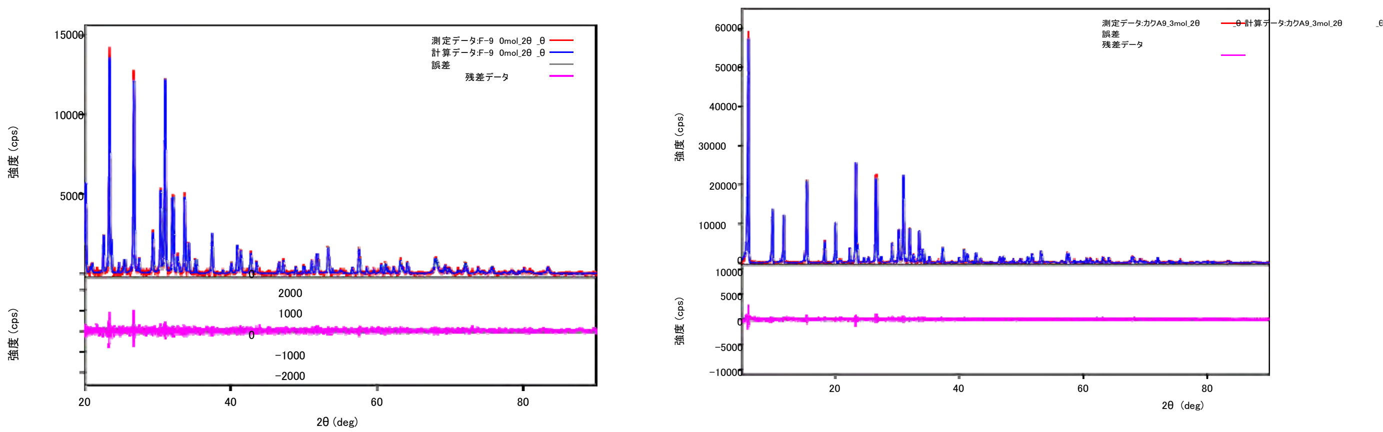
(a)Before preparation (b) After preparation for 3mol

Figure 5. X-ray diffractomete result for A4



(a)Before preparation (b) After preparation for 3mol

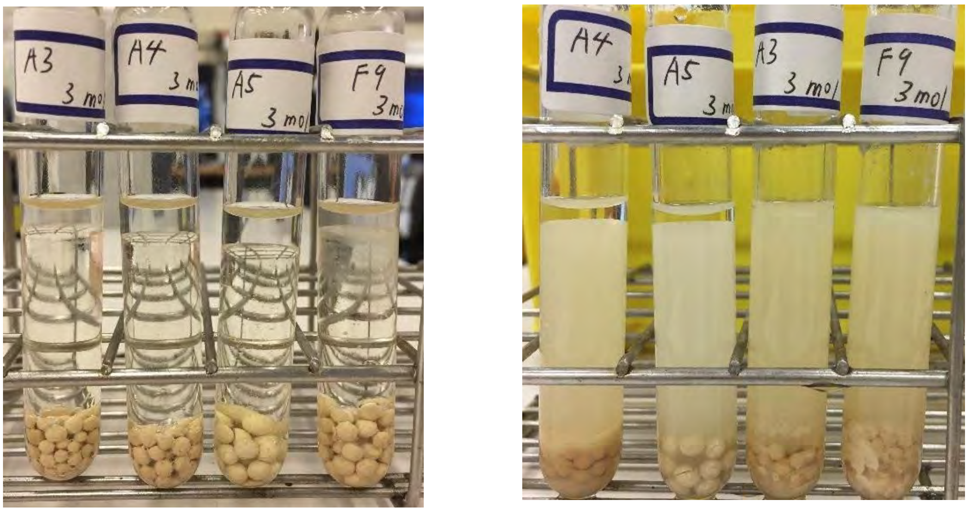
Figure 6. X-ray diffractomete result for A5



(a)Before preparation (b) After preparation for 3mol

Figure 7. X-ray diffractomete result for F9

4.2. BDF synthesis



(a) Before synthesis (b) After synthesis

Figure 8. BDF synthesis result

BDF synthesis result is shown in Figure 8. It is observed that BDF was obtained by using A4, A5 and F9 while less BDF was obtained by using A3. This could be explained that A4, A5 and F9 has the strong base strength after preparation.

4.3. Influence of ultrasonic irradiation on zeolite

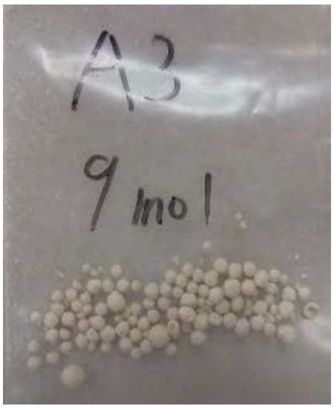
Since all the zeolite will be irradiated by ultrasound during BDF synthesis reaction, the influence of the ultrasonic irradiation on each zeolite is investigated. The result is shown in Figure 9.
After ultrasonic irradiation, the shape of A3, A4 and F9 changed to powder while only A5 kept its original shape. This is very important because after BDF synthesis, it is easy to separate the solid catalyst from the reaction liquid.

4.4. Simulation result

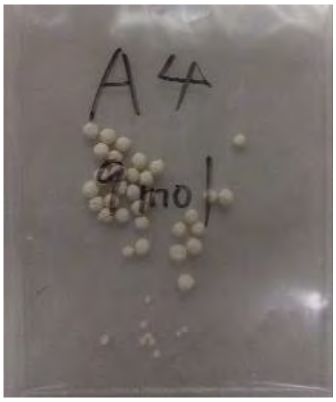
Influence of reaction temperature on the velocity distribution inside the U-type reactor is shown in Figure 10. As the temperature increased, the velocity distribution will increase because the increased temperature will lower the oil viscosity, this could properly lead to the higher BDF synthesis efficiency.

5. Conclusion

(1) Zeolite A5 is better for BDF synthesis reaction;
(2) Increased reaction temperature will properly increase the BDF synthesis efficiency.



(a) A3

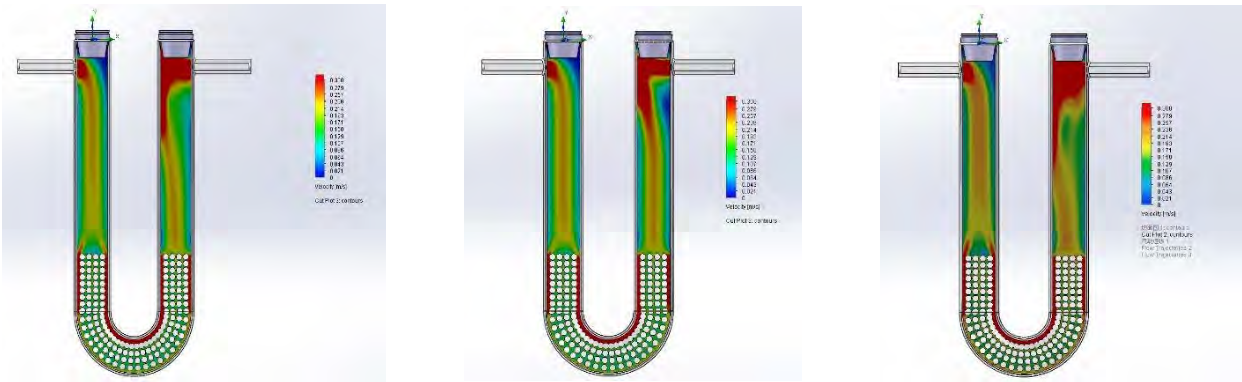


(b) A4

(c)A5

(d) F9

Figure 9. Influence of ultrasonic irradiation on the shape of zeolite



a 20℃ b 40℃ c. 60℃

Figure 9. Influence of reaction temperature on the velocity distribution

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