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# A novel method for the synthesis of biodiesel from soybean oil and urea

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## ABSTRACT

The increasing demand for energy has encouraged the development of renewable resources and environmentally benign fuel such as biodiesel. In this study, ethyl fatty esters (EFEs), a major component of biodiesel fuel, were synthesized from soybean oil using sodium ethoxide as a catalyst. By-products were glycerol and difatty acyl urea (DFAU), which has biological characteristics, as antibiotics and antifungal medications. Both EFEs and DFAU have been characterized using Fourier transform infrared (FTIR) spectroscopy, and <sup>1</sup>H nuclear magnetic resonance (NMR) technique. The optimum conditions were studied as a function of reaction time, reactant molar ratios, catalyst percentage and the effect of organic solvents. The conversion ratio of soybean oil into pure EFEs was 76% after 10 h of reaction. The highest conversion yield of EFEs is obtained when the urea/soybean oil ratio was from 6.2 mmol to 1 mmol, while the highest production of DFAU is obtained when the ethoxide (as a catalyst)/soybean oil ratio is from 6.4 mmol to 1 mmol in hexane as the reaction medium.

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## 1. Introduction

Biodiesel as a biomass-based diesel fuel is a non-fossil fuel alternative to the traditional fuels used in diesel engines. Generally, methyl or ethyl fatty esters synthesized from vegetable oil or animal fats are considered a biodiesel [1,2]. Using biodiesel in diesel engines sustains air quality, since its usage produces 78% less CO<sub>2</sub> emissions, which is the major gas causing greenhouse effect, compared to petroleum diesel, while it reduced CO<sub>2</sub> emissions by 15% when mixing petroleum with 20% biodiesel [3].

Many studies have been carried out to obtain biodiesel from the transesterification of different vegetable oils, like

soybean, sunflower, peanut, rapeseed, palm, olive, cottonseed, linseed, coconut, etc. Transesterification at 60 °C in addition to vigorous stirring at 600 rpm for Jojoba oil-wax with methanol was carried out to yield biodiesel in the presence of 1 wt% of sodium methoxide as a catalyst. A quantitative conversion of the oil was developed after 4 h for a molar methanol/oil ratio of 7.5:1 [4]. Transesterification using alkali-catalysts was widely utilized for biodiesel fuel production [5]. In the same manner, non-edible oil was used to produce biodiesel [6].

EFEs can be used as a major component of biodiesel fuel, which is a considerable alternative energy resource to petroleum [7,8]. DFAU was produced as a by-product. DFAU has attracted much attention because of its activity against bacteria, yeasts, molds, in addition to its industrial applications as a surfactant, lubricant, disinfectant, and in cosmetics, shampoos, detergents and antifoams [9]. Many

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researchers have studied the synthesis of fatty acyl urea from fatty acids or their esters with various amine compounds, either at elevated temperature and pressure [10] or by enzymatic synthesis [11]. The presence of long-chain fatty acids from soybean oil containing O and N donor sets suggests that DFAU should be very useful as an organic reagent for the extraction and the separation of metal ions from aqueous solutions [12]. Additionally, DFAU offers potential applications as surfactants, modifying natural clays used to produce polymer nanocomposites [13,14].

Clay is hydrophilic in nature because it contains inorganic cations on the basal planar. Its particles can only disperse on the micro-scale in a polymer matrix. Clay modification is required to improve its reinforcing ability [15]. A recent way of enhancing this ability is by making clay an organophilic material via ionic exchange of its interlayer cations with organic cations, such as the amidonium ions of DFAU.

The present study describes the production of EFEs from soybean oil by refluxing it with urea using sodium ethoxide as a catalyst.

## 2. Experimental

### 2.1. Materials

Commercial soybean oil (Nacalai Co., Kyoto, Japan) was used, as well as urea, ethanol, and sodium metal (Merck, Germany).

### 2.2. Synthesis of EFEs and DFAU

In a 250-mL round-bottom flask fitted with a reflux condenser and a magnetic stirrer, finely cut sodium (0.50 g) was dissolved in 100 mL of absolute ethanol. After the reaction of all sodium, 4.00 g of soybean oil were added into the solution, which was followed by the addition of 1.38 g of dried urea (pre-dried at 60 °C for 4 h) dissolved in 50 mL of hot (70 °C) ethanol. After thorough shaking, the mixture was refluxed for 8 h in an oil bath at 70 °C. The mixture was left to cool at room temperature, then was transferred into a separation funnel, and allowed to settle overnight. The thick phase of glycerol was removed from

the bottom layer. The top layer, which comprises the products, was poured into a beaker and mixed with 100 mL of hot distilled water (60 °C) and 10 mL of concentrated hydrochloric acid, and stirred for 15 min.

The white mass of ethyl fatty ester was filtered out of the DFAU solution, while the clear solution containing DFAU was cooled in an ice bath. The pale yellow precipitate was collected on a Buchner funnel to be washed with 50 mL of cold water and then dried in a vacuum desiccator over phosphorous pentoxide. The conversion ratio of soybean oil into DFAU was 76%. The preparation reaction is shown in Fig. 1.

The proposed mechanism for soybean oil conversion into DFAU and ethyl fatty ester is shown in Fig. 2. Catalysis by sodium ethoxide initiates proton withdrawing from ( $-NH_2$ ) in the urea molecule, as shown in Fig. 2A. This pathway is feasible from a physical organic point of view [10]. While Fig. 2B demonstrates it, the urea ion attacks one of the triglyceride groups and forms an *N*-carbamoyl fatty amide that attacks the second group of triglyceride after withdrawal of a proton by the basic catalyst. In two consecutive reactions, 2,3-dihydroxypropyl fatty ester, 1,3 dihydroxypropan-2-fatty ester (as intermediates), and DFAU are formed. The final step is displayed in Fig. 2C, which shows the alkoxy group attacking the third group of triglyceride to form ethyl fatty ester and glycerol [12].

## 3. Results and discussion

### 3.1. Characterizations of EFEs and DFAU

#### 3.1.1. FTIR spectra

EFEs and DFAU were characterized using FTIR spectroscopy (APS-1650 Automated Prep Station, PerkinElmer, USA). The characteristic FTIR peaks of soybean oil were observed at 3010, 2922, 2855, 1742, 1461, 1161, and 720  $cm^{-1}$ ; they result from C–H stretching of  $CH=CH$ , C–H asymmetric stretching of  $CH_2$ , C–H symmetric stretching of  $CH_2$ , C=O stretching of ester (glyceride),  $CH_2$  scissoring, O–C–C stretching, and  $CH_2$  rocking, respectively [13], while DFAU spectra show new bands at 3348, 1623 and 1045  $cm^{-1}$  attributed to N–H stretching, C=O stretching, and C–N stretching of amide, respectively. The fading of peaks at 1742 and 1161  $cm^{-1}$  and the appearance of peaks

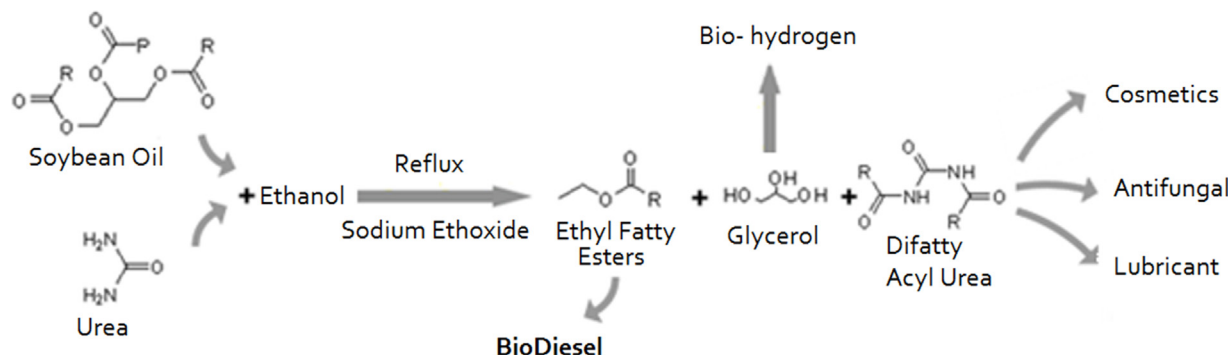


Fig. 1. Synthesis of ethyl fatty esters and difatty acyl urea from soybean oil and urea.

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