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Alkali catalyzed transesterification of safflower seed oil assisted by microwave irradiation

M. Zahir Duz [a,*], Abdurrahman Saydut [b], Gulsen Ozturk [a]

[a] Dicle University, Chemistry Department, Diyarbakir TR-21280, Turkey
[b] Dicle University, Mining Engineering Department, Diyarbakir, TR-21280, Turkey

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ABSTRACT

The safflower (*Carthamus tinctorius* L.) oil was extracted from the seeds of the safflower that grows in Diyarbakir, SE Anatolia of Turkey. Biodiesel has been prepared from safflower seed by transesterification of the crude oil under microwave irradiation, with methanol to oil molar ratio of 10:1, in the presence of 1.0% NaOH as catalyst. The conversion of *C. tinctorius* oil to methyl ester was over 98.4% at 6 min. The important fuel properties of safflower oil and its methyl ester (biodiesel) such as density, kinematic viscosity, flash point, iodine number, neutralization number, pour point, cloud point, cetane number are found out and compared to those of no. 2 petroleum diesel, ASTM and EN biodiesel standards. Compared with conventional heating methods, the process using microwaves irradiation proved to be a faster method for alcoholysis of triglycerides with methanol, leading to high yields of biodiesel.

1. Introduction

As the fossil fuels are depleting day by day, there is a need to find out an alternative fuel to fulfill the energy demand of the world. Biodiesel is one of the best available sources to fulfill the energy demand of the world. The petroleum fuels play a very important role in the development of industrial growth, transportation, agricultural sector and to meet many other basic human needs. However, these fuels are limited and depleting day by day as the consumption is increasing very rapidly. Moreover, their use is alarming the environmental problems to society. Hence, the scientists are looking for alternative fuels. Biodiesel is gaining more and more importance as an attractive fuel due to the depleting nature of fossil fuel resources [1]. It is a renewable, low carbon environmentally friendly fuel and has been regarded as a promising alternative fuel for transportation. Previous studies have shown that biodiesel produces substantially less CO, CO_2, particulate matter (PM), poly-aromatic hydrocarbons (PAH), nitrated poly-aromatic hydrocarbons (nPAH), sulfur oxides (SO_x) and HC emissions than fossil fuel [2–8]. A life cycle analysis of biodiesel showed that overall CO_2 emissions were reduced by 78% compared with petroleum-based diesel fuel. Its additional advantages include outstanding lubricity, excellent biodegradability, superior combustion efficiency and low toxicity, among others [5,9,10].

Carthamus tinctorius L. which belongs to the Composites family, is cultivated in several parts of the world due to its adaptability to different environmental conditions [11–13]. It is highly branched, herbaceous, thistle-like annual, usually with many long sharp spines on the leaves. Plants are 30 to 150 cm tall with globular flower heads (capitula) and commonly, brilliant yellow, orange or red flowers which bloom in July. Each branch will usually have from one to five flower heads containing 15 to 20 seeds per head. *C. tinctorius L.*, has been grown for centuries, primarily for its colorful petals to use as a food coloring and flavoring agent, for vegetable oil and also for preparing textile dye in the Far East, Central and North Asia, America, North Africa, Europe and Caucasia. The principle countries where safflower is grown are India, USA, Mexico, and in lesser extent Kazaghistan, Ethiophia, Argentina, China, Uzbekistan, Australia, Russian Federation, Pakistan and Spain [14]. Safflower is an oil-seed crop that originated from the eastern Mediterranean. It is adapted to relatively low rainfall areas receiving winter and spring rainfall with a dry atmosphere during flowering and maturation. It is a xeric crop tolerant to drought, because it has long roots that can take up water deep down in the soil profile. It is considered to be the most drought-resistant of all oil-seed annuals [13]. The safflower oil is extracted from the seeds of the safflower a perennial shrub that grows in semi desert areas in some parts of the world. This oil is flavorless and colorless, and nutritionally similar to sunflower oil. The seeds of *C. tinctorius* commonly named as *Safflower*, respectively, produce an oil with a favorable fraction of some very desirable fatty acids, among which predominate specifically the essential fatty acids linoleic acid (LA-C 18:2) and linolenic acid (LNA-C 18:3), which show a growing importance for nutritional and pharmaceutical purposes. This species was cultivated for more than two thousand years as a dye, carthamin, obtained from its flowers. However, safflower has lately gained importance because the seed oil has an important food-value, with high linoleic acid content. This polyunsaturated oil is similar to

* Corresponding author. Tel.: +90 412 248 85 50; fax: +90 412 248 83 89.
E-mail address: zahird@dicle.edu.tr (M.Z. Duz).

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purslane, soybean or walnut oil. In addition to the unsaturated FAs of the 18-carbon group, other unusual molecular species are present in varying quantities, e.g., arachidic (C 20:0), eicosenoic (C 20:1, *cis*-11), behenic (C 22:0) and lignoceric (C 24:0) acids [12,15].

Biodiesel is produced from animal fat, vegetable oil or waste cooking oil, and that can be used as the basis for a clean substitute for fossil fuel without any modification to diesel engines, boilers or other combustion equipments [5,10,16–20]. Biodiesel is a non-toxic and biodegradable substitute for petroleum-based diesel. Biodiesel is produced through a reaction known as transesterification. The transesterification is the key and foremost important step to produce the cleaner and environmentally safe fuel from vegetable oils [21]. There are three kinds of catalysts that can be used in transesterification reaction, a strong alkaline catalyst, a strong acid, and an enzyme. The main advantages of using a strong alkali as a catalyst are shorter reaction time and less amount of catalyst required in the manufacturing process of the transesterification reaction. Therefore, a strong alkaline catalyst is widely used in the industry for mass biodiesel production.

This process has been widely used to reduce the high viscosity of triglycerides. In a transesterification or alcoholysis reaction, 1 mol of triglyceride reacts with 3 mol of alcohol (molar ratio of methanol to vegetable oil of 3:1) to form 1 mol of glycerol and 3 mol of the respective fatty acid alkyl esters. The process is a sequence of three reversible reactions, in which the triglyceride molecule is converted step by step into diglyceride, monoglyceride and glycerol (Fig. 1).

For conventional heating, heat energy is transferred to the reaction through convection, conduction and radiation from the surfaces of the reactor, which is an inefficient heat transfer. Large amount of energy is used to heat the media. Thus, long reaction time (usually from 30 min to 8 h) is required to achieve a satisfactory conversion of oil to biodiesel. An alternative heating system "microwave irradiation" has been used in transesterification reactions in recent years. Microwave (mw) radiation, on the other hand, delivers energy directly to the reactants. Therefore, preheating step is eliminated. Heat transfer is more effective than conventional heat. Consequently, transesterification with mw radiation can be completed in much shorter time [22,23]. Mw irradiation activates the smallest degree of variance of polar molecules and ions such as alcohol with the continuously changing magnetic field. The changing electrical field, which interacts with the molecular dipoles and charged ion, causes these molecules or ions to have a rapid rotation and heat, is generated due to molecular friction. Hence, mw irradiation accelerates the chemical reaction and high product yields are achieved within a short time [17,19,24–33].

This study supports the production of biodiesel from safflower seed oil under mw irradiation, using homogeneous alkali catalysis as a viable alternative to the diesel fuel. Properties of safflower seed oil and its methyl ester were within the limits of ASTM and EN standards.

2. Experimental

Safflower seeds used in the present study were purchased from Agricultural Faculty of Dicle University in Diyarbakir, situated in SE Anatolia of Turkey during the summer season of 2005. The seeds were cleaned manually to remove all foreign matter such as dust, dirt, stones and chaff as well as immature, broken seeds. In order to preserve its original quality, the sample was stored at an ambient temperature of 25 ± 3 °C in sealed plastic bags prior to any

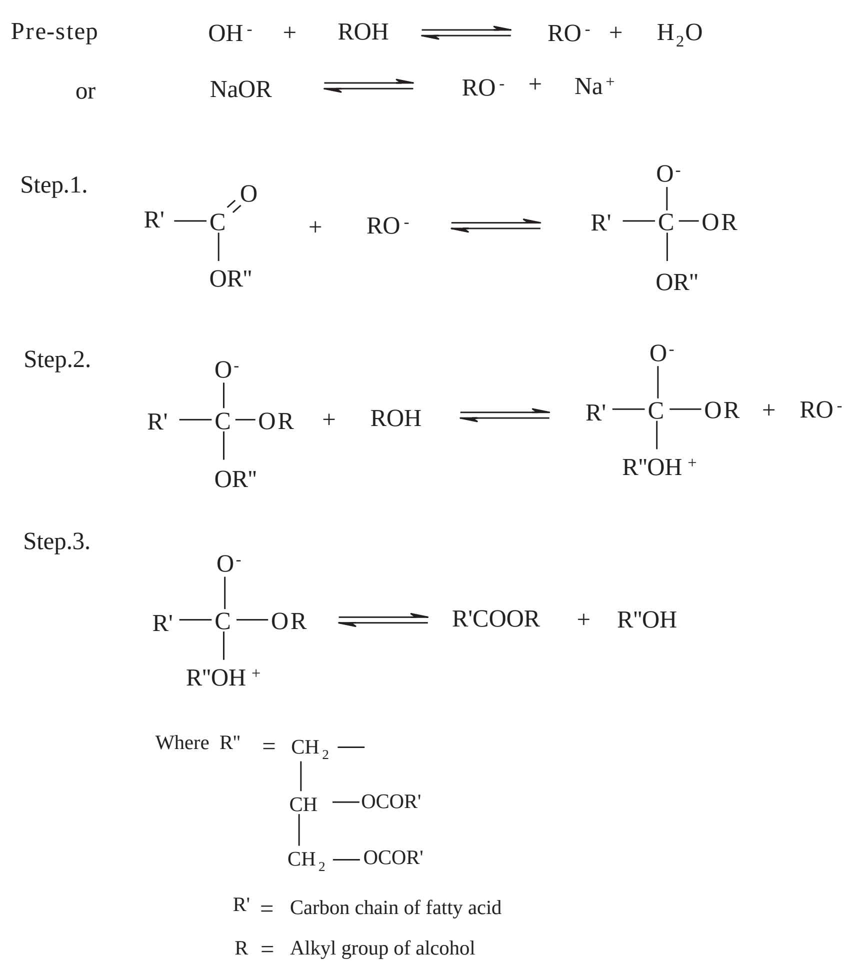

Fig. 1. Mechanism of base catalyzed transesterification.