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Research Article

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Determination of Optimum Reaction Conditions in Biodiesel Production from Microalgae Oil Using Microwave Irradiation

Mehmet Recai DURGUT[1]*, Türkan AKTAŞ[1], Serap KAYİŞOĞLU[2], Çetin YAĞCILAR[2]

[1]University of Namık Kemal, Faculty of Agriculture, Department of Biosystem Engineering, Tekirdag-Turkey
[2]University of Namık Kemal, Vocational School of Technical Sciences, Tekirdag-Turkey

Abstract Demands for alternative fuels such as biodiesel are increasing because of decreasing of fossil fuel sources and its environmental concerns. However, the resulting biodiesel plant origin safflower, canola, peanut oil, etc. at the same time hold an important place in the food sector, the biodiesel production is the most important limiting factor. One of the most important vegetable oil sources is microalgae oil, because of high oil content of the seeds such as 31-68%. The yield of transesterification reaction is too high because microalgae oil can be dissolved in alcohol easily. In this study, parametric experiments were done using microwave heating system in order to obtain biodiesel by transesterification reaction of microalgae oil using KOH as a catalyst. Effect of catalyst ratio, reaction temperature and time on transesterification of microalgae oil were investigated. Microwave assisted transesterification of cottonseed oil under the conditions of 1.5% catalyst-oil ratio, 60^{o}C temperature and 6 minutes reaction time, resulted in a biodiesel yield of 91.7%.

Keywords Microalgae, microwave, biodiesel, biomass, transesterification, biofuels

Introduction

Biodiesel, as an alternative fuel, has many benefits. It is biodegradable, non-toxic and compared to petroleum-based diesel, has a more favorable combustion emission profile, such as low emissions of carbon monoxide, particulate matter and unburned hydrocarbons.

In biodiesel production, the transesterification reaction is carried out using different heating systems. In all conventional heating methods, heat is transferred to the walls of the reaction chamber and from there to the reaction medium. This process is quite slow [1]. In the case of microwave heating, substances in the reaction medium absorb the microwave energy under certain conditions and a warming occurs from the inside to the outside of the system. This heating is inversely related to the transport-convection, which allows the heating of the system from outside to inside [2]. If the microwave radiation has an even distribution in the reaction medium and the reactants are thought to be effectively mixed, there is no temperature change without heating by microwave. Thus, unwanted side reactions are minimal and, when other conventional heating systems are used, are advantageous over systems where the source is not directly contacted with the reaction mixture and the temperature change between the hot reactor walls and the cooler reaction medium occurs. In microwave heating reactions, the reactor wall is cooler than the reaction mixture [3].

Rapid heating is the heating of the catalyst or any chemical by absorbing the microwave regardless of low thermal conductivity. Microwave heating increases the reaction speed because there is no low heat transfer in the microwave, as in conventional heating. Volumetric heating means heating the entire volume of the sample in

the microwave oven from the center, that is, from the opposite side of the conventional heating (from the reaction medium to the reaction). Thus, the sample is heated faster [4].
The reaction time, which takes about 1 hour with the conventional heating system, is reduced to 3 to 7 minutes in case of using the microwave heating system [5-6].
For this reason, the research involves obtaining biodiesel from microalgae oil using a laboratory scale-modified standard microwave oven, and determining the factors that will affect the quality of the biodiesel to be obtained.

Materials and Methods

Nannochloropsis oculata was obtained from the Ege Biotechnology Inc.. Algae were grown in Erlenmeyer flasks and then transferred to a 50-L flat panel photobioreactor (PBR) made of clear transparent glass at 90 cm long, 10 cm wide, and 60 cm high (shown in Fig.1) [7-8]. Continuous illumination by cool white fluorescent lamps of 100–120 μmol photons m^{-2} s^{-1} at 12:12 h light/dark period was provided. The culture temperature was regulated at 24 ± 2 °C by indoor air conditioning. Aeration of 0.2 vvm was provided by air bubbling to the bottom of the PBR using an 60 cm air stone connected to an air pump [9-10]. Standard F/2 medium was used to culture the microalgae.
At the end of the production period, algae reaching the stagnation phase were sediment. The sedimentation was carried out by adjusting the pH of 250 L culture medium ranging from pH 7.5 to pH 12.5 using 5M sodium hydroxide and 1N hydrochloric acid. The medium was mixed rapidly (800rpm) until the required pH value was achieved and then slowly (250rpm) for 1 minute using mixer [11].
The culture suspension having been flocculated, the microalgal cells were then washed with distilled water to remove coagulants. The cells were then dried at 70 ºC for 24 h and triturated to create dry microalgae powder for experiment. The lipid extraction was performed using modified Bligh and Dyer method [12].
Experiments were carried out in a commercial microwave oven with minor modification. An Arçelik model MD574S commercially available, domestic microwave oven was chosen. The oven was equipped with continual power transfer and adjustable output up to 700 W. Small hole was created at the side of the oven for insertion of a type K thermocouple. They were installed directly into the raw material mixture inside the oven. Small hole was created at the bottom of the oven for insertion of a motor-driven stirrer. A digital controller model REX-C100 was employed to monitor and control reaction temperature. At the start of each experimental campaign, irradiation leakage of the microwave reactor system was thoroughly checked to ensure that no leak took place (shown in Fig. 2).

Figure 1: Designed photobioreactor, harvested and dried microalgae samples

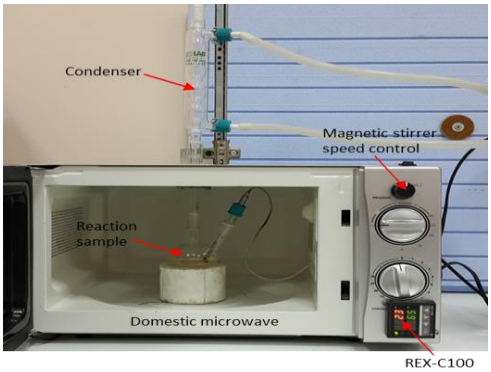

Figure 2: Modified domestic microwave oven

Experiments to determine the optimum reaction conditions of biodiesel production by microwave method to reduce the energy input of microalgae biodiesel production were carried out by keeping the ratio of 6:1 methanol / oil molar constant; (3, 4, 5, 6, 7, 8 minutes) and catalyst (KOH) ratio (0.5%, 1.0%, 1.5%), reaction temperature (50 °C, 55 °C, 60 °C, 65 °C) the effects on transesterification yield have been examined [13]. In the studies, firstly, the catalyst was dissolved in alcohol and then added to the oil, allowing the reaction at the determined temperature and time under the reflux in the microwave unit. During the reaction, stirring was maintained at 600 rpm [14].

After completion of the reaction, the reaction product was cooled to room temperature in an ice bath, subjected to centrifugation at 4000 rpm for 10 minutes, and methyl ester and glycerin phases were separated. The methyl ester was washed with distilled water to remove the impurities contained in the phase and continued to wash until pH reached 7. The excess water and methanol remaining after washing were removed in the rotary evaporator [15].

Determination of Fuel Properties

Biodiesel yield, defined as the weight percentage of the final product relative to the weight of microalgae oil at the beginning, was estimated [16-17]. The flash point of the biodiesel were determined as per the ISO 3679 method. The pour point determinations were made using the ISO 3016method. The kinematic viscosity was determined using a viscometer and according to the ISO 3104method. The density of the fuel was determined using the ISO 12185 method. All experiments were carried out in triplicate and mean values were reported.

Results and Discussion

The biodiesel efficacy of the transesterification of microalgae oil by microwave heating at 60^{o}C temperature and 6 min reaction time was investigated and the results obtained are given in Table 1.

Table 1: Biodiesel yield of catalyst ratio at 60^{o}C temperature and 6 minutes reaction time

Catalyst Ratio (%)	Biodiesel Yield (%)
0,5	88,3
1	91,2
1,5	91,7

When the effect of catalyst ratio on the product yield was examined (Table 1), it was seen that the highest yield value was reached by using 1.5% KOH. The rate of 1.5% catalyst in the transesterification in the microwave unit of microalgae oil was determined as optimum value.

The biodiesel yield values obtained as a result of experimental studies carried out at different temperatures using this catalyst ratio are given in Table 2.

Table 2: Catalyst ratio of 1.5%, biodiesel yield values of temperature at 6 min reaction conditions

Temperature (^{o}C)	Biodiesel yield (%)
50	91.3
55	91.2
60	92.3
65	91.4

Studies using 1.5% KOH at different temperatures have shown that the optimum working temperature is 60 °C. In this study, biodiesel yield of 92.3% was reached (Table 2).

The result of the work done at different temperatures using 60 °C temperature, 1.5% KOH catalyst is given in Table 3.

Table 3: Biodiesel yield values at reaction conditions of 60 °C, 1.5% KOH

Reaction time (min)	Biodiesel yield (%)
3	91.3
4	91.3
5	92.2
6	92.5
7	91.6
8	90.4

It was determined that the optimum reaction time was 6 minutes for the determined reaction conditions (Table 3).

Some fuel properties of the biodiesel sample with the yield of 92.5% at the reaction conditions of 1.5% KOH, 60 °C, 6 min were determined and compared with the studies (Table 4).

Table 4: Fuel properties of the obtained biodiesel

Properties	**Unit**	**European Committee for Standardization**		**Biodiesel Experiments**
		min	**max**	**KOH**
Efficiency	%	90	-	92.5
Density, 25°C	kg/m^3	860	900	892
Viscosity, 40°C	mm^2/s	3.5	5.0	4.37
Flash Point	°C	120	-	152
Cloud Point	°C	-	-	3
Pour Point	°C	-	-	-1

Density is the measure of the fluidity of biodiesel. The density is expected to be between 860-900 kg/m^3. This value is between 892 kg/m^3 and the desired values.

Viscosity is one of the most important characteristics of biodiesel. High viscosity fuel causes poor atomization, bad combustion, clogging of the injectors and carbon buildup in the segments. For biodiesel this value should be between 3.5-6 mm^2/s. The viscosity value is in the range of 4.37 mm^2/s.

Flow and Cloud point; is the point where the crystal clouds are first seen when the liquid is cooled under the prescribed conditions. The cloud point is a critical factor for the performance of diesel fuels in cold weather. This is problematic in the use of fuels in very cold weather conditions.

The raw material from which the cloud point biodiesel is obtained varies according to the properties of the oil. For example, in the measurement made with ASTM D2500 standards, the cloud point in biodiesel obtained from rapeseed oil is -3 ^oC whereas in food quality frost oil this value is 19 ^oC [18]. In this study, where biodiesel was obtained using microalgae, the flow and cloud point were -1 ^oC and 3 ^oC respectively.

Conclusion

In this project, the effect of microwave heating on biodiesel from microalgae oil was investigated.

The effect of catalyst ratio at constant temperature (60 °C) and reaction time (6 minutes) was investigated in microwave heating experiments (Table 1). Biodiesel purity increased with increasing catalyst ratio and this ratio was considered as the optimum value in experimental studies since the highest purity was achieved with a catalyst ratio of 1.5 %.

When the studies carried out at different temperature and reaction times with microwave heating were examined (Table 2), the best results were reached at 60 ^oC temperature and 92.5 % at the end of 6 minutes. This condition is determined as the best condition for biodiesel elution with microwave heating, since the highest yield is obtained at the highest value of biodiesel purity above the specified minimum value.

The results of the analyzes made on the biodiesel sample obtained under these conditions show that the biodiesel sample meets the standards as a result of comparison with European Biodiesel Standard values.

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