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Effect of fractional winterization of beef tallow biodiesel on the cold flow properties and viscosity

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HIGHLIGHTS

- ▶ Fractional winterization of beef tallow biodiesel was investigated.
- ▶ Saturated fatty acid content of beef tallow biodiesel was reduced by winterization.
- ▶ Cloud point, pour point, viscosity and density of biodiesel winterized were monitored.
- ▶ CP, PP and cold flow properties of the beef tallow biodiesel significantly improved.

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ABSTRACT

Biodiesel can cause performance problems at cold temperatures because of its fatty acid composition. Flow characteristics of biodiesel such as viscosity, density, cloud point and pour point increase as saturated fatty acid content of the lipid material used as a feedstock increases. Beef tallow, due to its high saturated fatty acid content, is not preferred as a feedstock in biodiesel production. In this study, it is aimed to reduce saturated fatty acid content of biodiesel obtained from beef tallow by fractional crystallization process also known as winterization. The fatty acid methyl ester contents of the filtrates obtained from crystallization were determined. In addition, cloud point, pour point, viscosity and density of biodiesel winterized were monitored. It has been detected that saturated fatty acid methyl ester content of winterized biodiesel from beef tallow reduced from 86.91% to 73.38% while unsaturated ones increased from 12.00% to 19.95% until the lowest crystallization temperature which is 16.3 °C in six steps. It was observed that density, viscosity and cold flow properties of the beef tallow biodiesel significantly improved by the removal of saturated methyl esters from the biodiesel.

1. Introduction

As an alternative diesel fuel, biodiesel is obtained from lipid materials such as vegetable oils and animal fats. It is renewable, biodegradable and nontoxic, has low emission profiles and so is environmentally advantageous [1]. Biodiesel, due to the crystallization of highly saturated fatty acid methyl esters (FAMEs), has some performance problems in pipes and filters of the vehicles at low temperatures [2]. When biodiesel is exposed to lower temperatures the solid wax crystal nuclei begin to form. Further decreases in temperature cause the crystal nuclei to grow [3] and become visible, which is known as cloud point (CP). Further decrease results in appearance of a waxy structure at the known pour point temperature (PP) beyond which the flow of mixture stops. There is an ongoing effort to improve the cold flow properties of biodiesel [4–6].

Vegetable oils are usually used in biodiesel production because of their low cold flow properties and viscosity, but it contributes to higher cost of biodiesel production when they are used as a feedstock. Therefore, alternative feedstock is an important research topic in biodiesel research. Animal fats may be an important alternative biodiesel feedstock because of its relatively lower cost. Although beef tallow is rich in saturated fatty acid content, it can be used after blending with vegetable oil based biodiesel in certain proportions [7]. Another method towards beef tallow usage as the feedstock can be the separation of saturated fatty acid methyl esters (FAMEs) and other lipid components that has higher crystallization temperatures than the beef tallow biodiesel through crystallization process known as winterization. There are two stages in a winterization process: (1) Crystallization in which selective nucleation and crystal growth occurs under slow agitation and controlled cooling. (2) Filtration in which resulting slurry is filtered to separate solid and liquid fractions [8]. Different winterization techniques of peanut biodiesel to improve the cold flow

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properties were examined and the best technique was found to be crystallization filtration [9].

This study is aimed to improve the cold flow properties of biodiesel obtained from beef tallow by fractional crystallization process. An evaluation of the crystallization behavior of beef tallow biodiesel was performed by measuring the saturated/unsaturated FAMEs of the filtrates. In addition, effect of saturated FAME separation by winterization on the cloud point (CP), pour point (PP), kinematic viscosity and density were examined. The amount (%wt.) of crystallized biodiesel (FAMEs) from each crystallization/filtration process has been determined.

2. Materials and methods

2.1. Materials

The beef tallow was obtained from a local slaughterhouse in Erzurum, Turkey. A typical composition and some characteristic physical properties of beef tallow are shown in Table 1. The saturated fatty acid content of beef tallow used in this work accounts for almost 87% of the total fatty acids in the beef tallow. The higher stearic and palmitic acid contents of beef tallow give high melting point and high viscosity properties of beef tallow [10]. Methyl alcohol and potassium hydroxide used in the experiments has purity of 99.9% and 99.9% respectively and were purchased from Merck and Flake.

2.2. Equipments

Transesterification experiments were carried out in a 1000 mL jacketed glass reactor. The reactor was equipped with a condenser to avoid methanol losses. A mixer and a constant temperature circulator were used to provide required agitation and constant temperature. The experiments were carried out under the normal atmospheric pressure and at temperature of 60 °C to prevent beef tallow from freezing. The constant parameters in the experiments were chosen as reaction time, 60 min; oil/alcohol ratio, 1:6 (in weight); catalyst ratio, 0.75% KOH (in weight), and reaction temperature of 60 °C.

Winterization experiments involving crystallizations/filtrations were carried out in a 1000 mL jacketed glass reactor. This reactor was equipped with a stirrer and a constant temperature circulator with a cooling rate 0.6 °C/min. The filtration process was performed in a sintered-glass funnel using a vacuum flask that was connected to a vacuum pump and thermally insulated sufficiently to maintain the filtration temperature.

Table 1
Typical compositions and some physical properties of crude beef tallow [10].

Characteristics	Beef tallow (range)
Iodine number	35–48
Saponification number	193–202
Melting point (°C)	47–50
Fatty acid composition (wt.%)	
Myristic (C-14:0)	2–8
Palmitic (C-16:0)	24–37
Stearic (C-18:0)	14–29
Oleic (C-18:1)	40–50
Linoleic (C-18:2)	1–5
Triglyceride composition (%)	
Trisaturated (GS_3)	15–28
Disaturated (GS_2U)	46–52
Monosaturated (GSU_2)	20–37
Triunsaturated (GU_3)	0–2

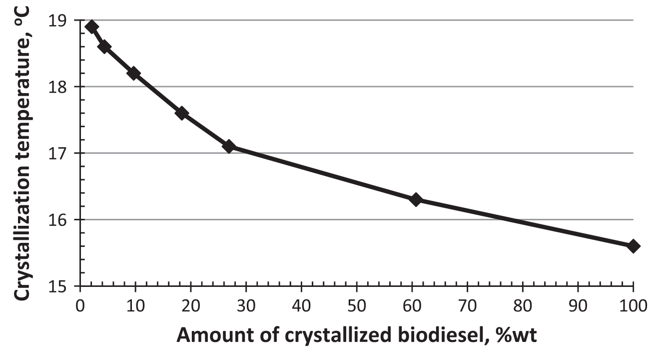

Fig. 1. The cumulative amount of crystallized biodiesel after each crystallization step (±2 average of standard deviation and R^2 = 0.98).

3. Experimental procedure

Experiments were repeated three times at different times. Averages of standard deviations calculated were given along with the labels of figures.

3.1. Transesterification reaction

The reactor was initially filled with the beef tallow and heated to the desired temperature. The catalyst (KOH) was first dissolved in the methanol and this solution was added to the stirred reactor. The reaction started as soon as the catalyst and methanol solution had been added to the reactor. After the reaction the mixture was taken to a separator funnel and the glycerol phase was separated at the bottom. Thereafter the mixture was washed three times with deionized water. The residual methanol was separated from the biodiesel by a rotary evaporation under vacuum at 80 °C for a period of 1 h.

3.2. Winterization process

The biodiesel was agitated at 250 rpm during the winterization. The winterization of the beef tallow biodiesel begun from CP temperature and reduced by about 0.5 °C intervals at a cooling rate of 0.6 °C/min. The resulting crystals were then separated by vacuumed filtration at the same temperature. Samples obtained from each filtration were analyzed in terms of cold flow properties, viscosity and density. Filtered liquid phase was also used for the next crystallization stage. This process was continued until whole biodiesel sample was crystallized. In addition, the amount of crystallized biodiesel was determined after each experiment in weight basis.

3.3. Analytical methods

Fatty acid methyl esters included in beef tallow were analyzed by a gas chromatograph (Agilent 5973 N gas chromatograph, Waldbronn, Germany) with a capillary column (DB23, 60 m × 250 μm × 0.15 μm), temperature (increasing from 100 to 200 °C with a rate of 5 °C/min and from 200 to 250 °C to with a rate of 4 °C/min), mass selective detector at 280 °C, helium gas (1.2 mL/min), and injection block temperature of 250 °C.

After each experiment, pour point (ISO 3016), cloud point (EN 23015), density at 15 °C (EN ISO 3675) were analyzed according to related EN standards. Kinematic viscosity of the samples at 40 °C was measured by using Koehler kV 4000 series viscosimeter (EN ISO 3104).