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Research Article

Optimization of the Production of Biodiesel from Beef Tallow Applying Ultrasound Technology

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Abstract

Biodiesel is one of the renewable energy sources that can be produced from vegetable and animal sources. Biodiesel has many advantages such as being environmentally friendly, non-toxic, low emission profile and biodegradable. Contrary to its advantages, one of the most important disadvantages is its high cost. The cost of biodiesel increases considerably when it is produced from edible crude oils. Therefore, a suitable feedstock should be used for biodiesel production. Beef tallow is a fat that is not preferred much in the food sector and can be called as waste. When biodiesel is produced from this fat, the contribution will be made both to the environment and to the economy. In this work, the optimization of ultrasound assisted biodiesel production from beef tallow was investigated. Taguchi method was used to optimize the biodiesel yield. Catalyst amount (0.75-2 wt.% of oil), methanol/oil molar ratio (5/1-9/1), reaction time (30-120 min), ultrasound time (5-20 min), ultrasound power (10-40 W) were selected as parameters. The optimum conditions were found to be 1.5 wt.% of oil for the catalyst amount, 7/1 methanol/oil molar ratio, 40W ultrasound power, 60 minute reaction time and 10 minute ultrasound time. The fuel properties of biodiesel obtained under these conditions accomplished the requirements of both the EN 14214 and ASTM D 6751 standards. The biodiesel yield under optimum conditions was 99.8%. In addition, according to the ANOVA results, it is found that the influence of reaction time, ultrasound time, methanol/oil molar ratio, catalyst amount and ultrasound power on production yield are obtained as 34.56%, 29.72%, 23.68%, 9.18% and 2.17%, respectively.

Keywords: Optimization, Biodiesel, Beef Tallow, Ultrasound.

Ultrases Teknolojisi Uygulayarak Sığır İç Yağından Biyodizel Üretiminin Optimizasyonu

Öz

Biyodizel, bitkisel ve hayvansal kaynaklardan üretilebilen yenilenebilir enerji kaynaklarından birisidir. Biyodizel, çevre dostu olması, toksik olmaması, düşük emisyon profiline sahip olması ve biyolojik olarak bozunabilir olması gibi pek çok avantaja sahiptir. Onun bu avantajlarının aksine en önemli dezavantajlarından birisi yüksek maliyetli oluşudur. Biyodizelin maliyeti, yemeklik ham yağlardan üretildiğinde önemli ölçüde artar. Bu nedenle, biyodizel üretimi için uygun bir hammadde kullanılmalıdır. Sığır iç yağı, gıda sektöründe fazla tercih edilmeyen bir yağdır ve atık olarak adlandırılabilir. Bu yağdan biyodizel üretildiğinde, hem çevreye hem de ekonomiye katkı sağlanacaktır. Bu çalışmada sığır iç yağından, ultrases destekli biyodizel üretiminin optimizasyonu araştırıldı. Biyodizel verimini optimize etmek için Taguchi yöntemi kullanıldı. Katalizör miktarı (ağırlıkça % 0.75-2), metanol/yağ molar oranı (5/1-9/1), reaksiyon süresi (30-120 dk), ultrases süresi (5-20 dk), ultrases gücü (10-40 W) parametre olarak seçildi. Optimum koşullar % 1.5 (ağırlıkça) katalizör miktarı, metanol/yağ oranı 7/1, ultrases gücü 40 W, reaksiyon süresi 60 dk ve ultrases süresi 10 dk olarak bulundu. Bu koşullar altında elde edilen biyodizelin yakıt özellikleri, hem EN 14214 hem de ASTM D 6751 standartlarının gerekliliklerini yerine getirmiştir. Optimum koşullar altında biyodizel verimi % 99.8 dir. Ayrıca, ANOVA sonuçlarına göre, ürün verimi üzerine reaksiyon süresi, ultrases

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süresi, metanol / yağ molar oranı, katalizör miktarı ve ultrases gücünün etkisinin sırasıyla % 34,56, % 29,72, % 23,68, % 9,18 ve % 2,17 olduğu bulunmuştur.

Anahtar Kelimeler: Optimizasyon, Biyodizel, Sığır iç yağı, Ultrases.

1. Introduction

Due to the increasing population and developing technology in the world, the energy demand is increasing. Although fossil fuels are the most important energy sources, they are harmful to the environment and can be consumed. For this reason, interest in biodiesel, which is one of clean and renewable energy sources, is increasing day by day. Although biodiesel is not economically viable when compared to fossil fuels, it can be advantageous when a suitable raw material is used. In this context, animal fat (beef tallow) has shown as a good raw material of biodiesel. Because, the central production in the slaughter of the beef tallow is very high, the price is low and it can be considered as a waste because it is not preferred much as food (Teixeira et al., 2009).

Biodiesel is traditionally produced by the transesterification of crude/waste vegetable and animal fats with a primary alcohol, such as methanol or ethanol, in the presence of an acid or base catalyst. Ultrasound-assisted transesterification is one of the alternative methods used to improve the reaction time, substance consumption and conversion during biodiesel production (Gupta, Yadav, & Rathod, 2015; Martínez et al., 2017; Parida, Sahu, & Misra, 2016).

The ultrasound increases the mass transfer between the immiscible reactants. Ultrasound waves include both expansion and compression pressure waves. Therefore, it generates microbubbles that are filled with reactants vapors in the liquid. The mass transfer into the microbubbles increases with the effect of ultrasound. The event called cavitation ocurs when microbubbles grow and collapse (Sajjadi, Aziz, & Ibrahim, 2015). The collapse of cavitation bubbles increases the interaction between phases and the reaction rate. Therefore, higher conversion is achieved in a shorter time during transesterification of fatty acids by ultrasonic energy (Korkut & Bayramoglu, 2016; Mohod, Gogate, Viel, Firmino, & Giudici, 2017; Nakayama, Imai, & Woodley, 2017; Shinde, Nohair, & Kaliaguine, 2017).

Many studies have been conducted on the effect of ultrasound on biodiesel production from vegetable oils (Brasil, Oliveira, & Franca, 2015; Martinez-Guerra & Gude, 2015; Parida et al., 2016; Sáez-Bastante et al., 2014) However, ultrasonic-assisted biodiesel production from animal fats (especially beef tallow) has received little attention. Since the melting point, viscosity and density of the beef tallow are high, the effect of ultrasound energy may be more important on the production of biodiesel from this oil (Teixeira et al., 2009). Thus, the subject of this work was to investigate to ultrasonic-assisted biodiesel production from beef tallow and determine the optimum parameters of this process.

As is known, the optimization of the parameters used in biodiesel production is very important to obtain maximum yield (Saravanakumar, Avinash, & Saravanakumar, 2016). The Taguchi method can be used to find the optimum parameter with the least number of experiments. Taguchi can examine the parameters that affect an experiment as controllable and uncontrollable. In addition, it can be applied to an experimental design that includes many design factors. These are the most important advantages of the Taguchi method according to other statistical methods (Kackar, 1985). The Taguchi method was used to optimize the production of biodiesel from various oils in many studies. In these studies, the optimum parameter conditions of the process and the amount of maximum biodiesel yield in these conditions were determined (Adewale, Vithanage, & Christopher, 2017; Akhtar, Tariq, Iqbal, Sultana, & Wei, 2017; N. Kumar, Mohapatra, Ragit, Kundu, & Karmakar, 2017; R. S. Kumar, Sureshkumar, & Velraj, 2015; Sasikumar, Balamurugan, Rajendran, & Naveenkumar, 2016).

2. Materials and Methods

2.1. Materials

The beef tallow was obtained from a local slaughterhouse in Erzurum, Turkey. The properties of the beef tallow are given in Table 1. Methanol (Merck reagent of 99.9% purity), phosphoric acid (Merck reagent of 85% purity) and potassium hydroxide catalyst (Flake reagent of 99.9% purity) were used in the experiments.

Table 1. Properties of crude beef tallow

Properties	Value
Iodine number	45.2
Saponification number	163
Acid value (mgKOH/g)	1.09
Free fatty acids (as oleic acid, %)	0.55
Fatty Acid Composition (wt %)	
Myristic (C14:0)	2.13
Palmitic (C16:0)	25.11
Stearic (C18:0)	15.34
Oleic (C18:1)	40.21
Linoleic (C18:2)	2.20

2.2. Orthogonal Array and Experimental Design

L_{16} orthogonal array was chosen to optimize the effect of five important parameters on the production of ultrasound assisted biodiesel from beef tallow. The L_{16} orthogonal array is shown in Table 2.

Table 2. Taguchi L_{16} (4^5) Experimental plan

Experiment number	Parameters				
	A	B	C	D	E
1	1	1	1	1	1
2	1	2	2	2	2
3	1	3	3	3	3
4	1	4	4	4	4
5	2	1	2	3	4
6	2	2	1	4	3
7	2	3	4	1	2
8	2	4	3	2	1
9	3	1	3	4	2
10	3	2	4	3	1
11	3	3	1	2	4
12	3	4	2	1	3
13	4	1	4	2	3
14	4	2	3	1	4
15	4	3	2	4	1
16	4	4	1	3	2

Catalyst amount (wt. % of oil), methanol/oil ratio (mol/mol), ultrasound electric power (W), reaction and ultrasound time (minute) were taken into account in the experimental plan. The experimental parameters and their levels are listed in Table 3.

Table 3. Selected parameters and their levels

	Parameters	Levels			
		1	2	3	4
A	Catalyst amount (wt. % of oil)	0.75	1	1.5	2
B	Methanol/oil ratio (mol/mol)	5/1	6/1	7/1	9/1
C	Ultrasound power (W)	10	20	30	40
D	Reaction time (minute)	30	60	90	120
E	Ultrasound time (minute)	5	10	15	20

2.2.2. Experimental Setup and Procedures

The experimental setup used for ultrasound assisted transesterification experiments is shown in Figure 1. An ultrasonic generator (Cole Parmer CPX Ultrasonic Homogenizer, 750W, 20 kHz) equipped with a horn type probe was used to send ultrasound energy to the reactor content.

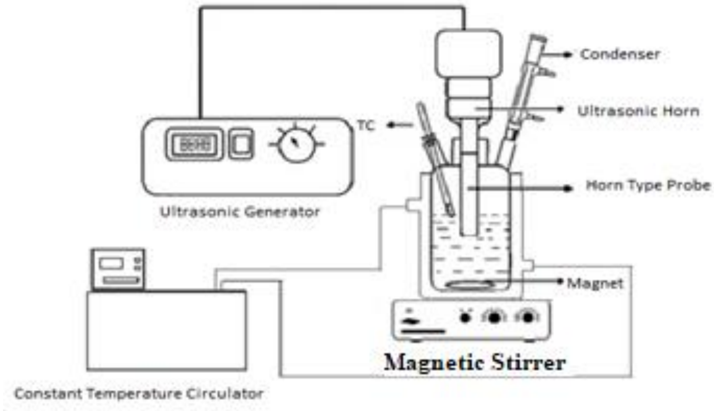


Figure 1. The experimental set-up

All transesterification experiments were carried out using the same reaction volume (700 mL) in a 1000 mL jacketed glass reactor. The reactor was equipped with a condenser, constant temperature circulator and a magnetic stirrer. The reaction temperature was kept constant at 60 °C to prevent beef tallow from freezing.

The melted beef tallow was initially filled into the reactor and heated to 60 ºC. Known weight the catalyst (KOH) was first dissolved in the methanol and added to the reactor. The reactor content was stirred magnetically at 650 rpm. The ultrasonic energy was sent at the determined power and time to reactor. The reaction was stopped when the predetermined reaction time was over. Phosphoric acid was added to the mixture until the pH was neutral to completely stop the reaction. The mixture was taken to a separator funnel and the glycerol phase was seperated at the bottom. Then the biodiesel (methyl esters phase) was washed three times with deionized water and the residual methanol was separated by rotary evaporator under vacuum. Finally, biodiesel samples were analyzed.

The fatty acid methyl ester (FAME) content of the biodiesel phase was analyzed by a gas chromatograph system equipped with an auto injector (Perkin Elmer Clarus 680, USA) and the BPX-7 capillary column (SGE, Melbourne, Australia, 60m x 0.25 mm id., 0.25). The initial oven temperature was 50 ºC for 4 minute, and was increased to 230ºC at a rate of 4 ºC/minute, then maintained for 10 minute. Methyl ester yields were calculated based on EN 14103.

Standard analyses for the ester content, acid value (ISO 660), viscosity (ISO 3104), density (ISO 3675), iodine value (ISO 3961), pour point (ISO 3015) and cloud point (ISO 3016) carried out to evaluate the quality of the biodiesel. All experiments and analysis were repeated twice and average values were described.

3. Results and Discussion

3.1. Performance Statistics, S/N Ratios

In this study, since the highest ester yield is important to us, larger-the-better type of performance characteristic was used in calculating the signal-to-noise (S/N) ratio. The S/N ratios of all the experiment are calculated using Eq. (1).

$$\frac{S}{N} = -\frac{10log(\sum 1/Yi^2)}{n} \quad (1)$$

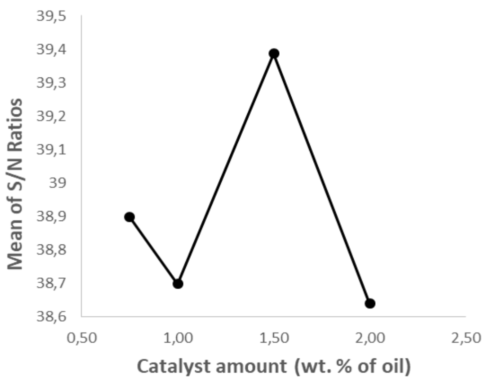
Where *S/N* is the performance statistics, *n* the number of repetitions done for an experimental combination, and Y_i performance value of *i*th experiment. The S / N ratios calculated from Eq. (1) are shown in Table 4.

Table 4. S/N ratio of the experiments

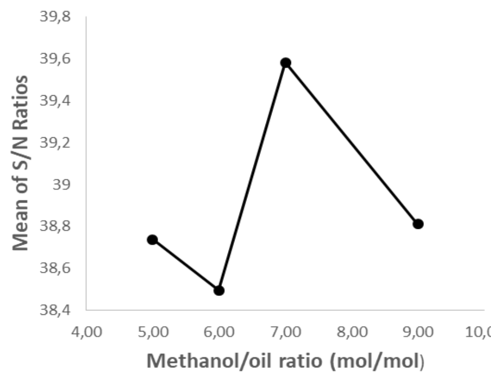
Experiment number	S/N ratio
1	38.1332
2	39.3350
3	39.9486
4	38.1922
5	38.0727
6	38.1922
7	39.2805
8	39.2663
9	39.9547
10	39.8987
11	39.8050
12	37.9877
13	39.3247
14	36.1372
15	39.8245
16	39.2918

3.2. Effect of Parameters on Biodiesel Yield

The results obtained from the experimental study are analyzed using the Minitab-13 computer software package program to examine the effect of each parameter on biodiesel yield and the mean of S/N ratios is shown in Figure 2.



(a)



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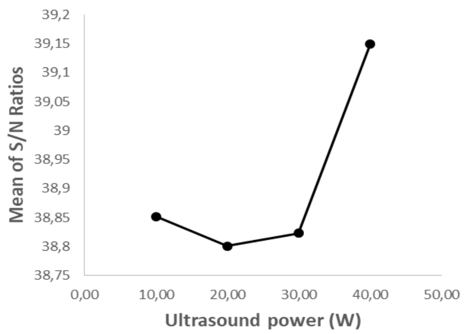

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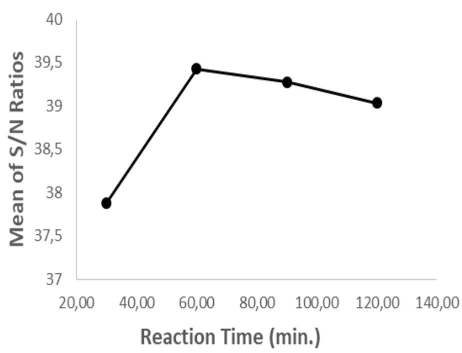

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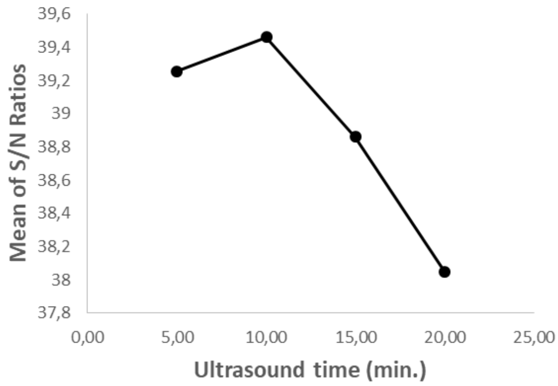

(e)

Figure 2. The mean effects plot for S/N ratios

Figure 2 shows the influence of parameters on the optimization criteria. For example, Figure 2a shows the variation of the performance statistics with catalyst amount. The catalyst amount for the first data point in Figure 2a is 0.75 (wt. % of oil) which is level 1 for this parameter. As Table 1 shows, experiments at level 1 of the catalyst amount (column A) are experiments 1,2,3, and 4. For this reason, the first data point in Figure 2a is created by taking averages of experiments 1, 2, 3 and 4. The other data points in Figure 2a are formed in the same way. The highest S/N value of each parameter in Figure 2 shows the optimum value for that parameter. Accordingly, the optimum conditions for this process are determined to be 1.5 (wt. % of oil) catalyst amount, 7/1 methanol/oil molar ratio, 60 minute reaction time, 10 minute ultrasound time and 40W ultrasound power (see Table 5).

Table 5. Optimum working conditions, calculated and observed biodiesel yield and confidence interval

Parameters		Optimum levels	Optimum value
A	Catalyst amount (wt. % of oil)	3	1.5
B	Methanol/oil ratio (mol/mol)	3	7/1
C	Ultrasound power (W)	4	40
D	Reaction time (minute)	2	60
E	Ultrasound time (minute)	2	10
Calculated optimum biodiesel yield (%)		100	
Observed optimum biodiesel yield (%)		99.8	
Confidence interval (confidence level α=95%)		98.36-100	

When Table 2 is examined, it is seen that the experiment corresponding to optimum conditions is not included in the experimental plan. Therefore, the experiment result (calculated biodiesel yield) at the optimum conditions is calculated using Eq. (2).

$$Y_i = \mu + X_i + e_i \quad (2)$$

where μ is the overall mean of performance value at the optimum conditions, X_i the fixed effect of the parameter level combination used in *i*th experiment, and e_i the random error in *i*th experiment. % transformation of Y_i calculated by Eq.(2) is made using Eq.(3) and this value is given in Table 5.

$$\Omega\ (\text{db}) = -Log\left(\frac{1}{P} - 1\right) \quad (3)$$

where Ω *(db)* is the decibel value of percentage value subject to omega transformation and *P* percentage of the product obtained experimentally.

The result calculated from Eq.(2) is an estimated result. For this reason, the confidence interval (CI) must be calculated to test this value. CI can be calculated using Eq. (4) and given in Table 5.

$$CI = \mu \pm \sqrt{\frac{F(1,n_2)V_e}{N_e}} \quad (4)$$

where $F(1,n_2)$ is the F value from the F table in any statistical book at the required confidence level and at degrees of freedom (DOF) 1 and error DOF n_2; Ve is the variance of error term (from ANOVA); and Ne is the effective number of replications

$$N_e = \frac{Total\ number\ of\ results\ (or\ number\ of\ \ S/N\ ratios)}{DOF\ of\ mean\ (=1\ always) + DOF\ of\ all\ factors\ included\ in\ the\ estimate\ of\ the\ mean}$$

The confirmation experiments in the optimum conditions have been carried out three times, the result obtained (99.8%) is in the confidence interval calculated at 95% confidence level (see Table 5) and the experimental results are within ±5% error.

3.3. Analysis of Variance (ANOVA)

Analysis of variance (ANOVA) is performed to statistically analyze the experimental results and to determine the effective parameters (Mahamuni & Adewuyi, 2010). Also, it is used to estimate the% activity of each parameter in the L16 test plan. In this study, ANOVA is used to identify the most important parameter to reach the maximum biodiesel yield. The ANOVA table containing the values as sum of squares, degrees of freedom, F, etc. was created and given in Table 6.

Table 6. ANOVA table

Parameters		Sum of Squares (SS)	Degrees of freedom (DOF)	Mean of Squares (MS)	*F*	Percent of the contribution (%)
A	Catalyst amount (wt. % of oil)	268.26	3	89.42	138.78	9.18
B	Methanol/oil ratio (mol/mol)	688.99	3	229.66	356.44	23.68
C	Ultrasound power (W)	64.98	3	21.66	33.62	2.17
D	Reaction time (minute)	1004.70	3	334.90	519.77	34.56
E	Ultrasound time (minute)	864.07	3	288.02	447.02	29.72
	Error	10.31	16	0.64		0.69
	Total	2901.3	31			100

As shown in Table 6, the least important contribution (2.17) is ultrasound power while the most important contribution (34.56) is reaction time. It can be said that the reaction time is the most important parameter for this process. According to Table 6, the power of ultrasonic energy has very little effect on the process. However, the ultrasound time seems to be very effective. In the process, effectiveness of the parameters are reaction time, ultrasound time, methanol/oil molar ratio, catalyst amount and ultrasound power, respectively.

3.4. Characterization of Biodiesel Product

The obtained product in optimum conditions was characterized by means of some of the EN 14214 and ASTM D 6751 standard specifications. The biodiesel synthesized at optimum conditions meets these standard specifications (Table 7). The maximum biodiesel yield obtained at optimum conditions was 99.8%. In previous studies, homogeneous one-step transesterification of beef tallow (base catalyzed) showed that biodiesel yield was 91% in 1 hour (Teixeira et al., 2009), 87.4% (Hoque, Singh, & Chuan, 2011), 90.8% (Mata, Cardoso, Ornelas, Neves, & Caetano, 2011) in 2 hours and 96.4% in 3 hours (da Cunha et al., 2009). Accordingly, the yield (99.8%) of biodiesel obtained from present work at the optimum condition is quite high when compared to earlier reported yield.

Table 7. Characterization of obtained biodiesel under optimum conditions

	Experimental values	Europe EN 14214	ASTM D 6751
Acid value (mg KOH / g oil)	0.4	< 0.5	< 0.8
Density at 15ºC (g/cm^3)	0.874	0.86-0.90	0.575-0.900
Kinematic viscosity at 40ºC (mm^2/s)	3.67	3.50-5.00	1.9-6.0
Iodine value (gI_2/100 g sample)	40.7	≤ 120	-
Cloud poind (ºC)	+11	-	-3 to +12
Pour point (ºC)	+ 9	-	-15 to +10

4. Conclusion

This study shows the application of the L16 parameter design (Taguchi method) in the optimization of the production of biodiesel from beef tallow with ultrasound energy. The results obtained in the study are given below:

- The most effective parameter on the production of biodiesel from beef tallow with ultrasound energy is reaction time. While the ultrasound power has little effect, the ultrasound time has a great effect on the process.
- The maximum biodiesel yield of around 99.8% is noted in this experimental work for optimum control parameters of 1.5 wt.% of oil for the catalyst amount, 7/1 methanol/oil molar ratio, 40W ultrasound power, 60 min reaction time and 10 min ultrasound time.
- The biodiesel produced under optimum conditions meets the EN 14214 and ASTM standard specifications.
- The Taguchi method can be used to optimize the biodiesel yield with lesser number of experiments.

5. Acknowledgements

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