Optimization of Production Parameters of Tobacco Seed Oil Methyl Ester using Multi-Response Taguchi Method and MANOVA

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Summary: There is a growing recognition that using of biodiesel in large commercial systems based on sustainability, existing resources and residues can help to natural resources. Tobacco seed oil (TSO) is also used for biodiesel production as a non-edible vegetable oil. A crude oil was obtained from tobacco seeds (TS) and then tobacco seed oil methyl ester-TSOME (biodiesel) was obtained by a transesterification process. In this study, we aimed to achieve optimal biodiesel properties based on different factors using multi response Taguchi method and multivariate analysis of variance (MANOVA). The purpose of the process was to meet a European Biodiesel Standard EN 14214. Properties of the biodiesel (responses) were determined as methyl ester quantity, kinematic viscosity, density, flash point of methyl ester and freezing point of methyl ester. The factors (production parameters) were chosen such as catalyst type, alcohol/oil molar ratio, reaction temperature and catalyst amount for experiment design. The factors that influenced the desired properties were determined using MANOVA. The factors' level was obtained using a multi response Taguchi method. We found that catalyst type and catalyst amount have a significant effect on the responses and their levels must be level 1(KOH) and level 3 (1.5%) respectively. Thereby the methods provided to produce biodiesel meet requirements of the standard with minimum cost and in short time.

Key words: Tobacco seed oil, Biodiesel, Optimization, Multi response Taguchi method, Multivariate analysis of variance (MANOVA)

Introduction

Biodiesel is an alternative renewable and biodegradable fuel with properties similar to petroleum diesel. Scarcity of traditional petroleum fuels, its over-dependence by nations, increasing emissions of combustion-generated pollutants and their increasing costs have made renewable energy sources more attractive. Currently, biodiesel is mainly prepared from conventionally grown edible oils such as rapeseed, soybean, sunflower and palm thus leading to alleviate food versus fuel issue [1-3]. Extensive use of edible oils may cause other significant problems such as starvation in developing countries. The use of non-edible plant oils when compared with edible oils is very significant in developing countries because of the tremendous demand for edible oils as food which are too expensive to be used as fuel at present.

Biodiesel is made up of fourteen different types of fatty acids, which are transformed into fatty acid methyl esters (FAME) by transesterification [4, 5]. Different fractions of each type of FAME in various feedstocks influence some properties of fuels. Vegetable oil is converted into biodiesel through a chemical process that produces methyl or ethyl ester. Commonly used alkali catalysts are potassium hydroxide (KOH) or sodium hydroxide (NaOH). Alkali-catalyzed transesterification is much faster than acid-catalyzed reaction [6, 7]. The chemical process is called transesterification which produces biodiesel and glycerin.

In this study, TSO was chosen in order to produce a biodiesel. The oil extraction process from tobacco seed and transesterification process for biodiesel production were considered. Hence, in biodiesel production in this study, the multi response Taguchi method developed by Tong et al. [8] and MANOVA were employed. It was expected that multi response Taguchi method and MANOVA together could provide optimal production conditions for biodiesel. Effective factors on responses were identified using MANOVA. If each response was separately analyzed with analysis of variance (ANOVA), it could be expected that each response affected by different factors. MANOVA provided to reach a general solution in case of consideration of all the responses at together. Levels of effective factors were determined by Multi response Taguchi method. If we used only multi response Taguchi method we could determine effective factors according to multi response signal to noise (MRSN) value. But in using MANOVA, responses were used instead of MRSN.

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In the literature, Taguchi experimental design was employed for transesterification of soybean oil with methanol by Mahamuni and Adewuyi [9], rapeseed methyl ester by Kim and Park [10] and biodiesel from sunflower by Antolin et al. [11]. There are also some studies related with applications of multi response Taguchi technique in the literature [12-14]. For example, Muqeem et al. (2017) also interested in multi response optimization for diesel engine using Taguchi approach with combining grey relation and principal components analyses [15]. Sharma et al. studied an optimization of diesel engine input parameters based on Polanga biodiesel to improve performance and exhaust emission using MOORA technique and standard deviation [16]. Muqeem et al. (2018) optimized diesel engine input parameters using Taguchi method to reduce hydrocarbon emission and smoke [17]. Saravanan et al. (2012) used Taguchi's orthogonal array in multi response to optimize NO_X emission parameter of diesel engine [18]. According to our limited literature review, no study in biodiesel production with using MANOVA and multi response Taguchi method.

Parametric studies were performed by using transesterification method for catalyst type, alcohol/oil molar ratio, and reaction temperature and catalyst amount factors. These factors and its levels were chosen based on the earlier experts' opinions and literature study [7, 9, 10, 14, 19]. Methyl ester was obtained during the experiments based on L18. Methyl ester quantity, kinematic viscosity, density, freezing point of methyl ester and flash point of methyl ester were measured in the experiments. We found that catalyst type and catalyst amount were significant effect on the responses and their levels must be level 1 (KOH) and level 3 (1.5%) respectively. Thereby the methods were appropriate to produce biodiesel meet requirements of the standard with minimum cost and in reasonable time.

Experimental

Oil extraction of TSO

In this study, TS (Sarıbağlar-407) were provided Aegean Agricultural Research Institute, Menemen districts of Izmir city in Turkey. n-hexane (96% analytical grade) and methanol were received from a local chemical store, Labkim in Istanbul, Turkey. Analytical grade potassium hydroxide (KOH) and sodium hydroxide (NaOH) were obtained from Merck, Germany. TSO was extracted using n-hexane solvent and the oil was separated from the solvent using a rotary vacuum evaporator in the laboratory condition. Fatty acid composition of TSO was determined by Gas Chromatography (GC) analysis. A Hewlett Packard 5890 series II Gas Chromatograph, with a SP2340.

Production procedure for biodiesel

Transesterification process was carried out using the Buchi rotary vacuum evaporator. Several factors may affect production processes of a biodiesel. In this study, the most important four factors and their levels such as catalyst type (KOH and NaOH) (factor A), alcohol/oil molar ratio (1/4, 1/5 and 1/6) (factor B), reaction temperature (40, 50 and 60 ºC) (factor C) and catalyst amount (0.5, 1 and 1.5%) (factor D) were chosen.

The prepared methanol-catalyst mixture was added to TSO to start the transesterification reaction by using the thermostatic bath and flask of rotary evaporator. For each parametric study, 100 grams of TSO sample was used. The mixture was stirred for one hour at 600 rpm. After completion of the transesterification, the reaction mixtures were allowed to cool down to room temperature to produce two phases: crude ester phase and glycerol phase. The bottom layer of glycerol was removed, and the upper layer of the ester (tobacco seed oil methyl ester-TSOME) was washed with warm pure distilled water in order to remove the impurities like unreacted methanol, unreacted oil and catalyst. The final methyl ester was separated from particles by using NUVE mark NF400 type centrifuge.

Multi Response Taguchi method

Multi response Taguchi method was employed in order to simultaneously evaluate multi responses. Application procedure of method is as follows. The quality loss for each response characteristic is computed based on following equations [8].

$$L_{ij} = k_3\left(\frac{s_{ij}}{\bar{y}_{ij}}\right)^2$$ for nominal the best response (1)

$$L_{ij} = k_2\frac{1}{n_i}\sum_{k=1}^{n_i}\frac{1}{y_{ijk}^2}$$ for the larger the better response (2)

$$L_{ij} = k_1\frac{1}{n_i}\sum_{k=1}^{n_i}y_{ijk}^2$$ for the smaller the better response (3)

where

L_{ij} quality loss for *i*th response at *j*th trial,
y_{ijk} observed data for *i*th response at *j*th trial *k*th repetition
n_i replication for *i*th response

$$\bar{y}_{ij} = \frac{1}{n_i}\sum_{k=1}^{n_i} y_{ijk}, \quad S_{ij} = \frac{1}{n_i - 1}\sum_{k=1}^{n_i}\left(y_{ijk} - \bar{y}_{ij}\right)^2$$

k_1, k_2 and k_3: quality loss coefficients.

Total normalized quality loss (TNQL) values were found based on the experiments values in Eq.4

$$TNQL_j = \sum_{i=1}^{5} w_i C_{ij} \qquad j = 1, \dots, 18 \qquad (4)$$

where w_i is weight of *i*th normalized response and assumed as 0.2. C_{ij} is normalized the quality loss of jth trial for ith response.

TNQL was transformed into a multi response signal noise ratio (MRSN) for each trial by using the Eq. 5.

$$MRSN_j = -10\log\left(TNQL_j\right) \qquad (5)$$

The estimated mean MRSN ratio for optimum experiment set can be calculated as the following

$$\bar{\eta} = \eta_m + \sum_{i=1}^{q}\left(\overline{\eta_i} - \eta_m\right) \qquad (6)$$

where, η_m is mean MRSN ratio, $\overline{\eta_i}$ is mean MRSN ratio corresponding to i^{th} factor and j^{th} level and q is number of factors.

MANOVA

Multivariate analysis of variance (MANOVA) is an extension of the univariate analysis of variance (ANOVA). The MANOVA essentially tests whether or not the factors simultaneously explains a statistically significant amount of variance in the responses. In other words it provides to determine effective factor(s) on the all responses. Two conditions must be ensured in order to apply a MANOVA test for an experimental result. First one is homogeneity condition test. The condition can be tested with Levene's. There is no homogeneity among variances of responses. Second condition is an equality of variance of responses in a covariance matrix test. It can be tested with Box's M. It is expected there is not covariance among dependent variables.

If significant value of factor is greater than 0.05 it is accepted be effective factor. ETA-squared (η^2) can be used as another measure. It is used to determine effect size of factor in MANOVA. If ETA-squared (η^2) value is range [0, 0.1], [0.1, 0.2] and [0.2, 1], the variance is entitled as "small", "medium" and "big" respectively.

Setting up experiments

Single-stage transesterification procedure was carried out in laboratory condition. Five responses which were properties of a biodiesel such as a methyl ester quantity (g), kinematic viscosity value (mm^2/s), density (g/L), freezing point of methyl ester (ºC), and flash point of methyl ester (ºC), were named y_1, y_2,y_3,y_4 and y_5 respectively. Their signal noise ratios were calculated according the larger the better for y_1 and y_5, nominal the best for y_2 and y_3, the smaller the better for y_4. L18 orthogonal array was chosen because one of the four factors described in section production procedure for biodiesel was 2, others were 3-level and the total degree of freedom was 7. Experiments were performed with repeated as twice. All required calculations based on the eq. 1-5 were summarized at Table 1.

Results and Discussion

TSO Characterization

The oil content of tobacco seed ranged from 40% on dry weight basis. The crude TSO consisted of a high proportion of unsaturation (87.96%), comprised primarily of linoleic acids (75.58%) and oleic (11.24%) and a lower proportion of saturation (11.98%), comprised mainly of palmitic acid (8.46%).

Analysis of Taguchi method and MANOVA

Before applying MANOVA, two needed tests, Levene's test and Box's M test, were applied to experiment data. Both of them indicated that required conditions of applying a MANOVA test were satisfied. Four different, Pillai's Trace, Wilks' Lambda, Hotelling's Trace and Roy's Largest Root tests were applied (Table 2).

Table-1: Design of the experiments and MRSN.

	Factors				Ester quantity (y_1)		Kinematic viscosity (y_2)		Density (y_3)		Freezing point (y_4)		Flash point (y_5)			
Experiment	A	B	C	D	1	2	1	2	1	2	1	2	1	2	TNQL	MRSN
1	1	1	1	1	100	99	3.865	3.765	833.333	834.500	-18	-19	148	150	2.081557	-3.18388
2	1	1	2	2	95	96	3.903	3.911	830.769	829.563	-12	-11	150	149	2.128377	-3.28048
3	1	1	3	3	85	85	2.941	2.945	826.923	827.373	-12	-13	150	151	1.996939	-3.00365
4	1	2	1	1	101	99	3.387	3.375	806.450	806.563	-10	-11	150	150	2.119504	-3.26234
5	1	2	2	2	99	100	3.731	3.630	812.500	811.365	-13	-14	144	145	2.984503	-4.74872
6	1	2	3	3	88	90	2.500	2.715	828.125	826.963	-19	-18	156	155	2.28637	-3.59146
7	1	3	1	2	102	99	3.581	3.641	818.181	818.236	-16	-17	146	148	7.198144	-8.57221
8	1	3	2	3	93	94	2.373	3.110	847.826	845.968	-12	-13	152	153	2.28840	-3.59532
9	1	3	3	1	94	93	4.021	3.989	838.709	838.863	-17	-17	142	142	2.703734	-4.31964
10	2	1	1	3	52	53	3.288	3.541	821.428	822.369	-16	-18	148	149	3.581643	-5.54082
11	2	1	2	1	93	94	3.089	3.560	819.444	820.324	-12	-13	156	158	2.349782	-3.71028
12	2	1	3	2	88	90	2.763	2.840	807.692	806.987	-16	-17	152	151	2.726127	-4.35546
13	2	2	1	2	94	94	3.325	3.630	827.586	828.658	-14	-15	148	148	2.309498	-3.63518
14	2	2	2	3	61	62	4.407	4.208	835.820	836.456	-11	-11	156	157	3.010047	-4.78573
15	2	2	3	1	95	94	2.672	2.840	812.500	812.874	-23	-24	148	148	3.94592	-5.96148
16	2	3	1	3	90	92	9.951	8.452	833.333	831.456	-10	-11	154	155	2.444433	-3.88178
17	2	3	2	1	94	95	3.044	3.254	854.166	854.235	-11	-12	150	149	2.812655	-4.49117
18	2	3	3	2	90	91	2.577	2.618	807.692	808.286	-24	-24	148	150	2.495916	-3.97230

Table-2: MANOVA results.

Effect		Value	F	Hypothesis df	Error df	Sig.	Partial Eta Squared
Intercept	Pillai's Trace	1.000	3,1602.113	5.000	32.000	0.000	1.000
	Wilks' Lambda	0.000	3,1602.113	5.000	32.000	0.000	1.000
	Hotelling's Trace	4,937.830	3,1602.113	5.000	32.000	0.000	1.000
	Roy's Largest Root	4,937.830	3,1602.113	5.000	32.000	0.000	1.000
A	Pillai's Trace	0.372	3.790	5.000	32.000	0.008	0.372
	Wilks' Lambda	0.628	3.790	5.000	32.000	0.008	0.372
	Hotelling's Trace	0.592	3.790	5.000	32.000	0.008	0.372
	Roy's Largest Root	0.592	3.790	5.000	32.000	0.008	0.372
B	Pillai's Trace	0.177	0.642	10.000	66.000	0.772	0.089
	Wilks' Lambda	0.830	0.625	10.000	64.000	0.787	0.089
	Hotelling's Trace	0.196	0.607	10.000	62.000	0.802	0.089
	Roy's Largest Root	0.122	0.803	5.000	33.000	0.556	0.108
C	Pillai's Trace	0.280	1.074	10.000	66.000	0.394	0.140
	Wilks' Lambda	0.738	1.049	10.000	64.000	0.414	0.141
	Hotelling's Trace	0.330	1.023	10.000	62.000	0.435	0.142
	Roy's Largest Root	0.216	1.426	5.000	33.000	0.241	0.178
D	Pillai's Trace	0.328	1.293	10.000	66.000	0.253	0.164
	Wilks' Lambda	0.682	1.348	10.000	64.000	0.225	0.174
	Hotelling's Trace	0.451	1.398	10.000	62.000	0.202	0.184
	Roy's Largest Root	0.416	2.746	5.000	33.000	0.035	0.294

The MANOVA results indicated that factor A and D have a significant effect on the responses accordingly Roy's Largest Root test. But, B and C factors have no significant effect on the responses. Other tests such as Pillar's Trace, Wilks'Lambda and Hotelling's Trace gave that only factor A is effective factor. It was accepted that results of Roy's Largest Root test for that was powerful test than others. Likewise, according to the ETA-squared results on Table 2, factors A and D have a big effect whereas factor B and C have a medium effect on responses.

It was found means of MRSN ratios of levels for determining level of effective factor (Fig. 1). It was found as difference among maximum and minimum MRSN values of levels. The factors set were found as level 1 (KOH) and level 3 (1.5%) for effective factors respectively catalyst type (A) and catalyst amount values (D) respectively. There was not significant difference between level 1, level 2 and 3 for not effective factors (alcohol/oil molar ratio and reaction temperature) instead of level 1 using level 2 or level 3 does not alter the results. It was decided that alcohol/oil molar ratio (B) and reaction temperature (C) levels must be 1/4 and 50°C respectively. The optimum combination of factor level was defined as $A_1B_1C_2D_3$

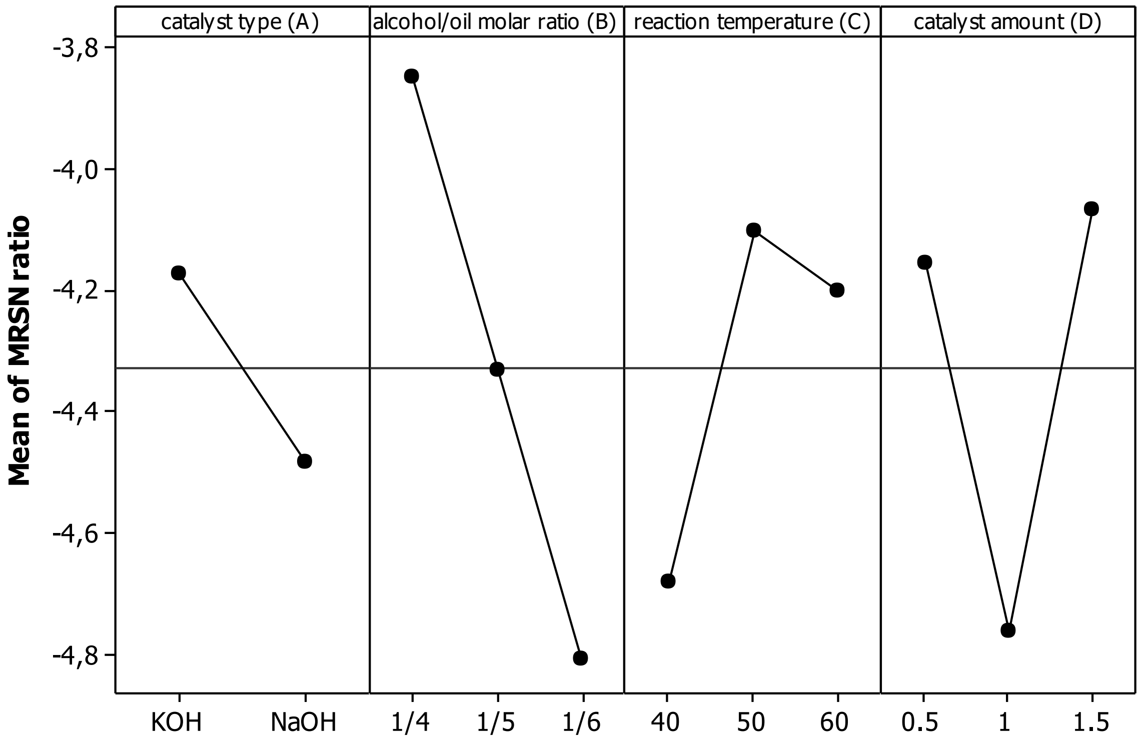

Fig. 1: MRSN ratio levels of factors.

The predicted mean MRSN ratio for each combination of factor levels in the L_{18} orthogonal array was calculated by using equation 6. The predicted mean MRSN ratio (-3.20652) of the determined optimal combination ($A_1B_1C_2D_3$) was found to be greater than that found for 18 trials (Table-3). This result shows that the $A_1B_1C_2D_3$ yields optimal results.

Table-3: Predicted mean MRSN ratios.

Exp No	Predicted mean MRSN ratio	Exp No	Predicted mean MRSN ratio
1	-3.87139	10	-4.09118
2	-3.90134	11	-3.60299
3	-3.30453	12	-4.30747
4	-4,35730	13	-5.27034
5	-4,38568	14	-3.99899
6	-3.78885	15	-4.18532
7	-5,43663	16	-5.04994
8	-4.16528	17	-4.56175
9	-4,35161	18	-5.26622

When each response was separately analyzed from each other by Taguchi technique, it might expected that different levels of factors could possibly be an effective response. ANOVA tests were repeated based on every single response (y), different effective factors become significant on chosen response. For example, in case of y_1 chosen as response, first level of factor A becomes significant. However, second level of factor A become significant in case of y_5 was chosen as response. Therefore, it was difficulty to reach a general solution in case of consideration of all the responses at same time. Therefore, it was believed that the multi response Taguchi method and MANOVA produced a better choice in order to find out a general solution that optimized all the responses. In spite of the fact that the technique did not provide "the best" value of the every responses, but, it produced "better results" with considering of total responses. There was also other difficulty in order to reach a common judgement about effective factors on all responses at the same time. The MANOVA provided an answer of this difficulty. The results are given at Table 4. It was used Minitab15 for analyzing experiment result and MANOVA, respectively.

Table-4: Comparison of single and multi-responses.

Method	Response(s)	Factors				Taguchi Technique
		A	B	C	D	Multi responses
MANOVA	y_1, y_2,y_3,y_4,y_5					A_1D_3
		Single response				
	y_1					A_1
	y_2					C_3D_2
ANOVA	y_3			-		C_3D_2
	y_4					B_3
	y_5					A_2D_3

Table-5: Fuel properties of TSOME.

Analyses	Unit	Method	TSOME	EN 14214	Diesel No 2
Ester content	%(m/m)	EN 14103	96.5	min 96.5	-
Density, 15 °C	g/cm^3	EN ISO 3675	0.88	0.86 - 0.90	0.82 – 0.86
Kinematic viscosity	mm^2/s	EN ISO 3104	4.88	3.5-5	2.5 – 3.5
Carbon residue	%(m/m)	EN ISO 10370	0.17	-	-
Acid value	mgKOH/g	EN 14104	0.25	max 0.50	-
Iodine value	g iodine/100 g	EN14111	122	max 120	-
Pour point	°C	ASTM D97	-6	max 0	- 33
Flash point	°C	ASTM D93	135	min 120	> 55
Heating value	MJ/kg	ASTM D 240	39.16	min 35	42.7
Copper band corrosion (3 hours at 50 °C)	-	EN ISO 2160	1a	1a	-
Total contamination	mg/kg	EN12662	20	-	-
Cetane number	-	EN ISO 5165	54.5	min 51	49 - 55
Total glycerol	%(m/m)	EN14105	0.02	max 0.25	-

TSOME Characterization

After the optimum combination of factor level ($A_1B_1C_2D_3$) was determined, tobacco seed oil methyl ester was produced according to this combination. Fuel properties of TSOME results as compared with those of the European biodiesel standard EN 14214 is shown in Table 5. It was seen that the analysis results satisfied all the values of specified in EN 14214.

Conclusions

For batch transesterification of tobacco seed oil with methanol, the influence of four processing factors on the methyl ester quantity, kinematic viscosity, density, freezing point and flash point were simultaneously analyzed employing the multi response Taguchi method. Factors' levels were found by employing MRSN ratio. In addition that the MANOVA test was carried out determine effective factors with considering five responses at the same time. The following conclusions could be deduced as follows:

i) According MANOVA test, only factor A (catalyst type) and D (catalyst amount) had significant effect on the responses. Multi response Taguchi method provided these factors levels such as A_1D_3.
ii) MANOVA test indicated that two factors B (alcohol/oil molar ratio) and C (reaction temperature) were insignificant. Experts can decide these insignificant factors' levels as 1 or 2 or 3. (it can be chosen any level). It was decided be B_1C_2 according to mean MRSN ratio value in this study. Further improvement of experimental investigations can be done with considering only significant factors. Therefore, considerably less number of experimental investigations and possible more less experimental cost may come out.
iii) Tobacco seed oil methyl ester produced in accordance with the optimum combination determined by the methods satisfied the EN 14214 biodiesel standards.

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