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Effective Parameters on Biodiesel Production from Feather fat oil as a Cost-Effective Feedstock

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ABSTRACT: Using low-cost feedstocks such as rendered animal fats in biodiesel production willreducebiodieselexpenditures. One of the low-cost feedstocksfor biodiesel production could be the fat extracted from poultry feathers producedin slaughterhouses abundantly. This paper describes a new and environmentally friendly process for developing biodiesel production technology from feather waste produced in poultry industry. In this research the crude oil of poultry feather fat was extracted by soxhlet method using hexane as a solvent. The data resulted from gas chromatography (GC) revealed these percentages for fatty acid compositions: myristic acid (3%), palmitic acid (30%), stearic acid (22%), oleic acid (8.1%), linoleic acid (3%) and arachidonic acid (7%).In this experimental research, the effects of some parameters such as alcohol to oil molar ratio (4:1,6:1, 8:1), catalyst concentration (0.75,1 and 1.25% w/w) and the transesterification reaction time(40,60 and80min) on the percentage offatty acids conversioninto methyl ester(biodiesel) are studied. The results show increasing catalyst concentration up to 1% causes the oil to biodiesel conversion percentage having an upward trend and then adownward trend byincreasing catalyst concentration up to 1.25%. With increasing molar ratio from 4:1 to 6:1 and then 8:1, oil to biodiesel conversion percentage increased16% and2%, respectively. Ultimatelythe optimum point defined by response surface method (RSM) forproducing biodiesel from feather fat is calculated catalyst concentration of 1 wt%, 7.24:1 molar ratio and in 75 minutes resulting in conversion percentage of97.62%.

Key words: Feather,Biodiesel,Optimization,RSM,Transesterification

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INTRODUCTION

Energy is the single most important resource capable of sustaining life on earth. Energy not only is the engine of economic growth but also the cause of important life threatening outcomes (Karbassi *et al.*, 2008).The strong interest in liquid biofuel is due to the fact that it can be used as a supplement, or alternative, to gasoline or diesel fuel derived from petroleum fossil fuel (Ghobadian, 2012a).About 100 years ago, RadolfDiesel invented diesel engine which worked with vegetable fuel (Knothe *et al.*, 2005, Najafi *et al.*, 2009). Over time with entrance of oil as new and cheap fuel, tendency to this fuel increased. Vegetable oils such as canola oil have been used in diesel engines. When it is used in IC (internal combustion) engines, this oil owes some problems such as low ignition quality. All oils have high viscosity and need specific injection pumps and injectors. Mixing this oil with oil derivatives, could partially solve the high viscosity problem. Therefore, because of this fuel advantages a lot of researchers have attempted to produce it. Several ways have been used to produce biodiesel fuel and there are three common methods including Micro emulsion, pyrolysis and Transesterification(Schwab *et al.*, 1987and Balat and Balat, 2008).Among the mentioned methods, transesterification is commonly used for biodiesel production because of its higher yield and lower energy consumption.Transesterification is a chemical process of reacting triglycerides with alcohol in the presence of a catalyst. If the reaction is not completed, then there will be mono-, di- and tri-glycerides left in the reaction mixture. Alcohols such as methanol, ethanol or butanol can be used in the transesterification (Canakci, 2007).

Natural oils transesterification reaction with appropriate alcohol in presence of acid or alkaline catalyst about 1 to 2 hours inside reactor is discrete. Because of the lack of amalgametion between oil and alcohol, mixture efficiency is one of the determining factors in reaction yields (Hanh *et al.*, 2008).

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Various studies and investigations have revealed that about 70 to 75 percent of the biodiesel fuel cost goes for the feedstock. The feedstock for biodiesel production differs from place to place and from country to country (Ghobadian, 2012b).

Biodiesel is usually produced from high quality vegetable oils. These feedstocks have high cost, which currently accounts for over 85% of biodiesel production expenses(Encinar*et al.*, 2011). In the United States, soy, corn, canola, and cotton seed oil are the primary sources for biodiesel production. The use of these feedstocks for a prolonged time is potentially detrimental to society and the environment. According to the U.S. Department of Agriculture (USDA), “Industrial consumption of vegetable oil, which is dominated by biodiesel production, accounts for 40% of the annual vegetable oil demand growth yet represents only 20% of the overall vegetable oil consumption. Demand for non-food use of oil is expected to grow 10% annually”. This growth causes the line between food and fuel economies to be blurred as both of these stocks are competing for the same oil resources. A successful biofuel industry will not be based on digestible starch from staple crops such as corn. The main problem the biodiesel industry frequently faces is the availability of cheap and abundant, high-quality feedstock. Thus, finding alternative, nonfood, feedstocks such as waste vegetable oil, grease, and animal fats (beef tallow) is considered a necessity for the industry. Through continued research to produce biofuels from nonfood sources, it has been discovered that poultry feather offers another promising feedstock source for biodiesel production(Kondamudi *et al.*, 2009). Feather fat is a low-cost feedstock for biodiesel production compared to high-grade vegetable oils.

Feathers are byproducts of poultry processing plant and produced in large amount. Worldwide 24 billion chickens are killed annually and around 8.5 billion tones of poultry feather are produced. Currently the poultry feathers are treated in some ways such as dumping, landfilling, composting and incinerating,which involve problems in storage, handling, emissions control and ash disposal (Fukuda *et al.*, 2001 and Abduliand Azimi, 2010). Moreover feather meal is used as an animal feed, given its high protein content, and also as a fertilizer because of its high nitrogen content (Kondamudi *et al.*, 2009). The utilization of feather fats for biodiesel production is a good alternative to recycle these wastes (Encinar *et al.*, 2011).

In 2010, chicken was the most common and widespread domestic species, with a consumption of more than 8.6 million tonsthat year, and according to FAO[1]of the United Nations, the production and consumption of chicken meat arepersistently growing. In Europe, the chicken consumption reached 20 kg/ capita/year in 2007,according to FAO, while in the USA; the consumption of chicken has surpassed 50 kg/ capita/year.Deeming a mature chicken to weigh 1.8–1.9 kg (1.5 kg of meat) (Salminen and Rintala, 2002), with 5–7 % of its bodyweight comprising feathers (Gessesse *et al.*, 2003),the generation of chicken feather waste is easily estimated.

Municipal Solid Waste (MSW) disposal has always been an important issue for governments all over the world (Abdoli *et al.*, 2012).In this research the feasibility of biodiesel production from feather fat and also the effects of factors such asalcohol to oil molar ratio, catalyst concentration and time of reaction on methyl ester production from feather fat areinvestigated.

MATERIALS & METHODS

Acid catalysts are too slow to be suitable for converting triglycerides to biodiesel. However, they appear to be quite effective at converting FFAs to esters. Because of these reasons, an acid catalyst can be used to esterify the FFAs to esters. The acid-catalyzed process is called as pretreatment. FFAs are converted to monoesters through the pretreatment of the feedstock with high FFA and thereby the FFA level reduces. The major handicap for the acid-catalyzed esterification of FFAs is the water formation. The water formation is the primary mechanism limiting the completion of the acid catalyzed esterification reaction with FFAs. After pretreatment, the pretreated feedstock can be transesterified with an alkalin catalyst to convert the triglycerides into esters. Many researchers have investigated the availability of animal fats and waste oils for biodiesel production. However, few researchers have studied on the feather fat especially with high FFA (Kondamudi *et al.*, 2009).

In order to obtain feather fat for biodiesel production, the feather should be supplied from slaughterhouse. The test samples were selected from slaughterhouse in Tehran randomly (Tehran is the capital of Iran, at 35°412 463 N latitude and N 51°252 233 E longitude).

Feathers are composed of 90–95 % of proteins (Onifade, 1998 and Haddar, 2009) and the fat content of the feather varies from 2 to 12% depending on the kind of used feathers (Dale, 1992).For example, chicken feathers contain approximately 11% fat content, while turkey and duck feathers contain approximately 6.7% fat content (Kondamudi *et al.*, 2009).

Most of the studies reported the use of non-polar (hexane) solvent as an extraction solvent. The extraction of oil with hexane gave highest oil yield(Nwabueze and Okocha, 2008).Hexane is a non-polar solvent. Fats are also non-polar substances and thus highly and rapidly soluble in hexane as compared to any other solvent. Therefore, if a total fat content determination is required, hexane is the best while not suitable for polar and partially polar lipids. Fat content was determined by soxhlet extraction with hexane for 8 hoursat boiling point of the solvent(Kumar *et al.*, 2012).Hexane's boiling point at 760mm HG is 69°C.Hexane was used, instead of ether, for the solvent extraction to minimize the extraction of unwanted polar compounds, such as free fatty acids found in the feather samples(Kondamudi *et al.*, 2009).Oil was extracted with boiling hexane in the soxhlet extractor for 8 hours at 69°C. Then feather samples were dried in an oven at a temperature of 70 °C for 24 hours to remove water content, and then hexane wasdeleted (Abraeva *et al.*, 2011).

According to data resulted from soxhlet method, the oil content in the samples were 5.80% (I),6.55% (II) and 6.05% (III). It was revealedthat supplied feathersamples contain averagely 6.13% fat content. Increasing the time of extraction and/or using ultrasonic technic might further improve the fat extraction capabilities.

Chemical composition of oils and fats used in the biodiesel synthesis can influence processing and storage conditions, due to the percentage of unsaturated fatty acids(Ramalho *et al.*, 2011).Therefore, the Metcalf method was used to metilize the extracted oil. Fatty acid profile was determined using a gas chromatography. However, feather fats often contain significant amounts of free fatty acid (FFA) which cannot be converted to biodiesel using an alkaline catalyst due to the formation of soap(Alptekin *et al.*, 2011). Fatty acid composition of poultry fat has been shown in table 1 and the chromatogram of poultry fat oil used in this research work is shown in fig. 1.

Table 1. Fatty acid composition of poultry fat

Fatty acid	Amount(%)
C 14:0 myristic	3 %
C 16:0 palmitic	30 %
C 18:0 stearic	22 %
C 18:1 oleic	8.1 %
C 18:2 linoleic	3 %
C 18:3 linolenic	25 %
C 20:0 arashidic acid	7 %
Other fatty acids	1.8%

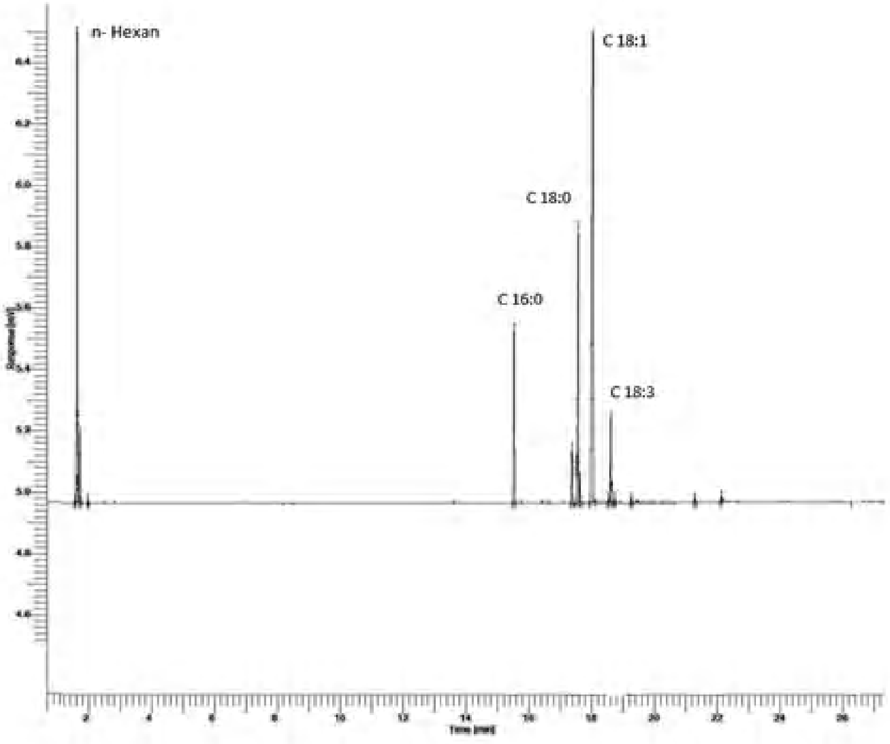

Fig. 1. chromatogram of poultry fat oil used in this research work

High free fatty acid content of the oil caused transesterification reaction to fail and led to soap production. For free fatty acid (FFA) content of more than 1%, the following equations (based on weight ratio) were used for titration and calculation of catalyst rates(Gerpen *et al.*, 2004):

$$\%FFA = \frac{0/5 \times A \times N \times W_{cat}}{W} \tag{1}$$

$$KOH\,(gr) = \frac{[\%FFA] \times 0/197}{0/86} + \%1 \tag{2}$$

Where:
A=Catalyst volume for oil titration (mL)
W= The sample value (g)
N= Normality
W_{cat}= Molecular mass of catalyst (g) which is 56.1 for KOH

The free fatty acid (FFA) level of the feather fat was determined 24% using titration method, which is not sufficient to complete the reaction with alkaline catalysts.The reduction of FFA esterification reaction was carried out using methanol and sulfuric acid.After reactions proceeded to completion, the level of FFA was reduced to less than 1%. Thetransesterification reaction started in the presence of methanol, alkaline catalyst and the feather oil with free fatty acid levels of less than 1%.

Methanol (99.7%) and KOH (99%) used in this study, were provided from Merck company products. The most preferred alcohol used in biodiesel production is methanol because of its low price, physical and chemical advantages (polar and the shortest chain alcohol). It can easily react with triglycerides and catalysts (Alptekin and Canakci, 2011).Potassium hydroxide (KOH, 99% assays) in the form of solid tablets was used as catalyst.

In this research work the effect of alcohol to oil molar ratio (4:1, 6:1 and 8:1), catalyst concentration (0.75, 1 and 1.25%) and reaction time (40, 60 and 80 min) on oil conversion into methyl ester (biodiesel) were investigated. Gas chromatography (GC) set, Perkin Elmer clarus-580 based on BS-EN 14103 standards and equations (3 and 4) were used to determine biodiesel yield and percentage of methyl ester content in the produced biodiesel.Therefore:

$$C = \frac{\sum A - A_{IS}}{A_{IS}} \times \frac{M_{IS}}{M} \times 100 \tag{3}$$

Where:
Σ A= Total area under pick for fatty acids C_6 to C_{14}, µV*sec.
A_{IS} = Area under thrinternal standard (methyl heptadecanoate) pick, µV*sec.
M_{IS} = Mass of applied internal standard (mg).
M = Mass of biodiesel sample (mg).

Conversion percentage of oil to methyl ester (biodiesel) obtained from following equation(4) (Thanh *et al.*, 2010):

$$\text{FAME}(\%) = \frac{w_{FAME} / M_{FAME}}{3w_{WCO} / M_{WCO}} \times 100 \tag{4}$$

Where:
W_{FAME}= Produced biodiesel (mg)
W_{WCO}= mass of feather fat(mg)
M_{FAME}= Mean molecular mass for biodiesel
M_{WCO}= Mean molecular mass for feather fat oil

For the transesterificationto give maximum yield, the alcohol should be free of moisture and the FFA content of the oil should be less than 0.5% (Geise, 2002). The absence of moisture in the transesterification reaction is important because according to the equation (shown for methyl esters),

R-COOCH3 + H2O '!R-COOH + CH3OH
(R = alkyl)

hydrolysis of the formed alkyl esters to FFA can occur. Similarly, because triacylglycerols are also esters, the reaction of the triacylglycerols with water can form FFA. At 32°C, transesterification was 99% complete in 4 hours when using an alkaline catalyst (NaOH or NaOMe) (Gerpen and Knothe, 2005).Two phases could be identified after the pre-treatment.The upper phase consisted of methanol, catalyst, H_2O, resulted soap and impurities. This layer should be separated to reach acceptable levels for fuel parameters. The lower phase mainly consisted of fats and the esterified fatty acids.For this aim, the biodiesels were obtained by transesterification reaction, via methylic routes. Samples and reaction final mixture were placed in decantation funnels and allowed to stand overnight and centrifuged to ensure the complete phase separation (methyl esters and glycerol).After reaction, the glycerol phase (bottom phase) was separated by decantation and the biodiesel phase (upper phase) was heated at 85°C(with anhydrous sodium sulfate, before filtration) to eliminate methanol. The biodiesel was neutralized with KOH (20% needed KOH to neutralize the added H_2SO_4 as catalyst) and it was washed with distilled water to remove catalyst and reach neutral pH.The collected fat was mixed with a basic solution of potassium hydroxide (KOH) to remove the free fatty acids (FFA) in the form of soap. The soap was separated from the fat content by centrifugation. The purified fat was processed to the next step, transesterification.

Transesterification of the purified fat described above was conducted to convert triglycerides to biodiesel. In this process, the recovered fat content waspreheated to 100°C and cooled to room temperature to remove the tracesof water content.A solution of and KOH and methanol (as a catalyst) were added to the fat. The reaction mixture wasrefluxed at 60°C for 1 hour. Optimization of the transesterification reactionwas achieved by varying the amounts of methanol and potassium hydroxide.After the transesterification process, the reaction mixture was allowed to cool to room temperature overnight. The glycerol layer, which contains un-reacted alcohol and catalyst, was separated from the biodiesel. The top layer was then washed twice with warm water (40-45 °C) and with acidified water (0.5 wt%tannic acid) to remove the excess methanol and the traces of catalyst (Kondamudi *et al.*, 2009).

This two-step protocol typically gives high degrees of transesterification (>98%), with negligible amounts of remaining unreacted (complete or partial) acylglycerols. The final ester product separates readily from the polar liquid phase, which contains unreacted alcohol, the glycerol coproduct, and the catalyst.

The water washing step is intended to remove any remaining catalyst, soap, salts, methanol, or freeglycerol from the biodiesel Neutralization before washing reduces the amount of water required and minimizes the potential for emulsions to form when the wash water is added to the biodiesel. After the wash process, any remaining water is removed from the biodiesel by a vacuum flash process (Gerpen and Knothe, 2005).

After drying, the produced feather fat methyl esters (biodiesel) were characterized by determining their viscosity, density, flash point and acid value and the purified biodiesel was characterized, Gas chromatography (GC) set Perkin Elmer clarus-580 based on BS-EN 14103 standards(Fig. 2).

In this research work the effect of alcohol to oil molar ratio (4:1, 6:1 and 8:1), catalyst concentration (0.75, 1 and 1.25%) and reaction time using ultrasonication (40,60 and80 min) on oil to methyl ester (biodiesel) conversion were investigated. All data were analyzed by analysis of variance and mean comparison by SAS 10 and MiniTAB16 software and the process optimized using response surface methodology (RSM).

In this research response surface method and box-behnken design were used. First dependent variable's level according to Table 2 was coded and based on selected method, required tests were chosen according to Table 3. Finally, due to the depicated diagrams and the domain for independent variables, the optimal point was found and this point was evaluated practically by software. Software results were then compared with experimental data.

Fig. 2. The Gas Chromatograph used for oil and produced biodiesel analysis

Table 2. Coded and actual values of variables used for the experimental design

Independent variable	Coded variables levels		
	-1	0	1
Catalyst (%)	0.75	1	1.25
Molar ratio (alcohol to oil)	4:1	6:1	8:1
Time(min)	40	60	80

Table 3. Experimental coded conditions for biodiesel production

Random	Run	Catalyst	Molar ratio	Time
15	1	0	0	0
13	2	0	0	0
8	3	1	0	1
4	4	1	1	0
9	5	0	-1	-1
10	6	0	1	-1
11	7	0	-1	1
14	8	0	0	0
1	9	-1	-1	0
2	10	1	-1	0
7	11	-1	0	1
3	12	-1	1	0
6	13	1	0	-1
12	14	0	1	1
5	15	-1	0	-1

Table 4. Analysis of variance (ANOVA) for the biodiesel production

Source	DF	Sum of squares	MSE	F
Catalyst	2	11550.50	5775.25	1220.59**
Molar Ratio	2	3311.98	1655.99	349.99**
Time	2	1878.79	939.39	198.54**
Catalyst* Molar Ratio	4	65.71	16.43	3.47*
Catalyst* Time	4	119.38	29.85	6.31**
Molar Ratio* Time	4	63.08	15.77	3.33*
Catalyst* Molar Ratio* Time	8	17.26	2.16	0.46^{ns}
Error	54	255.50	4.73	
Total	80	17630.18		

**and * significant in 1% and 5% level, respectively, ns: non significant

RESULTS & DISCUSSIONS

Analysis of variance indicated the effect of factors including catalyst concentration, molar ratio, time and interaction of catalyst concentration and time had significant effect ($p<0.01$), interaction of catalyst concentration and molar ratio and interaction ofmolar ratio and time had significant effect ($p<0.05$) on biodiesel production (Table 4).

As Fig. 3 shows, by increasing catalyst concentration from 0.75% to 1% (w/w) in different molar ratios, biodiesel conversion rate increases 19.01% averagely and up to 1.25% catalyst concentration rate decreases 20.76% averagely. Fig. 3 shows mean comparison (LSD) between different levels of molar ratio and catalyst concentration interaction ($p<0.01$) on molar ratios 4:1, 6:1 and 8:1. But between two molar ratios (6:1 and 8:1) there wasn't significant effect for

0.75% catalyst concentration level. Therefore, there would be an optimum Interval for catalyst concentration on these effective molar ratios that should be investigated by response surface method.

By increasing catalyst concentration percentage from 0.75% to 1%, additional amount of catalyst runs the transesterification reaction forward, breaking triglyceride structure and increasing biodiesel production. Saponification could be the reason of conversion reduction from 1% to 1.25%. In higher percentages, due to presence of the small amount of free fatty acids and saponification in presence of alkaline catalyst, viscosity of mixture increases. This leads to effective catalyst concentration rate reduction in reaction and as a result conversion percentage will reduce (Hingu et al, 2010). Catalyst cost would be a considerable part of biodiesel production expenses. Therefore considering reduction of catalyst concentration would be so much effective in reducing the process expenses and response surface method could be usable for this purpose.

Obtained results show that increasing molar ratio of alcohol to oil leads to conversion percentage rate increase. As this ratio increases from 4:1 to 6:1 and 8:1 conversion percentage increases 12.31% and 2.22%, respectively (Fig. 4).

This can be related to equilibrium of transesterification reaction that with increasing alcohol molar ratio to oil, transesterification reaction progresses and increase biodiesel production. It should be mentioned this increase in biodiesel conversion percentage resulted from molar ratio has some restrictions. If this ratio rises above a certain level, biodiesel production will reduce. The principle reason of this occurrence might be related to the amount of methanol increase in mixture, which leads to more dissolution of glycerin and alcohol in biodiesel and this will considerably influence its purity.Several studies have reported similar results about catalyst concentration increase and molar ratio (Encinar *et al.*, 2005, Anwar and Rashid, 2007).

By increasing transesterifcation reaction time, biodiesel production rate increases but increase rate differs in different catalyst percentages(Fig. 5). For different levels of catalyst concentrations (0.75%, 1% and 1.25%) with increasing time, biodiesel conversion increases 5.78% and 6.01%, respectively and the significant effect of interaction of these twofactors is because of this difference. As fig. 6 shows, saponification rate in 1.25% catalystconcentrationfor all three reaction times (40, 60 and 80 min) has caused the best biodiesel conversion to be in 1% catalyst concentration. Mean comparison (LSD) of data showed there is significant effect ($p<0.01$) between different levels of catalyst in different reaction times from40 min to 80 min of timereaction considerably advances. Besides, low amount of biodiesel production in 1.25% catalyst concentration in all three time levels with respect to other catalyst concentration levels is a sign of saponification. Other researchers have reported similar results (Safieddin *et al.*, 2011).

Fig. 7 shows with increasing reaction time from 40 to 60 minutes significantly effects ($p<0.01$) on all molar ratios. On the other hand with increasing molar ratio from 4:1 to 8:1 at all three reaction time levels have significant effect except on 80 min reaction time level between 6:1 and 8:1 molar ratios.

Therefore it could be found between different test levels of molar ratio, considering low consumption of alcohol; 6:1 molar ratio is the best molar ratio. Fig. 8 shows biodiesel conversion rate changes versus molar ratio and reaction time. Hingu et al also has reported similar results (Hingu *et al.*, 2010).

According to the fitted regression equations for tests data, the best objective function is defined as following:

$$\text{Yield}= -120.15667+187.36167*C+21.72167*M+1.07128*T-0.17833*C*M-0.095111*C*T-95.55333*C^2-1.51792*M^2$$

In which:
C: Catalyst concentration
M: Molar ratio
T: Time (minute)
Y: biodiesel conversion

Then using boundary conditions optimal point is defined by response surface method. As fig. 9 shows, with increasing catalyst concentration up to 1% biodiesel conversion increases and afterwards it gets downward slope. Besides with increasing reaction time, biodiesel conversion rate increases. Increasing molar ratio from 4:1 to 6:1 has greater slope on biodiesel conversion with respect to increasing molar ratio from 6:1 to 8:1. According to the biodiesel conversion changes versus other mentioned independent variables optimal point for biodiesel conversion was found to be 97.62%, when catalyst concentration, molar ratio and reaction time were 0.96%, 7.23:1 and 75 minutes respectively.

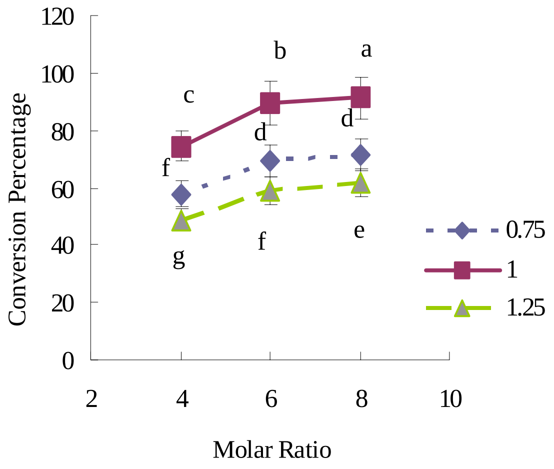

Fig. 3.The relationship between molar ratio and biodiesel conversion for different catalyst concentrations

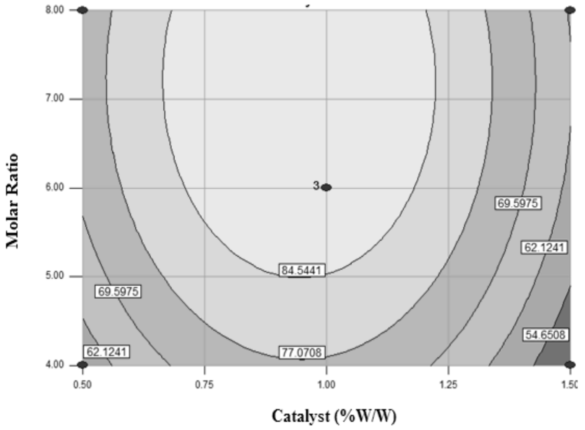

Fig. 4. conversion percentage variation versus catalystconcentrationand molar ratio variation

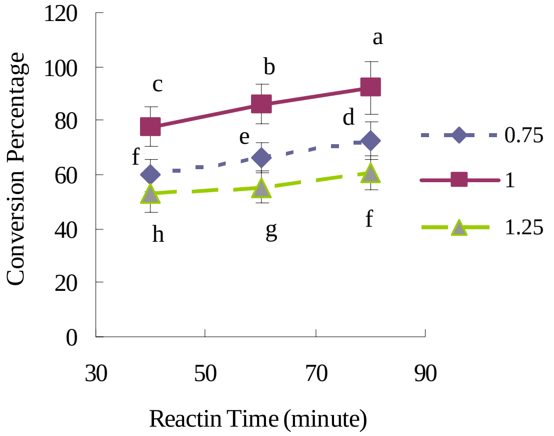

Fig. 5.The relationship between elapsed reaction time and biodiesel conversion for different catalyst concentrations

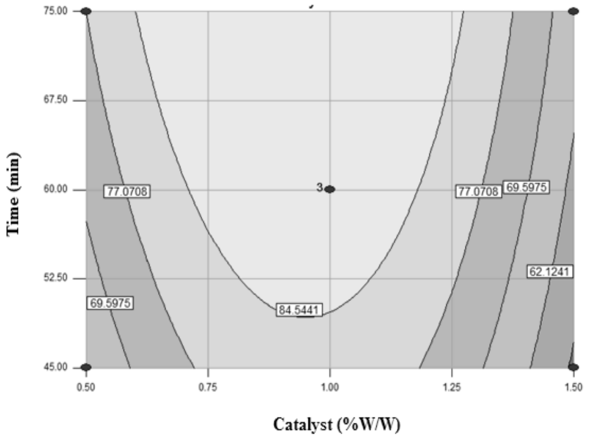

Fig. 6.Conversion percentage variation versus catalystconcentration and reaction time variation

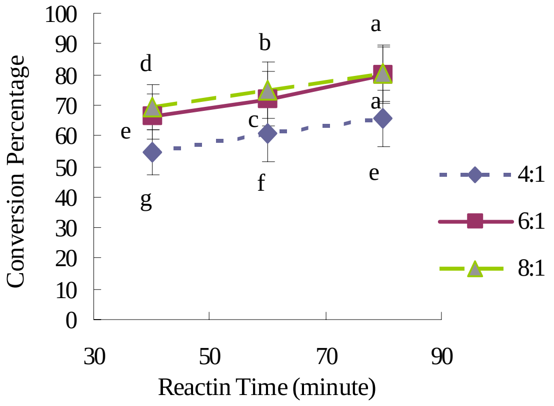

Fig. 7. The relationship between elapsed reaction time and biodiesel conversion for different molar ratios

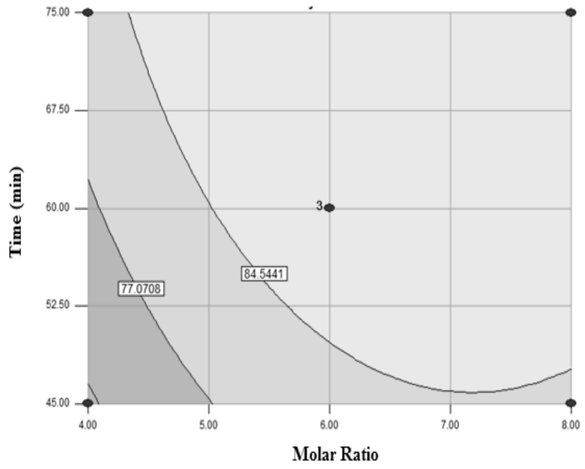

Fig. 8. conversion percentage variation versus molar ratio and reaction time variation

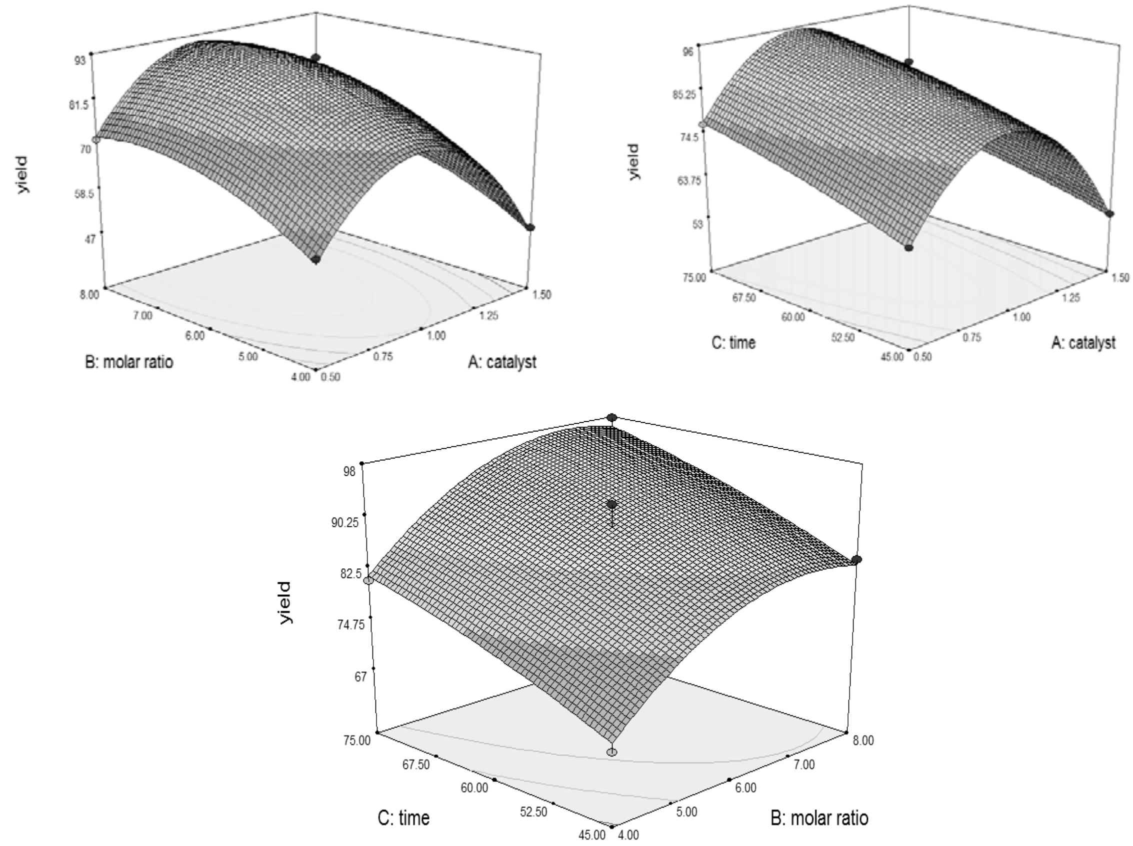

Fig. 9. conversion percentage variation versus catalyst percentage, molar ratio and reaction time variation

CONCLUSION

- For biodiesel production from feather fat, with increasing catalyst concentration up to 1% at first conversion percentage increased and then decreased.
- With increasing molar ratio with respect to oil leads to transesterification reaction progress and biodiesel production increase, with increasing molar ratio form 4:1 to 6:1 and 8:1 conversion percentage increased 16% and 2% respectively.
- Optimal point for biodiesel production from feather fat in 1% catalyst concentration, 7.23:1 molar ratio and 75 minutes reaction time was found to be 97.62%.

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