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Research Article

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## Cerium(III)Phosphotungstate: an Efficient Catalyst in Esterification of Fatty Acids

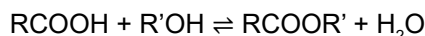
### ABSTRACT

In this report, a known heteropolyacid salt (HPAs) cerium (III) phosphotungstate was synthesized in a volume ratio 2:1:2 with a molar ratio 1:1:1 of each ingredient. These HPAs are further utilized in form of a catalyst to generate biodiesel through the esterification of variable carbon chain length alcohols (methanol, ethanol, n-propanol, isopropanol, n-butanol) and stearic acid at different conditions of reaction. FTIR of the produced biodiesel was also done for the assurance of ester peaks in it. Analysis of some important biodiesel properties such as density, dynamic viscosity, acid value, aniline point, cloud, pour point, flash and fire point etc., done to differentiate and validate the results. A large surface area of the catalyst i.e. 121.427 m<sup>2</sup>/g determined using the BET surface area analyser, supports the fact of outrageous catalytic action in the esterification reaction. The effect of additives was also studied on the properties of resultant biodiesel. The calorific value of the samples was measured as 7320 Kcal/kg without additive and 7512 Kcal/kg after adding toluene (as an additive) in the biodiesel generated in the study. The pour point temperature of the biodiesel with additives was observed even < 20°C.

**Keywords:** Cerium (III) phosphotungstate, biodiesel, butyl stearate, fuel value, additives

### 1. INTRODUCTION

Catalysts may be of different varieties but the one that gives the best outputs with a higher reaction rate is the heteropolyacid salts [1] which have been many times used in the reactions based upon amended lipids, esters with some additional features, and free fatty acids which has characteristics like; emulsification, and aroma [2,3]. Given below is a general form of esterification reaction:



The present study investigates the esterification of stearic acid with different alcohols, such as; n-butanol, iso-propanol, n-propanol, ethanol, and methanol, using cerium (III) phosphotungstate as a catalyst. The literature is rich in studies where heteropolyacid salts as catalysts were used in biodiesel production.

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Researchers have developed various methods to produce pure and degradable fuel using transesterification and esterification processes. For instance, Sahu et al. carried out the study related to the kinetic aspects of homogeneously catalysed esterification of a sequence of aliphatic acids with different types of alcohols [4]. In another study, de Lima and his team immobilized six lipases sustained on a styrene-divinylbenzene and estimated the batch esterification reaction parameters of stearic acid with fusel oil, which resulted in a high conversion rate of 90-93% [5]. Ibrahim et al. used Tin zirconium oxide catalysts (Sn-supported TMOs) to obtain a 74% quantitative result of methyl stearate under various conditions as mentioned [6]. Additionally, Ahmed et al. used interlinked amidoximated polyacrylonitrile ion-swapping web protonated by sulfuric acid as a catalyst for the conversion of stearic acid to methyl stearate (biodiesel) and achieved a conversion rate of 94.1%, which is almost equivalent to the yield achieved with 1wt% H<sub>2</sub>SO<sub>4</sub> [7]. Mahmoud et al. produced biodiesel through the esterification of C<sub>18</sub>H<sub>36</sub>O<sub>2</sub> (stearic acid) with C<sub>2</sub>H<sub>5</sub>OH catalyzed

by mesoporous material ( $ZrO_2/SiO_2$ ). The catalysts displayed the maximum conversion of 69.2% at 120°C following 3 hrs of reaction time [8]. In another study, biodiesel was produced by the reaction of fatty acid (stearic acid) with ethanol, where organophosphonic acid supported on NaY molecular sieve, having Arrhenius coefficient and activation energy of  $6.394 \times 10^3$  and 70.51 kJ/mol, respectively [9]. Vinod and team performed the steps for the synthesis of alkyl stearates from stearic acid within a closed batch reactor by means of heteropolyacid catalysts, available in the market [10]. Saravanan et. al demonstrated that an increase in the ratio of Zr:Si from 1:2 to 2:1 leads to the increase in yield of methyl stearate from 71% to 91% keeping a constant temperature of 60°C for 7 hrs, due to an elevation in acid spot concentration [11]. In another study, an environment-friendly carbon-based catalyst developed from extracted bagasse lignin was exploited in the esterification of stearic acid and methanol under most favourable conditions, i.e. 260 °C temperature for 5 min with a 9:1 methanol:stearic acid molar proportion, 5% wt catalyst, and 10% v/v toluene yielding 91.1% methyl stearate [12]. Kastratovic et al. were able to secure a maximum yield of

99% with  $H_2SO_4$  as a catalyst while reacting stearic acid and 1-butanol with an acid/alcohol/catalyst in a mole ratio of 1/15/0.75 at 65°C. The observed order of reactivity of different alcohols was: 1-butanol ( $CH_3CH_2CH_2CH_2OH$ ) > 1-propanol ( $(CH_3CH_2CH_2OH)$ ) > 2-methyl-1-propanol ( $CH_3CH(CH_3)CH_2OH$ ) > ethanol ( $CH_3CH_2OH$ ) > 2-butanol ( $CH_3CH_2CH_2OHCH_3$ ) > 2-propanol ( $CH_3CH_2OHCH_3$ ) > 2-methyl -2-propanol ( $CH_3C(CH_3)_2OH$ ) [13]. A detail of efficiency of different catalysts is shown in table 1. Han and his team synthesised ethyl palmitate using tungstophosphoric acid ( $H_3PW_{12}O_{40}$ ; HPW) with keggin geometry incorporated with ionic liquids having  $SO_3H$  as functional units, and the observed yield was 97.2%. This report indicates that heteropolyacid salts can be useful in obtaining a high yield of biodiesels. In this study, cerium (III) phosphotungstate is used for the first time to produce biodiesel from stearic acid with different alcohols. Several studies were conducted to optimize different parameters of the biodiesel, resulting in high calorific value, pour and cloud point, etc. This report's centre of attraction is the comparison between the properties of pure biodiesel obtained and the blended form of this biodiesel with additive (toluene) in 2:8, 1:9 ratios.

Table 1. Esterification of stearic acid using a variety of catalysts to obtain diverse yield.

Sr. no.	Reaction conditions/ Reactants	Catalyst	Biodiesel produced	yield	Reference no.
1.	Lipases which were assayed on a packed-bed system	Sulfuric acid	Isoamyl stearate	90-93 %	5
2.	T=120°C Time=60 minutes, Methanol: stearic 150 M: 1	Tin zirconium oxide (TMOs) 0.2 g	Methyl stearates	74 %	6
3.	Methanol: stearic acid 35.5:1 T=90°C	Protonated and crosslinked amidoximated polyacrylonitrile	Methyl stearate	94 %	7
4.	Time=3hr T=120 °C Ethanol: stearic acid 120:1	Protonated mesoporous $ZrO_2/SiO_2$ (0.4 g)	Ethyl stearate	76.9 %	8
5.	Time=4hour Alcohol: acid. 4:1, T=95°C and 100°C.	organophosphonic acid with Na exchanged zeolite (2.0 g)	Ethyl stearate	69.10 %	9
6.	Time=4 hours T= 110°C Slight excess of alcohols	Heteropoly acid; phosphotungstic acid (PTA) as catalyst (1 mol%)	Alkyl stearates	>95 %	10
7.	T= 60°C Time=7 hours Stearic acid: methanol	Aerogel-sulfated $ZrO_2-SiO_2$ blended with oxide solid acid catalyst	Methyl stearate	91%	11

8.	Stearic acid: methanol 1:9, T=240°C Time=10 minutes	sulfonated carbon derived catalysts from organosolv- lignin with methane sulfonic acid 5 wt%	Methyl stearate	89.2%	12
9.	Acid/alcohol/H <sub>2</sub> SO <sub>4</sub> 1/15/0.75, T=65°C	Sulfuric acid	Alkyl stearates	99%	13
10.	EtOH/PA = 7.7 mol/mol, catalyst % = 4.4 wt%, Time = 2.5 h, T = 363 K,	Keggin tungstophosphoric acid with ionic liquids having SO <sub>3</sub> H functional unit	Ethyl palmitate	97.2%	14
11.	Stearic acid: Methanol 1:10, T. = 90°C, Time = 2.5 hours	Cerium (III) Phosphotungstate	Butyl Stearate	98.7%	Proposed work

## 2. EXPERIMENTAL PROCEDURE

### 2.1 Required materials

Cerium nitrate (CDH), Orthophosphoric acid (HI media), and sodium tungstate (CDH), methanol, ethanol, iso-propanol, n-propanol, n-butanol, and stearic acid (Qualikems), Ostwald's viscometer, Pensky Martin's closed cup flash point apparatus with standard: IP 34, ASTM D 93, IS 1448 (P-21), ISO 2719, Bomb calorimeter (ASTM-D5865-98a), Pour point analyser (IS 1448, P:10), BET surface area analyser (AutosorbiQ Station 1).

### 2.2 Procedure followed

#### 2.2.1 Synthesis of catalyst (HPAs)

A known catalyst, cerium (III) phosphotungstate as reported by Raj et al [15] was prepared in the present report in different chemical proportions. Cerium nitrate, Orthophosphoric acid, and sodium tungstate were the compounds used in the preparation of this catalyst. All these compounds were mixed in their respective volumetric ratios 2:1:2 and their respective concentrations were 108.6 gm/dm<sup>3</sup>, 24.5 gm/dm<sup>3</sup>, 82.5 gm/dm<sup>3</sup>. The molar ratio was Ce:P:W::1:1:1. The resultant solution was heated at a temperature of 60°C after adjusting its pH at 1.08, by pouring an appropriate quantitative volume of dilute HCl/NaOH in the solution. Hence, mixture obtained was refluxed for 3 hours, keeping the reaction temperature at 40–45°C. The product obtained after this reaction have been filtered and, washed multiple times with de-ionized water. After drying the compound at room temperature in a desiccator, it was further immersed into distilled water to absorb the water molecule in the channel of the structure. Then, it was activated with dilute HCl acid in volume 25 ml.

Then, again washing, and drying at room temperature bestowed the desired heteropolyacid salt cerium (III) Phosphotungstate as a which appears as a greyish-white compound; the same was further used in form of a catalyst during the esterification reaction for synthesis of biodiesel.

#### 2.2.2 Esterification reaction

Stearic acid is mixed with five different types of alcohols; methanol, ethanol, iso-propanol, n-propanol, and n-butanol followed by an esterification reaction using a catalyst cerium (III) phosphotungstate. A generalized form of esterification reaction is as mentioned below;

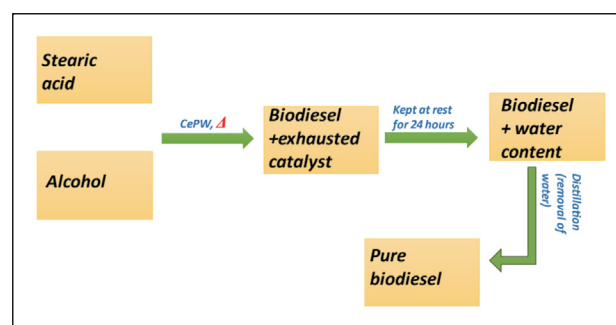
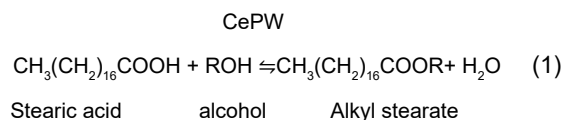


Figure 1. Generalised experimental procedure followed to produce biodiesel.

By following this procedure, varieties of biodiesels were produced through the esterification of stearic acid with varieties of alcohols (methanol, ethanol, isopropanol, n-propanol, n-butanol) keeping constant value for catalyst loading 3% (w/v), reaction temperature (90°C) and reaction time (1.5 hours).

The %yield of produced biodiesel was measured in every half an hour by titrating a liquefied reaction mixture against standardised NaOH.

To identify the sample with the most optimal results, the following conditions were implemented during the study:

### 2.3 Effect of variable reaction parameter

#### 2.3.1. Effect of different alcohols

In this study, different types of alcohol were tried during esterification of stearic acid. Following reaction was preceded for 1.5 hours. Catalyst quantity was kept 3% w.r.t to stearic acid. The whole setup was subjected to a reaction temperature of 90°C. After half an hour the reaction mixture has been filtered for separation of the heterogeneous catalyst. At this stage, this resultant mixture contains only the ester product, and water as by-product, and, also the alcohol, which was used as a solvent. The mixture obtained in this way, was reserved in a separating funnel and then both fractions were collected separately.

#### 2.3.2 Effect of reaction temperature

In this case, n-butanol was selected for further analysis, as already investigated in the previous study, and the reaction time was performed for 1 hour, while the temperature acted as a variable factor, i.e. from 60°C to 90°C. This study analysed the impact of reaction temperature by determining physicochemical properties.

#### 2.3.3 Effect of reaction time

The present study helped to optimize the contact time of ingredients to get a quantitative yield of the esterification reaction. In this study, the reaction temperature was fixed at 90°C. Different reaction times ranging from one hour, 1.5 hours and 2.5 hours were tried and tested to conclude the most optimal reaction temperature and most optimal time for the reaction to proceed to its maximum.

### 2.4 Biodiesel properties over adding additive

We have tried to enhance the fuel value and other parameters of synthesized biofuel by mixing the resultant product with the additives in different ratios and optimizing the physicochemical properties. So, the addition of an additive enhanced these properties in a very good manner. In the present study, Toluene is used as an additive in different volumetric ra-

tios with the biodiesel (butyl stearate). Studies were done to standardize conditions to produce good quality biodiesel in recognizable yield.

### 2.5 Properties discussed in this study

Density, dynamic viscosity, acid value, aniline point, flash point, fuel point, cloud and pour point, were measured to compare biodiesel characteristics while using different alcohols (methanol, ethanol, isopropanol, n-propanol, and n-butanol) during esterification.

During measurement of all the physicochemical properties, the room temperature was noted to be approximately 13°C. The following properties were measured for the biodiesel samples:

- Density: The density of each sample was assessed using a Pycnometer. The density was calculated in terms of  $\text{g/cm}^3$ .
- Dynamic viscosity: The Ostwald's viscometer was used to measure the dynamic viscosity of the biodiesel samples. The viscosity was calculated using the formula provided by Anzueto and his team [16]. The units are  $\text{mPa} \cdot \text{s}$ .
- Acid value: This parameter of the biodiesel samples was determined by titration with a standard NaOH solution. The formula utilized for this property was the same as used by Bashiri et al [17] and was used to calculate the acid value in terms of  $\text{mg NaOH/g stearic acid}$ .
- Aniline point: The aniline point of the samples is determined as the temperature at which there is uniformity in the mixture of aniline and oil [18]. The aniline point of any fuel indicates its aromatic content.
- Cloud and pour point: These characteristics of the fuel determine its stability in very cold weather. Pour point indicates the temperature at which the liquid stops flowing, while the cloud point indicates the presence of cloudiness at a particular temperature [19]. A lower value of these properties indicates better quality of the fuel.
- Flash and fuel point: This value of a fuel is the temperature required to catch fire by a fuel, while the fire point represents the temperature needed to burn the sample [20]. The Pensky Marten's flash point instrument was used to determine these properties.

## 3. RESULT AND DISCUSSION

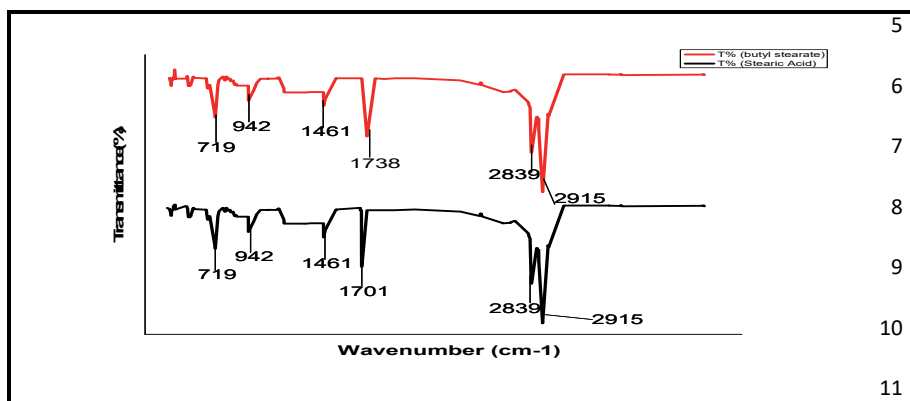


Figure 2. FTIR of butyl stearate and stearic acid.

**FTIR analysis:** Although the progress of this reaction was monitored via titration with a standardized alkaline solution but, the final confirmation of the product formation was done with the help of IR study of the desired product after separation.

FTIR peak analysis was conducted on butyl stearate and stearic acid. The peak at  $1738\text{ cm}^{-1}$  denotes the formation of ester. Figure 2 compares the FTIR spectra of butyl stearate ( $1738\text{ cm}^{-1}$ ) and stearic acid ( $1701\text{ cm}^{-1}$ ) and it confirms the formation of biodiesel (ester formed).

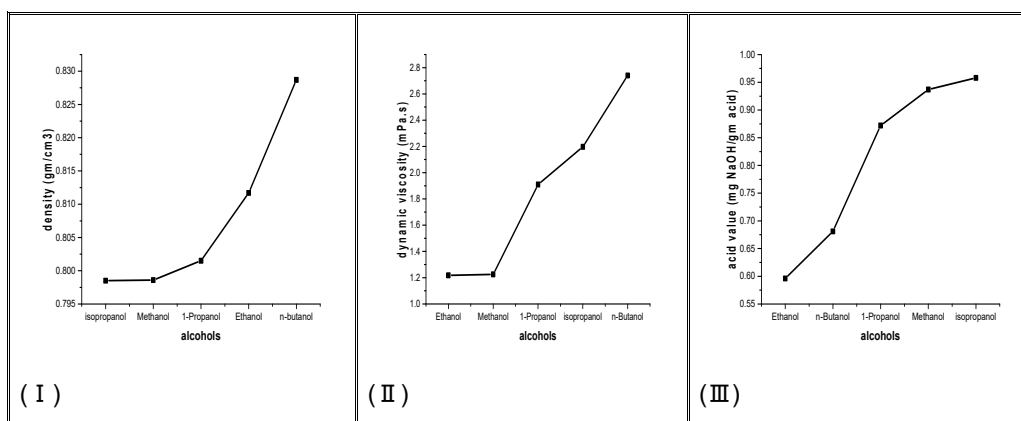


Figure 3. Properties of synthesized biodiesel using different carbon chain length of alcohols ( I ) density, ( II ) dynamic viscosity, ( III ) acid value.

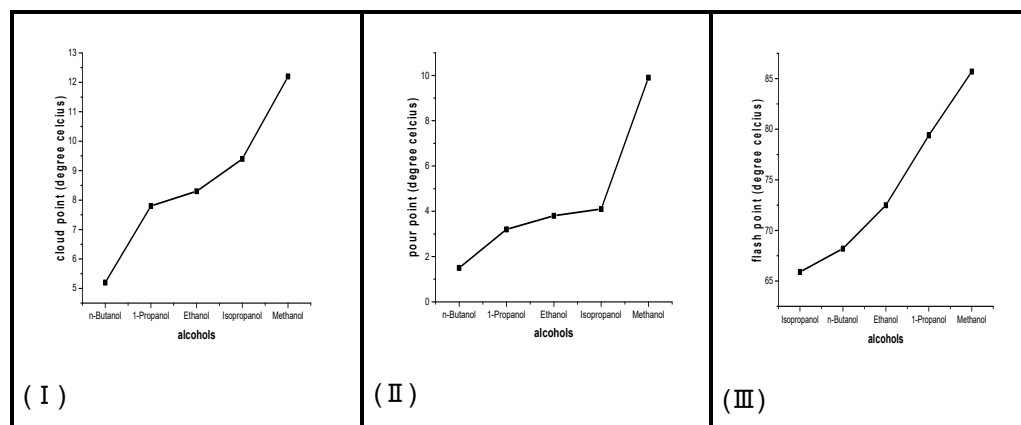


Figure 4. Properties of synthesized biodiesel using different carbon chain length of alcohols ( I ) pour point, ( II ) cloud point, ( III ) flash point.

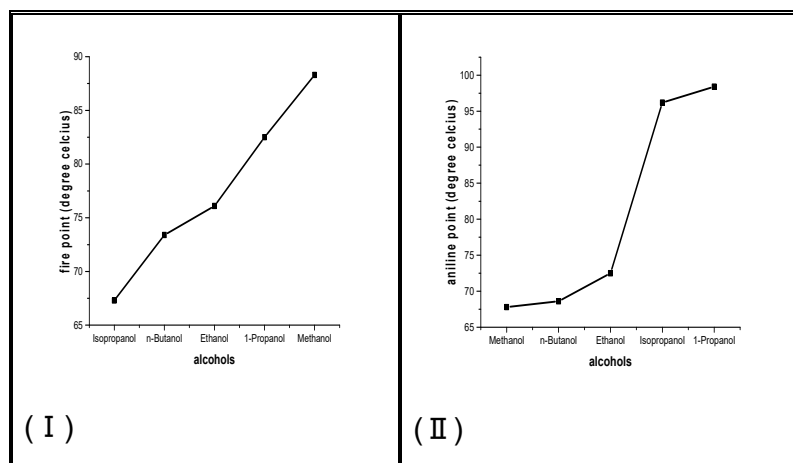


Figure 5. Properties of synthesized biodiesel using different carbon chain length of alcohols ( I ) fire point, ( II ) aniline point.

### 3.1 Study of several physicochemical properties with variation of types of alcohols

Effect of alcohol carbon chain length: From the above results mentioned in Figure (3-5), it was concluded that n-butanol gave the best results like; a low acid value, low but above room temperature flash and fire point, and very low temperature values for cloud and pour points which is considered as one of the best features for a fuel. So, all these factors favour the suitability of biodiesel obtained via esterification of stearic acid with butanol and hence chosen for further studies.

Therefore, n-butanol is mixed with stearic acid in vicinity of catalyst cerium (III) phosphotungstate to produce the required biodiesel through an esterification reaction. In the present section, the varying factors were as; reaction temperature (90°C), reaction time (1.5 hours), and effect of additive (toluene), etc. The % yield, we obtained in the case of alcohols methanol, ethanol, 1-propanol, isopropanol, and n-butanol was calculated as 86.2, 88.5, 90.3, 92.8, and 96.5 % respectively at 90°C with reaction time 1.5 hours.

#### 3.1.1 Effect of reaction temperature

Evolved from the results of this study, it was culminated that n-butanol produced the highest percentage yield compared to other alcohols. Therefore, this subsequent study used n-butanol as the alcohol in the esterification process. The discussion of the results obtained under the variable reaction temperature condition (60°C, 70°C, 80°C, 90°C) for reaction time 1.5 hours. Percentage yield, we obtained in

case of butyl stearate (biodiesel) was calculated at different reaction temperatures 60°C, 70°C, 80°C and 90°C as 74.8, 76.2, 88.3, and 96.5 % respectively.

#### 3.1.2 Effect of reaction time

Now the maximum %yield we obtained at reaction temperature 90°C (kept fixed) during this study. The percentage yield in the case of n-butanol was calculated at different reaction times 1, 1.5, 2.5 hours as 94.2, 96.5, and 98.7 % respectively.

### Analysis of biodiesel properties over adding additive (Toluene)

Additives are chosen based upon their various physicochemical characteristics like; density, dynamic viscosity, flash and fire point, acid value, aniline point, cloud and pour point, % yield, and calorific values etc [21-24]. By using additives, the lifetime of a machine/engine can be increased as additives prevent machines from many kinds of technological issues. The trend shown by the physicochemical properties of butyl stearate are expressed in terms of their successive plots in Figure (6-9). All these results obtained, in case of different blends of biodiesel and toluene (used as an additive here) viz; pure biodiesel, toluene: biodiesel::1:9, and toluene: biodiesel::2:8 are arranged in the ascending values as mentioned in these graphs.

Conclusively, it can be expressed here that the incorporation of toluene as an additive brought excellent changes in all these properties as compared to the pure biodiesel that have been clearly expressed below in these respective plots.

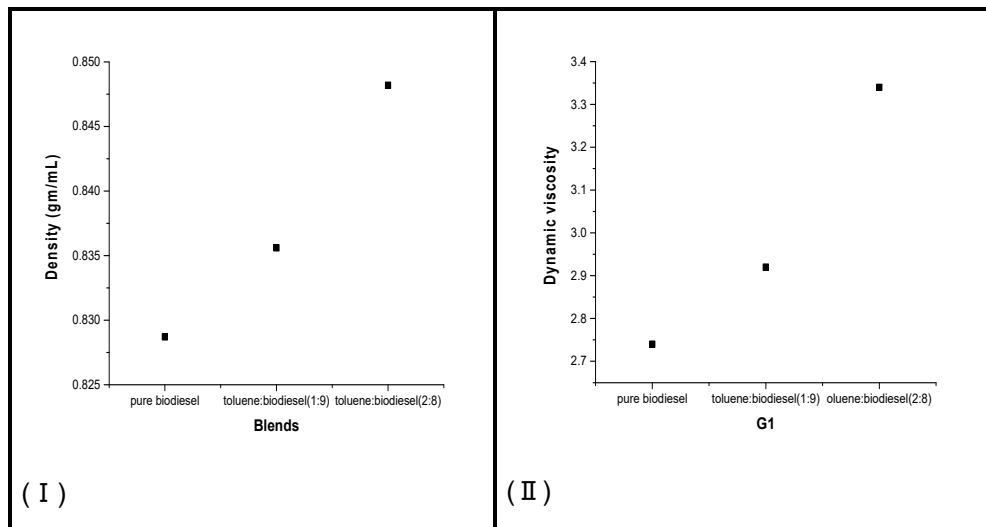


Figure 6. Observations of results: density ( I ) and dynamic viscosity ( II ) for pure biodiesel and additive:biodiesel:: 1:9 and 2:8 with different alcohols .

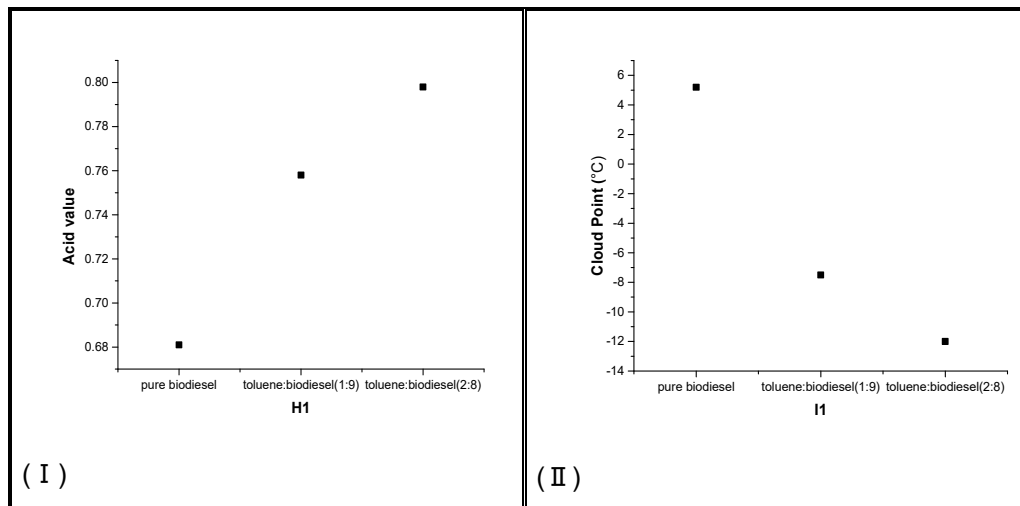


Figure 7. Observations of results: acid value ( I ) and cloud point ( II ) for pure biodiesel and additive:biodiesel:: 1:9 and 2:8 with different alcohols .

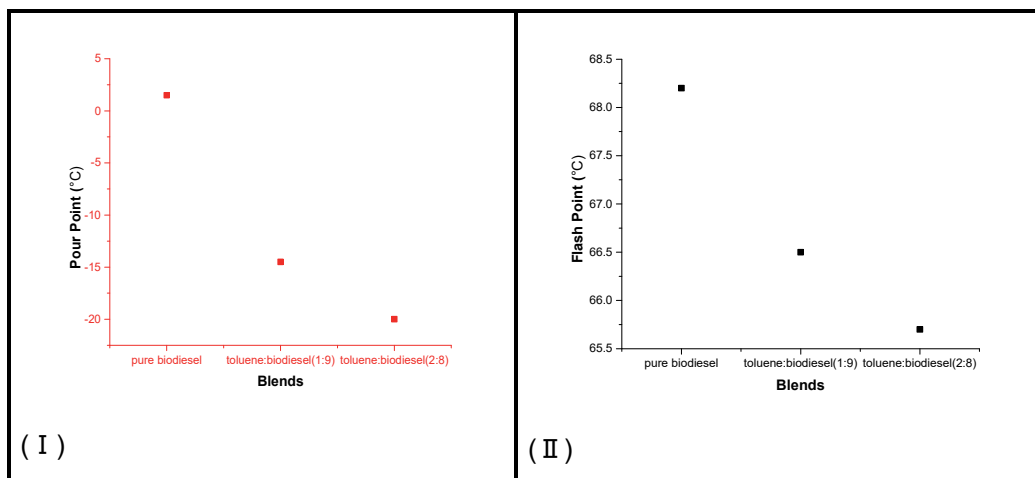


Figure 8. Observations of results: pour point ( I ) and flash point ( II ) for pure biodiesel and additive:biodiesel:: 1:9 and 2:8 with different alcohols .

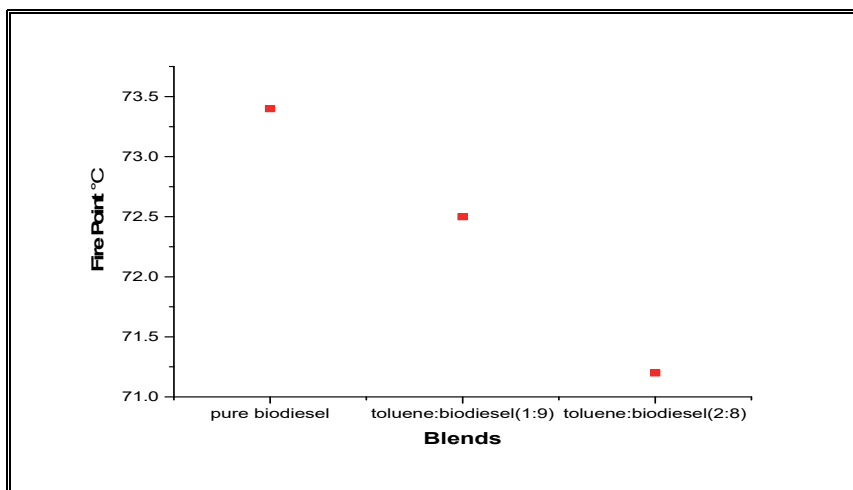


Figure 9. Observations of results: Fire point for pure biodiesel and additive: biodiesel:: 1:9 and 2:8 with different alcohols .

**Calorific value & surface area**

The test method ASTM-D5865-98a was used to measure calorific values of selected pure biodiesel and biodiesel with additive, i.e. butyl stearate and butyl stearate with toluene (additive). 7320 Kcal/kg without additive (pure biodiesel) and 7512 Kcal/kg with additive were observed through the technique as mentioned. These results showed the enhancement in fuel calorific value which further supports the

applicability of the fuel. Higher values point towards the better efficiency of biodiesel.

BET surface area analyzer (AutosorbiQ Station 1) [25] was utilized to find the surface region of the catalyst which helps in measurement of adsorption rate of the catalyst. The surface area of the catalyst cerium (III) phosphotungstate was determined as 121.427 m<sup>2</sup>/g, the original plots obtained for this property are mentioned in Figure 10.

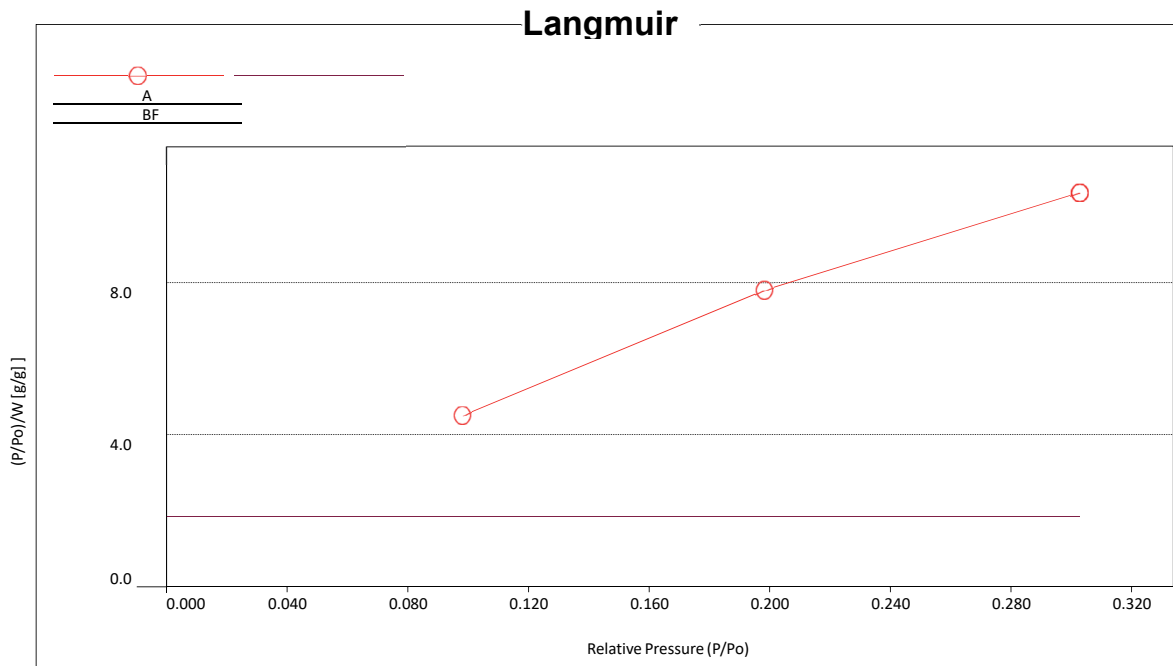


Figure 10. Langmuir plot for surface area of catalyst.



### % yield of biodiesel

The values for percentage yield point the total biodiesel we obtained from its starting material per hundred; following formula applied for required calculations [26]:

Biodiesel % yield = (weight of biodiesel/weight of initial stearic acid) × 100

Different % values correspond to all these alcohols with stearic acid and CePW as the catalyst. A graphical comparison of the % yield obtained is given in Figure 11, and Table 2 depicts optimised reaction conditions. Based upon this observation, n-butanol-derived biodiesel was prioritized over other alcohols.

*Table 2. % age yield obtained in esterification reaction with the variation of carbon chain length of alcohol*

Alcohol	% age yield	Reaction Temperature	Reaction Time
Methanol	89.2%	90°C	2.5 hours
Ethanol	90.8%		
n-Propanol	92.5%		
Isopropanol	94.3%		
n-Butanol	98.7%		

*Figure 11. Variation of % yield with different alcohols used during esterification process.*

### Rejuvenation of catalyst

On completion of this reaction, the catalyst was separated from the reaction mixture and was washed properly with de-ionized water before drying to make it suitable for reuse. The catalyst was used three times for the same procedure, and the percentage yields of biodiesel obtained were measured each time. A small decrease in yield percentage was observed, and by the third cycle of catalyst use, the final decrease was approximately 92.6%.

### Conclusion

The synthesis of catalyst cerium (III) phosphotungstate was done in a volumetric ratio of 2:1:2 and their corresponding molar ratio was 1:1:1 for the respective ingredients. The colour of the catalyst was recognized as greyish-white. This catalyst was utilized during esterification of steric acid with variable carbon chain length alcohols. Using the same catalyst, we have found that the properties of n-bu-

tanol-derived biodiesel were best suited than other alcohols. So, n-butanol was selected for further studies in which the addition of toluene as an additive was done which resulted in remarkable changes in the properties of biodiesel than its pure form without an additive. Maximum %yield of biodiesel was found to be ~98.7% in the case of butyl stearate (biodiesel) which was reduced to 92.6% by 3<sup>rd</sup> cycle of its use. Hence, butyl stearate is a competent biodiesel obtained through esterification using stearic acid, n-butanol and cerium (III) phosphotungstate (HPAs) as catalyst.

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## IZVOD

### CERIJUM(IV)FOSFOTUNGSTAT: EFIKASAN KATALIZATOR U ESTERIFIKACIJI MASNIH KISELINE

*U ovom radu, poznati fosfotungstat cerijuma (III) soli heteropoli kiseline (HPAs) sintetizovan je u zapreminskom odnosu 2:1:2 sa molarnim odnosom 1:1:1 svakog sastojka. Ovi HPA se dalje koriste u obliku katalizatora za generisanje biodizela kroz esterifikaciju alkohola promenljive dužine ugljeničnog lanca (metanol, etanol, n-propanol, izopropanol, n-butanol) i stearinske kiseline u različitim uslovima reakcije. FTIR proizvedenog biodizela je, takođe, urađen za osiguranje estarskih pikova u njemu. Analiza nekih važnih svojstava biodizela kao što su gustina, dinamički viskozitet, kiselinska vrednost, tačka anilina, oblak, tačka stinjanja, tačka paljenja i plamena itd., urađena su da bi se razlikovali i potvrdili rezultati. Velika površina katalizatora, tj. 121.427 m<sup>2</sup>/g, određena korišćenjem BET površinskog analizatora, podržava činjenicu nečuvanog katalitičkog delovanja u reakciji esterifikacije. Takođe je proučavan uticaj aditiva na svojstva dobijenog biodizela. Izmerena je kalorijska vrednost uzoraka na 7320 Kcal/kg bez aditiva i 7512 Kcal/kg nakon dodavanja toluena (kao aditiva) u biodizel generisan u studiji. Temperatura tečenja biodizela sa aditivima je primećena čak i < 20°C. Ključne reči: cerijum (IV) fosfovolframat, biodizel, butil stearat, vrednost goriva, aditivi.*

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