

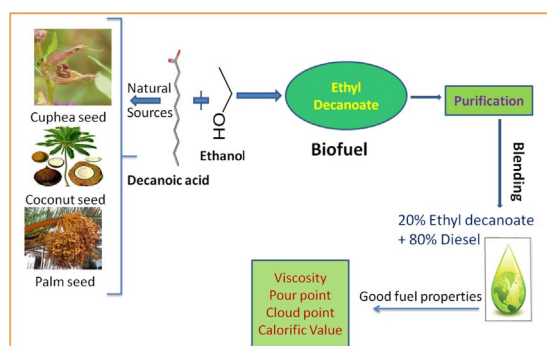


Full Length Article

Solid acid catalyst supported synthesis and fuel properties of ethyl decanoate

Ruchi Lamba^{a,*}, Sudipta Sarkar^b, Surendra Kumar^c^a Centre for Transportation System, Indian Institute of Technology Roorkee, Utrakhand 247667, India^b Department of Civil Engineering, Indian Institute of Technology Roorkee, Utrakhand 247667, India^c Chemical Engineering Department, Indian Institute of Technology Roorkee, Utrakhand 247667, India

GRAPHICAL ABSTRACT



ARTICLE INFO

Keywords:

Amberlyst 15
Decanoic acid
Esterification
Ethyl decanoate
Calorific value

ABSTRACT

Decanoic acid, found in naturally occurring coconut and palm kernel oils, and in genetically modified cuphea plant species, is gaining increasing importance as platform chemical for the development of many useful products. Although perceived to be an important starting molecule for production of biodiesel, the process of production of and fuel properties of its ethyl ester have never been explored before. In this article, we have reported about laboratory-based studies for esterification of decanoic acid with ethanol in presence of solid acid catalysts. Activation energy of the esterification reaction was found to be 38.5 KJ/mol. The studies showed that the conversion of decanoic acid to its ethyl ester can be performed at optimal temperature 348 K in presence of solid acid catalyst. We have also investigated salient fuel properties of ethyl decanoate and its several blends with conventional fossil-derived diesel and found that use of ethyl decanoate with conventional diesel could become economically attractive.

1. Introduction

Over the last few decades, biodiesel has found its acceptability and importance in the market as a sustainable replacement to petroleum. Biodiesel is defined as monoalkyl esters of oils and fats from vegetable or animal sources. Due to its renewable nature, biodegradability, and

relatively high flash point, it is technically competitive and environmentally more sustainable than conventional fossil fuels. Generally, vegetable oil that is most abundantly found in a region is used as the most common feedstock for the production of biodiesel [1]. The plant variety in a particular region changes depending on the local climate and soil conditions [2]. Therefore, the feedstock for biodiesel

* Corresponding author.

E-mail addresses: rochidet@iitr.ac.in (R. Lamba), srkarf@iitr.ac.in (S. Sarkar).

changes depending on the climatic condition of a region. Many times the local supply of the vegetable oils is not enough to replace significant amount of petroleum as fuel; so, there is a look out for alternative feedstock. *Jatropha* oil and algae are possible feedstocks that are gaining popularity [3]. *Pongamia*, rubber seed, *madhuca indica* oil, used cooking oil and moringa are some other less popular oils that might be used as feedstock for production of biodiesel [4–7].

While biodiesel is considered to be the most sustainable fuel, the commercial success, apart from price, is also limited by the two properties: first, reduced flow ability due to the presence of long-chain saturated fatty esters with high melting points, and second, compromised oxidative stability due to the presence of polyunsaturated fatty esters [8]. Recently, it was discussed that esters of saturated medium chain acids, especially esters of decanoic acid, or esters of monounsaturated acids, especially palmitic, oleic acid, may be especially suitable as major components of biodiesel [9,10]. Coconut and palm kernel oils contain high amounts of medium-chain fatty acids which mainly consists of lauric (dodecanoic) acid [11]. Traditionally, coconut and palm-kernel oils are used as a good source of decanoic acid. It is reported that the lighter fraction of biodiesel derived from palm and copra oil contains significant amount of ethyl decanoate [12]. Hence, ethyl decanoate can find potential application as biodiesel. Newer and wider applications of decanoic acid are being developed. Ethyl decanoate, an ester of decanoic acid, is traditionally used as artificial aroma in many applications such as alcoholic drinks, fruit juices and drinks, perfumes, etc [13]. Lubricants such as estolides also are made from decanoic acid [14,15]. The sources of decanoic acid are natural and are limited to plant sources such as cuphea plant [16–18]. Recently, genetically modified cuphea plant with codename PSR-23 has been developed which is high in C-10 content [19]. The increasing use of decanoic acid is in turn motivating the commercialization of cuphea as a new oil seed and there is an impetus for cultivation of cuphea as a valuable rotation crop in different countries such as USA. Soon, other countries might follow the same trend because self reliance in the domestic production of it can curb the dependence on foreign imports of coconut and palm kernel oils.

Above discussions suggest that decanoic acid can make excellent biofuel both in terms of raw material availability and quality of biodiesel produced. The fuel property of methyl decanoic ester has been studied and reported [20,21]. Ethanol can be commercially cheaper than methanol because many agricultural residues such as sugarcane, sugar beets, grass and trees, rice stem, corn leaves and corn cobs, wood chips and cellulosic bio-waste materials etc are used for ethanol production [22,23]. Fatty acid ethyl esters have better fuel properties such as flash point, viscosity, cloud point, lubricity, high calorific value and thermal efficiency and they emit lesser greenhouse gases in comparison to methyl esters [24,25]. Blending of petroleum with biodiesel or ethanol is a norm in many countries and the target is to increase the amount of blending as much as possible to attain optimum operating conditions in terms of fuel efficiency, environmental protection as well as economics. Although ethyl decanoate is an important compound in terms of the future of biofuel, key fuel properties of it has not yet been reported.

The esterification reaction requires H^+ ions as catalysts [26].

Therefore, in presence of acid, the reaction leading to the formation of alkyl esters enhances. Although homogeneous catalysis using strong acid solutions are traditionally used to favor the esterification reactions, nowadays heterogeneous catalysis on solid acid catalysts such as ion exchange resins, zeolite, acidic clay particles, etc. are more popularly being used [27,28]. In contrast to homogeneous catalysts, in heterogeneous catalysts the acidic functional groups attached to a solid phase or matrix, produces H^+ ions that catalyze the reaction. The functional groups containing H^+ ions are fixed to the solid matrix or phase from where they cannot diffuse out into the bulk liquid phase containing the reaction mixture. Therefore, separation of solid catalysts from the liquid product become easier compared to the homogeneous catalysts which are also liquid materials. The heterogeneous catalysis process for biodiesel production is also much more efficient than that through the homogeneous catalysis route [29].

The available body of open literature does not provide any applicable details of heterogeneous catalysis for the esterification of ethanol and decanoic acid to produce ethyl decanoate. In this work, we have studied in detail the esterification of decanoic acid with ethanol carried out in presence of strong acidic ion exchange polymer such as Amberlyst 15 as catalyst. The study included the reaction kinetics, effect of different parameters such as molar ratio of reactants, temperature, catalyst loading, etc. on the conversion of decanoic acid into ethyl decanoate. Various fuel properties of ethyl decanoate and various percentages of blends with commercial diesel have been found out and have been compared with relevant ASTM standard specifications for standard diesel.

2. Materials and methods

2.1. Materials

AR grade Decanoic acid and ethanol were purchased from Merck. Amberlyst 15 dry, Indion 225 and Indion 236 acid resin catalyst were obtained from Rohm and Haas Company and Ion exchange India Limited. Ethyl decanoate standards were purchased from Sigma-Aldrich for preparation of calibration curves. Prior to its use, the catalyst was dried in an oven at 353 K for three hours. The characteristics of the catalysts are provided in Table 1.

2.2. Methods

2.2.1. Experimental methods

Batch experiments of esterification reaction were performed in 500 ml round bottom glass flask. Ethanol and decanoic acid were heated separately to the required reaction temperature using a hot plate before it was added to the glass flask. The catalyst was simultaneously added into glass flask along with both the reactants and stirring was continued at the desired speed. The time at which ethanol, decanoic acid and catalyst were added was noted as the starting time of reaction. Samples of 0.5 ml volume were withdrawn at predetermined time intervals for further testing. After withdrawal, the sample was allowed to cool down to room temperature before it is passed through HPLC for analysis. All reported data are arithmetic averages of experimental

Table 1
Salient characteristics of cation exchange resins used as catalysts.

Properties	Amberlyst 15H	Indion 225H	Indion 236
Matrix	Macroreticular copolymer Styrene-DVB	Microreticular copolymer Styrene-DVB	Gel polyacrylic copolymer
Functional groups	Sulphonic acid group	Sulphonic acid group	Carboxylic acid group
Ion exchange capacity ^a	3.9 meq H^+ /g (dry) (± 0.5)	3.1 meq H^+ /g (dry) (± 0.3)	8.8 meq H^+ /g (dry) (± 0.5)
Operating temp	120 °C	120 °C	120 °C

^a Calculated from the acid-base titration of dry resin (see subsection 2.2.2).

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