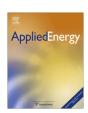
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# A multi-variant approach to optimize process parameters for biodiesel extraction from rubber seed oil

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#### ABSTRACT

Biodiesel is biodegradable, non-toxic and has the capacity for sustainable development, energy conservation and environmental preservation. Apart from yielding high value latex, the rubber plant supply large amount of rubber seed, which are currently underutilized. Extracting biodiesel from rubber seed is a viable option which demands attention for research to consolidate and optimize the process parameters. Design of experiments (DOE) is a powerful statistical approach which is used for optimizing the process parameters through two stage esterification process, relating acid and alkaline as catalyst. Reducing the acid value is the primary objective for process optimization in acid esterification process, whereas, maximizing the monoester yield is the objective for the alkaline-esterification process. Different saturated and unsaturated monoesters present in the biodiesel were quantified using gas chromatograph in order to determine the yield percentage, which ensures the quality of the biodiesel. The fuel was tested for properties such as viscosity, calorific value and carbon residue using standard test procedures and found to be analogous with diesel, which makes it possible to use this alternate fuel in the existing engine without any modification.

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#### 1. Introduction

The present world is confronted with twin crises of fossil fuel depletion and environmental degradation. Indiscriminate extraction and lavish consumption of fossil fuels have led to reduction in underground-based carbon resources. Increasing industrialization and motorization has led to a steep rise in the demand of petroleum-based fuels. With the exception of hydroelectric power and nuclear energy the majority of world's energy needs are supplied through petrochemical sources such as coal, oil and natural gas [1,2]. The search for alternative fuels, which promise harmonious correlation with sustainable development, energy conservation, efficiency and environmental preservation, has become highly pronounced in the present context.

The fuels of bio-origin can provide a feasible solution to this worldwide petroleum crisis. Gasoline and diesel-driven automobiles are the major sources of greenhouse gas (GHG) emission [3]. Scientists around the world have explored several alternative energy resources, which have the potential to quench the everincreasing energy thirst of today's population. Various bio-fuel energy resources such as biomass, biogas, primary alcohols, vegetable oils and biodiesel are largely environment-friendly but they need

to be evaluated on case-to-case basis for their advantages, disadvantages and specific applications [4]. Some of these fuels can be used directly while others need to be formulated to bring the relevant properties closer to conventional fuels.

To-date biodiesel research is carried out in more than 28 countries of which Germany and France are the pioneers. The United States department of energy has assessed that up to 50% of the total conventional diesel fuel could be potentially replaced by biodiesel [1]. At present bio-fuels are produced from edible crops like cotton seed, palm nut, linseed, and groundnut and also from non-edible crops such as *Jatropha curcas*, algae, eucalyptus, orange skin, Eruca sativa gars, neem, and rubber seed. In India, biodiesel production using edible oil is not much realistic because of the gap in its demand and supply, thereby limiting the use of edible oil in the production of biodiesel [5]. In fact, the price of crude oil is the most dominant factor in the cost of biodiesel fuel, and determines the competitiveness of biodiesel with fossil fuel in the fuel market [6,7]. Hence, search for a low-cost raw material with adequate fuel characteristics for biodiesel production is an important step towards establishing a successful biodiesel industry. Typically, rubber seed oil, which is non-edible, is considered as a prospective feedstock for biodiesel production.

The present study is the continuation and consolidation of the work initiated by researchers [8–11]. The computer software, Design Expert 7.0 is used in this work to design the prominent

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process parameter and there response is statistically analyzed and validated to derive objective inferences. Accordingly, acid and alkaline-esterification process parameters for biodiesel extraction were designed and the optimum process characteristics were found. The relative amount of different monoesters was quantified for the first time using gas chromatograph (GC) to estimate the percentage yield. Spate of research has been done in Jatropha based biodiesel [12–20], however rubber seed based biodiesel extraction is rather sparse and needs strenuous attention as they lend a hand both in economic and ecological aspects of insatiable demand for energy.

#### 2. Experimental procedure

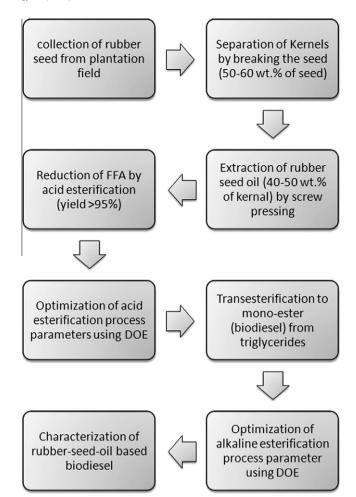
#### 2.1. Potential and extraction of rubber seed oil

India is one among the top ten rubber producing countries in the world and Kerala state is the leading rubber plantation state of India. It originally belongs to Amazon basin (Africa) and later in the 19th century introduced to other countries in the tropical belts of Asia. The Para rubber tree (*Hevea brasiliensis*), belongs to the family Euphrobiaceae and the most economical member of the genus *Heavea*. The tree is deciduous in nature having trifoliate leaves with long stalks (see Fig. 1). Rubber plantation is of great economic value because of its sap-like extract, known as latex collected on the daily basis from its bark which is the primary source of natural rubber. In India they shed their leaves during December–February and refoliation starts quickly followed by copious flowering. Flowers are small but appear in large clusters. The main yield of rubber plantation is latex. However, the rubber tree also produces large volumes of seed, which is underutilized.

In India alone there are around 2 lakhs hectares of rubber plantations. On an average 160 kg of rubber seed is produced per hectare of rubber plantation. The estimated availability of rubber seeds in India is about 30,000 tons per annum, which can yield oil to the tune of about 5000 tons per annum. The oil content in the rubber seed is roughly around 40–60 wt.% [10]. The seeds are three lobed, each holding three seeds, looks like castor seed in appearance but much larger in size (see Fig. 1). Rubber seeds are ellipsoidal in size varying from 25 to 30 mm long, mottled brown in colour, lustrous in look, weighing 2–4 g each. Rubber seeds are collected and kernels are separated by breaking them. These kernels are crushed and oil is extracted by screw pressing and then filtered. The rubber seed cake, which is the leftover after oil extraction is rich in protein and can be used as cattle and poultry feed. The entire process



**Fig. 1.** Photograph of rubber plantation in Kerala state of India along with rubber seed (inside picture).



**Fig. 2.** Block diagram describing the entire process in the extraction of biodiesel from rubber seed.

involved for the extraction of biodiesel from rubber seed is shown in a block diagram (Fig. 2).

The rubber seed oil varies in colour from light yellow to brown, depending on the Free Fatty Acids (FFA) content, yellow being on the lower side. Vegetable oils generally have different grades of fatty acids in the form of triglycerides. The fatty acids vary in their hydrocarbon chain length and the number of double bonds. The fatty acid profile and other important properties of rubber seed oil in comparison with other vegetable oil is given in Table 1 [8]. The type and percentage of fatty acids content in vegetable oil depends on the plant species and on the growth conditions of the tree. Rubber seed oil contains 18.9% saturated acids (palmitic and stearic) and 80.5% unsaturated acids (oleic, linolic and linolenic). Saturated fatty acid alkyl esters increase the cloud point, cetane number and improve stability. Rubber seed oil extracted and used for this analysis had an acid value of 35 mg KOH/g, which is equivalent to 17.5% FFA. The presence of high FFA hinders the transesterification process by single stage using a base catalyst due to its preference for saponification thereby forming soap [21]. Hence the amount of FFA must be reduced to its minimum by acid esterification before alkaline esterification.

#### 2.2. Acid esterification to reduce FFA

Vegetable oil has the potential to supplement petroleum-based fuels. However their high FFA, thereby its high viscosity deters its usage in internal combustion engine directly. The viscosity of the

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