Fuel 150 (2015) 636–644

Contents lists available at ScienceDirect

Fuel

journal homepage: www.elsevier.com/locate/fuel

Extraction of oil from rubber seeds for biodiesel application: Optimization of parameters

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HIGHLIGHTS

- Soxhlet extraction of rubber seed to yield vegetable oil.
- Optimization of the process parameters using response surface methodology (RSM).
- Compositional analysis of extracted oil using ^{1}H NMR and FTIR.
- Characterization of oil to measure its suitability for biodiesel synthesis.

ARTICLE INFO

Article history:
Received 7 November 2014
Received in revised form 13 February 2015
Accepted 17 February 2015
Available online 26 February 2015

Keywords:
Rubber seed
Solvent extraction
RSO
RSM
Optimization

ABSTRACT

Rubber seed comprises of 40–48% shell and 52–60% kernel by weight of seed was subjected for oil extraction study. The effects of process parameters such as extraction time (3–12 h), kernel size range (0.5–3 mm), ratio of kernel to solvent (0.03–0.09 g/ml), and variety of solvents (polar and non-polar) on Soxhlet extraction process were studied. Design of experiment (DOE) schemes was considered to prepare an experimental matrix using central composite design (CCD) approach. Response surface methodology (RSM) was applied to optimize the process parameters to achieve maximum oil yield. The maximum oil recovery, 49.36 wt% was obtained during the experiment conducted with hexane as solvent, 0.08 g/ml solute to solvent ratio, average kernel size of 1 mm and 8 h extraction time. Physico-chemical properties of oil obtained from the rubber seed were estimated to measure its suitability for biodiesel production. Proton nuclear magnetic resonance (^{1}H NMR) spectra of the obtained rubber seed oil (RSO) revealed 13.17% linolenic, 39.86% linoleic, 27.06% oleic and 19.91% saturated fatty acid in its composition. These compositional data were qualitatively confirmed with Fourier transform infrared (FT-IR), thermal gravimetric (TG) and differential scanning calorimeter (DSC) analyses of extracted oil samples.

1. Introduction

Energy demand is steadily increasing along with the growth of population and industrialization [1,2]. Currently the world is facing energy crisis due to the excessive demand of the fossil fuels and their limited availability [3–5]. Environmental concerns are also related to excessive usage of fossil derived fuels [6,7]. Production of biofuels from biomass is one way of substituting of non-renewable energy sources as well as reduction of emission of greenhouse gases. Biofuels are produced from source such as corn, soybean, flaxseed, rapeseed, sugarcane, palm oil, sugar beet, raw sewage, food scraps, animal fats, and rubber seed [8]. Biodiesel is a mixture of fatty acid alkyl esters obtained from trans-esterification (ester exchange) of feedstock esters [9]. Various raw materials are available for biodiesel production such as oil seed (vegetable oil), animal fats, algae and waste such as waste cooking oil, and greases [10]. The use of edible raw materials for biodiesel production has recently been of great concern on food versus fuel dispute and sustainability [11]. According to report published in 2012 [12], India is importing 10.3 million tons of vegetable oil to cater its need for edible purposes and thus it cannot affords providing edible oil for non-edible applications. Non-edible oils for example rubber seeds, soapnut, cassava, sal, mahua, neem, karanja, kusum, jatropha, etc. have great role and contribution for production of biodiesel [1]. Some of the non-edible oil sources are listed in Table 1.

Rubber tree (*Hevea Brasiliensis*) belongs to the family of euphorbiaceous [10]. It is originated from South America (Amazon) and cultivated as an industrial crop since its introduction to Southeast Asia in 1876 [21]. The rubber trees growth is most rapid at altitudes below 200 m with temperatures about 27 or 28 °C. Natural rubber tree producer in the world are Thailand (35%),

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http://dx.doi.org/10.1016/j.fuel.2015.02.058
0016-2361/

Table 1
Non-edible sources of vegetable oil for biodiesel production.

Non-edible vegetable source	Plant type	Plant part	Oil content		Refs.
			Seed (%wt.)	Kernel (%wt.)	
Rubber	Tree	Seed, kernel	40–60	40–50	[1–2,10,13,17–20]
Neem	Tree	Seed, kernel	20–30	25–45	[1,13,15,19–20]
Milk weed	Herbaceous perennial	Seeds	20–25		[1,13]
Ethiopian mustard	Herbaceous annual	Seed, kernel	42	2.2–10.8	[1,13–14,19]
Desert date	Tree	Kernel		36–47	[13]
AnnonaSquamosa	Tree	Seed	15–20		[13]
Cotton tiglium	Herbaceous perennial	Seed, kernel	30–45	50–60	[13]
Cuphea	Herbaceous annual	Seed	20–38	–	[13]
Garciniaindica	Tree	Seed	45.5	–	[13]
Jatropha	Tree	Seed, kernel	20–60	40–60	[1,11,13,15–16,19–20]
Koroch	Tree	Seed	33.6		[13]
Sapindus	Tree	Seed	51.8	–	[13]
Ximenia	Tree	Kernel	–	49–61	[13,16]
Terminaliacatappa	Tree	Seed	49	–	[13]
Tung	Tree	Seed	35–40	–	[13]
Linseed	Herbaceous annual	Seed	35–45	–	[1,13,15,20]
Karanja	Tree	Seed, kernel	25–50	30–50	[11,13–15,19–20]
Castor	Tree	Seed	45–50	–	[1,11,15,20]
Kusum	Tree	Kernel	–	55–70	[13,20]
Mahua	Tree	Seed, kernel	35–50	50	[11,13–15,20]

Indonesia (23%), Malaysia (12%), India (9%), and China (7%). Rubber seed provides 40% to 60% of kernel by weight [22]. Mechanical and chemical (solvent) extraction techniques are commonly used for extracting the oil from seeds kernel. In chemical extraction technique via Soxhlet extractor, the parameters such as extraction time, amount of solute to solvent ratio, particle size, drying time, extraction temperature, and solvent type affect the oil yield [2]. Design of experiment technique is useful to prepare an experimental matrix within the range of process parameters to optimize process conditions [23].

Sayyar et al. [3] obtained optimum oil yield from jatropha seed using hexane solvent at 8 h extraction time and 0.16 ratio of solute to solvent. They reported that optimum oil yield varies (46–47.3%) with particle size studied, 0.5–0.75 mm. Kostic et al. [24] reported that optimization of hemp seed oil extraction using RSM and artificial neural network-genetic algorithm (ANN-GA) technique. In this study, they consider the effect solvent to seed ratio (3:1, 6.5:1 and 10:1 ml/g), extraction temperature (20, 45 and 70 °C) and extraction time (5, 10 and 15 min.) on oil yield of hemp seed for RSM technique whereas 8 time levels were considered for ANN model fitting. The optimum extraction condition obtained from both techniques RSM and ANN-GA were similar. They found that optimum oil yield of 29.56% under extraction condition: solvent to seed ratio (10:1) and extraction time (10 min.) at near boiling temperature of solvent hexane. Similar observations were reported by Bokhari et al. [2] for RSO extraction. They performed RSM analysis on RSO extraction processe and obtained optimal oil yield (33.56%) with hexane as solvent at 0.027 ratio of solute to solvent (g/ml) and 4.5 h extraction time. RSM was also applied for oil extraction process from neem seed using hexane solvent [25]. The optimal oil yield 37.2% was reported at 6 h extraction time, 0.12 ratio of solute to solvent and particle size 0.4–0.7 mm.

In this study, non-edible RSO (seed collected from Assam, India) was extracted using Soxhlet-solvent extraction technique. The parameters such as extraction time, kernel to solvent ratio, particle size and types of solvent were considered to optimize oil recovery experimentally. The effect of particle size and solvent type were screened experimentally and, in further study, the process parameters such as extraction time and kernel to solvent ratio were optimized by DOE using of RSM. In general the RSM studies based on three (or more) parameters are useful to understand the interaction of parameters on desired response. However, the effects of two process parameters on response were also reported for other systems [26,27]. Mathematical model for extraction of oil were developed to describe the effects and relationship between the process parameter. Collected oil samples were characterized for physico-chemical values, thermal properties and its fatty acid composition to measure its suitability for biodiesel production.

2. Materials and methods

2.1. Materials

Rubber seeds were collected from Assam, India. The rubber seeds were de-shelled manually and kernels were collected. The average weights of a rubber seed and a kernel were observed to be 2.90 g and 1.73 g respectively. Kernels were dried for 12 h at 50 °C before oil extraction and, 3.25% (average) weight loss was observed. Further, the dried kernels were grinded to increase the contact surface area during extraction.

2.2. Methods and apparatus

The oil from seed was extracted using Soxhlet apparatus according to AOAC standard method [28]. Ibemesi, Attah [29] reported that the maximum oil yield from rubber seed can be achieved near the boiling point of the solvent used. Thus, in this study all the extraction experiments were carried out near the boiling point of the respective solvents used. Extraction time was counted when the first drop of extracting solvent recycled back into the thimble. The oil was concentrated in a rotary evaporator under vacuum by removing the solvent. The residual oil was cooled and weighed. The oil yield was calculated using following expression,

$$\text{Yield}(\%) = \frac{\text{mass of extracted oil}}{\text{mass of kernel before exrtraction of oil}} \times 100 \qquad (1)$$

All the experiments were duplicated and average values are used for the analysis.