



Experimental and modelling studies on the solvent assisted hydraulic pressing of dehulled rubber seeds



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ABSTRACT

A systematic study on the expression of rubber seed oil from dehulled rubber seeds in a hydraulic press was performed in the presence and absence of ethanol. The effect of seed moisture content (0–6 wt%, w.b.), temperature (35–105 °C), pressure (15–25 MPa) and ethanol to seed ratio (0–21%v/w) on the oil recovery was investigated. An optimum oil recovery of 76 wt%, d.b. was obtained (1.6 wt% moisture content, 14%v/w ethanol, 20 MPa, 75 °C, 10 min pressing time). The experimental dataset was modeled using two approaches, viz (i) the Shirato model and (ii) an empirical model using multi-variable non-linear regression. Good agreement between models and experimental data was obtained. Relevant properties of the rubber seed oil obtained at optimum pressing conditions (free fatty acid content, viscosity, density, water and P-content, cold flow properties and flash point) were determined. The pressed rubber seed oil has a relatively low acid value (2.3 mg KOH/g) and is suitable for subsequent biodiesel synthesis.

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1. Introduction

The rubber tree (*Hevea brasiliensis*) is a perennial plantation crop which has been cultivated mainly as a source of natural rubber. However, the tree also produces a rubber seed, of which the valorisation has received limited attention till now. The yield of rubber seeds is reported to be in the range of 100–1200 kg/ha/yr (Stosis and Kaykay, 1981; Abdullah and Salimon, 2009). From a biorefinery perspective, the identification of high added value outlets for the rubber seeds is highly relevant as it increases the overall value of the value chain from rubber plantation to processed latex (Abduh et al., 2013).

The seeds consist of a kernel surrounded by a hard shell. The kernel contains 40–50% of oil (Ramadhas et al., 2005; Njoku et al., 1996) embedded in a protein rich matrix. The oil, also known as rubber seed oil (RSO), may be a valuable source for biofuel production (Ikwaagwu et al., 2000; Morshed et al., 2011). In addition, it may find applications as lubricants, ingredient in soaps and alkyl resins (Aigbodion and Pillai, 2000). The protein rich matrix may be used as cattle feed, as a feed for biogas production, for binderless

board production (Hidayat et al., 2014) and as a feed for thermochemical processes like pyrolysis (Vaz et al., 2005; Kootstra et al., 2011).

A number of studies have been reported on the expression of RSO from the rubber seeds and these are summarized in Table 1. Most studies involve solvent extraction using a hydrocarbon solvent (hexane, petroleum ether) or a chlorinated solvent. The oil yields cover a wide range and are between 5 and 49%.

Three studies have been performed using mechanical pressing, involving either a hydraulic or screw press. The yields in this case are typically lower than for solvent extraction and between 5.4 and 28.5%. Improved yields are possible by using solvent assisted hydraulic pressing. For instance, Morshed et al. (2011) showed that the use of hexane in mechanical pressing increased the yield from 5.4% to 49% yield (Table 1). Addition of a solvent during oil pressing has also been applied successfully to increase oil yields for the extraction of cotton, sunflower and soybean seeds (Abraham et al., 1993; Dufaure et al., 1999).

Table 1 show that the free fatty acid (FFA) content of the product oils varies from 2 to 38 wt% for the reported studies. For biodiesel synthesis, an FFA value of 3 wt% (Meher et al., 2006) is acceptable. The high FFA values are not necessary an intrinsic feature of the RSO but will depend on the processing conditions and technology,

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Table 1
Overview of literature studies on oil isolation from rubber seeds.

Isolation technique	Conditions	Oil Yield ^a	FFA content (wt%) ^c	Reference
Solvent	68–69 °C, <i>n</i> -hexane, 6 h	41.6%	7.6	Abdullah and Salimon (2009)
Solvent	40–60 °C, petroleum ether	45.6%	2	Ikwuagwu et al. (2000)
Solvent	27 °C, carbon tetrachloride, overnight	38.9% ^b	–	Haque et al. (2009)
Solvent	68–69 °C, <i>n</i> -hexane	49%	4	Zhu et al. (2011)
Solvent	68–69 °C, <i>n</i> -hexane, 4 h	45%	–	Ebewele et al. (2010)
Mechanical (hydraulic)	70 °C, 8 MPa, 10 wt% m.c	28.5%	38	Ebewele et al. (2010)
Mechanical	27 °C	5.4%	–	Morshed et al. (2011)
Mechanical + solvent	27 °C, hexane/seed wt. ratio: 0.8%	49%	2	Morshed et al. (2011)

^a Kernel (dehulled seed) unless stated otherwise.

^b Seed estimated from acid value (mg KOH/g).

^c Estimated from acid value (mg KOH/g).

and also by the storage conditions of the seeds (Zhu et al., 2011; Ebewele et al., 2010).

This paper presents a systematic study of the influence of pressure, temperature, moisture content and the use of a solvent on the pressing behaviour of dehulled rubber seeds in a laboratory scale hydraulic press. These process variables have shown to be of high importance for both oil yields and product quality (Venter et al., 2007; Willems et al., 2008; Subroto et al., 2015). A large number of experiments were performed and modeled using appropriate models. Ethanol was selected as the solvent of choice as it may be obtained from renewable resources.

2. Theory: the Shirato model

Several different mathematical models for the expression of oilseeds have been developed, which may be categorised as (i) models based on the nature of cell structures (Mrema and McNulty, 1985), (ii) empirical models (Fasina and Ajibola, 1990), and (iii) Terzaghi-type models (Shirato et al., 1986). The first model type provides fundamental insights in the expression process. However the information of the cell structure and cell dimensions is not easy to obtained, which limits the applicability.

Empirical models enable the prediction of oil yields but are often limited to specific seeds and processing equipment. Terzaghi models allow for a good description of the expression process, however, the model assumes that the cake thickness remains constant during pressing which is often not a good assumption. The Shirato model is a modified Terzaghi-type model. It has been previously applied successfully to model the hydraulic pressing of dry cocoa nibs and several oilseeds (Venter et al., 2007; Willems et al., 2008). It is a dynamic model which uses the cake thickness of a sample as a function of time and processing parameters as input. The cake thickness is expressed as the consolidation ratio (U_c), defined as the difference between cake thicknesses at the start of the process and the cake thickness at time t divide by the maximum difference in cake thickness (before and after the process). This consolidation ratio can be described as a function of time, pressure and material properties (Eq. (1)).

$$U_c(t) = \frac{L(0) - L(t)}{L(0) - L(t_{\text{end}})} = (1 - B) \left\{ 1 - \exp \left(\frac{-\pi^2 C_e t}{4\omega_0^2} \right) \right\} + B \left\{ 1 - \exp \left(- \left(\frac{E}{G} \right) \cdot t \right) \right\}$$

where

$$C_e = \frac{P}{\mu_1 \rho_s \alpha \frac{\partial e}{\partial P}} \quad (1)$$

with

U_c consolidation ratio (–)

L_0 cake thickness at t_{initial} (m)

$L(t)$ cake thickness at t_{initial} (m)

L_{end} cake thickness at t_{end} (m)

B relative contribution of secondary consolidation

C_e consolidation coefficient (m^2/s)

ω_0 volume of solids per unit area (m^3/m^2)

t time (s)

E/G creep constant (s^{-1})

P pressure (Pa)

μ_1 liquid viscosity (Pa.s)

ρ_s solids density (kg/m^3)

α filtration resistance (m/kg)

e void ratio (–)

The Shirato model consists of the sum of two terms, primary and secondary consolidation (creep). The relative contribution of the second term is given by the (fit) parameter B . For an individual pressing experiment, the consolidation ratio *versus* time is determined at a constant value of the pressure P . Parameter fitting allows calculation of the value of α , B and E/G for this particular experiment. When performing experiments at different pressures, the values for the individual cake resistances α may be correlated using the following relation:

$$\alpha = \alpha_0 \cdot \left(1 + \frac{P}{P_a} \right)^B \quad (2)$$

where

α_0 material constant for filtration resistance (m/kg)

β material constant for filtration resistance (–)

Parameter fitting for the experiments at different pressures allows calculation of α_0 , the pressure independent filtration resistance and the value of β , which is a measure for the pressure dependence of the cake resistance. A large value for β is indicative for a hard material.

Besides the cake thickness, the oil yield in the form of an oil recovery is also determined for each pressing experiment. The oil recovery can be related to material properties using Eq. (3).

$$\text{Oil recovery (wt\%)} = \left(\frac{(1 - F_0) \varepsilon \rho_o}{(1 - \varepsilon) \rho_s F_0} - 1 \right) \times 100\% \quad (3)$$

With:

ε final average porosity (m^3 non solid/ m^3 total)

F_0 original oil content of the seeds (wt%, d.b.)

ρ_o oil density (kg/m^3)

ρ_s solid density (kg/m^3)

The values for F_0 , ρ_o and ρ_s were determined experimentally in separate experiments (see experimental section for details). In combination with the experimentally determined oil recovery for an individual experiment, the cake porosity ε for each individual experiment may be determined. ε values for experiments at different pressures may be correlated using Eq. (4):

$$(1 - \varepsilon) = (1 - \varepsilon_0) \cdot \left(1 + \frac{P}{P_a} \right)^n \quad (4)$$

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