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Optimization of *Jatropha curcas* pure plant oil production

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Optimization of *Jatropha curcas* Pure Plant Oil Production

Erna Subroto

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PhD Thesis
University of Groningen
The Netherlands

The work described in this thesis was conducted at the department of Chemical Engineering, University of Groningen, The Netherlands and Research and Application Center, Bandung Institute of Technology, Indonesia

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Optimization of *Jatropha curcas* Pure Plant Oil Production

PhD thesis

to obtain the degree of PhD at the
 University of Groningen
 on the authority of the
 Rector Magnificus Prof. E. Sterken
 and in accordance with
 the decision by the College of Deans.

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Summary

Fossil fuels such as petro-diesel, LPG and kerosene are the main energy sources within the developing countries. However, fossil fuel is becoming more depleted and the price steadily increased over the last decade to values between 100 and 110 USD per barrel at this moment. Moreover, most developing countries are depending on fossil fuel imports and government's subsidization of those fuels becomes a burden for government's budgets. In addition, many negative environmental impacts are caused by the combustion of fossil fuel, i.e. the greenhouse effect associated with CO₂ emissions. Therefore, utilization of alternative fuels is strongly recommended.

The use of pure plant oils as fuel, either directly or after conversion of the oil to bio-diesel (FAME), is considered as a sustainable and environmental friendly option. *Jatropha curcas* L. is an example of an oil-bearing plant that produces non-edible oil, unsuitable for human consumption. The plant is well known in many tropical countries like Indonesia. It has potential to become an important oil producing plant in the future. The plant oil extracted from its seeds is a potential feed for biofuels or other oleochemicals.

The research described in this thesis is therefore aimed at developing this optimum pathway from harvesting seeds, drying, through storage, pre-treatment, oil extraction, purification and stabilization of that oil (see Figure 1).

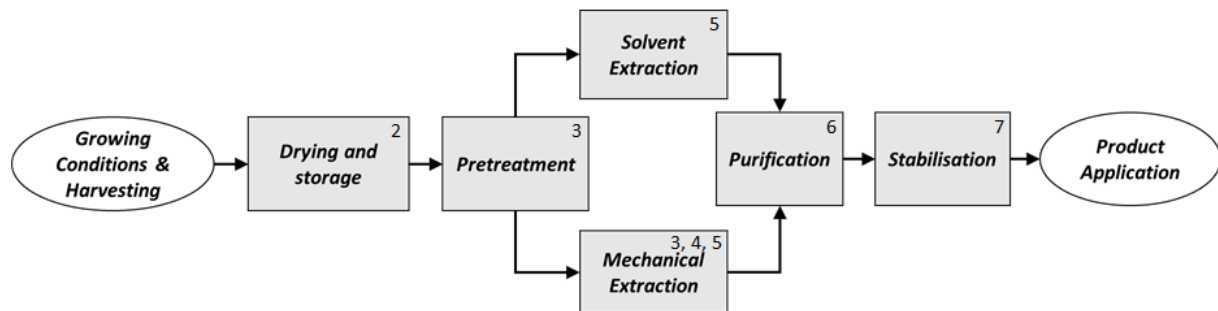


Figure 1 Schematic representations of “Jatropha PPO production”; superscripts: the corresponding chapter numbers in this thesis

In **Chapter 1**, a general overview of the use of vegetable oil for energy generation and chemical production will be provided. It will be shown that Jatropha oil is an interesting vegetable oil source for energy resources. The extraction technology developed for the production of vegetable oil is reviewed, including refining process and stabilization during storage. Subsequently, an extensive overview of process-product relations is discussed. Finally, an outline of this thesis is provided.

In **Chapter 2**, the drying characteristics of Jatropha in both seed and kernel are compared and discussed. The effect of drying parameters, including air-drying temperature, on several quality indicators of crude Jatropha oil were evaluated in terms of acid value, phosphorus content and oxidative stability.

In **Chapter 3**, the influence of process parameters on oil recovery from Jatropha kernel are investigated in more detail. The rate of pressure build-up, applied pressure, moisture content of sample, pressing temperature, duration of pressing, feedstock size reduction, shell removal and preheating time were studied as variables, and the quality of the obtained oils was evaluated.

In **Chapter 4**, the most important pressing parameters obtained from chapter 3 were further optimized to maximize the oil yield. This approach used the face centered central composite design response surface method. The non-linear model was generated and predicted the best condition to maximize oil yield. The experimental validation was conducted and the quality of the obtained oils was evaluated.

In **Chapter 5**, the effect of solvent assisted pressing was evaluated in order to maximize oil yield. Renewable solvents were used: bio-ethanol and bio-butanol. The purity of solvent, solvent to feed ratio, pressure, temperature and pressing time were studied as variables, and the quality of the obtained oils was evaluated.

In **Chapter 6**, purification of Jatropha oil was studied. The effect of purification parameters including purification agent, concentration and temperature on several quality parameters of crude jatropha oil were evaluated in terms of acid value, phosphorus content and oxidative stability on batch scale. The best condition was applied in a continuous process in a Centrifugal Contactor Separator (CCS) unit, and the quality of the obtained oils was evaluated.

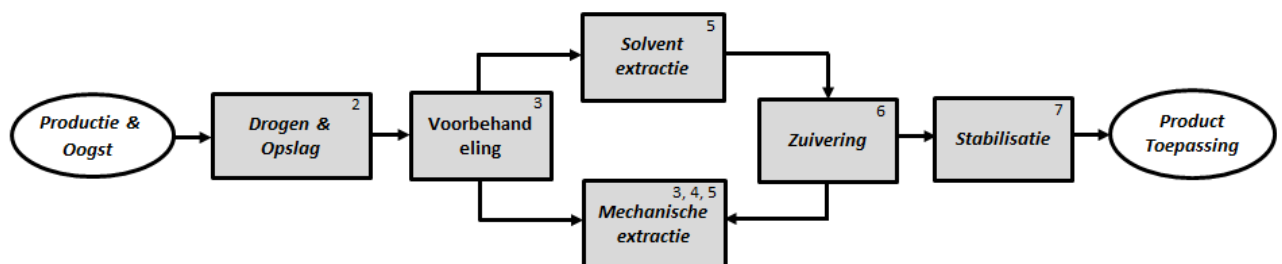
In **Chapter 7**, the stabilization of Jatropha oil was evaluated. Various antioxidants were studied to postpone the oxidation of the oil. The Oxidative Stability Index was analyzed and maximized as a quality parameter for the stabilization of Jatropha oil.

Samenvatting

In ontwikkelingslanden zijn brandstoffen zoals kerosine, benzine, diesel en LPG, die worden gewonnen uit fossiele bronnen, nog steeds de belangrijkste vormen van energie. Echter, deze fossiele bronnen zijn eindig, en de prijzen, van bijvoorbeeld olie, bleken instabiel en bereikten in de laatste 10 jaar waarden van 100 tot 110 USD per vat. Bovendien zijn veel ontwikkelingslanden afhankelijk van import en de door overheden verstrekte subsidies vormen een zware belasting voor de overheidsuitgaven. Tot slot leidt het gebruik van fossiele brandstoffen tot negatieve milieu effecten zoals de uitstoot van broeikasgassen. Al deze factoren hebben tot gevolg dat het gebruik van alternatieve brandstoffen wordt gestimuleerd.

Het gebruik van plantaardige olie als brandstof, hetzij direct of na omzetting tot biodiesel (FAME) wordt gezien als een milieuvriendelijke en duurzame optie. *Jatropha curcas* L. is een voorbeeld van een oliehoudende plant waaruit een olie gewonnen kan worden die niet geschikt is voor menselijke consumptie. De plant komt voor in tropische streken zoals, bijvoorbeeld, Indonesië. De plant kan in de toekomst een belangrijke bron worden voor de productie van olie. De olie wordt gewonnen uit de zaden en is een geschikte voeding voor de productie van brandstoffen en oleo-chemicaliën.

Het onderzoek dat is beschreven in dit proefschrift is gericht op de ontwikkeling van de optimale productieketen die loopt van het oogsten van de zaden, via drogen, opslag, voorbehandeling, olie extractie en olie-zuivering, tot het stabiliseren van de olie (Figuur 1).



Figuur 1. Schematisch overzicht van “*Jatropha* PPO productie”; cijfers verwijzen naar nummering van de hoofdstukken

Hoofdstuk 1 geeft een algemeen overzicht van het gebruik van plantaardige olie, inclusief *Jatropha* olie, voor energie toepassingen en de productie van chemicaliën. Extractie technologie, het raffinageproces en de uiteindelijke product stabilisatie zijn gebundeld en aangevuld met een discussie met betrekking tot de relatie tussen proces parameters en product eigenschappen.

Hoofdstuk 2 worden de droogkarakteristieken van Jatropha zaden en zaadkernen met elkaar vergeleken en besproken. Het effect van parameters in het droogproces, zoals bijvoorbeeld temperatuur tijdens de droging, op de product kwaliteitscriteria van de ruwe olie is bestudeerd. De belangrijkste onderwerpen voor evaluatie waren: het zuurgehalte in de olie, het finale fosfor gehalte en de oxidatieve stabiliteit.

Hoofdstuk 3 behandelt de invloed van proces parameters tijdens de olie extractie uit de zaadkernen van de Jatropha noot. Parameters die werden bestudeerd met betrekking tot proces optimalisatie en olie-kwaliteit zijn: snelheid van druk-opbouw tijdens de persing, toegepaste druk, vochtgehalte van het materiaal, temperatuur tijdens persing, totale perstijd, granulatie van de zaden, verwijderen van de schil, en het voorverwarmen van het materiaal.

Hoofdstuk 4 beschrijft de optimalisatie van de parameters die in het vorige hoofdstuk zijn geïdentificeerd. De optimalisatie is uitgevoerd met behulp van een face-centered central composite design response surface. Het design heeft geresulteerd in een lineair model voor de productie van de olie. De gemodelleerde maximaal haalbare situatie is praktisch getoetst en de kwaliteit van de olie is geëvalueerd.

Hoofdstuk 5 beschrijft de proces optimalisatie met bio-ethanol en bio-butanol als hulpmiddel tijdens de extractie. Als variabelen werden bestudeerd: type oplosmiddel, zuiverheid van het oplosmiddel, oplosmiddel tot grondstof verhouding, persdruk, temperatuur en perstijd. Het effect van de variabelen werd getest in termen van opbrengst en product kwaliteit.

De zuivering van Jatropha olie is beschreven in **Hoofdstuk 6**. Het effect van de proces parameters in termen van toegevoegde agentia, concentratie en temperatuur werden getest met betrekking tot hun effect op de volgende olie kwaliteitscriteria: zuur gehalte, fosforgehalte en oxidatieve stabiliteit. De beste resultaten werden verkregen bij het gebruik van een CCS – Centrifugale Contactor Separator – eenheid.

De stabilisatie van Jatropha olie is beschreven in **Hoofdstuk 7**. Verschillende anti-oxidanten werden getest op hun vermogen om de oxidatie van de olie te vertragen. Dit werd getest en geoptimaliseerd middels de bekende “Oxidatieve Stabiliteit Index – OSI” oxidatiesnelheids-indicator.