



## Full Length Article

# Optimizing the physical parameters to achieve maximum products from co-liquefaction using response surface methodology



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## HIGHLIGHTS

- A Design of Experiment (DOE) for co-liquefaction parameters optimization was proposed.
- Optimization of process parameters using Box-Behnken design in RSM.
- The products concentration of co-liquefaction relies on physical parameters used.

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## ABSTRACT

In this study, the influence of rubber seed kernel (RSK) on Mukah Balingian (MB) coal was investigated. Co-liquefaction was performed in a tubing reactor with tetralin as the solvent at various physical parameters. The study focused on the effects of RSK weight ratio, temperature and reaction time on conversion and products distribution. This paper aims at developing an effective methodology to find the optimum conditions that maximize the co-liquefaction conversion and products by using response surface methodology (RSM). Experiments were planned as per Box-Behnken design experiments. The mathematical model for the co-liquefaction products was developed using RSM with blending the ratio of RSK, temperature, and reaction time as process parameters. The developed RSM model is to determine the optimal process conditions that affect each of the outcomes. The results of this study revealed that the proposed RSM technique managed to find the optimum physical condition, which leads to the maximum value of conversion performance.

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## 1. Introduction

Rubber tree (*Hevea brasiliensis*) is a traditional plantation crop which has been cultivated mainly as an origin of natural rubber. The crop belongs to the family of euphorbiaceae and is native to rain forest in the Amazon region of South America. Nowadays, the tree is distributed throughout low latitude tropical region such as Malaysia and Thailand. According to the rubber seed production in India which is about 150 kg per hectare, the production of rubber seed in the country is expected at 300 kilotons per year. The use of non-edible rubber seed is a good solution to avert any fuel-food crisis. In addition, RSK can provide 40–50 wt% of oil yield which is a promising feedstock and could produce sufficient amount of biodiesel in Malaysia [1]. However, liquefaction of RSK has received limited attention till now.

The source of biomass such as RSK for liquefaction was usually affected by season, years or producing sites, typically has a relatively larger distributing area comparing with its total amount [2] and storage and harvesting of bulk density biomass. [3]. All these factors enhance the difficulty in supply and limit its large scale utilization. On the other hand, direct coal liquefaction (DCL) requires severe conditions to perform a considerable productivity [4]. The method to solve the above mentioned problems encountered in the individual liquefaction of coal and biomass is by co-liquefaction of them. Co-liquefaction technology of coal and biomass waste in preparation of oil has gained rapid increase in research interest [5].

Previous studies suggested a few benefits of the process. Firstly, the process can make use of hydrogen in biomass completely and thus reducing the hydrogen consumption and moderating the conditions of DCL [6]. Hence, the mild operation of DCL could be realized. Secondly, the addition of biomass to a coal conversion process reduces greenhouse gas emission. The carbon footprint of that process could be curtailed because biomass thermal conversion is

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inherently carbon neutral. Thirdly, biomass can promote the thermal disintegration of coal macromolecular, thus alleviate the severe reaction condition [7]. Lastly, the co-liquefaction of biomass and coal can ease the conversion and enhance the quality and quantity of oil yield. Large molecular fractions are produced from coal decomposition while biomass can produce smaller molecular fragments. These smaller fragments are able to combine with fractions produced from coal and terminate the cross-linking reactions between them [8]. Till today, very limited studies of co-liquefaction on coal and biomass have been carried out and the optimized conditions are not entirely clear.

Optimization by classical method famously known as optimization one parameter at a time is an option that involves a lot of experiments which will lead to the use of more materials, time consuming and labor intensive. In addition, this approach is only valid if the variables are totally independent of each other. However, there is other option of optimization which involves statistical techniques that currently being used in many studies known as Design of Experiment (DOE). This design could minimize cost by reducing the number of experiments to be conduct, minimize process variation, and rework. DOE may determine the individual and interactive effect of many factors that could affect the output of a process in any design simultaneously and understand the relationship between them [9].

RSM is one of the popular DOE techniques. This method implements statistical and mathematical techniques for development, improvement and optimizing process, in which the response of interest is affected by several variables. This statistical process works by defining the effect of independent variables, individually or in combination, thus allows the determination of the significance of each parameter studied, along with the significant interaction between parameters. Plackett-Burman design, Box-Behnken design and Central Composite design are among the typical designs in RSM. Thus, an optimization attempt should be made for co-liquefaction of low rank MB coal and RSK. In this study, Box-Behnken design as a model for multivariate optimization of analytical procedure was implemented because it is more efficient [10], has the lowest number of experiments and cost effective.

To the best of our knowledge, optimization of co-liquefaction of rubber seed kernel and Mukah Balingian coal has not been reported. The high percentage of oil + gas and low percentage of asphaltene and preasphaltene is the main target for this study. In order to attain products of high conversion, optimization of parameters such as blending ratio of RSK, temperature, and reaction time are crucial since the co-liquefaction technique was performed. Optimization of the bio-crude composition percentage by co-liquefaction technique using Box-Behnken approach is reported in this work.

## 2. Experimental

### 2.1. Feedstocks

All feeds were acquired locally. The low rank coals used in this study were a sub-bituminous MB coal from Sarawak, which were received in bulk form. The biomass feedstocks used were rubber seeds, supplied by local rubber plantation in its raw form. The rubber seed was then separated between the shell and kernel. MB coal and RSK were pulverized and all feeds were separately sieved. The particle size feeds used in this study were  $\sim 212 \mu\text{m}$ . Feeds were dried at  $105^\circ\text{C}$  for approximately 24 h and stored in an air-tight container. Solvents used were tetralin, tetrahydrofuran (THF), hexane and toluene. Tetralin and THF were analytical grade reagents, purchased from Merck. Hexane and toluene were industrial grade reagents, purchased from a local supplier. All solvents were used without further purification.

### 2.2. Experimental setup and procedure

The liquefaction experiments were carried out in a 25 mL tubing reactor shaken by using an air vibrator. 1.0 g of a mixture of MB coal and a certain blending ratio of RSK was loaded into the reactor together with 5 mL of tetralin and a 5 mm diameter steel ball. Before the liquefaction experiment, the reactor was sealed. The reactor was placed into a furnace and heated to the desired temperature and maintained for the desired reaction time. Then, the reactor was quenched to ambient temperature in an ice bath. The liquefaction mixture in the reactor was transferred completely to a clean flask by washing with THF. The THF soluble yield was further separated while the THF insoluble yield was accounted as liquefaction residue.

### 2.3. Separation of liquefaction product

The liquefaction product was separated by Soxhlet solvent extraction with hexane, toluene, and THF in turn. The hexane insoluble but toluene soluble fraction was defined as asphaltene (As), and the toluene insoluble but THF soluble fraction was defined as preasphaltene (PAS). The liquefaction conversion of feedstock and yields of liquefied product were defined according to Eqs. (1)–(3):

$$\text{Conversion (\%)} = \frac{W_f - W_r}{W_f} \times 100\% \quad (1)$$

$$W_{as} (\%) = \frac{(W_{ts} \times 100\%)}{W_f} \quad (2)$$

$$W_{pas} (\%) = \frac{(W_{ti} \times 100\%)}{W_f} \quad (3)$$

where  $W_f$  is the mass of the feedstock;  $W_r$  is the mass of residue,  $W_{ts}$  is the mass of toluene soluble fraction; and  $W_{ti}$  is the mass of the tetrahydrofuran soluble fraction.

### 2.4. Experimental design

The effect of adding RSK on the liquefaction of MB coal was studied. In response surface methodology (RSM), the Box-Behnken design with 3 levels and 3 factors were used for optimization of the co-liquefaction process condition. For three factors, the design offers some advantage in requiring a fewer number of runs. The software used for the Box-Behnken design was Design Expert Software (Stat-Ease Inc., MN, USA). The variables studied in this design were RSK blending ratio, temperature and reaction time. 17 sets of experimental runs in random order were carried out in order to optimize the process based on the response data.

## 3. Results and discussion

The proximate and ultimate analysis results of MB coal and RSK is listed in Table 1. Analysis of both samples indicated that MB coal have lower content of volatile matter, carbon, hydrogen and nitrogen than RSK. The H:C mass ratio for MB coal was 0.9, relatively low with comparison to that of RSK with 1.5.

The reaction time for all the individual liquefaction experiments was kept constant at 30 min (Fig. 1). It was observed that the conversion increased from  $360^\circ\text{C}$  to  $380^\circ\text{C}$  and decreased at  $400^\circ\text{C}$ . The behavior may be due to increase rupture of carbon structure into radicals of smaller molecular weight at  $380^\circ\text{C}$  [11]. A similar behavior to the conversion occurred for oil + gas production: oil increased with the increase of temperature from  $360^\circ\text{C}$  to  $380^\circ\text{C}$ . The oil + gas yield then decreased with the increasing of temperature from 27.1% at  $380^\circ\text{C}$  to 22.9% at  $400^\circ\text{C}$  and 19.9% at

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