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Research paper

Effect of storage time on moisture content of *Reutealis trisperma* seed and its effect on acid value of the isolated oil and produced biodiesel

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A B S T R A C T

Reutealis trisperma has been studied as an alternative source of producing biodiesel. Moisture content of *Reutealis trisperma* seed is very important to determine the quality of biodiesel produced from the oil isolated from *Reutealis trisperma* seed. This study investigated the time profile of moisture content for four different fractions of trisperma seed (PT-3, PT-7, PT-14, NPT-21) for four months. The moisture content of trisperma seeds was modelled using instationary diffusion equation. Total oil content in trisperma seed as well as acid value of the extracted oil from stored trisperma seed were periodically measured for four months. Acid value of trisperma oil obtained from the stored seed experienced an increase from 1.1 mg KOH/g to 2.8 mg KOH/g for PT-3, 1.9 mg KOH/g to 9.9 mg KOH/g for PT-7, 3.4 mg KOH/g to 11.6 mg KOH/g for PT-14 and 4.7 mg KOH/g to 25.4 mg KOH/g for NPT-21. The acid value of trisperma oil and biodiesel that had been stored for four months (27 °C, closed container) was also determined. Upon storage, the acid value of trisperma oil only slightly increased from 1.1 to 1.3 mg KOH/g. The acid value of stored biodiesel from trisperma oil also only slightly increased from 0.4 to 0.43 mg KOH/g.

1. Introduction

The demand of fuel for transportation sector is predicted to increase by 38% from 2010 to 2040. Many studies have been carried out to investigate the suitable natural resources that can be valorised to produce biodiesel as an alternative fuel for vehicles (Balat, 2007; Hoekman et al., 2012; Silitonga et al., 2013). It is reported that more than 350 natural resources such including plant oils and animal fats have been studied for producing biodiesel.

Palm, sunflower and rapeseed oil are currently the major source for biodiesel production (Atabani et al., 2012). However, these oils are edible, and various studies have been carried out to investigate non-edible sources that may be valorised as an input to produce renewable fuel such as jatropha and rubber seeds (Silitonga et al., 2011; Abduh et al., 2013, 2015, 2016a,b,c; Kusomo et al., 2017). Several studies have showed that trisperma oil is considered as a promising feedstock due to its high oil content (38%–40%) and productivity (3.8–8.7 ton/ha/yr) (Kumar et al., 2015; Silitonga et al., 2017; Abduh et al., 2018; Silitonga et al., 2019).

The synthesis of biodiesel from trisperma oil have been previously studied (Kumar et al., 2015; Holilah et al., 2015; Silitonga et al., 2017, 2019). Two-steps transesterification was commonly applied in the previous studies because acid value of the trisperma oil used in the studies was relatively high (5 to 14 mg KOH/g). These values exceeded the typical acid value of plant oil required for the synthesis of biodiesel (Zhu et al., 2011; Abduh et al., 2016a,b,c). Such high acid value may be due to improper storage and processing conditions as found by Abduh et al. (2016a).

Studies on the effect of storage conditions on the quality of trisperma seed, oil and biodiesel have not yet been reported. In addition, studies on one step transesterification of trisperma oil for producing biodiesel are very scarce. Hence, this study was carried out to investigate the moisture content of trisperma seed as well the acid value of trisperma oil and biodiesel as a function of storage time

2. Materials and methods

2.1. Materials

Reutealis trisperma seeds were received from Majalengka and kept at 27 °C (room temperature) before experimental

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studies were conducted at Institut Teknologi Bandung. Methanol, n-hexane (99%, for analysis) and KOH were purchased from Brataco, Bandung.

2.2. Experimental conditions for the effect of storage on trisperma seeds, oil and biodiesel

Upon receipt, trisperma seeds were divided into for fractions (NPT-21, PT-14, PT-7 and PT-3) and subjected to different pre-treatments as shown in Fig. 1. Trisperma seeds in fraction PT-14 were sun-dried for 7 days until the seeds had a moisture content of approximately 14.3%. Trisperma seeds in fraction PT-7 were dried in an oven at 60 °C for 1 day whereas the seeds in fraction PT-3 were dried at similar conditions for 3 days until the seeds had a moisture content of approximately 6.5% (PT-7) and 3.4% (PT-3). Trisperma seeds in fraction NPT-21 were not pre-treated and had an initial moisture content of 21%.

Each fraction had an average weight of 10 kg and were kept in baskets (with a similar dimension of 50 cm × 30 cm × 18 cm) at 27 °C. Conditions in the storage room particularly relative humidity and temperature were recorded daily. Oil and moisture content of the stored trisperma seeds were determined regularly for a period of 4 months. Trisperma oil isolated from the stored seed were also analysed in terms of acid value. In addition, acid value of stored trisperma oil and biodiesel (27 °C) were also measured during the study period of 4 months.

2.3. Analysis of moisture and oil content of trisperma seeds

Moisture content of trisperma seed was determined by heating the seed at 103 °C in an oven for at least 24 h until the weight remains constant (Abduh et al., 2016a,c). Total oil content in the trisperma seed was extracted using a Soxhlet extractor with an n-hexane as a solvent. A detailed procedure to determine the total oil content is described elsewhere (Abduh et al., 2016a,c).

2.4. Hydraulic pressing of trisperma oil

All trisperma oil used in this study were isolated from the seed using a mechanical method except for the case of determining the total oil content in trisperma seed that used a solvent extraction method. A laboratory-scale hydraulic pressing machine as described by Abduh et al. (2016c) was used in this study to isolate trisperma oil from the seed. Approximately 7 g of sample was pressed at 20 MPa (27 °C) inside a pressing at chamber for 10 min. The extracted trisperma oil was stored at room temperature for further analysis.

2.5. Synthesis of biodiesel from trisperma oil

Trisperma oil that was isolated from trisperma seed that had been dried to a moisture content of 3.4% (PT-3) was used in the transesterification to produce biodiesel. The transesterification was carried out by mixing the trisperma oil with methanol and KOH as a catalyst in a batch reactor at 60 °C and mixed at 600 rpm for 120 min. A 6:1 molar ratio of methanol to oil and 1% weight of catalysis with respect to the oil were applied in this study. The synthesised biodiesel was separated from the glycerol layer and washed with water (Abduh et al., 2016a,b,c).

2.6. Analysis

Biodiesel yield is defined as the amount of crude biodiesel produced divided by the amount of trisperma oil used in the transesterification as in Eq. (1)

$$\text{Yield}\,(\%\ \text{weight, dry basis}) = \frac{\text{amount of crude biodiesel (g)}}{\text{amount of trisperma seed oil (g)}} \times 100 \quad (1)$$

An acid–base titration was carried out to determine the acid value of the samples with phenolphthalein as an indicator as suggested by Abduh et al. (2013). The density of the sample was determined at 27 °C using a standard pycnometer. Calorific value of trisperma oil and biodiesel was measured using a bomb calorimeter at the Physical Chemistry Laboratory, Institut Teknologi Bandung.

2.7. Modelling the moisture ratio of trisperma seed with respect to time

Eq. (2) was used to model the moisture ratio of trisperma seed (assumed to be spherical with an average radius of 0.0019 m) with respect to time and to determine the effective diffusion coefficient as suggested by Kang and Delwiche (2000). Other detailed assumptions for the model are described elsewhere (Abduh et al., 2016a)

$$Moisture\ ratio\ (wt\%) = \frac{M - M_0}{M_e - M_0} = 1 - \frac{6}{\pi^2}\sum_{m=1}^{\infty}\frac{1}{m^2}e^{\left[\frac{-m^2\pi^2 D_{eff}\,t}{r_0^2}\right]} \quad (2)$$

where

M_e Moisture content at equilibrium (%)
M_0 Moisture content at initial time (%)
M Moisture content at time t (%)
D_{eff} Effective diffusion coefficient (m^2/s)
t time (s)
r_0 radius (m)

3. Results and discussion

3.1. Relative humidity and moisture content of trisperma seed

Initially, trisperma seed received from Majalengka had a moisture content of 21% weight on a wet basis (w.b.). The seed comprises of 35% weight on a dry basis (d.b.) shell and 65% d.b. kernel. The kernel contains approximately 60.3% d.b. oil which is equivalent to 39.2% d.b. on a seed basis. As such resembles the total oil content of trisperma seed found by Kumar et al. (2015) which lies in the range of 38%–40% d.b.

Effect of time on the relative humidity and moisture content of trisperma seed was investigated for four different fractions of trisperma seed with different initial moisture content viz. (i) 21% (no pre-treatment, NPT) (ii) 14.3% (pre-treatment by sun-drying for 7 days, PT-14) (iii) 6.5% (pre-treatment by oven drying at 60 °C for 1 day, PT-7) (iv) 3.4% (pre-treatment by oven drying at 60 °C for 3 days, PT-3).

The parameters of storage conditions particularly storage temperature and relative humidity were determined twice daily for 4 months. The relative humidity of the surrounding varied between 50 to 60% whereas the temperature was stable between 26–28 °C. In addition, the relative humidity of all the seed fractions (NPT, PT-14, PT-6, PT-3) were also monitored twice daily and the average values were reported as illustrated in Fig. 2.

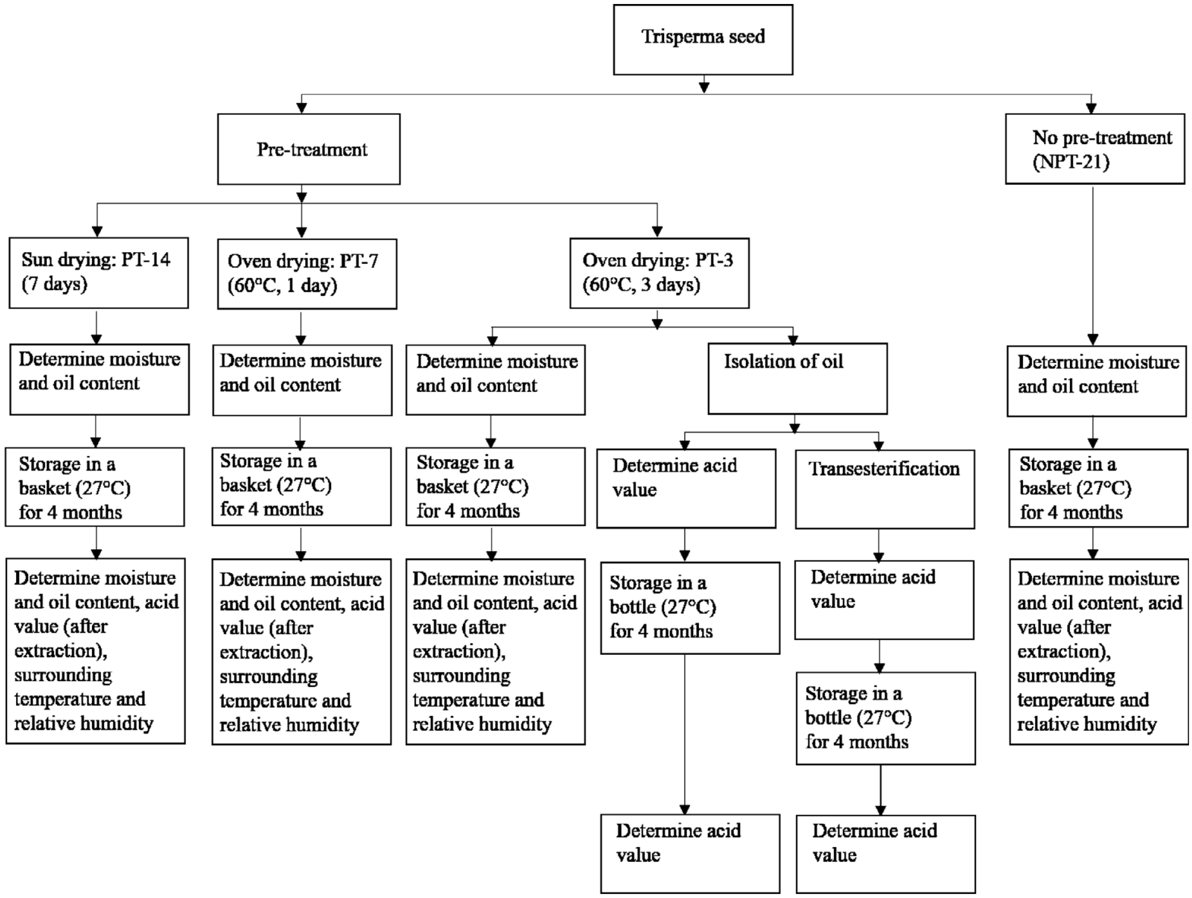

Fig. 1. Flowchart for the influence of storage on seed, oil and biodiesel from *Reutealis Trisperma*.

Initially, the relative humidity of the samples from NPT-21 and PT-14 was approximately 83% and decreased rapidly with time in the first month of storage before it reached an equilibrium value of approximately 57% after 40 days. The initial relative humidity of PT-7 was approximately 33% and increased rapidly with time in the first month of storage before it reached an equilibrium value of approximately 57% after 40 days. In contrast, the relative humidity of PT-3 increased slowly with time and equilibrium was almost achieved after 120 days.

The moisture content of all the seed fractions (NPT, PT-14, PT-7, PT-3) were also determined periodically. From Fig. 3, it can be observed that the moisture content of samples from NPT-21 and PT-14 decreased rapidly with time whereas the moisture content of samples from PT-7 and PT-3 increased slowly with time before the seed achieved an equilibrium moisture content of approximately 8%–9%. This value resembles the equilibrium moisture content for rubber seed (9%) and jatropha seed (8.6%) as described in previous studies (Subroto et al., 2015; Abduh et al., 2016a).

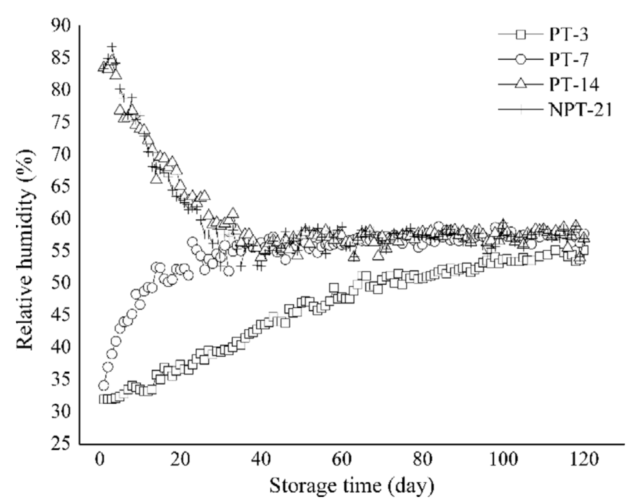

Fig. 2. Influence of storage time on the relative humidity of trisperma seeds (27 °C).

3.2. Modelling the moisture ratio of trisperma seed with respect to time

Moisture content of trisperma seed at equilibrium as well as water diffusion coefficient in trisperma seeds (at 50%–60% relative humidity and 27 °C) were obtained by fitting the experimental data (shown in Fig. 3) using Eq. (2). From Fig. 4, it can be observed that the developed model shows a relatively good agreement with the experimental data as illustrated in Fig. 4. From Table 1, it can be observed that the equilibrium moisture content for all trisperma seed fractions as estimated by Eq. (2) was approximately 9%. As such resembles the results reported in a previous study for other oilseeds (Abduh et al., 2016a). As for estimated diffusion coefficients of water in the trisperma seed, the values vary in the range of 1.18×10^{-11} m^2/s to 5.40×10^{-11} m^2/s which are slightly higher in comparison to the diffusion coefficient of water in rubber seed ($0.49–0.78 \times 10^{-11}$ m^2/s, storage temperature of 26–28 °C) and wheat ($0.22–0.31 \times 10^{-11}$ m^2/s, storage temperature of 27–31 °C) (Fan et al., 1961; Abduh et al., 2016a).

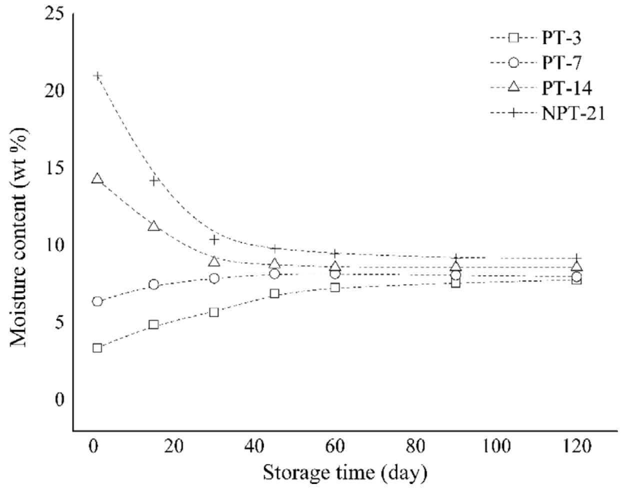

Fig. 3. Influence of storage time on the moisture content of trisperma seeds (27 °C, 50%–60% relative humidity).

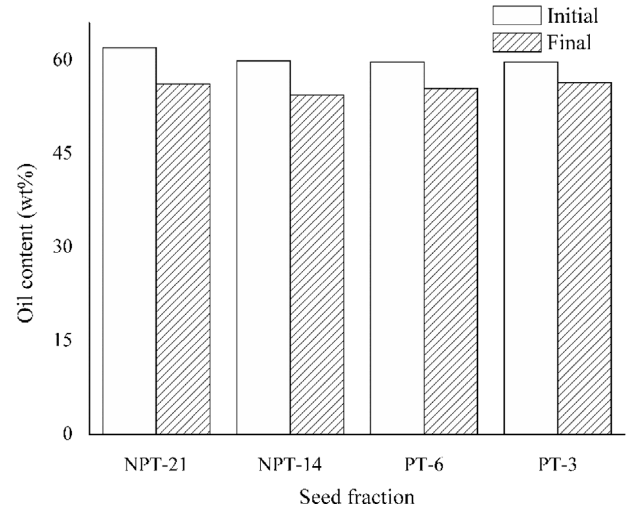

Fig. 5. Initial and final total oil content for different trisperma seed fractions.

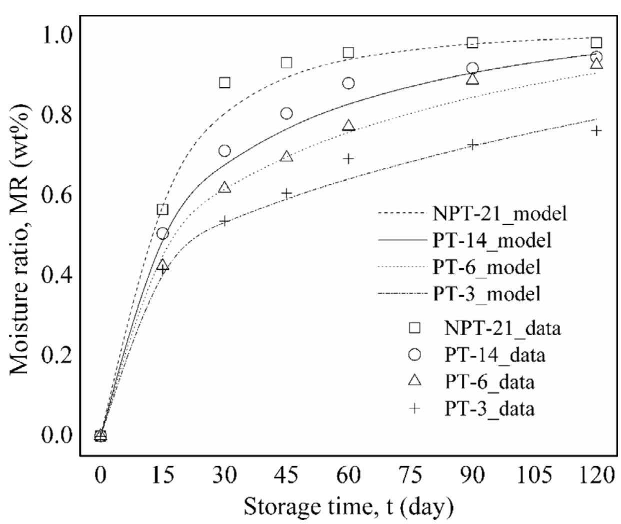

Fig. 4. Comparison of experimental and modelled moisture ratio versus time for different trisperma seed fractions.

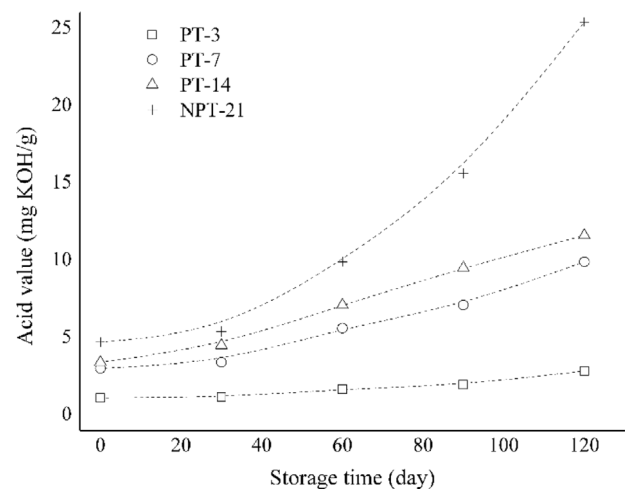

Fig. 6. Influence of storage time on the acid value of pressed trisperma oil from stored seeds (PT-3, PT-7, PT-14, NPT-21).

Table 1
Modelling results on different fractions of trisperma seed: equilibrium moisture content of and effective diffusion coefficient of water.

Seed fraction	M_e (%)	D_{eff} (m^2/s)
NPT-21	8.92	5.40×10^{-11}
PT-14	8.97	2.89×10^{-11}
PT-6	8.99	2.09×10^{-11}
PT-3	9.17	1.18×10^{-11}

3.3. Oil content of and acid value from stored trisperma seeds

Initially, dehulled trisperma seeds from all seed fractions had an oil content in the range of 60 to 62% d.b. The oil content for all seed fractions was determined again after being stored for 4 months, the total oil content decreased to 54%–56% d.b. as shown in Fig. 5. As such indicates that the oil content is slightly affected by storage.

The oil contained in the stored seeds were isolated and the acid value of the isolated oil increased from 1.1 mg KOH/g to 2.8 mg KOH/g for PT-3 (stored trisperma seed with an initial moisture content of 3.4%); 3.0 mg KOH/g to 9.9 mg KOH/g for PT-7 (stored trisperma seed with an initial moisture content of 6.5%) and 3.4 mg KOH/g to 11.6 mg KOH/g for PT-14 (stored trisperma seed with an initial moisture content of 14.3%). Formation of free fatty acid in the trisperma oil is more pronounced for the seed fraction NPT-21 which had the highest initial water content (21%). Fig. 6 shows that the acid value of the isolated oil increased from 4.7 mg KOH/g to 25.4 mg KOH/g. These findings suggest that drying trisperma seeds at 60 °C to reduce the moisture content of the seed (PT-3, PT-7) may deactivate the enzymes and consequently lower the rate of free fatty acid formation as reflected with lower acid values upon storage (Zhu et al., 2011).

3.4. Acid value from stored trisperma oil

Trisperma seed that had not been pre-treated with an initial moisture content of 21% (NPT-21) and trisperma seeds that had been dried to a moisture content of 3.4% (PT-3) were dehulled and pressed using a manual hydraulic press. The average oil yield was 27% d.b. The hydraulic pressing of trisperma seed was carried out at room temperature (27 °C) to minimise the effect of elevated temperature that may affect the acid value of trisperma oil. A previous study by Abduh et al. (2016c) demonstrated that increasing the temperature of hydraulic pressing from 35 °C to

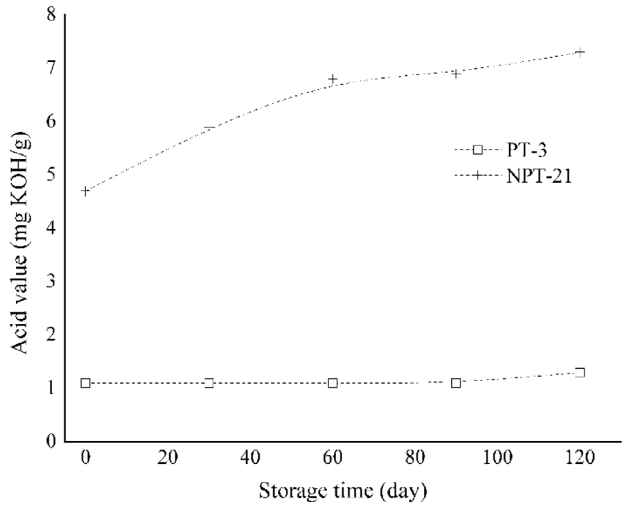

Fig. 7. Effect of storage time on the acid value of stored trisperma oil (PT-3 and NPT-21).

Table 2
Properties of crude oil and biodiesel from *Reutealis trisperma* seed.

Property	Trisperma oil	Biodiesel
Acid value (mg KOH/g)	1.1	0.4
Density (kg/m^3) (27 °C)	926	891
Calorific value (MJ/kg)	37.5	35.7

85 °C increased the acid value of isolated rubber seed oil from 1.5 to 2 mg KOH/g.

The pressed trisperma oil was kept in a glass bottle at room temperature (27 °C) for four months. Acid value of the isolated oil was measured on a regular basis and the profiles are illustrated in Fig. 7. Initially, trisperma oil obtained from PT-3 and NPT-21 had acid values of 1.1 mg KOH/g and 4.7 mg KOH/g, respectively. After being stored for four months, the trisperma oil experienced an increase in acid values to 1.3 mg KOH/g and 7.3 mg KOH/g for PT-3 and NPT-21, respectively.

A lower increase in acid value was recorded for stored trisperma oil in comparison to a higher increase of acid value for trisperma oil obtained from the stored trisperma seeds (Fig. 6). Similar trend was also obtained by Zhu et al. (2011) and Abduh et al. (2016a) who observed a rapid rise in the acid value of stored rubber seed oil as compared to the rubber seed oil isolated from stored rubber seed. Zhu et al. (2011) suggested that enzyme activity may be considered insignificant in the isolated oil whereas Abduh et al. (2016a) explained that isolated oil has a lower water content in comparison to the seed which influence the rate of hydrolysis of triglycerides.

Relevant properties of freshly pressed trisperma oil obtained from PT-3 such as acid value, density, and calorific value were determined as shown in Table 2. The acid value and density (at 27 °C) of trisperma oil was 1.1 mg KOH/g and 926 kg/m^3, respectively. The calorific value of the trisperma oil was 37.5 MJ/kg which resembles the results obtained by Kumar et al. (2015).

3.5. Acid value of biodiesel from trisperma oil

Trisperma oil that was isolated from trisperma seed that had been dried to a moisture content of 3.4% (PT-3) was used in the transesterification to produce biodiesel. Unlike the two steps transesterification procedures that had been investigated in previous studies by Holilah et al. (2015) and Kumar et al. (2015), one step transesterification was carried out in this study because the acid value of the trisperma oil was relatively low (1.1 mg KOH/g). The transesterification was carried out in a batch reactor for approximately 1 h. After that the mixture was allowed to settle to separate the crude biodiesel and glycerol layers. The yield of crude biodiesel was approximately 0.91 g/g whereas the yield of the crude glycerol was 0.19 g/g. The crude biodiesel was washed to remove the catalyst and excess methanol followed by drying to remove the water content. The yield of the biodiesel was approximately 0.78 g/g.

Relevant properties of the produced biodiesel such as calorific value, acid value, and density were determined as shown in Table 2. Biodiesel from trisperma oil has a calorific value of 35.7 MJ/kg which is similar to the values reported by Kumar et al. (2015) and Holilah et al. (2015). The biodiesel has a density (at 27 °C) of 891 kg/m^3 with an acid value of 0.4 mg KOH/g. After four months of storage in a glass bottle at room temperature (27 °C), acid value of the stored biodiesel only slightly increased from 0.4 mg KOH/g to 0.43 mg KOH/g. As such resembles the findings in previous studies that the acid value of biodiesel is relatively stable upon storage at room temperature (Leung et al., 2006; Abduh et al., 2016a)

3.6. Comparison of the acid value after four months of storage

Comparison of initial and final acid value of trisperma oil obtained from stored trisperma seed, as well as the initial and final acid value of trisperma oil and biodiesel after four months of storage at 27 °C are presented in Table 3. The relative increase of acid value for trisperma oil obtained from stored seed increased with a higher moisture content in the seed. The pre-treated seed fraction (PT-3, PT-7, and PT-14) had a lower relative increase of acid value in comparison to the non-pre-treated seed fraction (NPT-21). As such highlights the importance of pre-treatment to reduce the moisture content prior to long term storage to avoid excessive formation of free fatty acid that may increase the acid value (Abduh et al., 2016a,c). Trisperma oil obtained from seed fraction PT-3 had a lower increase in acid value than the trisperma oil obtained from seed fraction NPT-21. This again suggests that it is crucial to reduce moisture content of stored seed. The acid value of biodiesel from trisperma oil after four months is about constant.

4. Conclusions

Acid value of trisperma oil isolated from stored trisperma seeds augmented with time. Pre-treatment of the seeds by drying at 60 °C reduced the increase of acid value upon storage as compared to seeds that have not been subjected to a drying procedure. Reducing the initial moisture content of trisperma seed upon storage increased the equilibrium moisture content (from 8.92% to 9.17%) while at the same time decreased the effective diffusion coefficient of water in trisperma seed (from 5.40×10^{-11} m^2/s to 1.18×10^{-11} m^2/s). Trisperma seed that had been dried to a moisture content of 3.4% (PT-3) had the lowest effective diffusion coefficient of water and produced trisperma oil with lowest acid value; 1.1 mg KOH/g and the derived biodiesel had an acid value of 0.4 mg KOH/g. After four months of storage, the acid value of stored trisperma oil and biodiesel slightly increased to 1.3 mg KOH/g and 0.43 mg KOH/g, respectively.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Table 3
Initial and final acid values for oil from stored seed, oil and biodiesel originating from rubber and trisperma seed.

Stored material	Acid value (mg KOH/g)		Relative percentage of increase (%)
	Initial	Final	
Seed			
- PT-3[a]	1.1	2.8	155
- PT-7[a]	3.0	9.9	230
- PT-14[a]	3.4	11.6	241
- NPT-21[a]	4.7	25.4	540
- Rubber seed [b]	0.8	4.2	425
- Rubber seed [b]	0.5	2.1	320
Oil			
- Trisperma oil[c]	1.1	1.3	18.1
- Trisperma oil[d]	4.7	7.3	55.3
- Rubber seed oil[b]	0.52	0.6	15.3
Biodiesel			
- Trisperma oil methyl ester[e]	0.4	0.43	7.5
- Rubber seed ethyl ester[b]	0.32	0.33	3.1

[a]This study, after 4 months of storage.
[b]Abduh et al. (2016a,b,c) after 2 months of storage.
[c]This study, trisperma oil (isolated from seed fraction PT-3) after 4 months of storage.
[d]This study, trisperma oil (isolated from seed fraction NPT-21) after 4 months of storage.
[e]This study, after 4 months of storage.

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