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BATCH SYSTEM CLARIFICATION OF TWO VARIETIES OF PALM OIL: KINETICS AND THERMODYNAMICS

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ABSTRACT

The kinetics and thermodynamics of palm oil clarification, a major and critical unit operation along the palm oil chain especially at local levels, were studied in this work. The effects of temperature, oil/water ratio and stirring speed on oil yield were investigated. No significant difference in oil yield with respect to temperature was observed for the dura *variety while for the* tenera *variety oil yield decreased with an increase in temperature. Increase in oil water ratio significantly* ($p<0.05$) *increased oil yield for both varieties. Optimum clarification conditions for both varieties were established as temperature of 80°C, oil/water ratio of 1:2, 30 min settling time and a stirring speed of 95 rpm. These conditions gave oil clarification yields of 73.33% and 90.39% for* dura *and* tenera*, respectively. The kinetics of palm oil clarification was modelled using a modified Newton and Lewis equation and the Peleg's model. The thermodynamic study gave enthalpy change* (ΔH) *values of -1809.87 and -17182.54 kJ/mol for* dura *and* tenera *variety respectively showing that the clarification process is exothermic. The corresponding values for entropy change* (ΔS) *were negative while free energy changes* (ΔG) *were positive indicating the process is random, non-spontaneous and irreversible.*

Keywords: clarification, kinetics, oil yield, palm oil, thermodynamics.

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INTRODUCTION

The oil palm (*Elaeis guineensis*) is one of the most important species in the genus *Elaeis* which belongs to the family Palmae grown in the tropics. It originated from the Guinea Coast of West Africa (Henson, 2012). World's demand for cooking oils is expected to go well above 240 million tonnes by 2050 for both industrial and domestic consumption and palm oil would contribute to more than 50% of global world vegetable production (Xin *et al.*, 2022). Three main varieties are generally recognised, *i.e.*, *dura* (thick shelled), *tenera* (medium or thin shelled) and *pisifera* (shell-less). It is the cross between *dura* and *pisifera* that results in *tenera* variety that makes up the basis of modern plantations (Henson, 2012; Teoh, 2002).

One interesting feature of oil palm cultivation is the production of two different types of edible oils; palm and palm kernel oils with distinct physicochemical properties from the pulp and the kernel, respectively. Palm oil is extracted from fresh fruits which on reception are washed, sterilised, and cooked to soften the pulp and facilitate detachment from the fresh fruit bunches and subsequent oil flow during extraction. The oil is then extracted by press followed by clarification and subsequent drying and bottling. Palm kernel oil is extracted from the kernel after cracking of the nuts, a byproduct from the palm oil process. The nuts are crushed, cooked and extracted by press and/or solvent extraction. Palm kernel oil is then clarified in a filter press or by sedimentation, refined and packaged. In addition, the empty fruit bunches and husks left behind

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after oil processing can be used as combustion fuels and in pyrolysis to produce bio-oils and char for various applications (Claoston *et al.,* 2014; Omar *et al.,* 2011; Sukiran *et al.,* 2011; Sulaiman & Abdullah, 2011). These make oil palm cultivation very appealing because all parts of the plant have income-generating potential. The oils extracted from the oil palm are used in pharmaceuticals and cosmetics and recently for biodiesel production (Ayanlowo *et al.,* 2022; Suryani *et al.,* 2020).

Clarification is the last but most important unit operation of the palm oil process. Clarification is aimed at separating the sludge consisting of water, fibrous material, debris and other unwanted solids that are left in the oil after extraction (Nurulhuda, 2009). Several parameters such as the volume of oil to water ratio, operating temperature, stirring speed and retention time affect oil clarification yields. Hence, mastery of how these parameters affect oil clarification yields is important in the design of clarifiers for palm oil, to reduce oil losses, energy use and the overall cost of the clarification process.

Kinetic studies are useful to identify factors (temperature, reactants, catalysts) that influence a reaction significantly and how they can be controlled to maximise the amount of product produced and to determine the mechanism of a reaction. Thermodynamics determines if the reaction is endothermic or exothermic as well as the entropy changes that are involved. Thermodynamics thus explains the driving force for a reaction. There is an inherent relation between the kinetics and thermodynamics of a reaction (Van Boekel & Tijsken, 2001).

Modelling batch kinetic data is important for clarification process scale-up and design of clarifiers as it provides information that could be used to compare different modelling approaches under different operating conditions for the process.

Although a substantial amount of palm oil is produced industrially in most palm oil-producing countries, there is a non-negligible contribution that comes from smallholder farmers. For example, in Cameroon, it is estimated that smallholder farmers contribute about 30% of the total palm oil production (Frank *et al.,* 2011). Smallholder farmers, therefore, derive substantial incomes from oil palm production to partially or completely fulfil their household needs which is essential for poverty reduction in their communities (Koczberski 2007; Qaim *et al.,* 2020). Industrial palm oil processing is elaborate, mastered and already vulgarised for more than a century but is very expensive and beyond the reach of smallholder farmers. Because industrial processing techniques are not directly transposable to smallholder levels, there is a need to adapt them to those of smallholder farmers to reduce the drudgery that is involved in processing at this level. Processing at this level produces doubtful or variable quality oil because of the difficulties encountered at the level of clarification. For the smallholder farmers to meet with expected production targets that will contribute significantly to global palm oil production, processing techniques at their level need to be optimised.

Crude palm oil (CPO) contains water, cell debris, fibrous material, non-oily solid and oil as it is released from the press. This mixture undergoes clarification to obtain pure palm oil with little or no moisture. In small-scale palm oil processing units, clarification is done manually after the release of press liquor from the press using locally fabricated equipment. Two clarification methods are popular at this level. In the first case, during pressing, a small amount of water is added to the mashed mixture and the press liquid is left to settle in drums beneath the press at the temperature at which the oil was pressed. In the second case, the oil is boiled in drums using uncontrolled amounts of water and allowed to settle after boiling and then the top oil layer is scooped out (Nchanji *et al.,* 2013). Clarification of palm oil by local farmers is therefore a cause for concern as the process conditions are not mastered and most of the oil is left in the sludge leading to visible but unquantified oil losses. These farmers require well-designed and clearly defined processing conditions to enable them to produce high yield clarified oils of recommended quality to attract high prices for their produce. Palm oil clarification methods reported in the literature include the use of ultrasound assisted clarification (Juliano *et al.,* 2013a; 2013b) and the simultaneous clarification and dehydration of CPO using specially designed membranes (Wenten *et al.,* 2019). These methods are expensive and not easily adaptable to local processors working on small household units. Unfortunately, therefore, there is a dearth of information in the literature on studies that can facilitate the modification of existing local clarification methods or clarifiers and/or the development of new ones entirely. A major step towards achieving this is to carry out kinetic and thermodynamic studies on the clarification process of the different varieties in order to put at the disposal of the stakeholders, reliable information that can be exploited to design and construct clarifiers locally for small-scale processing of palm oil. This study examines the processing parameters that must be mastered in order to reduce the losses encountered by smallholder farmers during CPO clarification. The work lays down a reliable basis for defining parameters that will subsequently be used in the design of small-scale clarifiers for use by smallholder farmers to enhance oil yields and quality. The study examines the kinetics and thermodynamics of palm oil clarification, a critical unit operation in the palm oil process, as a major step to optimising oil clarification at smallholder processing levels.

MATERIAL AND METHODS

Sample Collection

Samples of the two varieties (*dura* and *tenera*) of CPO were bought from local women processors in Mbengwi (6.0126°N, 10.0192°E) Cameroon and transported in polyethylene containers to the Food Science Laboratory of the Biochemistry Department in the University of Dschang and stored in a refrigerator until use. The impurity of the oil was 0.12%. A block diagram for the work carried out is presented in *Figure 1.*

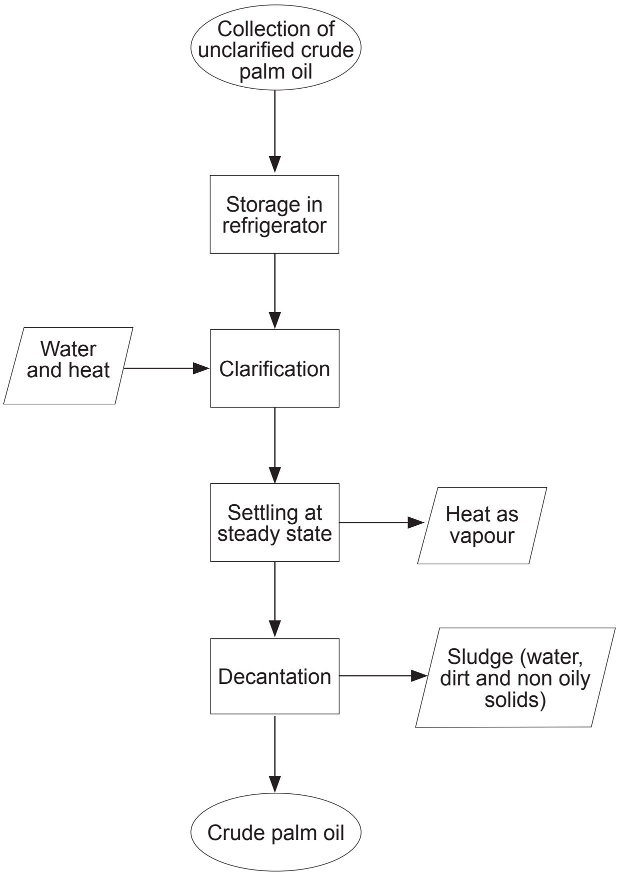

Figure 1. Block diagram for the clarification process.

The Experimental Set Up and Design

Laboratory beakers, 500 mL, were used as reactors for this study. Unclarified crude palm oil was accurately measured and transferred into the beaker. The beaker was covered with a polyethylene material and a hole with a diameter equal to that of the stirrer rod was made through the plastic such that when the stirrer was put in, the system functioned in a closed system. The system was then placed in a hot water bath and the temperature was adjusted to target values. The experimental setup is presented in *Figure 2.*

The effect of three parameters namely temperatures (80°C, 90°C, 100°C), agitation or stirring speeds (60, 95, 115 rpm), and oil/water ratio (1:1, 1:2, 1:3) were investigated at different settling times using a 3 x 3 x 3 experimental design. Oil yield from the clarification process was measured under different experimental conditions. The effect of mole ratios and agitation speed were first evaluated. To evaluate the effect of temperature, the mole ratio with the best yield was selected from the experiments at 80°C and carried out using a rotation speed of 60 rpm. While the effect of rotation speed was investigated at a constant mole ratio and temperature of 80°C.

The Clarification Process

A 500 mL laboratory beaker was used as a batch clarifier. The required quantity of water and CPO following the mole ratio was poured into the clarifier. The required stirrer speed and temperature were set. When the prescribed temperature was reached, the clarification process was allowed to go on for 20 min. The mixture was then allowed to sediment naturally for 0, 5, 10, 20, 30 and 40 min. After clarification, the mixture was separated by decantation and the weight of the oil and that of the sediment were determined using an electronic

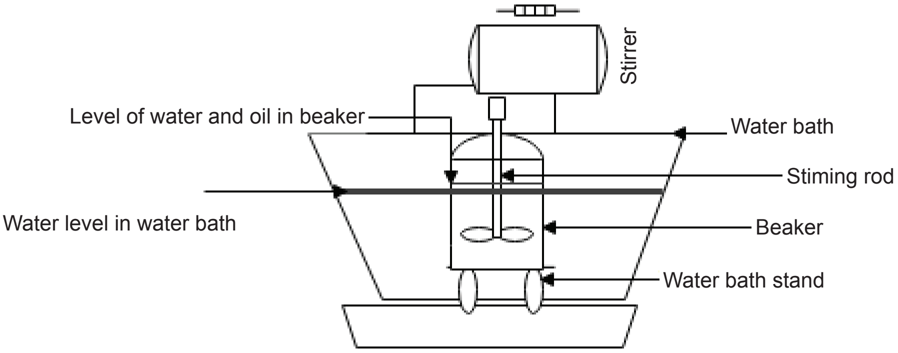

Figure 2. Sketch for clarifier setup.

balance. The percentage oil yield (Y) and quantity (Q) of sediment were calculated using Equations (1) and (2), respectively.

$$Y = \frac{W0}{W} \times 100 \tag{1}$$

$$Q = \frac{W1}{W} \times 100 \tag{2}$$

where, W, W_0 and W_1 are the weights of samples of press liquor or raw CPO, clarified oil (g), and sediment respectively. All experiments were carried out in triplicates.

Kinetics of Oil Clarification

The kinetics of oil clarification were modelled by two equations: A proposed modified Newton and Lewis equation and the Peleg model. For the proposed modified Newton model, it was assumed that once the reaction temperature is attained, palm oil clarification with respect to time occurred at a steady state and that there are no chemical reactions occurring at a steady state. For Newton's law, it was assumed that the rate of oil clarification is governed by the mass transfer of palm oil from sludge (non-oily solids and dirt particles) to the surface of the clarification medium (water). Therefore, following Newton's law, the rate of oil transfer to the surface of the clarification medium for the unsteady state can be written as (Liauw *et al.*, 2008; Westerman *et al.*, 1973):

$$Y = \exp(-kt) \tag{3}$$

A new term [*Y*/*t* which replaces Y in Equation (3)] representing the quantity of oil clarified per unit time was assumed to increase exponentially with sedimentation time and therefore followed an empirical relation given by a modified Newton and Lewis equation of the form [Equation (4)]. K_{nl} replaces -k in Equation (3) to depict the increase in the oil clarification rate with
time.

$$\frac{Y}{t} = e^{tK_{nl}} \tag{4}$$

Linearisation of Equation (4) gives Equation (5):

$$\text{In}\left(\frac{Y}{t}\right) = K_{nl}t \tag{5}$$

Y is the oil clarification yield (%), *t* is sedimentation or clarification time (min), *K* is the constant from the Newton equation and K_{nl} is clarification constant from the modified Newton and Lewis equation; the gradient of the graph of ln(*Y*/*t*) against *t*.

The linearised form of the Peleg model [Equation (6)] (Peleg, 1988) was also tested for the modelling of the clarification kinetics which can be rearranged to give Equation (7).

$$Y - Y_0 = \frac{t}{K_1 + K_p t} \tag{6}$$

$$\frac{t}{Y + Y_0} = K_p t + K_1 \tag{7}$$

Y_0 and *Y* are the initial and oil clarification yields at time *t* respectively. At $t = 0$, $Y_0 = 0$, and Equation (7) reduces to Equation (8).

$$\frac{t}{Y} = K_p t + K_1 \tag{8}$$

The clarification constant, K_p, from the Peleg equation is obtained from the gradient of the graph of *t*/*Y* against *t*. K_1 is the intercept.

Thermodynamics of Oil Clarification

Thermodynamic parameters were determined from Equation (9) and (11) (Liauw *et al.*, 2008).

$$\text{LnK} = \frac{-\Delta G}{RT} = \frac{-\Delta H}{RT} + \frac{-\Delta S}{R} \tag{9}$$

where, *K*, *ΔG*, *ΔH*, *ΔS* and *T* are clarification constant, Gibbs energy (KJ/mol), enthalpy change (KJ/mol), entropy change (KJ/mol.C) and temperature (K) respectively. *R* is the universal gas constant (8.314 KJ/Kmol).

K values determined from the modified Newton model and the Peleg equation did not give good fits for the thermodynamic Equation (9). A new value of K (K_{YQ}) was then defined by Equation (10) (Nwabanne, 2012).

$$K_{YQ} = \frac{Y}{Q} \tag{10}$$

Y is the percentage oil yield at temperature T, and *Q* is the percentage of sludge or sediment. The values of K_{YQ} for each treatment was then determined and used to plot (K_{YQ}) vs 1/T from which ΔH and ΔS were obtained from the slope and intercept respectively. The Gibbs energy (ΔG) was estimated from Equation (11) (Nwabanne, 2012; Santos *et al.*, 2015).

$$\Delta G = -\Delta H - T\Delta S \tag{11}$$

Statistical Analysis

The One-way ANOVA was used on JMP Pro 16 to detect differences between means and the DUNCAN multiple range test was used to separate the means.

RESULTS AND DISCUSSION

Effect of Process Parameters on the Clarification Yields

Effect of temperature on the yield of clarified palm oil. *Figure 1* present the temperature effect on the clarification yield of the *dura* variety of palm oil. For all three agitation speeds studied, no significant difference ($p<0.05$) was observed in the clarification yields because of the change in temperature. Oil yields for the *dura* variety ranged from 0.00% to 68.13%, 0.00% to 71.89% and 0.00% to 72.62% at 80°C, 90°C and 100°C respectively which were not significantly different ($p<0.05$). Unlike the *dura* variety, temperature had a profound effect on the clarification yields of the *tenera* variety *(Figure 2).* For the *tenera* variety, the oil yield decreased as the temperature increased from 80°C to 100°C when the clarification was carried out at an agitation speed of 60 rpm. Similar observations were made at higher stirring speeds of 90 rpm and 115 rpm where oil clarification yields were consistently lower at 90°C compared to 80°C. The ranges of oil yields were from 0.00% to 92.81%, 0.00% to 89.32% and 0.00% to 78.44% at 80°C, 90°C and 100°C, respectively at constant agitation speed of 60 rpm; the highest oil yield was obtained at 80°C, 60 rpm. The reduction in clarification yield with an increase in temperature is likely because the solubility of the oil in water increases as temperature increases. At high temperatures, more of the oil then dissolves in water which is removed as part of the sludge thereby leading to the observed decrease in oil clarification yields. These results suggest that for both varieties, clarification should be carried out at 80°C using a stirring speed of 60 rpm.

Effect of mole ratio on the yield of clarified palm oil. The effect of mole ratio on the clarification yields for the *dura* and *tenera* varieties at 80°C are presented in *Figure 3* and *4.* The oil yield for the dura variety decreased as the mole ratio increased from 1:1 to 1:3. The highest oil yield was obtained at 1:1 mole ratio. For the *dura* variety and at a lower stirring speed of 60 rpm, oil clarification yield decreased with an increase in CPO to water ratio from 1:1 to 1:3, which was contrary to our expectation. At higher rotation speeds of 95 and 115 rpm there was no significant difference in the oil clarification yields of the *dura* variety with respect to mole ratio (*Figure 3*). Therefore, for the *dura* variety, the optimum oil to water ratio which gives high oil yield after clarification is 1:1. For the *tenera* variety, significant differences ($p<0.05$) were observed between the three different mole ratios investigated in this study. Oil clarification yield increased with an increase in CPO/water ratio as expected when

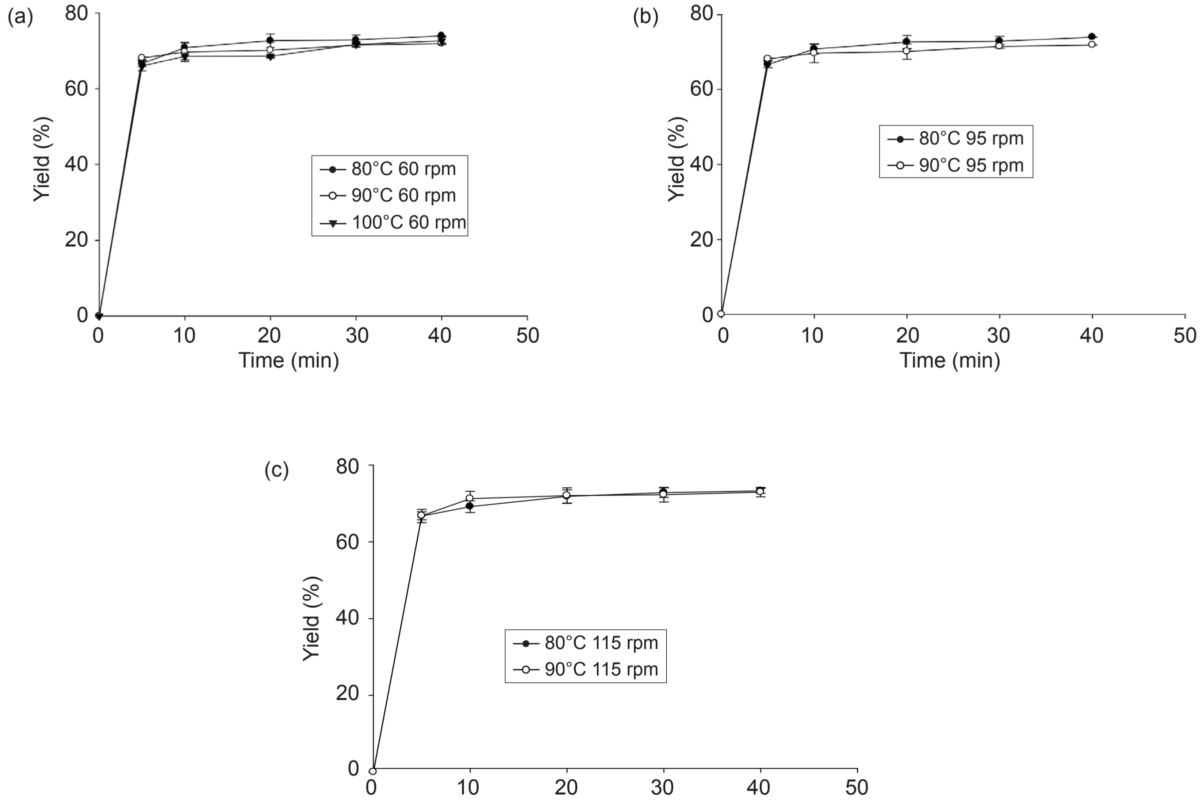

Figure 1. Effect of temperature on the clarification of palm oil dura *variety at (a) 60 rpm, (b) 95 rpm and (c) 115 rpm.*

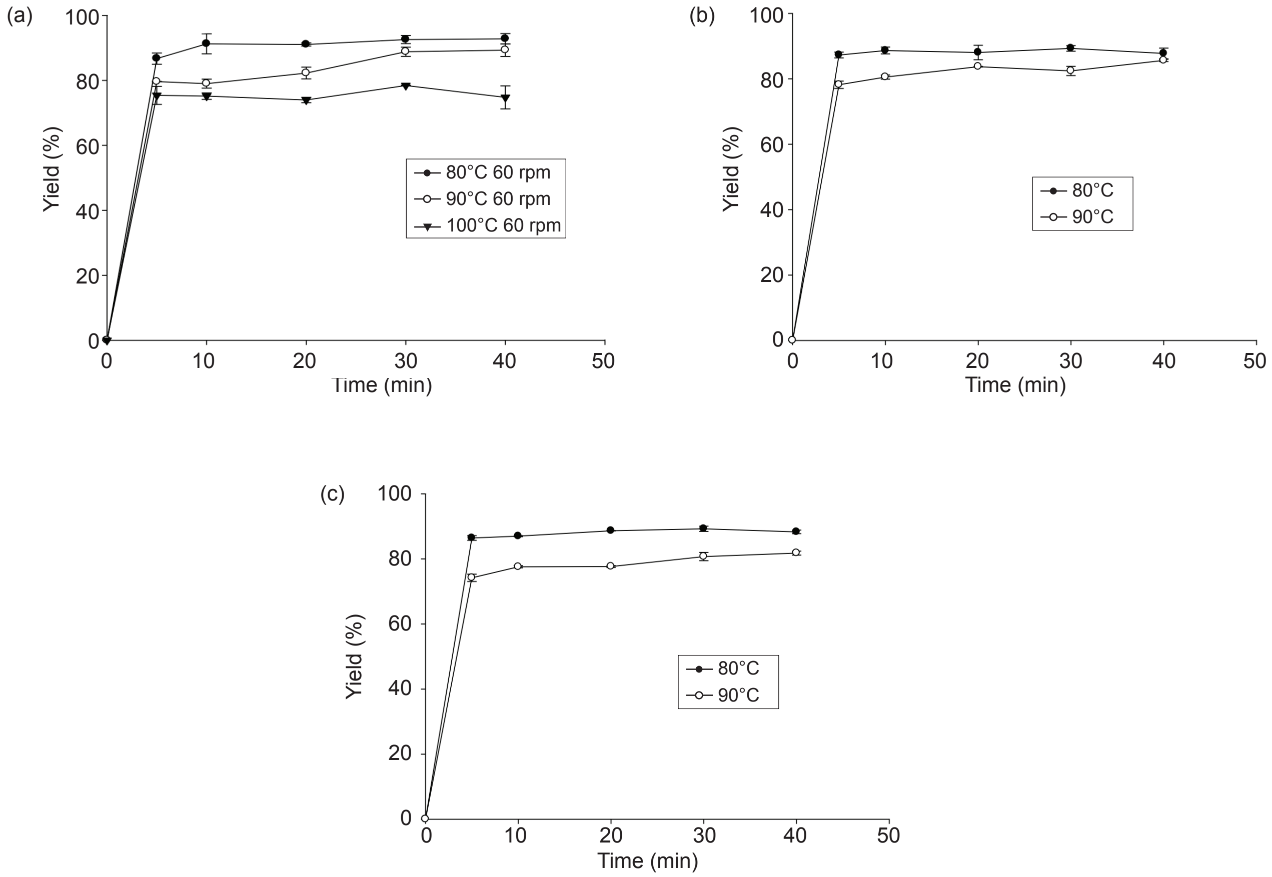

Figure 2. Effect of temperature on the clarification of palm oil tenera *variety at (a) 60 rpm, (b) 95 rpm and (c) 115 rpm.*

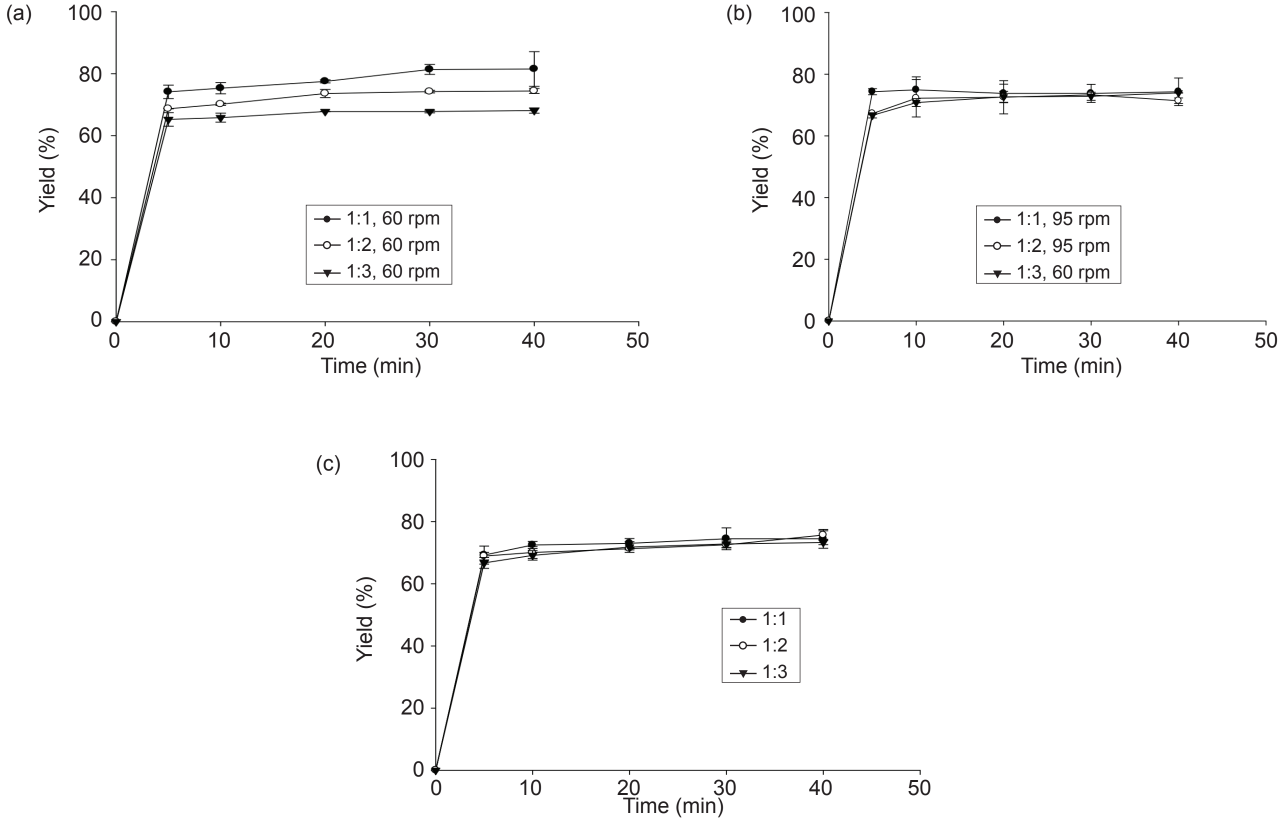

Figure 3. Effect of mole ratio on palm oil clarification dura *variety, at (a) 60 rpm and 80°C, (b) e 95 rpm and 80°C and (c) 115 rpm and 80°C.*

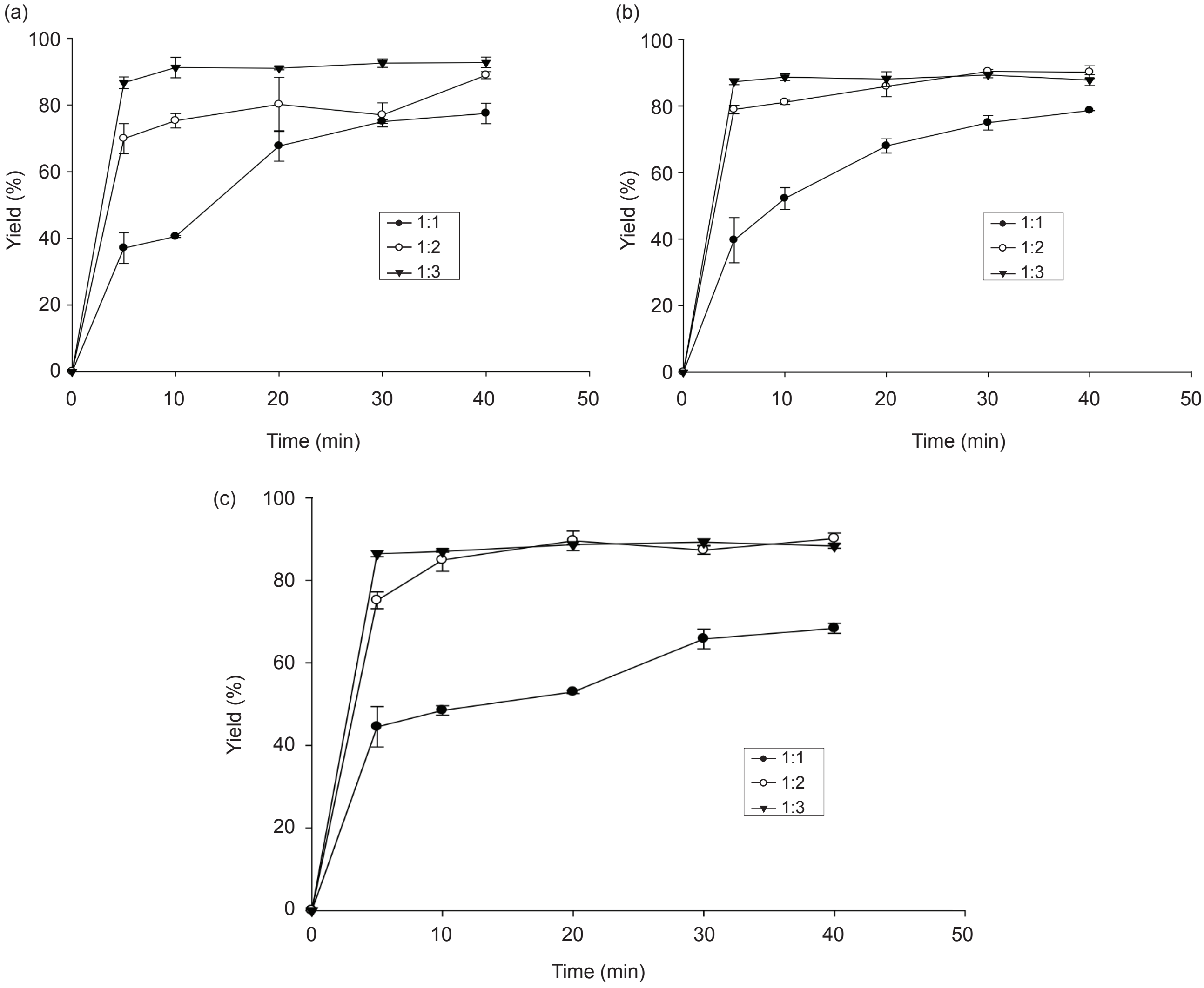

Figure 4. Effect of mole ratio on palm oil clarification tenera *variety, at (a) 60 rpm and 80°C, (b) 95 rpm and 80°C and (c) 115 rpm and 80°C.*

the temperature was kept constant. At constant temperature, more impurities are expected to dissolve in an increased quantity of water to form a sludge thereby facilitating the separation of sludge from the oil and hence the high oil clarification yields. At 95 and 115 rpm there was no significant difference ($p < 0.05$) between mole ratio 1:2 and 1:3. Therefore optimum mole ratio for the clarification of oil from the *tenera* variety was retained as 1:2.

Effect of stirring speed on the yield of clarified palm oil. The effects of stirring speed on the clarification of palm oil from the *dura* and *tenera* varieties are shown on *Figure 5* and *6*, respectively. Oil clarification yield decreases as speed increases from 60 to 115 rpm at mole ratio 1:1 and increases as stirring speed increases from 60 to 115 rpm at mole ratio 1:2 and 1:3 respectively for both varieties. One of the advantages of agitation is to cause the dirt particles to form aggregates which become heavier and then sediment to ease separation. The slight reduction in oil yield with increase in stirring speed was attributable to the fact that at low mole ratio (1:1), high agitation did not allow sufficient contact time for the particles to aggregate and sediment hence the observed decrease in the clarified oil yield. Adopted stirring speed for both varieties was 95 rpm.

Effect of variety on the yield of clarified palm oil. The effect of variety on the clarification of palm oil is shown in *Figure 7* and *8*. It can be observed that except for the treatments at mole ratio 1:1, temperature 80°C each at 60, 95 and 115 rpm where clarification yields for *dura* were significantly higher ($p < 0.05$) compared to *tenera* (*Figure 7*), all other treatments gave oil yields for tenera which were significantly higher than that of *dura*. The observed significant differences obtained for the two varieties can be linked to the oil composition, method of oil extraction, quantity of debris or dirt contained in the CPO.

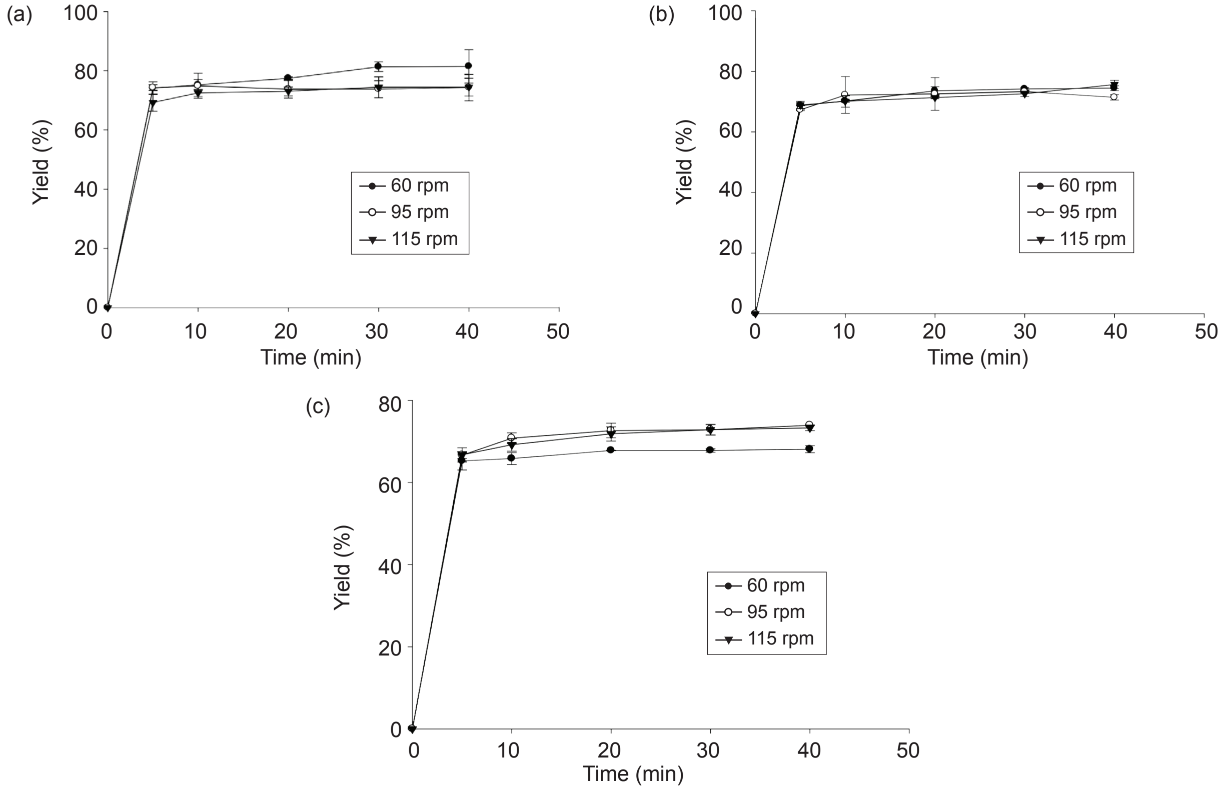

Figure 5. Effect of stirring speed on palm oil yield at temperature 80°C and mole ratio (a) 1:1, (b) 1:2 and (c) 1:3 for dura *variety.*

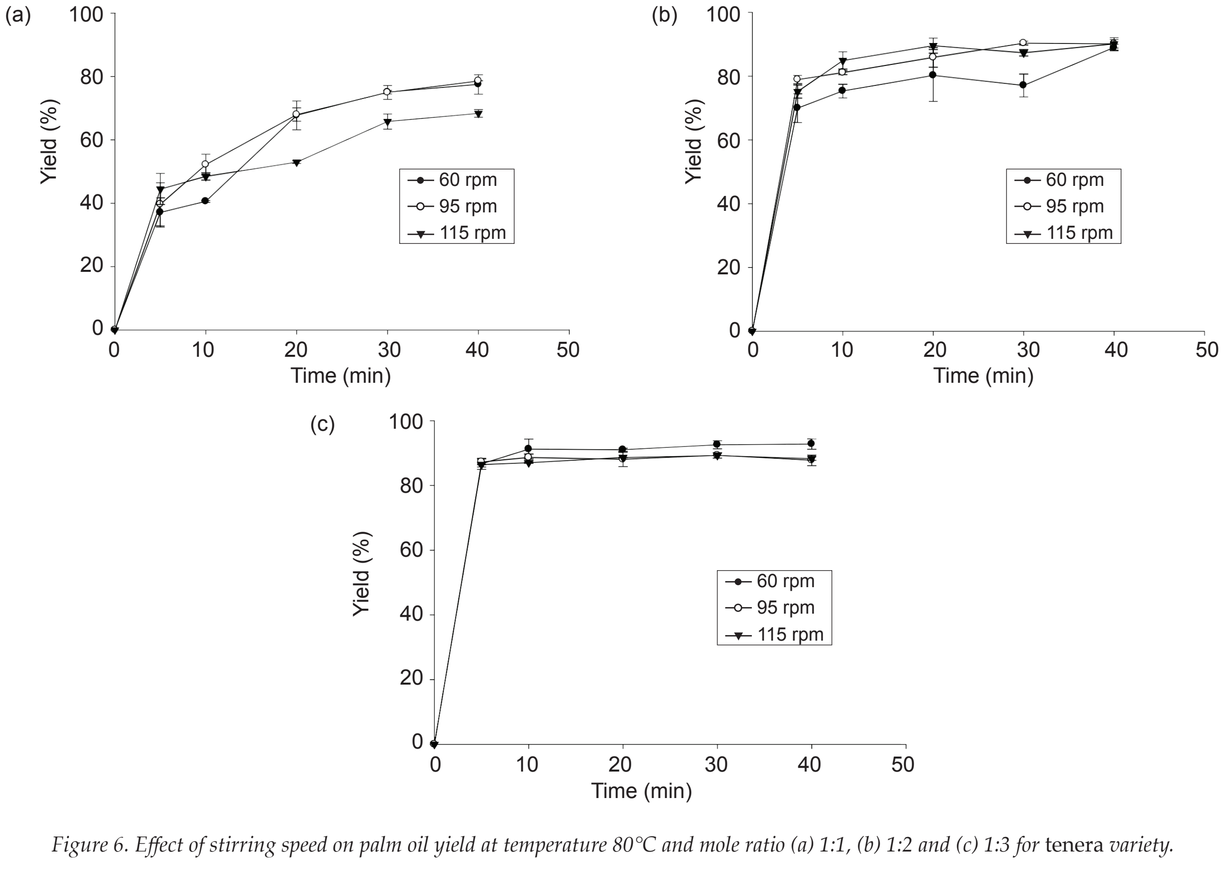

Figure 6. Effect of stirring speed on palm oil yield at temperature 80°C and mole ratio (a) 1:1, (b) 1:2 and (c) 1:3 for tenera *variety.*

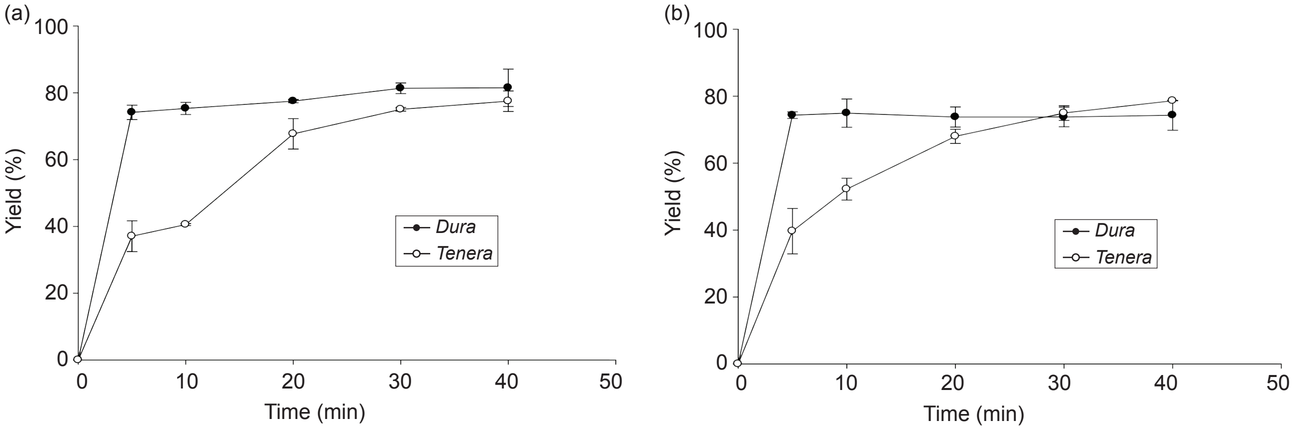

Figure 7. Effect of variety on oil yield at 80°C, 1:1 mole ratio, (a) 60 rpm and (b) 95 rpm.

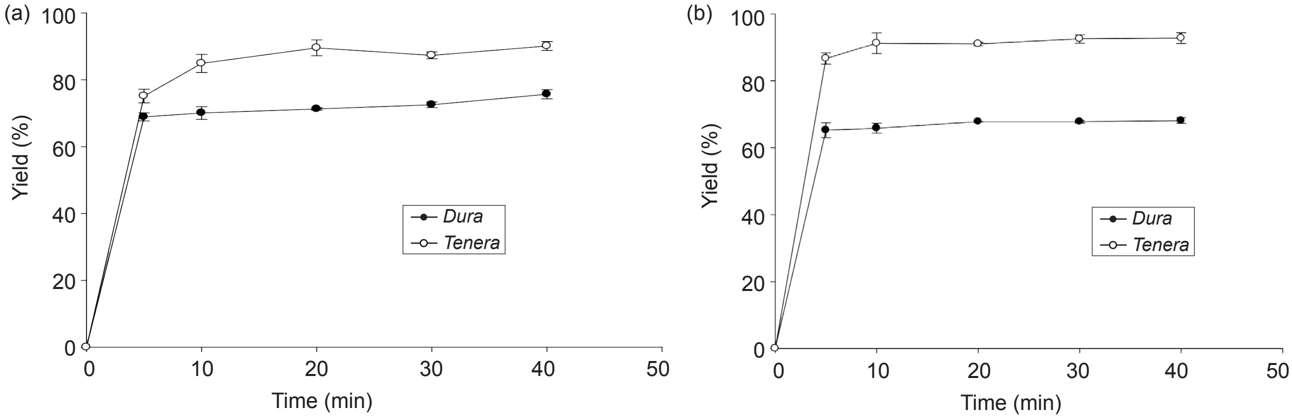

Figure 8. Effect of variety on oil yield at (a) 80°C, 1:2 mole ratio, 95 rpm, 115 rpm and (b) 80°C, 1:3 mole ratio, 60 rpm.

Palm oil separation generally adopts a vertical or horizontal settling tank. Depending on the preferred method, parameters (variables) to be considered include fluid flow rate, feed composition, settling volume, retention time, and temperature (Abikoye, 2014). Nurulhuda (2009) reported optimum oil clarification efficiency of palm oil at 95°C. However, crude palm oil obtained from the two varieties of oil palm treated was not taken into consideration. In this study, it was found that temperature significantly affected the clarification yields of CPO from two different varieties of oil palm. As temperature increases, oil yield in the *tenera* variety decreases while in the *dura* variety, the situation was the reverse even though no significant difference was observed at 90°C and 100°C. Nurulhuda (2009) and Abikoye (2014) found out that the feed flow rate and the settling volume are the major controlling factors in the separation of oil from sludge in a continuous system. A batch system was used in this work. John *et al.* (2019) studied the conventional method of clarifying CPO where the temperature in the clarification tank was set at 70°C. Most industrial palm oil processing units deal with the *tenera* variety of CPO commonly called the commercial variety. Kandiah and Batumalai (2013) used evaporation method to replace the centrifugation stage in the conventional clarification of palm oil. In this method, water addition to facilitate oil-sludge separation was avoided. Sulung and Tan (1996; 1999) used the membrane filter press to clarify palm oil. In this method, the diluted crude slurry produced by mechanical pressing of the cooked fruitlets is settled in a vertical or horizontal clarifier.

These studies were aimed at standardising the traditional clarification of palm oil which is very costly and unaffordable for local farmers. It was found out that at a temperature of 80°C, mole ratio of 1:2 and stirring speed of 95 rpm, maximum oil yield will be obtained for the two varieties.

Kinetic Parameters

Model constants for the modified Newton/Lewis and Peleg models are presented on *Table 1* and 2, respectively. All R^2 values for both models were close to 1; therefore the two equations can be used to describe the clarification process of palm oil for both varieties. These results are in accordance with that of Bup *et al.* (2012), who used Peleg's model to evaluate the water adsorption kinetics of cooked and uncooked shea nut kernels (*Vitellaria paradoxa* Gaertn).

TABLE 1. VALUES OF K_{NL} AND R^2 OBTAINED FROM THE PLOT OF THE MODIFIED NEWTON AND LEWIS EQUATION

Temperature (°C)	Mole ratio (g)	Speed (rpm)	*Dura*		*Tenera*	
			K_{nl}	R^2	K_{nl}	R^2
80	1:1	60	4.218	0.999	2.908	0.967
80	1:1	95	4.317	1.000	3.161	0.996
80	1:1	115	4.193	0.999	3.415	0.99
80	1:2	60	4.161	0.999	4.100	0.9969
80	1:2	95	4.184	0.999	4.246	0.999
80	1:2	115	4.162	0.999	4.224	0.998
80	1:3	60	4.141	1.000	4.427	0.999
80	1:3	95	4.138	0.998	4.468	0.999
80	1:3	115	4.129	1.000	4.437	0.999
90	1:3	60	4.101	0.999	4.265	0.998
90	1:3	95	4.183	1.000	4.299	0.999
90	1:3	115	4.156	0.999	4.239	0.999
100	1:3	60	4.120	0.999	4.311	0.999

TABLE 2. VALUES OF R^2 AND K_P OBTAINED FROM THE PELEG EQUATION FOR *Dura* AND *Tenera*

Temperature (°C)	Mole ratio (g)	Speed (rpm)	*Dura*		*Tenera*	
			K_p	R^2	K_p	R^2
80	1:1	60	0.011	0.999	0.108	0.972
80	1:1	95	0.001	0.999	0.078	0.999
80	1:1	115	0.006	0.999	0.071	0.981
80	1:2	60	0.008	0.999	0.023	0.986
80	1:2	95	0.001	0.999	0.013	0.999
80	1:2	115	0.001	0.99	0.010	0.999
80	1:3	60	0.004	1.000	0.004	0.999
80	1:3	95	0.008	0.9999	0.000	0.999
80	1:3	115	0.009	1.000	0.001	0.999
90	1:3	60	0.010	0.999	0.015	0.998
90	1:3	95	0.006	0.999	0.008	0.999
90	1:3	115	0.006	1.000	0.010	0.999
100	1:3	60	0.012	0.999	0.001	0.998

Thermodynamics Parameters

Entropy (ΔS) values were -9.333KJ/mol and -59.166KJ/mol for *dura* and *tenera* varieties respectively while the corresponding enthalpy change (ΔH) values were -1809.87 KJ/mol and -17182.54 KJ/mol. The negative values of enthalpy change indicate that the process is exothermic; energy is lost to the surrounding during the process, thus oil would clarify best at lower temperatures than at higher temperatures. This corroborates with the highest oil yields which were obtained at 80°C, compared to 90°C and 100°C. The Gibbs free energy at different temperatures is calculated using Equation (11). Gibbs free energy (ΔG) were respectively 1484.67, 1578.00, 1671.3 KJ/mol and 3702.95, 4294.61, 4886.26 KJ/mol for *dura* and *tenera* at 80°C, 90°C and 100°C. ΔG is positive indicating that this process is exothermic, irreversible and not spontaneous.

CONCLUSION

This study has shown that the traditional clarification process of palm oil can be controlled. Temperature, oil/water ratio and stirring speed significantly affect oil yield. The effect of these parameters varies with CPO from the different oil palm varieties. Best conditions to obtain high yield of palm oil (92%) were found to be temperature of 80°C, 1:2 mole ratio, settling time of 30 min and 95 rpm stirring speed for both varieties. Palm oil clarification followed the modified Newton's and Lewis Equation and the Peleg's Model. It was also found that ΔH and ΔS are negative for both varieties, and ΔG is positive indicating that this process is exothermic, irreversible and not spontaneous.

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