PAPER 33 – EFFECTS OF PROCESS PARAMETERS ON OIL AND BIODIESEL YIELD FROM SANDBOX (*HURA CREPITANS*) SEED

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ABSTRACT

Multiple applications of vegetable oil for food, industrial applications and biodiesel production have necessitated the need to boost product yield. Process factors that influence oil and biodiesel yield were investigated. Factors studied for oil extraction were, moisture content (%wb), roasting temperature (°C), roasting time (min), extraction temperature (°C) and time (min). Reaction time (min), reaction temperature (°C), catalyst concentration (%wt) and molar ratio were investigated for biodiesel yield. Central composite rotatable design of response surface methodology experimental design was used for the study. Results obtained were analysed at ($p \le 0.05$). The oil yield from the sandbox seed ranged from 20.9-53.6%. The maximum oil yield of 53.6% was obtained at the processing conditions of 10.0%wb moisture content, 95 °C roasting temperature, 15 min roasting time, 65 °C extraction temperature and 90 min extraction time. The biodiesel yield ranged from 80.00-96.12% and, maximum biodiesel yield of 96.12% was extracted at 72.0 min reaction time, 50.0 °C reaction temperature, 0.9%wt catalyst concentration and 1:6 molar ratio. Mathematical models to predict sandbox seed oil and biodiesel yields at varying process conditions were developed. Coefficient of determination (R^2) relating the oil extraction and biodiesel production were 0.81 and 0.74, respectively. The results from the study provide data for equipment and process designs for oil extraction and biodiesel production from sandbox and other oilseeds.

KEYWORDS: Hura crepitans, Oil extraction, oil yield, biodiesel production, biodiesel yield.

1. INTRODUCTION

Sandbox (Hura crepitans Linn.) is a dicotyledon tree of the (spurge) family Euphorbiaceae. It is a native tree of the tropical zones of the Amazon rainforest of North and South America (Adewuyi et al., 2014). Sandbox seed has been suggested to contain a high oil content in addition to a number of important properties that can be useful for food and feed production, paints, and cosmetics amongst others industrial application and pharmaceutical potentials (Olatidoye et al., 2010; Onwe and Bamgboye, 2020; Basumatary, 2013). It is however grouped under underutilized species of plant (Adewuyi et al., 2014). According to Idowu et al. (2012), sandbox is used as shade trees in many parts of the world because their wide spreading limb. In corroboration, Adewuyi et al. (2012) noted that sandbox is an underutilized plant in Nigeria and the seeds are unused. Solvent extraction has made it more profitable to recover most oil from seeds, nuts, and other materials with relatively low oil content, as up to 98% of oil can be recovered using solvent extraction (Matthäus, 2012). Oil is extracted from oil-bearing matters using crude liquids like acetone, ether, chloroform, petroleum, spirit, carbon tetrachloride, diethyl ether, and carbon tetra chloride. Various oilseeds have been studied to determine the best extraction conditions for the highest oil yield: sesame seed (Hashim et al., 2014; Elkhaleefa and Shigidi, 2015); rubber seed (Awais et al., 2015); soybean seed (Lawson et al., 2010); dried Bitter Gourd seed (Umamaheshwari and Dinesh Sankar Reddy, 2016); canola seed (Blake, 1982); un-sieved rice bran and sieved rice bran respectively (Sivala et al., 1991); sunflower kernels (Southwell and Harris, 1992) and grated coconut (Hammonds et al., 1991); Neem (Azadirachta indica) seed (Awolu et al., 2013); Sorrel (Hibiscus sabdariffa) seed (Betiku and Adepoju, 2013); almond seed (Akubude et al., 2017); Dika nut (Abidakun et al., 2012); groundnut (Olajide et al., 2014); Shea kernel (Olaniyan and Oje, 2007); African star apple seed (Ajala and Adeleke, 2014); Moringa seed (Adejumo et al., 2013). The global concern of the rapid depletion of the petroleum and its potential extinction, coupled to environmental pollution emissions from its combustion exhaust, has made biodiesel from organic lipids an available substitute to petroleum diesel (Reyes and Spulveda, 2006; Goering et al., 1982). Organic lipids like vegetable oils, animal fats, waste oils, and recycled lubricants are combined with alcohol, primarily methanol or ethanol, in a displacement reaction known as transesterification to create fatty esters like methyl/ethyl ester (biodiesel). Factors that influence methyl ester yield from lipids have been studied (Zhang et al., 2003; Ma and Hanna, 1999; Meher et al., 2006; Van, 2005 and Schuchardta et al., 1998). Oil extraction by solvent extraction method from sandbox seed have been reported (Okolie et al., 2012; Muhammed et al., 2013; Adewuyi et al., 2014; Shonekan and Ajayi, 2015; Nwanorh, 2015; Ottih et al., 2015). Biodiesel was produced from sandbox seed oil (Adewuvi et al., 2012; Adepoiu et al., 2013; Igbum et al., 2012). The present work is focused on investigating the effects of process parameters on sandbox seed oil and biodiesel yield respectively.

2. MATERIALS AND METHODS

About 100 kg of mature sandbox fruits were collected from Uyo metropolis, Akwa Ibom State, Nigeria between (2016-2018). The fruits were cracked to remove the seeds and the seeds peeled to get the kernel (mesocarp) (Figure 2.1).



Figure 2.1 Sandbox seed processing

2.1. Solvent oil extraction from sandbox seed

Oil extraction was carried out according to the American Oil Chemists' Society (AOCS 5-04) standard procedure. A six-in-one Soxhlet apparatus was used for the extraction process. The experiments were carried out at varying processing conditions: moisture content, mc (4, 6, 8, 10 and 12% wet-basis), roasting temperature, v_{tp} (80, 85, 90, 95 and 100°C) and time, v_{tm} (0, 5, 10, 15 and 20 min), extraction temperature, ε_{tp} (60, 65, 70, 75 and 80°C) and time, ε_{tm} (60, 90, 120, 150 and 180 min). The sandbox seed oil (Figure 2.2) was weighed and taken as a fraction of the ground seed samples. The oil yield was calculated from Equation 2.1 as adopted by Bello and Daniel (2015) for determination of oil yield percentage.

$$Oil\ Yield\ (\%) = \frac{Wieght\ of\ oil\ extracted}{Wieght\ of\ sandbox\ seed\ sample\ used\ in\ the\ extraction} \times 100 \quad (2.1)$$



Figure 2.2 Biodiesel production

2.2. Biodiesel production from sandbox seed oil

The effects of process variables: reaction time (24, 48, 72, 96 and 120 min), reaction temperature (40, 45, 50, 55 and 60 °C), catalyst concentration (0.3, 0.6, 0.9, 1.2 and 1.5 %wt) and molar ratio (1:2, 1:4, 1:6, 1:8 and 1:10 methanol: oil ratio) on the methyl ester yield were investigated. Two hundred millilitres (200 ml) of sandbox seed oil, amounting to 184 g in weight was used in each of the experiment. Other parameters were calculated as percentage weight or volume of the oil. Methanol was used as the alcohol in the transesterification reaction, and potassium hydroxide was used as the catalyst for the reaction. The Central Composite Rotatable Design (CCRD) of Response Surface Methodology was used for both experiments. Experiments were replicated thrice.

3. RESULTS AND DISCUSSION

The results of the sandbox oil and biodiesel yields at various process conditions are presented in Tables 3.1 and 3.2 respectively. Charts showing the effects of process parameters on oil and biodiesel yield are shown in Figures 3.1-3.4.

Table 3.1 On Tield from Bandoon Beed at Various Treessing Conditions							
	Factor 1	Factor 2	Factor 3	Factor 4	Factor 5	Response 1	
Run	A: mc	B: γ_{tp}	C: γ_{tm}	D: ε_{tp}	E: ε_{tm}	Oil yield	
	(%)	(%)	(min)	(°C)	(h)	(%)	
1	8	90	10	70	2.0	45.76	
2	8	90	10	70	3.0	35.02	

Table 3.1 Oil Yield from Sandbox Seed at Various Processing Conditions

3	8	90	10	70	1.0	23.00	
4	6	85	5	65	2.5	38.33	
5	8	90	10	60	2.0	42.87	
6	10	85	15	75	1.5	40.00	
7	10	85	5	75	2.5	40.12	
8	4	90	10	70	2.0	20.86	
9	10	85	15	65	2.5	48.88	
10	8	90	10	80	2.0	44.00	
11	12	90	10	70	2.0	30.00	
12	8	90	10	70	2.0	45.00	
13	6	95	15	65	2.5	40.00	
14	8	90	10	70	2.0	50.00	
15	8	90	10	70	2.0	49.80	
16	10	95	15	75	2.5	46.54	
17	6	85	15	65	1.5	35.66	
18	10	95	5	65	2.5	36.00	
19	8	80	10	70	2.0	38.10	
20	8	90	10	70	2.0	47.77	
21	10	85	5	65	1.5	36.56	
22	6	95	15	75	1.5	39.00	
23	10	95	5	75	1.5	42.40	
24	6	85	5	75	1.5	39.60	
25	6	85	15	75	2.5	40.55	
26	6	95	5	65	1.5	40.06	
27	6	95	5	75	2.5	38.99	
28	8	90	10	70	2.0	46.44	
29	8	90	0	70	2.0	21.00	
30	10	95	15	65	1.5	53.60	
31	8	90	20	70	2.0	47.99	
32	8	100	10	70	2.0	51.33	

Where mc = moisture content of sandbox seed, $y_{tp} = Roasting$ temperature, $y_{tm} = Roasting$ time, $\varepsilon_{tp} = Extraction$ temperature and $\varepsilon_{tm} = Extraction$ time

The oil yield from the sandbox seed ranged from 20.86-53.60%. The highest oil yield of 53.60% obtained was similar to 53.61 and 53.81% obtained by Okolie *et al.*, (2012) and Muhammed *et al.* (2013), and higher than 37.75, 36.70 and 42.70% obtained by Adewuyi *et al.* (2014), Shonekan and Ajayi, (2015) and Nwanorh, (2015). The value was however lower than the 57.26% obtained by Ottih *et al.* (2015). According to Anwar *et al.* (2006); Orhevba *et al.* (2013), differences in oil yield during extraction are a function of extraction methods employed, and also biological and environmental conditions.

Increase in oil yield was observed as the moisture content increased from 4-10%wb, after which the yield declined (Fig. 3.1). The highest oil yield was obtained at 10% seed moisture; the decline in oil yield at moisture contents above 10%wb could be attributed to the observation that the ground sandbox seed forms a slurry paste at moisture contents above 10%, making further oil removal difficult. The trend is in agreement with reports on soybeans, and un-sieved and sieved rice bran (Lawson *et al.*, 2010; Sivala *et al.*, 1991) among others.

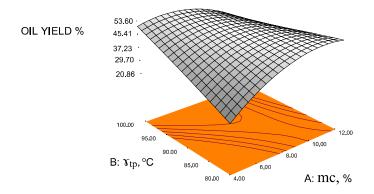


Figure 3.1. Oil yield against roasting time and moisture content

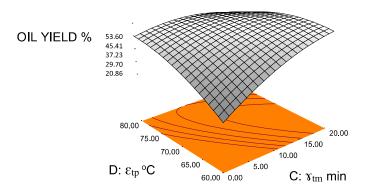


Figure 3.2. Extraction time and roasting temperature against oil yield

The sandbox oil yield increased as roasting temperature increased. The highest oil yield occurred at the roasting temperature region of 95-100°C (Figure 3.1). Due to the sandbox seed's softness, a seed similar to melon seed, roasting temperature around 100 °C was sufficient to penetrate the seed and release its oil. Similar roasting temperature of 100°C was obtained for maximum oil yield from Moringa oleifera (Adejumo et al. (2013). The unroasted samples at 0 min roasting time produced the lowest oil yield from the sandbox, and then the oil yield increased with an increase in roasting time up to 15 min beyond which the yield became constant (Figure 3.2). The sandbox oil yield increased with an increase in extraction temperature up to 75 °C, after which the oil yield decreased (Figure 3.2). Extraction temperature range of 60-90 °C has been reported to be suitable for maximum oil yield from seeds and nuts by solvent extraction. Solvent extraction temperature of 60 °C was reported by Asoiro and Akubuo (2012) as suitable for maximum oil extraction from Jatropha curcas L. Umamaheshwari and Dinesh Sankar Reddy (2016) obtaining maximum oil yield from bitter gourd at 98 °C extraction temperature and Elkhaleefa and Shigidi (2015) reported a lower extraction temperature of 40 °C for maximum oil yield from sesame seed. The sandbox oil yield increased with increase in extraction time up to 120 min, beyond which a decrease in oil yield was observed. It was observed that 2 hours reaction time was enough for complete oil removal from sandbox seed by solvent extraction. Lawson et al. (2010) obtained maximum oil yield from soybeans at an extraction time of 2½ hours.

Table 3.2. Biodiesel Yield from the Transesterification of Solvent Extracted Sandbox Seed Oil

S/N	Factor 1 A:Ftm (min)	Factor 2 B:ftp (°C)	Factor 3 C:ċcc (% of oil wt)	Factor 4 D:m _{ra} (% vol. of oil)	Response Fuel yield (%)
1	48	45	0.60	8	86.20
2	96	45	0.60	4	86.12
3	48	55	0.60	4	88.65
4	48	55	1.20	4	81.02
5	48	45	1.20	8	83.79
6	48	45	0.60	4	86.25
7	96	45	0.60	8	87.16
8	72	50	0.90	6	94.55
9	72	50	0.90	6	94.89
10	72	50	0.30	6	89.48
11	72	50	0.90	2	80.11
12	72	50	0.90	6	91.99
13	96	55	0.60	4	88.01

14	96	55	1.20	4	82.36
15	96	45	1.20	4	83.88
16	72	50	0.90	10	87.98
17	72	40	0.90	6	88.42
18	120	50	0.90	6	92.68
19	48	55	0.60	8	91.23
20	72	60	0.90	6	96.12
21	48	55	1.20	8	83.33
22	24	50	0.90	6	81.65
23	72	50	0.90	6	86.99
24	96	55	1.20	8	81.22
25	72	50	0.90	6	87.98
26	96	45	1.20	8	82.63
27	48	45	1.20	4	80.11
28	72	50	0.90	6	92.65
29	96	55	0.60	8	85.00
30	72	50	1.50	6	80.00

Where \mathbf{r}_{tm} = reaction time, \mathbf{r}_{tp} = reaction temperature, $\dot{\mathbf{c}}_{cc}$ = catalyst concentration (KOH) and η_{ra} = molar ratio

The biodiesel yield ranged from 80.00-96.12%. The maximum biodiesel of 96.12% was extracted at 72.0 min reaction time, 50.0 °C reaction temperature, 0.9%wt catalyst concentration and 1:6 molar ratio.

The biodiesel yield increased with increase in reaction time from 24 min to about 90 min before a decline in biodiesel yield was observed (Figure 3.3). This was an indication that transesterification reaction for sandbox seed oil requires about 1.5 hour to be completed. Similarly, Math *et al.* (2010) reported 90 min reaction time as optimum for maximum biodiesel yield from lipids with FFA. A lower reaction time of 40 min was obtained by Nakpong and Wootthikanokkhan (2010) for maximum biodiesel yield from crude *Jatropha curcas* oil. Reports have shown that transesterification process is spontaneous at the beginning of the reaction, converting approximately 80% of the reactants into ester fuels in the first 5 min and gaining approximately 93-98% conversion after 1 h of reaction period (Encinar *et al.*, 2005; Srivastava and Prasad, 2000; Alcantra, 2000; Shailendra *et al.*, 2008).

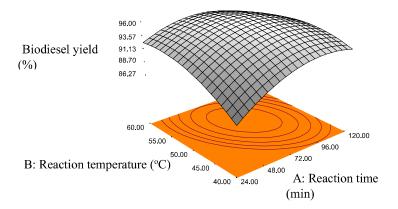


Figure 3.3. Reaction temperature and reaction time against fuel yield

The sandbox biodiesel yield increased with increase in reaction temperature from 40 °C to 50 °C after which the yield became steady (Figure 3.3). This indicates that 50-60 °C reaction temperature range is adequate for the

transesterification of sandbox seed oil. The reaction temperature was same with 60 °C obtained by Adepoju *et al.* (2013) for sandbox seed oil biodiesel and lower than 70 °C obtained by Adewuyi *et al.* (2014) for maximum biodiesel yield from transesterification of (H₂SO₄) acid pre-treated sandbox seed oil and but the. This difference in reaction temperature might be due to reaction process used. Similar reaction temperatures were reported by (Alhassan *et al.*, 2014 for *Gossypium Arboreum* seed oil; Anguebes-Franseschi *et al.*, 2016 for crude palm oil of African origin; Nakpong and Wootthikanokkhan, 2010 for *Jatropha* seed; Singh *et al.*, 2006 for canola oil; Mansourpoor and Shariati, 2012 for sunflower oil).

The biodiesel yield increased as the catalyst concentration increased from 0.3% to 0.9% after which the yield dropped sharply. The value 0.9% g wt/wt of oil represents 0.5% weight of volume of oil used in the reaction (Figure 3.4). The result is average to 0.4% and 0.55% obtained by Adewuyi et al., 2014 and Adepoju et al., 2013 for sandbox seed. Similar result was obtained by Alhassan et al., 2014 for Gossypium Arboreum seed oil, Anguebes-Franseschi et al., 2016 African palm oil and Mansourpoor and Shariati, 2012 for sunflower seed.

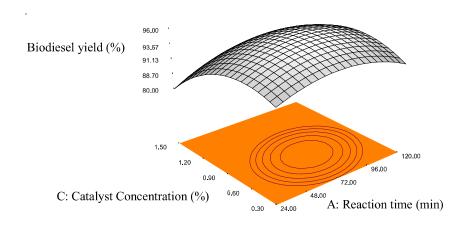


Figure 3.4. Catalyst concentration and reaction time against fuel yield

The sandbox biodiesel increased with increase in molar ratio from 1:2 up to 1:6 after which a decrease in yield was observed (Figure 3.4). Adewuyi *et al.* (2014) and Adepoju *et al.* (2013), obtained ratios of 1:6 and 1:5 respectively for maximum biodiesel yield from sandbox seed oil. Similar molar ratio was reported by Anastopoulos *et al.*, 2009; Alhassan *et al.*, 2014; and Nakpong and Wootthikanokkhan, 2010 as optimum for maximum biodiesel yield from four different vegetable oils, *Gossypium Arboreum* seed oil and *Jatropha curcas* seed oil respectively. The optimum molar ratio of 1:6 or less for other edible oils has been found to give the highest fuel yield (Encinar *et al.*, 2005; Vicente *et al.*, 2004; Dorodo, 2002).

a. Processes model equations

Mathematical relationship for predicting oil yield from sandbox relatively to the process factors is given in Equation 3.1. The coefficient of determination (R^2) relating the oil extraction process and oil yield was 0.81.

$$\bullet \quad \text{OY} = 45.89 + 2.09\text{mc} + 1.81\text{x}_{tp} + 3.59\text{x}_{tm} + 0.013\epsilon_{tp} + 1.10\epsilon_{tm} - 1.91\text{mc}^2 - 0.88\text{x}_{tp}^2 - 1.67\text{x}_{tm}^2 + 0.56\epsilon_{tp}^2 \\ - 3.05\epsilon_{tm}^2 + 0.57\text{mc}\text{x}_{tp} + 2.23\text{mc}\text{x}_{tm} - 0.63\text{mc}\epsilon_{tp} - 0.29\text{mc}\epsilon_{tm} + 0.70\text{x}_{tp}\text{x}_{tm} - 0.23\text{x}_{tp}\epsilon_{tp} - 1.85\text{x}_{tp}\epsilon_{tm} - 1.39\text{x}_{tm}\epsilon_{tp} + 0.80\text{x}_{tm}\epsilon_{tm} + 0.50\epsilon_{tp}\epsilon_{tm} \end{aligned}$$

Where, OY = Oil Yield (%), mc = moisture content of sandbox seed, v_{tp} = Roasting temperature, v_{tm} = Roasting time, ε_{tp} = Extraction temperature and ε_{tm} = Extraction time

Mathematical relationship for predicting biodiesel yield from sandbox relatively to the process factors is given in Equation 3.2. The coefficient of determination (R^2) relating the transesterification process and biodiesel yield was 0.74.

Where \mathbf{r}_{tm} = reaction time, \mathbf{r}_{tp} = reaction temperature, $\dot{\mathbf{c}}_{cc}$ = catalyst concentration (KOH) and η_{ra} = molar ratio

4. CONCLUSION

Effects of process factors on oil and biodiesel yield from sandbox seed were investigated. Among the process factors studied, the oil yield from the sandbox seed ranged from 20.9-53.6%. The maximum oil yield of 53.6%

was obtained at the processing conditions of 10.0%wb moisture content, 95 °C roasting temperature, 15 min roasting time, 65 °C extraction temperature and 90 min extraction time. The biodiesel yield ranged from 80.00-96.12%. The maximum biodiesel of 96.12% was extracted at 72.0 min reaction time, 50.0 °C reaction temperature, 0.9%wt catalyst concentration and 1:6 molar ratio. Mathematical models to predict sandbox seed oil and biodiesel yields at varying process conditions were developed with 0.81 and 0.74 coefficient of determination (R^2).

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