

Extraction and Quality Evaluation of Biodiesel from Six Familiar Non-Edible Plants Seeds

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Supplementary Materials

S1. Soxhlet Oil Extraction

The seed oil content (SOC) from all six seed sources was extracted using soxhlet and mechanical oil extractor (Fangtai Shibayoufang FL-S2017 China and Fangtai Shibayoufang J508, Guangdong, China) (Table S1, Supplementary Materials). The oil extraction occurred at 90 °C for 7 h and different solvents were also used during this process which comprised petroleum ether, acetone, dichloromethane, and ethyl acetate. The filter papers (pore size 30–50 µm) were used for removing the impurities and crude oil was removed at 80°C by employing rotary evaporator (Tokyo Rikakikai Co. Ltd. N-1210B, Tokyo Japan) under lower pressure. Finally, the oil extracted was stored and allowed to dry over anhydrous sodium sulphate prior to use.

S2. Mechanical extraction of non-edible seed oil procedure

The mechanical extraction of AF seeds was done by two different electric oil expeller machines, FANGTAI SHIBAYOUFANG FL-S2017 China (less power extractor) and FANGTAI SHIBAYOUFANG J508, China (high power extractor). Pre-treatment of seed is essential for mechanical extraction, which can increase the amount of oil recovery. After 2-3 revolutions, a large yield of crude AFSO was obtained. Through mechanical extraction, 8.7 wt.% oil content occurs. The oil removed from the seed by mechanical presses desires additional handling of extraction and filtration to produce a purer raw feedstock. The oil production of 6 sources was calculated by the following equation (1).

$$\text{Conversion\%} = \frac{\text{Obtained seed oil weight}}{\text{Total seed weight}} \times 100 \quad (1)$$

Further following steps were done to get the biodiesel from all 6 sources, filtration, rotary evaporation for access methanol, heating, transesterification, settling, separation, and washing.

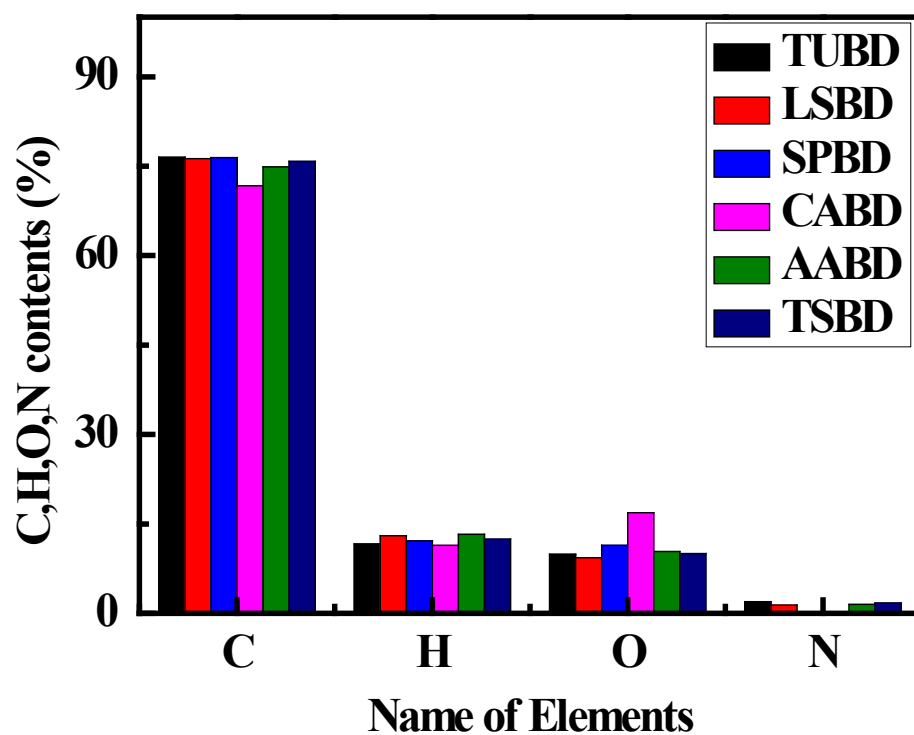


Figure. S1. Shows non-edible seed oil BD EA (Elemental analysis) for C, H, N, and O

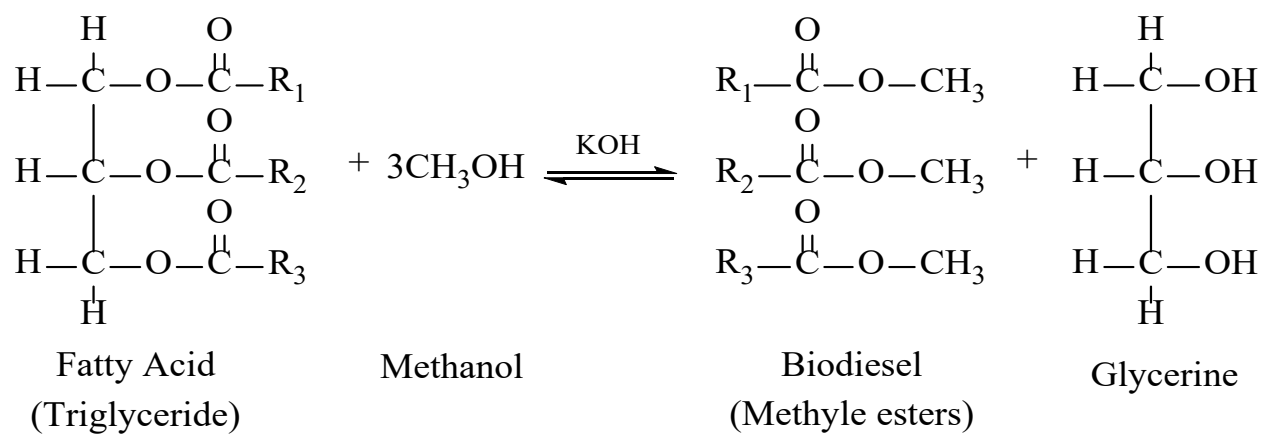


Figure S2. Transesterification reaction.

Table.S1. Oil, FFAs contents and Product yields

Source Name	Oil contents %	Soxhlet extraction %	Mechanical extraction %	FFAs contents %	Biodiesel%	Glycerine %	Soap%
Linseed (LS)	45	39-45	35	2.7	98	2	0
Tung tree (TO)	54.4	54.4	29	0.12	97.2	1.8	1
Heaven tree (AA)	38	38	21	1.9	93.9	6	0.1
Toonasinensis (TS)	35	28-35	19	2.1	95.2	3.8	1
Castor plant (CA)	48	45-50	27	0.8	96	4	0
Soapnut (SP)	51	51	30	1.1	96	3	1

LSBD=*Linum usitatissimum* L; linseed oil biodiesel; **TOBD**=*Vernicia fordii*; tung oil biodiesel; **AABD**= Heaven tree; *Ailanthus altissima* oil biodiesel; **TSBD**= *Toona sinensis* (Juss.) oil biodiesel; **CABD**=*Ricinus communis*; castor oil biodiesel; and **SPBD**= *Sapindus mukorossi*; soapnut oil biodiesel

Table.S2. AAOB Process of optimization

Amount of oil used (M/L)	Methanol to oil molar ratio	Temperature (°C)	Stirring intensity (rpm)	Reaction time (mins)	Amount of catalyst used		Percentage yield of various products		
					KOH (g)	CH ₃ OH (ml)	Biodiesel (%)	Glycerol (%)	Soap (%)
50	4:1	65	700	60	0.45	12.5	85	12	3
50	5:1	65	700	60	0.42	10	89	9	2
50	6:1	65	700	60	0.32	8.3	94	5	1
50	7:1	65	700	60	0.26	7.1	90	7	3
50	6:1	65	700	60	0.22	8.3	88	7	5
50	6:1	65	700	60	0.26	8.3	90	6	4
50	6:1	65	700	60	0.32	8.3	93.9	4	2.1
50	6:1	65	700	60	0.42	8.3	89	8	3
50	6:1	60	700	60	0.32	8.3	91	5	4
50	6:1	65	700	60	0.32	8.3	93.7	5	1.3
50	6:1	70	700	60	0.32	8.3	90.5	7	2.5
50	6:1	65	500	60	0.32	8.3	90.3	7	3.7
50	6:1	65	600	60	0.32	8.3	92	6	2
50	6:1	65	700	60	0.32	8.3	94	3	3
50	6:1	65	700	40	0.32	8.3	87	10	3
50	6:1	65	700	60	0.32	8.3	93.9	6	0.1
50	6:1	65	700	80	0.32	8.3	91.9	6	2.1
50	6:1	65	700	100	0.32	8.3	90	7	3

LSBD=*Linum usitatissimum* L; linseed oil biodiesel; **TOBD**=*Vernicia fordii*; tung oil biodiesel; **AABD**= Heaven tree; *Ailanthus altissima* oil biodiesel; **TSBD**= *Toona sinensis* (Juss.) oil biodiesel; **CABD**=*Ricinus communis*; castor oil biodiesel; and **SPBD**= *Sapindus mukorossi*; soapnut oil biodiesel

Table.S3. CAOB process of optimization

Amount of oil used (M/L)	Methanol to oil molar ratio	Temperature (°C)	Stirring intensity (rpm)	Reaction time (mins)	Amount of catalyst used		Percentage yield of various products		
					KOH (g)	CH ₃ OH (ml)	Biodiesel (%)	Glycerol (%)	Soap (%)
50	4:1	65	700	60	0.45	12.5	88	10	2
50	5:1	65	700	60	0.42	10	92	7	1
50	6:1	65	700	60	0.32	8.3	96	4	0
50	7:1	65	700	60	0.26	7.1	93	4	3
50	6:1	65	700	60	0.22	8.3	89	7	4
50	6:1	65	700	60	0.26	8.3	91	6	3
50	6:1	65	700	60	0.32	8.3	95.8	4.2	0
50	6:1	65	700	60	0.42	8.3	92.5	4.5	3
50	6:1	60	700	60	0.32	8.3	92	6	2
50	6:1	65	700	60	0.32	8.3	95.8	4.2	0
50	6:1	70	700	60	0.32	8.3	93	5	2
50	6:1	65	500	60	0.32	8.3	92	6	2
50	6:1	65	600	60	0.32	8.3	94	6	0
50	6:1	65	700	60	0.32	8.3	96	2	2
50	6:1	65	700	40	0.32	8.3	90	7	3
50	6:1	65	700	60	0.32	8.3	96	4	0
50	6:1	65	700	80	0.32	8.3	93	5	2
50	6:1	65	700	100	0.32	8.3	91	7	2

Table.S4. LSOB process of optimization

Amount of oil used (M/L)	Methanol to oil molar ratio	Temperature (°C)	Stirring intensity (rpm)	Reaction time (mins)	Amount of catalyst used		Percentage yield of various products		
					KOH (g)	CH ₃ OH (ml)	Biodiesel (%)	Glycerol (%)	Soap (%)
50	4:1	65	700	60	0.45	12.5	92	7	1
50	5:1	65	700	60	0.42	10	94	4	2
50	6:1	65	700	60	0.32	8.3	96.4	3.6	0
50	7:1	65	700	60	0.26	7.1	93	5	2
50	6:1	65	700	60	0.22	8.3	88	10	2
50	6:1	65	700	60	0.26	8.3	94.4	5.6	0
50	6:1	65	700	60	0.32	8.3	97.5	2	0.5
50	6:1	65	700	60	0.42	8.3	93.5	5	1.5
50	6:1	60	700	60	0.32	8.3	96	4	0
50	6:1	65	700	60	0.32	8.3	97.7	2	0.3
50	6:1	70	700	60	0.32	8.3	95.6	4.4	0
50	6:1	65	500	60	0.32	8.3	94	4	2
50	6:1	65	600	60	0.32	8.3	95.9	4	1.1
50	6:1	65	700	60	0.32	8.3	97.3	2	0.7
50	6:1	65	700	40	0.32	8.3	90	8	2
50	6:1	65	700	60	0.32	8.3	93	5.5	1.5
50	6:1	65	700	80	0.32	8.3	98	2	0
50	6:1	65	700	100	0.32	8.3	94.9	3.1	2

Table.S5. SPOB process of optimization

Amount of oil used (M/L)	Methanol to oil molar ratio	Temperature (°C)	Stirring intensity (rpm)	Reaction time (mins)	Amount of catalyst used		Percentage yield of various products		
					KOH (g)	CH ₃ OH (ml)	Biodiesel (%)	Glycerol (%)	Soap (%)
50	4:1	65	700	60	0.52	12.5	85	12	3
50	5:1	65	700	60	0.45	10	91	7	2
50	6:1	65	700	60	0.42	8.3	93	5	2
50	7:1	65	700	60	0.32	7.1	96	3	1
50	8:1	65	700	60	0.26	6.2	90	7	3
50	7:1	65	700	60	0.22	8.3	81	14	5
50	7:1	65	700	60	0.26	8.3	86	9	5
50	7:1	65	700	60	0.32	8.3	95.5	3.5	1
50	7:1	65	700	60	0.42	8.3	91	6.5	2.5
50	7:1	60	700	60	0.32	8.3	89	9	2
50	7:1	65	700	60	0.32	8.3	96	4	0
50	7:1	70	700	60	0.32	8.3	90	6	4
50	7:1	65	500	60	0.32	8.3	89	6	5
50	7:1	65	600	60	0.32	8.3	92	8	0
50	7:1	65	700	60	0.32	8.3	96	4	0
50	7:1	65	700	40	0.32	8.3	87	10	3
50	7:1	65	700	60	0.32	8.3	95.9	4.1	0
50	7:1	65	700	80	0.32	8.3	93	5	2
50	7:1	65	700	100	0.32	8.3	90	5	5

Table.S6. TOOB process of optimization

Amount of oil used (M/L)	Methanol to oil molar ratio	Temperature (°C)	Stirring intensity (rpm)	Reaction time (mins)	Amount of catalyst used		Percentage yield of various products		
					KOH (g)	CH ₃ OH (ml)	Biodiesel (%)	Glycerol (%)	Soap (%)
50	4:1	65	700	60	0.45	12.5	94	4	2
50	5:1	65	700	60	0.42	10	96.4	2	1.6
50	6:1	65	700	60	0.32	8.3	97	3	0
50	7:1	65	700	60	0.26	7.1	90	8	2
50	6:1	65	700	60	0.22	8.3	92	6	2
50	6:1	65	700	60	0.26	8.3	94.5	3	1.5
50	6:1	65	700	60	0.32	8.3	96.8	2.2	1
50	6:1	65	700	60	0.42	8.3	93	4	3
50	6:1	60	700	60	0.32	8.3	94	4	2
50	6:1	65	700	60	0.32	8.3	97.2	1.8	1
50	6:1	70	700	60	0.32	8.3	93	5	2
50	6:1	65	500	60	0.32	8.3	90	7	3
50	6:1	65	600	60	0.32	8.3	95	4	1
50	6:1	65	700	60	0.32	8.3	97	1	2
50	6:1	65	700	40	0.32	8.3	89	8	3
50	6:1	65	700	60	0.32	8.3	94	5	1
50	6:1	65	700	80	0.32	8.3	97	3	0
50	6:1	65	700	100	0.32	8.3	94.5	3.5	2

Table.S7. TSOB process of optimization

Amount of oil used (M/L)	Methanol to oil molar ratio	Temperature (°C)	Stirring intensity (rpm)	Reaction time (mins)	Amount of catalyst used		Percentage yield of various products		
					KOH (g)	CH ₃ OH (ml)	Biodiesel (%)	Glycerol (%)	Soap (%)
50	4:1	65	700	60	0.45	12.5	90	7	3
50	5:1	65	700	60	0.42	10	95.3	3.6	1
50	6:1	65	700	60	0.32	8.3	93.4	5.6	1
50	7:1	65	700	60	0.26	7.1	90.7	7.3	2
50	5:1	65	700	60	0.22	10	89	8	3
50	5:1	65	700	60	0.26	10	91	7	2
50	5:1	65	700	60	0.32	10	93.1	6.9	0
50	5:1	65	700	60	0.42	10	95.1	2.9	2
50	5:1	60	700	60	0.42	10	92	7	1
50	5:1	65	700	60	0.42	10	94.9	4	1.1
50	5:1	70	700	60	0.42	10	93.2	4.8	2
50	5:1	65	500	60	0.42	10	89	6	5
50	5:1	65	600	60	0.42	10	92.5	7.5	0
50	5:1	65	700	60	0.42	10	95	4	0
50	5:1	65	700	40	0.42	10	87	10	3
50	5:1	65	700	60	0.42	10	92	6	2
50	5:1	65	700	80	0.42	10	95.2	3.8	1
50	5:1	65	700	100	0.42	10	93.7	5	1.3

Table.S8. FTIR data presenting various functional groups in Non-edible seed oil FAMES

Peak no.	Wave number (cm ⁻¹)	Group attribution	Vibration type	Abs intensity	TSBD	TOBD	SPBD	LSBD	CABD	AABD
1	3464	-OH	Stretching	Weak	--	--	--	--	--	--
2	3007	=C-H	Stretching	Strong	3008	3008	3008	3008	3008	3008
3	2925	-CH ₂	Asymmetric stretching vibration	Strong	2927	2920	2927	2920	2920	2920
4	2854	-CH ₂	Asymmetric stretching vibration	Strong	2859	2852	2852	2852	2859	2859
5	1743	-C=O	Stretching	Strong	1741.30	1741.30	1741.30	1741.30	1741.30	1741.30
6	1641	-CH ₂	Shear type vibration	Middling	1641	1648	1641	1641	1641	1641
7	1435	Terminal Methyl	Stretching vibration	Higher	1435	1430	1435	1460	1435	1466
8	1361	-CH ₃	Bending vibration	Middling	1361	1361	1361	1361	1354	1367
9	1170	C-O-C	Symmetric stretching vibration,	Middling	1167	1167	1161	1166	1174	1163
10	1016	C-O-C	Vibration	Weak	1017	1093	1017	1024	1030	
11	723	-CH ₂	Plane rocking vibration	Weak	718	730	718	718	720	719

Table. S9. ¹H NMR spectroscopic data showing the chemical composition of various methyl esters (Methoxy proton) in non-edible seed oil, biodiesel (FAMEs)

Peak. No	Peak area/ region/ ppm	Identified compound	Chemical structure	TSBD	TOBD	SPBD	LSBD	CABD	AABD
1	0.89	Terminal methyl protons	-CH ₃	0.85	0.88	0.87	0.88	0.87	0.76
2	0.99	Terminal methyl Protons	-CH ₃	0.92	--	--	0.98	--	--
3	1.27	β-methyl protons	-CH ₂	1.26	1.29	1.28	1.29	1.28	1.19
4	1.31	β-methyl protons	-CH ₂	--	--	--	--		1.47
5	1.62	β-methyl protons	-CH ₂	1.55	1.59	1.61	1.62	1.61	1.89
6	2.04	α –methylene protons	-CH ₂	2.00	2.07	2.02	2.02	2.03	2.05
7	2.30	α-methylene proton	-CH ₂	2.25	2.28	2.29	2.30	2.29	2.19
8	2.80	α-methylene protons	-CH ₂	2.74	2.76	2.77	2.80	2.77	2.65
9	3.66	Methoxy proton	-OCH ₃	3.60	3.63	3.65	3.66	3.66	3.52
10	5.34	Olefinic protons	-HC=CH	5.30	5.34	5.34	5.35	5.34	5.23

Table.S10. ^{13}C NMR spectroscopic data showing the chemical shift values corresponding to various structural features in non-edible seed oil (Methoxy carbon) FAMES

Peak no:	Peak area/ region/ ppm	Identified compound	Chemical structure	TSBD	TOBD	SPBD	LSBD	CABD	AABD
1	14.12	Terminal methyl carbon	$-\text{CH}_3$	14.12	13.80	14.12	14.12	14.12	14.12
2	22.57	Methylene carbon	$-\text{CH}_2$	22.43	22.20	22.60	22.74	22.43	22.43
3	34.10	Methylene carbon	$-\text{CH}_2$	34.10	34.10	34.10	34.10	34.10	34.10
4	51.32	Methoxy carbon	$-\text{OCH}_3$	51.35	51.35	51.35	51.35	51.35	51.35
5	127.89	Olefinic carbon	$\text{C}=\text{C}$	128.00	126.68	128.00	127.68	127.68	128.00
6	130.21	Olefinic carbon	$\text{C}=\text{C}$	130.14	130.45	130.14	130.14	130.14	130.14
7	174.24	Carbonyl carbon of ester	$-\text{COOCH}_3$	174.15	174.14	174.14	174.46	174.14	173.23

Table.S11. Shows Non edible plant seed oil BD ICP-OES detail elements concentration (ug/g) in comparison with petrodiesel

Name of Elements	TOBD Elements Conc. ug/g	AABD Elements Conc. ug/g	SPBD Elements Conc. ug/g	TSBD Elements Conc. ug/g	CABD Elements Conc. ug/g	LSBD Elements Conc. ug/g	Petrodiesel ug/g
Zn	0.15	0.18	0.89	0.16	0.12	0.179	9.5
Cu	0.056	0.96	0.28	0.98	0.79	0.115	-
Mn	0.090	0.19	0.03	0.12	0.032	0.123	1.5
Ni	0.011	0.013	0.03	0.01	0.63	0.011	12.4
Fe	1.19	2.03	0.80	1.12	0.89	1.289	-
K	0.29	35.2	6.44	3.22	3.65	0.437	213.3
Na	5.83	13.2	42.2	22.6	24.5	6.008	868.3
Ca	11.26	29.4	15.6	45	21.9	18.38	21.4
Mg	3.60	6.68	0.30	5.94	1.02	4.136	35.6
S	1.26	1.94	22.1	15.7	17.8	1.143	-
P	0.35	0.88	0.10	14.7	0.22	0.269	-