Solid Lewis Acid Catalyzed Ultrasound Assisted Transesterification of Lesquerella Oil to Biodiesel

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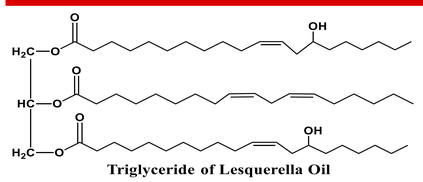
Abstract

Biodiesel, consisting of fatty acid methyl esters (FAMEs), is becoming a popular alternative to petroleum-based diesel fuels. It has been receiving great attention as a safe, biodegradable, and renewable fuel source. Moreover, it can either be used in pure form or in a blend with petroleum fuel in diesel engines. This study aimed to use direct immersion ultrasound at room temperature and shorter reaction time using different solid Lewis acid catalysts for the transesterification of Lesquerella oil (LO) fatty acids. Lesquerella oil is the only hydroxy fatty acids (Lesquerolic acid)-containing vegetable oil besides castor oil reported thus far. $AlCl_3$ has shown the most catalytic activity in transesterification of LO fatty acids. Tin (II) chloride and tin (II) acetate also showed catalytic activity. The amount of catalyst loading, solvent-to-oil molar ratio and reaction time are the main variables used in this study. Vibra-cell VC505 ultrasonic processor was used as the source of ultrasound. Thin-layer chromatography along with FTIR was used to observe the progress of reaction with time. FAMEs concentration and % conversion of fatty acids were analyzed by gas chromatography and 1H NMR spectroscopy respectively. Results have important indications that this is a green, energy-efficient and industrially applicable alternative for biofuel

Introduction



Triglyceride of Lesquerella Oil

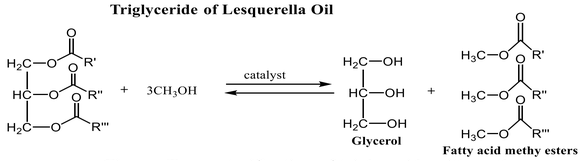


Figure: Transesterification of triglyceride with methano

Application of Ultrasound in Biodiesel Production

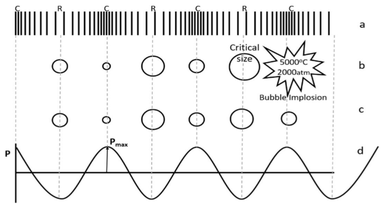


Figure: Ultrasonic wave propagation (a) compression and rarefaction, (b) transient ultrasonic cavitation, (c) stable ultrasonic cavitation, (d) pressure variation during compression and rarefaction

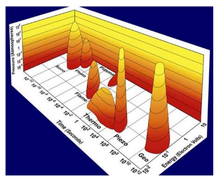
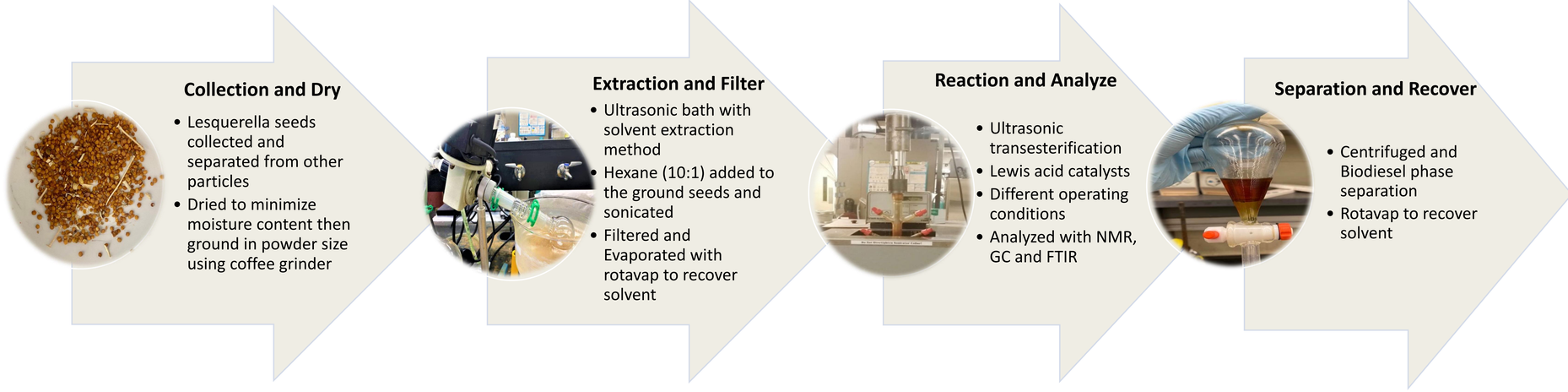
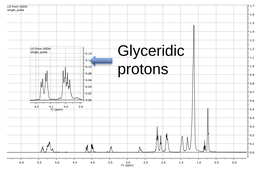


Figure: Difference of ultrasonic irradiation from traditional energy sources (such as heat, light, or ionizing radiation). The three axes represent duration of the interaction, pressure, and energy per molecule.

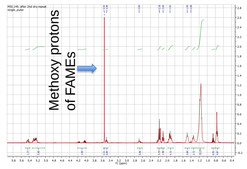


Results and Discussion

^{1}H NMR of Lesquerella oil before Transesterification



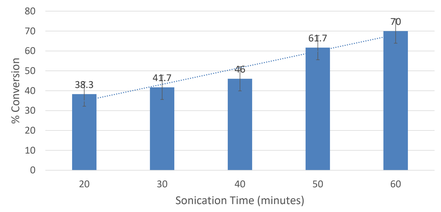
^{1}H NMR of Lesquerolic acid methyl esters



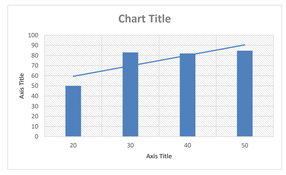
$$\%Yield_{FAME} = \frac{41/3}{42/2} \times 100$$

- A_1 is the area related to methoxy protons of the FAME which resonate at about 3.31 ppm
- A_2 is the area of methylene glyceridic protons resonated at 2.1 ppm

Lesquerella Oil % conversion to Biodiesel versus time with AlCl3 Lewis acid catalyst:



- 6 wt% $AlCl_3$ used as Lewis acid catalyst
- Methanol was used as solvent in the amount of 6:1 molar ratio (solvent: oil)



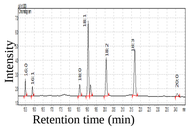
- 5 wt% $AlCl_3$ used as Lewis acid catalyst
- Methanol was used as solvent in the amount of 6:1 molar ratio (solvent: oil)

Results and Discussion

Table: GC Analysis of FAMEs Conversion using $SnCl_2$ catalyst

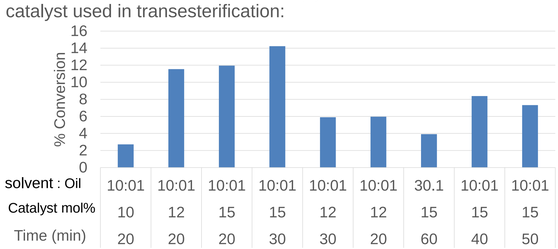
Entry	Catalyst loading mol%	Time (min)	Oil/MeOH (molar ratio)	Peak Area (μV.s) x10^3
1	0	20	1:06	0
2	2	20	1:06	0
3	5	20	1:06	50.71
4	10	20	1:06	59.54
5	12	20	1:06	63.03
6[a]	5	20	1:06	24.50
7[b]	5	0	1:06	0

[a] ultrasonic bath was used
[b] reaction without sonication

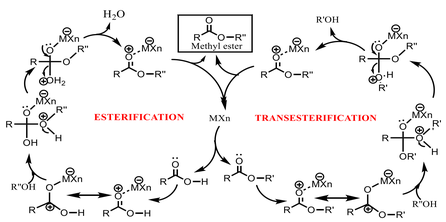


GC chromatogram of LO methyl esters using 12 mol% $SnCl_2$ catalyst.

% conversion of Biodiesel when Tin (II) acetate catalyst used in transesterification:



Reaction Mechanism for lewis acid catalyzed Transesterification and Esterification reaction



MXn = Lewis acid
R and R' = carbon chain of the fatty acid
R'' = alkyl group of the alcohol.

Conclusions

- The successful application of $AlCl_3$,$SnCl_2$, and Tin (II) acetate are reported where $AlCl_3$ showed the highest catalytic activity.
- Direct immersion ultrasound showed that it requires much less reaction time in transesterification with Lewis acid catalysts.
- The statistical analysis suggested that catalyst loading, solvent to vegetable oil molar ratio, and sonication time are the most significant parameters in this study.

Future Work

- More repetition of the reactions to collect repeated data.
- System design analyses and more biodiesel product quality analysis.
- Regeneration and reuse of the catalysts.
- Separation of the purified form of glycerin and study its application in chemical industries.

References

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Aknowledgements

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