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Comparative Analysis of Physicochemical Properties of Dimer Acids from *Jathropha Curcas* and *Thevetia Nerrifolia* Seed Oils

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Abstract

Thevetia Nerrifolia and *Jathropha Curcas* seed oils were extracted from their oil seeds by soxhlet extraction using petroleum ether (40-60 ^oC) as solvent. The oils were characterized and the oil yield, refractive index, acid value, saponification value, iodine value, colour and relative density were found to be 47.77%, 1.464, 4.365 (mg/KOH), 125.62 (mg/KOH), 98.48 (wij), 4^+ and 0.926 for *Thevetia Nerrifolia* seed oil and 46.56%, 1.496, 33.65 (mg/KOH), 175.12 (mg/KOH) 105.43 (Wij), 3 and 0.913 for *Jathropha Curcas* seed oil respectively. Dimer acids were prepared from these seed oils by heating 200g of each of the oils under nitrogen inert atmosphere in a four necked resin kettle at a temperature of 300^oC for 12 hours. The physicochemical properties of the dimer acids were analyzed and were found to compare favourably with each and other well known feed stocks of dimerization.

Keywords: Thevetia Nerrifolia seed oil, Jathropha curcas seed oil, Dimer acid, Sulphur catalyst, Polymerization, Physicochemical properties.

Contents

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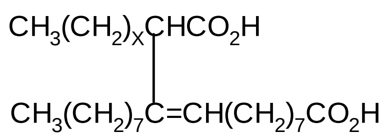
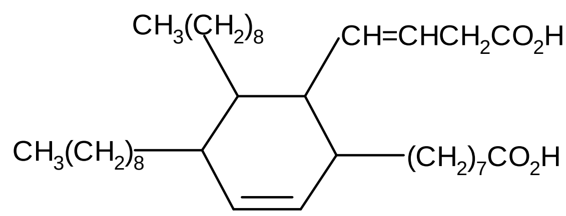
1. Introduction

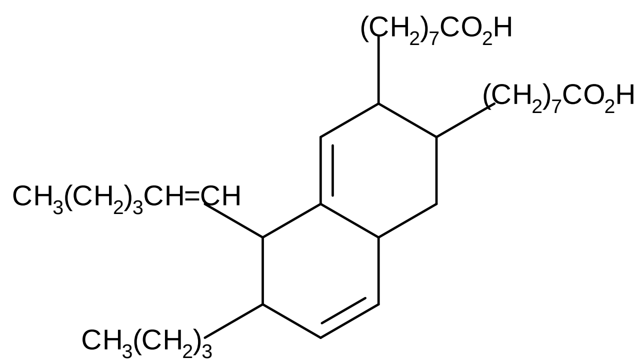
Dimer acids are products resulting from the thermal polymerization of C_{18} unsaturated fatty acids present in vegetable oils. The process of polymerization or dimerization involves heating the triglycerides or the mixture of the fatty acids of vegetable oils or their monoesters usually methyl-esters in an inert atmosphere to temperature of about 300°C for 12-20 hours. Under such conditions the mono-polyunsaturated C_{18} fatty acids dimerized to give mixtures of C_{36} compounds whose structures have been found to include acyclic, cyclic and bicyclic respectively as shown below [1].



Acyclic Dimer(1)



Monocyclic Dimer (2)



Bicyclic Dimer

Although the traditional method of dimerization is to heat the feedstock (vegetable oil, fatty acid mixtures, methyl esters or alkali metal salt of vegetable oils) in inert atmosphere to 300°C and above for long hours (12-20 hours), several other method of dimerization involving the use of catalysts which may act to reduce the process temperature and/or time. Among these catalysts is neutral clay and water mixture, usually added to the tune of 1-25% [2] of clay and 1-5% of water. This catalyst mixture is reported to reduce process conditions to 230-260°C and 2-4 hours when conducted under pressure [3, 4]. Suzuki et al, in catalyzing dimerization employed synthetic silica-alumina [5].

The largest volume of commercial application of dimer acids is in non-reactive polyamide resin. The dimer acids impart flexibility, corrosion resistance, chemical resistance, moisture resistance and adhesion to nonreactive polyamide [6]. They are also used extensively to react with epoxy or phenolic resins yielding adhesive that is useful in casting and laminating in structural work for patching and sealing compounds and for protective coatings [7].

2. Experimental

Thevetia Nerrifolia and *Jathroph acurcas* seeds were collected from Makurdi Local Government Area of Benue State. The seeds were sun dried and then oven dried at 45°C to constant weight and ground with porcelain mortar and piston to coarse particle size and stored in plastic containers for analysis. The oils were extracted using petroleum ether (40-60°C) on a soxhlet extractor for four hours [8]. The refractive index, acid value, saponification value, iodine value colour and relative density were determined using the method described by A.O.A.C [9]. Dimer acids from the *Thevetia Nerrifolia* and *Jathropha Curcas* seed oils were prepared by heating 200g each of the oils in a four necked resin kettle under nitrogen inert atmosphere at a temperature of 300°C for 12 hours using 0.5% sulphur (based on the weight of oil) as catalyst [10].

The physico-chemical properties of the *Thevetia Nerrifolia* and *Jathropha Curcas* seed oils were determined using the methods described by ASTM D 1639-90 for acid value [11] ASTM D 1962-67 for saponification value [12] ASTM D 1541-60 for iodine value [13] ASTM D 4052 for relative density [14] ASTM D 1747 for refractive index [15] and ASTM D 1544 for colour [16].

The physicochemical properties of the dimer acids were determined by methods described above for the seed oils. The molecular weight of the dimer acids were determined by cryoscopy.

FTIR spectra of the dimer acids were recorded to compare with other traditional dimer acids from well known feedstock of dimerization.

3. Results and Discussion

The physicochemical properties of *thevetia nerrifolia* and *Jathropha Curcas* are shown in Table 1.0. From Table 1.0, the oil yield of *Thevetia Nerrifolia* seed oil was found to be 47.47, which was slightly higher than that of *Jathropha Curcas* seed oil of 46.56 The yields are appreciable and shows that the oils has potential in surface coating industry and in the manufacture of oleochemicals.

The saponification value of *Thevetia Nerrifolia* seed oil was found to be 125.62. This value is lower than that for *Jathropha Curcas*of 175.12. Saponification value indicates the average molecular weight of the oil. A high saponification value indicates that the oil contained higher proportions of low molecular weight fatty acids [17]. This result shows that *Jathropha Curcas* seed oil has lower molecular weight fatty acids more than *Thevetia nerrifolia* seed oil.

Acid value of *Thevetia Nerrifolia* seed oil was found to be 4.365; this value is lower than that of *Jathropha Curcas* seed oil of 33.65. Acid value of oil measures the extent to which the glycerides had been decomposed by lipase action. The decomposition is usually accelerated by heat and light. The acids that are usually formed include free fatty acids, acid phosphate and amino acids. Free fatty acids are formed at a faster rate than the other acids [18]. The result indicated that *Jathropha Curcas* seed oil is more prone to this decomposition action.

The iodine value of *Thevetia Nerrifolia* seed oil was found to be 98.48 which is lower that of *Jathropha Curcas* seed oil of 105.43. This is an indication that *Thevetia Nerrifolia* seed oil is non-drying, whereas *Jathropha Curcas* seed oil is semi-drying. The closeness of the *Thevetia Nerrifolia* seed oil value to that of semi drying oils shows that there are high levels of poly unsaturated fatty acids [19].

The refractive index of 1.464, the relative density of 0.926 and the colour (Gardner) of 4^+ for *Thevetia Nerrifolia* seed oil and 1.496, 0.913, and 3 for *Jathropha Curcas* seed oil all conforms to the values given by literature for other vegetable oils. This is an indication that the oil extracted was of high degree of purity.

Table-1. Physicochemical Properties of ThevetiaNerrifolia and JathrophaCurcas seed oils

Property	ThevetiaNerrifolia	JathrophaCurcas Value
Oil yield (%)	47.47 ± 0.026	46.56 ± 0.26
Refractive index	1.464 ± 0.001	1.496 ± 0.02
Acid value (mg/KOH)	4.365 ± 0.017	33.65 ± 0.02
Saponification value (mg/KOH)	125.62 ± 0.31	175.12 ± 0.43
Iodine value (Wij's)	98.48 ± 0.02	105.43 ± 0.27
Colour (Gardner)	4^+	3
Relative density	0.926 ± 0.003	0.913 ± 0.03`

The results of the physicochemical characterization of dimer acids prepared from *thevetia nerrifolia* and *jathropha curcas* seed oils are shown from Table 2.0.

The molecular mass of the dimer acids were found to be 540.62 for *Thevetia nerrifolia* seed oil and 492.54 for *Jathropha curcas* respectively. These values are lower than what is expected when dimer methyl ester was used [20]. This is due to fact that crude dimers contain 30% monomeric acids which lowers the average molar mass [21].

Also the acid values of 119.26 for *Thevetia nerrifolia* seed oil and 122.85 for *Jathropha curcas* were lower than the expected values of 125.62 for *Thevetia nerrifolia* seed oil and 175.12 for *Jathropha curcas*, the saponification values of the respective oils. This is an indication that the oils were not completely hydrolyzed into acids.

The other parameters; saponification value, viscosity, refractive index, iodine value and colour of the dimer acids whose values are 192.68 (mgKOH/g), 5032.33 (mpa.s/25^{o}C), 1.4690, 98.56 (Wij's) and 12^+ for *Thevetia nerrifolia* dimer acid and 189.55 (mgKOH/g), 5081.47 (mpa.s/25^{o}C), 1.466, 97. 78 and 12^+ for *Jathropha curcas* dimer acid all compare favourably with each other and the dimer acids from well known traditional oils.

Table-2. Physicochemical Properties of Dimer Acids from *Theevetianerrifolia* and *Jathrophacurcas* Seed Oils

Analytical parameter	Value for TNSO	value for JCSO
Acid value (mg/KOH)	119.26 ± 0.03	122.85 ± 0.73
Molecular mass (g)	540.62 ± 0.02	492.54 ± 0.005
Colour (Gardner)	12^+	12^+
Refractive index	1.4690 ± 0.26	1.466 ± 0.053
Viscosity (mpa.s/25^{o}C)	5032.33 ± 0.31	5081.47 ± 0.23
Saponifcation value (mgKOH/g)	192.68 ± 0.02	189.55 ± 0.031
Iodine value (Wij's)	98. 56 ± 0.02	97.78 ± 0.001

The results of the physicochemical characterization of the dimer acids are corroborated by the FTIR spectra of the dimer acid from *Thevetia nerrifolia* seed oil and *Jathropha curcas* seed oil, Figure 1 and 2 respectively

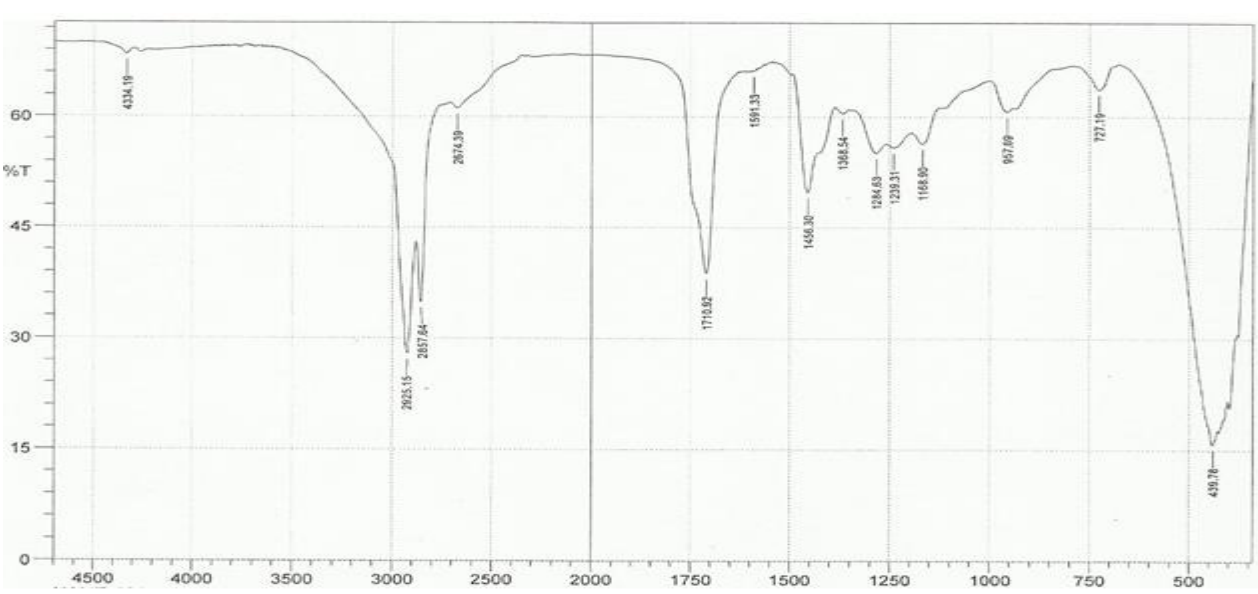


Figure-1. FTIR Spectrum of Dimer Acid from *Thevetia Nerrifolia* seed oil

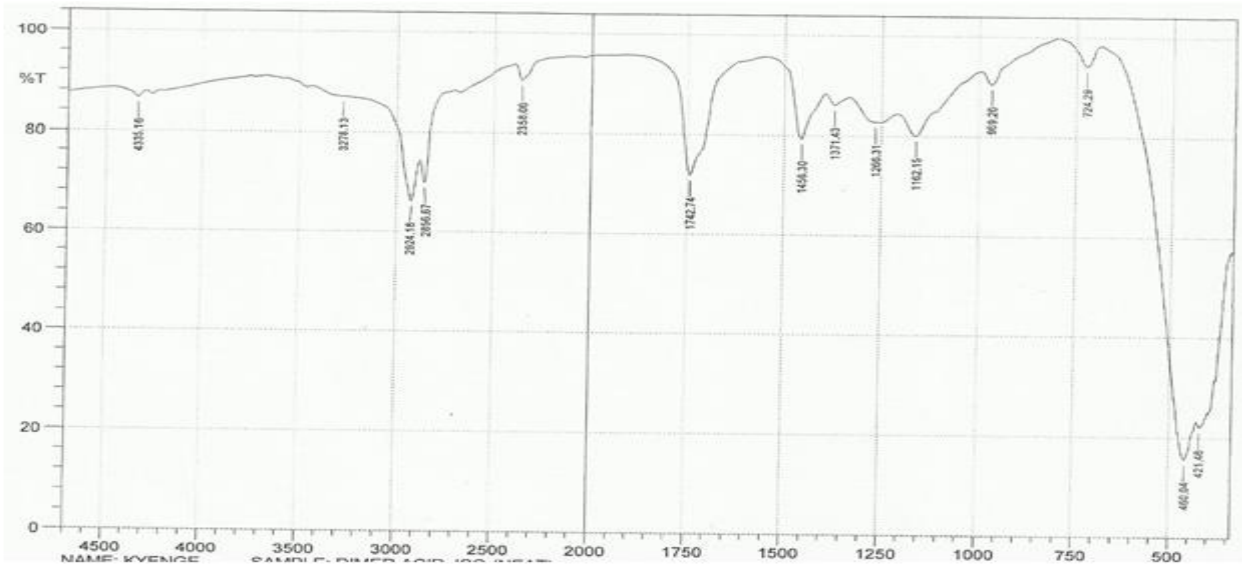


Figure-2. FTIR Spectrum of Dimer Acid from Jathropha Curcas Seed Oil

The FTIR shows a peak at 1710.92 cm^{-1} due to C=O stretch of carboxylic acid group for *Thevetia nerrifolia* seed oil dimer acid. This peak is however absent from *Jathropha curcas* dimer but there appear a peak at 1742.74 cm^{-1} due to C=O of an ester indicating incomplete hydrolysis of the oil to acid. A peak at 1280 cm^{-1} for *Thevetia nerrifolia* dimer was due to axial deformation of dimer C-O bond, this correspond to the peak at 1266.31 cm^{-1} for *Jathropha curcas* dimer.

4. Conclusion

Dimer acids prepared from *thevetia nerrifolia* and *Jathropha Curcas* seed oils shows comparable physicochemical properties as dimer acids prepared from other well known oils in the dimer acid synthesis and hence these non-edible vegetable oils can be used as replacements for those oils that are mostly edible for commercial production of dimer acids.

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