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# SEMECARPUS ANACARDIUM L.F. NUT OIL AS BIODIESEL FEED STOCK (ANACARDIACEAE -FAMILY)

#### \*Dr. Vustelamuri Padmavathi

Associate Professor, Department of Chemistry, Neil Gogte Institute of Technology, Kachavani Singaram, Hyderabad, India.

\*Corresponding Author: Dr. Vustelamuri Padmavathi

Associate Professor, Department of Chemistry, Neil Gogte Institute of Technology, Kachayani Singaram, Hyderabad, India.

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## **ABSTRACT**

As petrol is a fast depleting natural resource, it became a tremendous need for the scientists to search because another alternative renewable resource to petrol is a deemed necessity. Now serious efforts are being made on the production and utilization of biodiesel in India and other parts of the world. As an alternative invention of biodiesel from naturally growing herbal plants became the target. In this study, *Semecarpus anacardium* L.f. which grows naturally without any cost and care by the time, is analyzed and investigated as a proper feedstock in producing biodiesel for the first time. In order to prove its suitability, its seed and oil were experimented first. The tree is widely distributed through the hotter part of India. It is frequent in dry deciduous forests of Central India. Common in dry deciduous forests of Maharashtra spreading over Khandesi, Marathwada and East Maharashtra, in India. In the present study, biodiesel has been synthesised from *Semecarpus anacardium* L.f. oil. The acid value of this oil was found to be as 0.5 during these investigations, which leads us to convert it to biodiesel by the esterification followed by *trans*-esterification process. The methyl esters produced by these methods were analyzed and found that, some of them are suitable as biodiesel fuel to ascertain their suitability as diesel fuels.

**KEYWORDS:** *Semecarpus anacardium* L.f., Anacardiaceae Family; alkali *trans*-esterification; methyl ester; antimicrobial activity; alternative fuel.

### Abbreviations

ASTM: American Society for Testing and Materials; EN: European Norm; CP: cloud point; PP: pour point; PM: particulate matter; PAH: polycyclic aromatic hydrocarbons; Sox: sulphur oxides; EU: European Union; FFA: free fatty acids.

## INTRODUCTION

Biodiesel which is derived from triglycerides by transesterification and also from the fatty acids by esterification has attracted considerable attention during the past decade as renewable, biodegradable, ecofriendly and non-toxic fuel. Recently biodiesel is gaining prominence in global warming and the consequential changes in weather pattern manifested in the form of widespread flooding, gully erosion, massive mudslide, environmental degradation and increasing cost of fossil fuel have given the necessary impetus to switch to vegetable oils as alternative fuels for diesel engines. Vegetable oils, however, are not suitable for direct use in diesel engines because of high viscosity, poor cold flow properties and low cetane number, It can however, be made useable by either blending with diesel fuel or trans-esterification with alcohol as to methyl ester is called biodiesel.

As a substitute for petroleum based diesel due to environmental considerations and depletion of vital resources like petroleum and coal, the possible use of renewable resources as fuels and as a major feedstock for the chemical industry is currently gaining acceptance. Further, as petroleum is a fast depleting natural resource, an alternative renewable route to petroleum is a deemed necessity. Now serious efforts are being made on the production and utilization of biodiesel in India. Methyl esters are clean burning fuel with no sulfur emission. Although its heat of combustion is slightly lower than that of the petro-diesel, there is no engine adjustment necessary and there is no loss in efficiency. Methyl esters are non-corrosive and are produced at low pressure and low temperature conditions. Concentrated (about 80%) glycerin is obtained as a byproduct during transesterification process.

## **Plant History**

Semecarpus anacardium L.f.<sup>[1-4]</sup> (Anacardiaceae) is a deciduous tree distributed in the forests of the Western Ghats of India. In the Indian system of medicine, the plant is well known as Bhallatak (Sanskrit) and commonly known as 'marking nut' (English) and Kaadu geru (Kannada). Semecarpus anacardium L.f (Anacardiaceae) is reported to possess many medicinal

properties. Trees: up to 25 m tall; young branches: terete, tomentose, watery latex present, which on drying it turns black. The black corrosive juice of the pericarp contains tarry oil consisting of 90% of oxyacid anacardic acid and 10% of higher nonvolatile alcohol called cardol, also contains catechol and a mono-hydroxy phenol called as anacardol. The most significant components of the Semecarpus anacardium L.f. oil are phenolic compounds on exposure to air; phenolic compounds get oxidized to quinones. The oxidation process can be prevented by keeping the oil under nitrogen. Many of the well-known properties of marking nut oils are easily explainable by the catechol half and lipoid-soluble C<sub>15</sub> chain. During exposure to air, the catechol ring might be oxidized to an orthoguinone which might impart the dark color and also implies polymerization. The vesicant nature and the indelible pigmentation due to the rapid formation of the orthoquinonoid intermediate. The absorption of the oil by the skin is obviously due to the lipoid-soluble  $C_{15}$ chain. [5] In addition to this, Chemical and photochemical analysis of *Semecarpus anacardium* nuts revealed the presence of bhilawanol<sup>[5,6]</sup>, Anacardic acid<sup>[7,8&9]</sup>, I-4<sup>1</sup>, II-3<sup>1</sup>,4<sup>1</sup>,I-5, II5,I7hexahydroxy [I3,II8]biflavanone, 1-4<sup>1</sup> 3,4,1-5, II5,1/Hexanydroxy [15,Ho]ointavanone, 1-4, II-4<sup>1</sup>, I-5, II-5, II-7, II-7-hexahydroxy [I-3, II-8]biflavanone (3<sup>1</sup>, 8-binaringenin)<sup>[10]</sup>, I-4<sup>1</sup>, II-4<sup>1</sup>, I-7, II-7-tetrahexahydroxy [I-3, II-8] biflavanone (3<sup>1</sup>8-billiquiritigenin)<sup>[10]</sup>, Tetra hydrorobusta Flavanone<sup>[11]</sup>, Tetrahydroamento flavanone<sup>[11]</sup> Amentoflavone<sup>[11]</sup>, Calluflavanono<sup>[13]</sup> Semecarpuflavanone<sup>[12]</sup>, Galluflavanone<sup>[13]</sup>. Jeediflavanone[14,15]. Semecarpetine<sup>[16]</sup>. Anacarduflavanone [17]. Nallaflavanone<sup>[18]</sup> Anacardoside<sup>[19]</sup> Bhilwanol analogs<sup>[20]</sup>, Flavanoids.<sup>[21]</sup>

Pharmalogical activities of Semacarpus anacardium

Hypocholesterolemic activity<sup>[22]</sup>, Anti-inflammatory<sup>[23-26]</sup>, Immunomodulatory activity<sup>[27]</sup>, Antioxidant<sup>[28-29]</sup>, Adjuvant<sup>[30]</sup>, Antimicrobial activity<sup>[31-36]</sup>, Hypoglycemic and anti hyperglycemic<sup>[37]</sup>, Breast cancer <sup>[38-43]</sup>, Acetyl cholinesterase inhibitory activity<sup>[44]</sup>, Acute and subchronic toxicity study<sup>[45]</sup>, Anti mutagenic effect<sup>[46]</sup>, Propositivity<sup>[47-48]</sup>, Pages <sup>[49]</sup>, Activate and subchronic toxicity study<sup>[45]</sup>, Antimutagenic effect<sup>[46]</sup>, Propositivity<sup>[47-48]</sup>, Pages <sup>[49]</sup>, Propositivity<sup>[47-48]</sup>, Propositivity<sup>[48]</sup>, Pro Bioactivity<sup>[47-48]</sup>, Renal cortical necrosis<sup>[49]</sup>, Hair growth.[50]

#### MATERIALS AND METHODS

Semecarpus anacardium L.f. nuts were collected from field area very nearer to the village Nandgaon, Kolhapur City, Maharashtra, India. All plant material specimen's were identified by Dr Vatsavaya S. Raju, Plant Systematic Laboratory, Department of Botany, Kakatiya University, Warangal (A. P. State) and conformed as Semecarpus anacardium L.f. (syn: Anacardium latifolium Lam., A. orientale Steud.) of Anacardiaceae and plant specimen deposited at Kakatiya University Herbarium, Warangal (KUW) with accession number 1874. It is locally known as 'nalla jeedi' and popularly known 'marking nut/dhobi nut.

## Material and plant source Extraction and purification

Plant material: Semecarpus anacardium L.f. 3 kg nuts were collected and shade dried and six psyphons (What are these?) were done for each step. The nuts were directly percolated with cold petroleum ether 5-6 times, filtered and concentrated by vacuum evaporation.

**STEP 1:** Then the nuts were made in to small pieces and percolated with petroleum ether 5-6 times, filtered and concentrated by vacuum evaporation.

**STEP 2:** Again the powdered nuts were percolated with hot petroleum ether 5-6 times, filtered, and concentrated by vacuum evaporation.

STEP 3: After that, the same powdered nuts were extracted with cold acetone by changing the solvent for 3 hours in three intervals, filtered and concentrated by vacuum evaporation.

STEP 4: After cold acetone percolation, nuts were subjected to hot acetone extract, and concentrated by vacuum evaporation.

The above four concentrates were then fractionated using silica gel column with appropriate solvent gradient and the oil obtained from the fraction of CAPE (cold acetone petroleum ether extract) 30, 31, 33, 34, 35, 36, 37 cape 38 last, the combined oil (named CAPE 30) was concentrated by vacuum evaporation and given for spectral analysis. Basing on UV, FT-IR<sup>[22]</sup>, <sup>1</sup>H NMR<sup>[23-26]</sup>, <sup>13</sup>C NMR<sup>[23]</sup>, MASS, HPLC, LCMS, GCMS, GC<sup>[27-31]</sup> methods, it was found that it contains free fatty acids, triglycerides and others. Basing on spectral data and their physical properties Semecarpus anacardium L.f. nut oil was selected for biodiesel production.

#### FT-IR: CAPE 30

The compound displayed bands at 3473.09 cm<sup>-1</sup> (O-H, hydrogen bonded alcohol, phenols), 3006.16 cm<sup>1</sup> (hydrogen bonded alcohol, phenols), 2925.05 cm<sup>-1</sup>, 2855.42 cm<sup>-1</sup> (C-H, alkanes), 1746.10 cm<sup>-1</sup> (C=O, aldehydes, ketones, carboxylic acids, esters), 1460.52 cm<sup>-1</sup>, 1376.09 cm<sup>-1</sup> (C-H, alkanes), 1221.47 cm<sup>-1</sup>, 1163.38 cm<sup>-1</sup>, 1118.88 cm<sup>-1</sup> (C-O, alcohol, acids, ethers, carboxylic acids, esters.), 770.90 cm<sup>-1</sup>, 722.88 cm<sup>-1</sup> (C-H, alkanes).

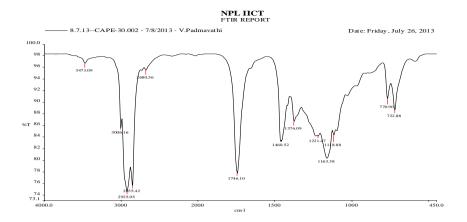


Fig 1: FT-IR of cape 30.

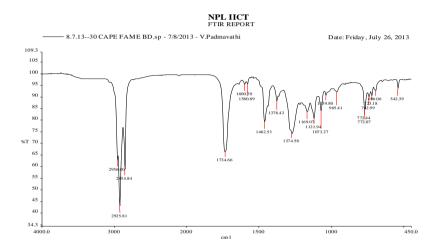
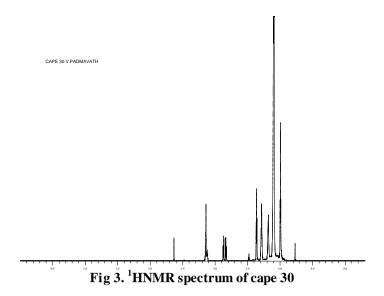


Fig 2: FT-IR FAME of cape 30.

## FIR: FAME CAPE 30

FT-IR of the compound displayed bands at 2956.36 cm<sup>-1</sup>, 2925.86 cm<sup>-1</sup>, 2855.09 cm<sup>-1</sup> (C-H, alkanes), 1734.45 cm<sup>-1</sup> (C=O esters.), 1378.23 cm<sup>-1</sup>, 1462.197 cm<sup>-1</sup> (C-H, alkanes), 1274.62 cm<sup>-1</sup>, 1169.06 cm<sup>-1</sup>, 1122.43 cm<sup>-1</sup>, 1073.67 cm<sup>-1</sup> (C-O, esters.), 1039.67 cm<sup>-1</sup> (methylene > CH<sub>2</sub> stretch), 966.06 cm<sup>-1</sup>, 772.30 cm<sup>-1</sup>, 743.38 cm<sup>-1</sup>, 723.85 cm<sup>-1</sup> (C-H, alkanes).



<sup>1</sup>HNMR spectrum of cape 30: (5.50-5.20) (ppm) [due to unsaturated acids. CH=<u>CH</u>: olefinic protons, the SN-2 protons (attached to the centre carbon; CHO) of the glycerol backbone of the triacylglycerols causes the small cluster of peak at 5.25 ppm]; (4.20-4.10,4.40-4.20) (ppm) SN-1 OR SN-3 [unsaturated fatty acids -<u>CH</u>-OCOR, (attached to the two terminal carbons; CH<sub>2</sub>O) of the glycerol back bone of the triacylglycerols]; (2.80-2.74) (ppm) [*bis*-allylic protons, *i.e.* protons attached to carbons situated directly next to two C=C double bonds, -CH=CH-<u>CH</u><sub>2</sub>-CH=CH-] (2.40-2.25) (ppm) [protons on

the second carbon in the fatty acid chains -O-C(=O)-CH2-]; (2.10-1.90) (ppm) [allylic protons, *i.e.* protons attached to H=CH-. carbons next to C=C double bonds, -CH<sub>2</sub>-CH<sub>2</sub>-CH=CH-.] (2.10-1.90) ppm) [allylic protons, *i.e.* protons attached to H=CH-. carbons next to C=C double bonds, -CH<sub>2</sub>-CH<sub>2</sub>-CH=CH-.] (1.75-1.50) (ppm) [methylene protons: (CH<sub>2</sub>)n-], (1.40-1.00) (ppm) [all fatty acids -(CH<sub>2</sub>)<sub>n</sub>-], (0.95-0.80) (ppm) terminal methyl protons -(CH<sub>2</sub>)<sub>n</sub>-CH<sub>3</sub>; the small peaks. J = 6.043; J = 6.043; J = 5.288; J = 11.331; J = 7.554; J = 15.108.

Table 1: Detailed <sup>1</sup>HNMR spectrum of cape 30.

_	Table 1: Detailed "HNMR spectrum of cape 30.										
S.NO	PEAK	$\delta$ (ppm) Values	Assignment of compound								
1		(5.50-5.20) δ(ppm)	unsaturated acids -CH= <u>CH</u> - olefinic protons, the SN-2 protons (attached to the centre carbon; CHO) of the glycerol backbone of the triacylglycerols causes the small cluster of peak at 5.25 ppm.								
2		(4.20-4.10, 4.40-4.20) $\delta$ (ppm)	SN-1 OR SN-3 unsaturated fatty acids - $\underline{CH}$ -OCOR, (attached to the two terminal carbons; $CH_2O$ ) of the glycerol back bone of the triacylglycerols.								
3		(2.80-2.74) <b>δ</b> (ppm)	<i>bis</i> -allylic protons, <i>i.e.</i> protons attached to carbons situated directly next to two C=C double bonds, -CH=CH-CH <sub>2</sub> -CH=CH								
4		(2.40-2.25) δ (ppm)	protons on the second carbon in the fatty acid chains -O- $C(=O)$ - $CH_2$								
5		(2.10-1.90) δ (ppm)	allylic protons, <i>i.e.</i> protons attached to H=CH carbons next to C=C double bonds, -CH <sub>2</sub> -CH <sub>2</sub> -CH=CH								

6	(1.75-1.50) <b>δ</b> (ppm)	methylene protons –(CH <sub>2</sub> )n
7	(1.40-1.00) δ(ppm)	fatty acids –(CH <sub>2</sub> ) <sub>n</sub> -
8	(0.95-0.80) <b>δ</b> (ppm)	terminal methyl protons - $(CH_2)_n$ - $CH_3$ ; the small peaks

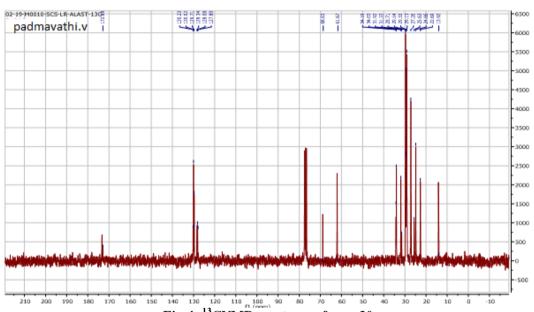


Fig 4: <sup>13</sup>CNMR spectrum of cape 30.

<sup>13</sup>CNMR(CDCl<sub>3</sub>): 172.88, 130.23, 130.02, 129.71, 128.34, 128.08, 127.89, 68.63, 61.67, 34.19, 34.03, 31.92, 31.53, 29.71, 29.54, 29.33, 29.13, 27.20, 25.63, 24.65, 22.69, 13.92.

#### GC

The fatty acid composition was determined by GC and the results are given below, the acid value and it was found that it is varied from 0.52-0.31.

For the analysis, the method followed for gas chromatography (GC) is as follows:

GC apparatus: Agilent Technologies, USA., Model no  $\rightarrow$ 6890 N, Column  $\rightarrow$  DB-225 [(50% cyanopropylphenyl)-dimethylpolysiloxane, 30M  $\times$  0.250mm 1D  $\times$  0.25 µm film thickness. inlet temperature  $\rightarrow$  230°C, oven prog  $\rightarrow$  160°C (2min) - 5°C/min-230°C (20min), detection temperature  $\rightarrow$  270°C, HY  $\rightarrow$  30ml/min, air  $\rightarrow$  300ml/min, inlet type  $\rightarrow$  split/split less, detection type  $\rightarrow$  FID, soft ware  $\rightarrow$  GC chem. station,

A.10.02 (1757). The fatty acid composition was determined by GC and the results are given below, the acid value and it was found that acid value varied from 0.52-0.31 (Fig. 5).



Fig. 5: GC of fatty acids in S. a L.f nut oil.

Table 2: Fatty acid composition of Semecarpus anacardium L.f. nut oil.

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Fatty acid	12:0	14:0	15:0	16:0	16:1	17:0	18:0	18:1	18:2	18:3	18.3	20:0	20:1	20:2	20:3	22:0	22:1	24:0	24:1
Wt%	0.54	1.08	0.4	16.1	0.4	0.2	9.8	45.8	0.8	7.3	0.5	3.1	0.4	0.6	3.2	4.0	1.03	0.95	2.6

Table 3: Properties of Semecarpus anacardium L.f.nut oil cape 30.

Properties	Semecarpus anacardium L.f. nut extracted oil					
acid value (mg KOH/g)	0.52					
iodine value	80.5					
density(mg/Lit)	0.872					
kinematics/viscosity	4.8 capillary suction time (cst)					
peroxide value	1.2					
saponification value (KOH/g)	165					
unsaponification value	sterols					
Phosphorous content	9.8					

After knowing the above acid value,s the sample was submitted for physical propertie.

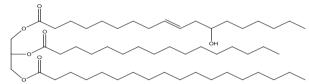


Fig. 6: (E)-2-(Palmitoxy)-3-(stearoyloxy)propyl 12-hydroxyocadec-9-enoate.

(**18:1** 45.8%, **16:0** 16.2% and **18:0** 18.99%). Basing on this lipid % and FT-IR, <sup>1</sup>H NMR, <sup>13</sup>C NMR, MASS, GC and GC-MS, CHN analysis.name of the compound is (E)-2-(palmitoxy)-3-(stearoyloxy)propyl 12-hydroxyocadec-9-enoate, chemical formula: C55H104O7, m/z 876.78, elemental analysis C,75.3; H,11.9; O,12.8.

## Trans esterification process<sup>[27-31]</sup>

In our investigation, *Semecarpus anacardium* nut oil cape 30 had found to have a free fatty acid (0.52) hence,

alkaline *trans* esterification was done by using anhydrous methanol at a molar ratio of 6:1 and 3g/Liter of sodium hydroxide as catalyst. The processor was stirred at 600 rpm and at a temperature of 60°C for 2 hours, after which the mixture was poured into a decanter and allowed to settle for 3 hours so that the reaction can be driven to completion. By following mechanism as in Figure 2 and found that the mixture has been separated in to its corresponding methyl ester.

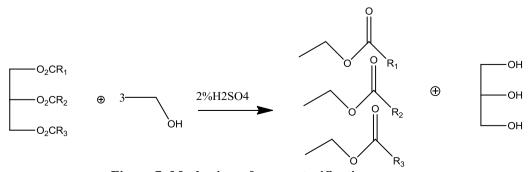


Figure 7: Mechanism of trans-esterification process.

(E)-2-(palmitoxy)-3-(stearoyloxy)propyl methyl ester glycerol 12-hydroxyocadec-9-enoate.

The glycerol at the bottom was drained off by gravity. The excess methanol in the ester was removed by using a flash rotary evaporator. The impurities were removed from the methyl ester by washing with distilled water of

volume ratio 3 to 1 three times. Finally, the washed methyl ester was dried by passing it through anhydrous sodium sulphate (Na<sub>2</sub>SO<sub>4</sub>).

Table 4: Comparison of Semecarpus anacardium L.f. nut extracted biodiesel with the standard diesel values.

Test property	Fatty acid methyl esters of S.a nut oil	Diesel (B100) blend stock ASTM <sup>1)</sup> D6751				
methyl ester conversion	99.010349%	94.5 - 100%				
acid value (mg KOH/g)	0.31	0.50 max mg KOH/g				
iodine value	80.5	115 max				
density(mg/Lit)	0.8720	0.875 - 0.90				
viscosity (cst) @40°C	5.96 cst	$1.9 - 6.0 \text{ mm}^2/\text{S}$				
saponification value(KOH/g)	165					
unsaponification value	sterols	not more than 1.5%				
phosphorous value	0.001 max	0.001 max				
kinematics η 40°C	5.96 cst	1.9 - 6.0 mm <sup>2</sup> /S				
copper strip corrosion 3 h 5 °C at max	1 a	1a				
flash point	140	130 - 170 <sup>o</sup> C				
pour point	-5	-15 to 10				
cloud point	-7	-3 to 12 <sup>0</sup> C				
heating value	37.12	42.9				
cetane number	48.99	47 to 65.				

1) ASTM: American Society of Testing Materials.

## RESULTS AND DISCUSSION

As the production of biodiesel from edible oils is currently much more expensive than diesel fuels due to relatively high cost Renewable energy such as biodiesel has the potential to replace petroleum derived transportation fuel in the future. The production s of Semecarpus anacardium L.f nuts were discussed by the transesterification of triglycerides of SA Nut Oil to yield Fattyb Acid Mthyl ester, and glycerine as a by-product and the Spectral Datas of Semecarpus anacardium L.f Nut Oil Which was presented in Table 1,2and Figs 1-5 and and their Deravative (FAME)Determine by Trans esterification process (fig 7) compared with the Varous Oil Spectral data & Various properties of Semecarpus anacardiun L.f. nut oil were determined by using standard methods and results are presented in Table 3. Properties of Semecarpus anacardium L.f. nut oil methyl esters were determined experimentally to ascertain their suitability as diesel fuel. The properties of Semecarpus anacardium L.f. nut oil methyl esters have been compared with the properties of biodiesel and petro diesel in table4. The fuel properties of Semecarpus anacardium L.f. nut oil methyl esters were within specifications. The properties of its oil, such as its saponification value, iodine value, and the amount of its free fatty acid content were 165, 80.5 and 0.52, respectively. Eventually, the methyl ester of its crude oil,

which was produced through alkali transesterification reaction with methanol alcohol and NaOH as catalyst was experimented and its physicochemical properties were determined. All of the determined characteristics, namely, density (0.8720 g/mL), kinematic viscosity (5.96 mm 2/s at 40 °C), flash point (140°C), cloud point (-7°C), pour point (-5°C), cetane index (48.99) meet the two accepted biodiesel standards (*i.e.* ASTM D6751 and EN 142140). Therefore, according to the results, *Semecarpus anacardium* L.f. which was not reported earlier [31-38], and feedstock in producing biodiesel for the first time. Among the very few resent investigations then on this field ours is the first report on the FAME *Semecarpus anacardium* L.f. nut oil (biodiesel).

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