

Comparative evaluation of polar and non-polar solvents in microwave-assisted extraction of cashew nut shell liquid

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Cashew nut shell liquid (CNSL) is a renewable, underutilised bioresource that offers a sustainable alternative to petrochemical-derived phenolic compounds. This study employs an open-vessel microwave-assisted extraction (MAE) method to extract CNSL from cashew nut shells (CNS) using a range of polar and non-polar solvents, aiming to enhance extraction efficiency and minimise environmental impact. The extraction process was conducted at microwave power levels ranging from 30 W to 300 W, with an extraction time of 30–40 minutes, using 150 mL of solvent. A Comparative analysis reveals that mid-polar and ketonic solvents achieve the highest yield (33–35%), beating the conventional extraction techniques in terms of selectivity, efficiency, and sustainability. Physicochemical characterization of the extracted CNSL showed pH values of 3.52–5.1, density of 0.86–0.98 g/cm³, moisture content of 0.52–1.04%, viscosity of 1780–3750 cP at 30°C, hydroxyl value of 86.55–96.11 mg NaOH/g, acid value of 12.52–16.01 mg NaOH/g, saponification value of 46.9–60.32 mg NaOH/g, and iodine value of 49.03–62.21 g I₂/100 g. High-Performance Liquid Chromatography (HPLC) analysis confirmed the characteristic phenolic composition of CNSL. These findings demonstrate that open-vessel MAE is a rapid, scalable, and environmentally benign approach for CNSL extraction, supporting the valorisation of agricultural waste and promoting CNSL as a viable feedstock for bio-based industrial applications.

Keywords: Anacardic acid, Cardanol, Cardol, CNS, CNSL, MAE

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Introduction

In the 21st century, most materials used in daily life are produced from petrochemical compounds derived from crude fossil fuels. The pharmaceutical, dye, polymeric, agricultural, and other bulk and chemical industries extensively use these compounds. However, the main disadvantage is that they are extremely expensive, harmful to the environment, and harmful to human health^{1–3}. The field of "Green Chemistry" was created in response to widespread concerns about the negative effects of increasing consumption, which include global warming and the depletion of these compounds. Green chemistry focuses on designing products and processes that reduce the use and production of hazardous materials in industry⁴. Researchers are now seeking natural oil based chemicals that are more affordable, pose less risk to human health, and are ecologically beneficial. They are derived from a variety of renewable sources, including flowers, seeds, and plant extracts. These chemicals have been recognised as a viable substitute

with significant economic, availability, and environmental advantages⁵. In addition to being used in the food industry to produce edible goods, oils and lipids are also key components of many non-food products, such as waxes, cosmetics, varnishes, adhesives, lubricants, soaps, synthetic resins, and biodiesel^{4,6}. To separate, identify, and use these chemicals, extraction is a crucial step. One of the most frequently encountered waste products from the agroindustry is cashew nut shells (CNS), which pique researchers' curiosity due to their 1/8-inch (3 mm) thick shells, soft honeycomb structure, and dark brown viscous liquid known as Cashew nut shell liquid (CNSL)^{5,7–12}. The CNS has been used to extract CNSL using various extraction techniques. Approximately 15–40% of CNSL is found in the CNS. CNSL is a major source of naturally occurring phenols. CNSL is frequently classified into two categories: Technical CNSL and Natural CNSL, depending on the extraction method used. CNSL extracted using organic solvents contains (70%) anacardic acid, (18%) cardol, (5%) cardanol, and trace amounts of 2-methyl cardol, and is called Natural CNSL^{12–14}. In contrast heat-extracted CNSL

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(Technical CNSL) contains a significant amount of cardanol (60–80%), cardol (10%), and polymeric material (20%). The reason for this is that anacardic acid is decarboxylated at higher temperatures, producing cardanol (Fig. 1)¹².

Anacardic acid, cardol, and cardanol are considered to be extremely valuable and versatile raw materials because these phenolic compounds exist as a mixture of saturated, mono, di, and trienes in the C15 alkyl side chain at the meta position (Fig. 2)¹⁴.

Traditional extraction methods, although widely used, often require long processing times (5–6 hours), large volumes of solvent, and may degrade heat-sensitive components. In contrast, microwave-assisted extraction (MAE) offers a faster and more energy-efficient alternative. However, the effectiveness of MAE depends heavily on operating conditions, including microwave power, extraction time, solvent type, and solvent polarity. In particular, there is a noticeable lack of studies that directly compare polar and non-polar solvents under the same MAE conditions. The microwave heating behaviour is strongly influenced by solvent polarity and dielectric properties; therefore, comparing different types of

solvents in a structured manner is crucial. Such a comparison can help improve extraction yield, reduce energy consumption, and tailor CNSL composition to specific industrial needs. At the same time, evaluating physicochemical parameters such as viscosity, density, acid value, hydroxyl value, iodine value, and saponification value is important to determine product quality, structural characteristics, and reactivity. These properties directly influence CNSL's performance in applications such as resins, coatings, adhesives, friction materials, and polyurethane systems. Therefore, this study aims to optimise MAE conditions for extracting major phenolic constituents from CNSL using both polar and non-polar solvents and to systematically assess their physicochemical properties in order to better understand their structure–property relationships and industrial potential.

Materials and Methods

Sample collection and experimental setup

CNS of *Anacardium occidentale* L. were obtained from Gujarat Cashew Industry (Valsad). The experiment was set up with a round-bottom flask, a standard condenser, a heating mantle with a temperature range of 0–200°C, a modified microwave oven from Mistry Electricals, Bilimora, India, and a simple distillation unit for solvent recovery from extracted CNSL. The solvents and chemicals were obtained from ATUL Ltd., Valsad, India, which also carried out quantitative and qualitative analyses of the isolated components.

Extraction of CNSL from CNS

To prevent contamination of the extracted CNSL, the CNS underwent cleaning, drying, shelling, and grinding procedures (Fig. 3). During cleaning, foreign matter, consisting of sand, stones and dried apple was removed by washed the raw nuts with water for 3–4 times. After cleaning, the nuts were dried to prevent fungal growth, this was carried out using either artificial drying (oven drying) and sun drying methods. After drying, shelling was performed to separate cashew kernels from shells, either manually or mechanically. Finally, grinding process was conducted, which is extremely important because extraction depends on the size of the crude particles. Larger particles will be poorly extracted, whereas smaller particles will be extracted more efficiently.

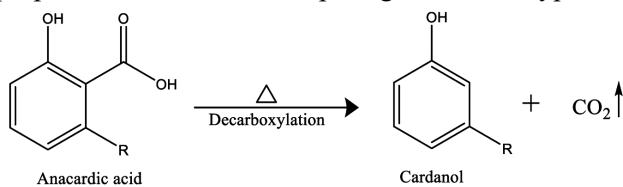


Fig. 1 — Decarboxylation of anacardic acid to cardanol^{12–14}.

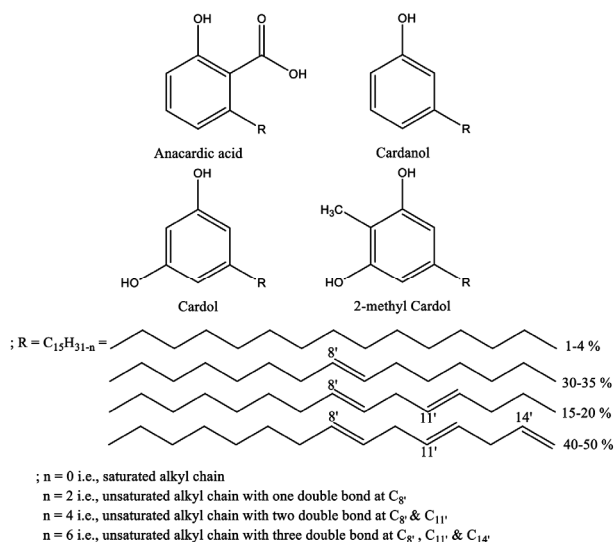


Fig. 2 — Chemical composition of CNSL.

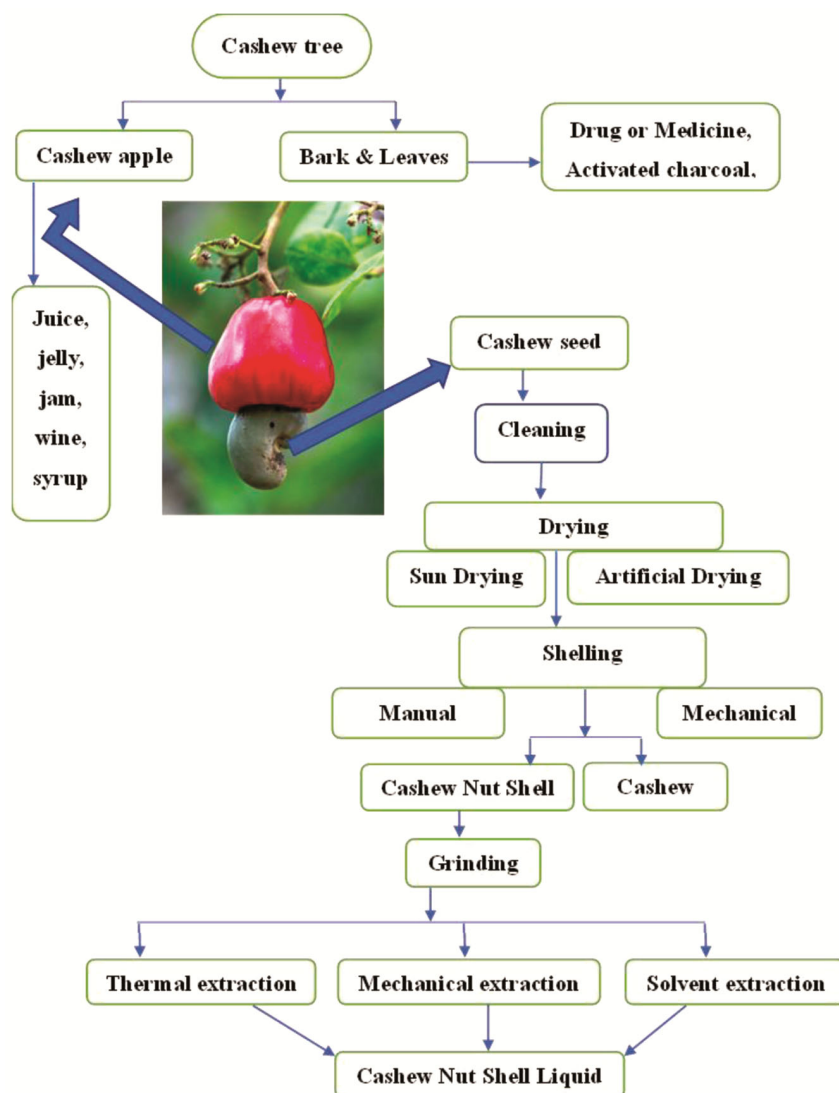


Fig. 3 — Flow diagram of the cashew nut processing process.

Microwave assisted extraction (MAE)

MAE has been effectively applied in several domains of chemistry¹⁵. The fundamentals of the MAE process differ from those of conventional methods like maceration because extraction is the final result of modifications in the cell structure induced by electromagnetic waves. Heat and mass gradients are two transport phenomena that work synergistically to accelerate the process and achieve a high extraction yield in MAE. In simple terms, microwave extraction—which combines microwave and conventional solvent extraction—is commonly referred to as microwave-assisted extraction (MAE)¹⁵. MAE is the technique of heating the solvent and plant tissue with microwaves to enhance the kinetics of extraction.

The extraction technique known as MAE uses microwave radiation to assist in separating analytes from a sample matrix into a solvent. The disruption of hydrogen bonds caused by the microwave electromagnetic field triggers dipole rotation in the molecules, which promotes the migration of dissolved ions and enhances solvent penetration into the sample matrix. Because it reduces extraction time, uses less solvent, and allows for temperature and pressure control during the extraction process, MAE has been considered superior to conventional solvent extraction techniques¹⁵. Microwave extraction has major disadvantages: to eliminate solid residues, an additional process (filtration or centrifugation) must be performed. When the targeted molecules are volatile, the efficiency of microwave extraction can be

extremely poor, and it is ineffective for heat-sensitive compounds because it may degrade them¹⁶⁻¹⁹. MAE can be carried out in closed or open extraction vessels. Closed vessels are suitable for high-temperature extractions, while open vessels are used for low-temperature extractions at atmospheric pressure²⁰.

In this research work, we used an open-vessel microwave with different polar and non-polar solvents, and the MAE method was used to extract CNSL from CNS. First, the round-bottom flask was loaded with 150 mL of solvent. Then, 50 g of crushed CNS powder was added to the flask. The CNS-to-solvent ratio was 1:3. Microwave power was set to 30–300 W to maintain gentle reflux conditions and prevent thermal degradation of CNSL. In an open-vessel reflux system, the extraction temperature is governed by the boiling point of the solvent rather than a direct microwave temperature controller. Cold water was circulated in and out of the extraction setup to condense the vapour generated. Once the solvent in the setup reached its boiling point, the reflux was continued for only 30–40 minutes. This heating and cooling process was maintained throughout the extraction. Throughout the extraction procedure, the solution in the round-bottom flask was filtered to remove the solid residue. The filtrate that remained

was transferred back into the round-bottom flask, and the distillation methodology was used to remove the solvent. All the extracted CNSL samples were dark brown in colour, exhibited a viscous consistency, and were stored in an airtight bottle at ambient temperature for a period of one year.

To evaluate the isolation and proportion of the major phenolic compounds present in CNSL, various methods have been extensively reviewed and described in previous literature. Here, we applied the method described in earlier studies^{13,21} (Fig. 4).

Isolation of anacardic acid

The isolation of anacardic acid from the extracted CNSL was carried out by precipitation as M+2 metal anacardate using metal hydroxide, such as Calcium hydroxide $[\text{Ca}(\text{OH})_2]$, Barium hydroxide $[\text{Ba}(\text{OH})_2]$, and Magnesium hydroxide $[\text{Mg}(\text{OH})_2]$. The precipitate of metal anacardate was filtered, washed it with acetone, and dried it in an oven at 75°C for 2 hours. The ketonic filtrate was preserved for the isolation of cardol and cardanol. The metal anacardate was subsequently treated with distilled water and concentrated hydrochloric acid. The resulting solution was further extracted 2–3 times with n-hexane, the organic layer was collected, and the n-hexane was

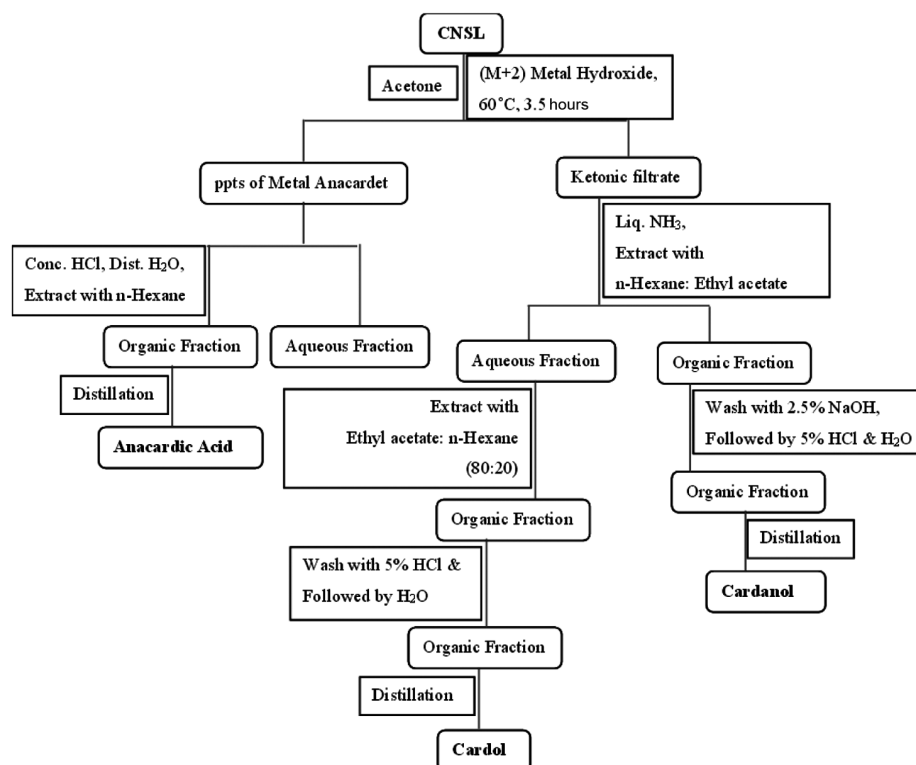


Fig. 4 — Flow diagram of process for isolation of Anacardic acid, Cardol and Cardanol.

distilled off. The residue obtained was a slightly brownish precipitate (26 g, i.e., 52%), called anacardic acid.

Separation of cardanol and cardol

The ketonic filtrate was first concentrated and then treated with the liquor ammonia solution, which was subsequently extracted with n-hexane: ethyl acetate (98:2) [3 x 100 mL]. The combined organic layer was washed with 2.5% NaOH solution, followed by 5% HCl solution and distilled water. The resulting solution was concentrated to yield cardanol (7 g, i.e., 14%).

The aqueous ammonia layer was extracted with n-hexane: ethyl acetate (20:80) (100 mL). The organic layer was washed with 5% HCl solution, followed by distilled water. The resulting solution was concentrated to yield cardol (0.75 g, i.e., 1.5%).

High performance liquid chromatography (HPLC)

The HPLC analysis of samples was conducted on Shimadzu high performance liquid chromatography (model: LC-2030) with the use of inertSustain C18 analytical column (High Purity ES Silica Gel, 5 μ m particle size, 350 m²/g surface area, 100 Å pore size, 0.85 mL/g pore volume, and a pH range 1-10). A mixture of acetonitrile:water:acetic acid (80:20:1) was used as the mobile phase at a flow rate of 1.80 mL/min, and the absorbance was monitored at 280 nm using UV-Visible detector, with the following gradient profile, which is suitable for CNSL Table 1. For sample preparation, weigh 25 mg of the sample was weighted, 5 mL of acetonitrile was added, the solution was sonicated for 2 minutes, and 20 μ L was injected. The total running time was 30 minutes.

Oil yield determination

At the end of the procedure of extraction, the CNSL yield was calculated as a percentage by simple calculation of the weight of extracted oil (g), divided by the weight of crushed CNS powder⁹.

$$\text{Percentage oil yield} = \frac{\text{Wt. of Extracted oil (g)}}{\text{Wt. of crushed CNS powder(g)}} \times 100$$

Table 1 — Gradient profile for CNSL

Time (minute)	80% Acetonitrile	20% Water + 1% Acetic acid
0.0	60	40
5.0	70	30
10.0	80	20
15.0	90	10
20.0	90	10
25.0	60	40
30.0	60	40

Characterisation of CNSL

The Karl Fisher moisture analyser (915 KF Ti-Touch, ATUL, Valsad) was used to determine moisture content after calibration. Density is defined as mass per unit volume.

$$\text{Density} = \text{Mass} \div \text{Volume}$$

A digital pH meter (Optima GOLD 533, ATUL, Valsad) was used to measure the sample's pH. The pH 4.0, 7.0, and 9.2 buffer solutions were used to calibrate the pH meter. The viscosity of the sample at 30°C was measured using a Brookfield viscometer with a small-sample adapter system (Firsers Prodig LR/Lamy Rheology, ATUL, Valsad). For Ash content determination, a porcelain crucible was weighed. A sample weighing between 1.0 and 1.5 g was placed in the crucible. The crucible containing the sample was placed in a muffle furnace and calcined at 600–650°C for 2 hours. The crucible with the contents was carefully removed, allowed to cool in a desiccator, and then weighed. The ash content was determined using the equation below.⁹

$$\% \text{ Ash} = \frac{(\text{Wt. of crucible + ash}) - \text{Wt. of crucible}}{\text{Wt. of sample}} \times 100$$

For the chemical analysis, all determinations were performed using standard procedures outlined by the Association of Official Analytical Chemists (AOAC). An acid value (AOAC 940.28 and ISO 660.2009) is a chemical quantity that is used to measure how acidic a certain chemical compound is. It is the amount of base, normally expressed in milligrams of potassium hydroxide (KOH) or sodium hydroxide (NaOH), needed to neutralise the acidic components in 1.0 g of a sample. The quantity of carboxylic acid groups in a chemical compound is expressed as the acid number. For the determination, 1.0 g of the sample was weighed into a flask and 20 mL of freshly neutralised methanol was added. The mixture was boiled for approximately 5 minutes. Then, added 2-3 mL of phenolphthalein indicator solution was added, and the mixture was titrated against 0.1 N KOH/NaOH solution with vigorous shaking until a pink colour appeared. The equations given below were used to compute the acid value (AV) and free fatty acid (FFA), respectively⁹.

$$\text{AV} = \frac{\text{Molecular wt. of (KOH/NaOH)} \times \text{Normality of (KOH/NaOH)} \times \text{Burette reading (mL)}}{\text{Wt. of sample (g)}}$$

$$\text{FFA} = \frac{\text{AV}}{2}$$

The term "saponification value" (SV) refers to the quantity of KOH (mg) required to saponify 1.0 g of an

oil sample under the specified conditions. To measure the saponification value (AOAC 920.160), two flasks were used: flask A and flask B. In flask A, 1.0 g of the oil sample was taken and 50 mL of 4% methanolic KOH solution was added; the flask was then refluxed in a water bath for 30 minutes. The mixture became transparent and clear, indicating that saponification was complete. The flask was cooled and titrated it against a 1.0 N HCl solution using phenolphthalein indicator; the endpoint changed from pink to colourless. The volume of acid used was recorded as mL of HCl required by the sample (V_s). In flask-B, follow the same process without the oil sample. The volume of acid used was recorded as mL of HCl required by blank (V_B). Make sure that the reflux time of both flasks was kept the same. The SV was calculated using the equation given below⁹.

$$SV = \frac{\text{Molecular wt. of KOH} \times \text{Normality of HCl} \times (V_B - V_s)}{\text{Wt. of sample (g)}}$$

The amount of KOH or NaOH milligrams needed to neutralise the acetic acid absorbed during the acetylation of 1.0 gm of a chemical compound containing free hydroxyl groups is known as the "hydroxyl value" (HV). To measure the hydroxyl value (AOAC 969.21), take two flasks, flask A and flask B. In flask A 1.0 g of oil sample was taken and 20 mL of acetic anhydride: pyridine (1:3) solution was added. The flask was refluxed in water bath for 30-45 minutes, the flask was Cooledown and 50 mL of distilled water and 2-3 mL of phenolphthalein indicator solution was added to the mixture and titrated against 1.0 N KOH/NaOH solution with vigorous shaking, end point changed from white turbidity to pink coloured solution. The volume of base used was recorded as mL of KOH/NaOH required by the sample (V_s). In flask B, follow the same process without an oil sample. The volume of base used was recorded as mL of KOH/NaOH required by blank (V_B). The HV was calculated using the equation given below.

$$HV = \frac{\text{Molecular wt. of (KOH/NaOH)} \times \text{Normality of (KOH/NaOH)} \times (V_B - V_s)}{\text{Wt. of sample (g)} + AV}$$

The mass of iodine in g that is used by 100 g of a chemical substance is known as the "iodine value" (IV). The degree of unsaturation in oils can frequently be determined using IV. In fatty acids, unsaturation occurs mainly as double bonds, which are very reactive towards halogens, such as iodine in this case. Therefore, the higher the iodine value, the more unsaturations are present in the oils. The "Hanus

method" is one of the most often used techniques for determining an oil's iodine value (AOAC 920.159). Take two flasks, flask A and flask B. In flask A, 1.0 g of oil sample was taken, 10 mL of chloroform and 20 mL Hanus solution were added (it is prepared by dissolving 18.2 g of iodine in 1 Liter of glacial acetic acid and then adding 3 mL of bromine water to increase the halogen content). The flask was closed with the help of a glass stopper, and kept in the dark for 30 minutes. After 30 minutes, 10 mL of 10% KI solution and 50 mL of distilled water were added. The mixture was titrated with 0.1 N Sodium thiosulfate. When a light-yellow solution was obtained, starch indicator was added; forming a blue solution. Continued the titration till the blue colour disappeared, record burette reading as (V_s). In flask B, followed the same process without an oil sample. Record burette reading as (V_B). The IV was calculated using the equation given below.

$$IV = \frac{\text{Molecular wt. of iodine} \times \text{Normality of sodium thiosulphate} \times (V_B - V_s)}{\text{Wt. of sample (g)}}$$

Results and Discussion

The extraction process in the solvent extraction method depends on the leaching of the solid sample into a liquid, which depends on the polarity of the material to be extracted. The physicochemical properties reported in the literature are summarized in Table 2. In this research work, the open-vessel MAE method was used with different solvents. The results of the extraction work are given in Table 3 and Fig. 5. A comparison of the percentage yields achieved by MAE using various solvents was carried

Table 2 — Physicochemical properties of CNSL in the literature

S. No.	Parameter	Observation	Reference
1	Yield (%)	15–40	24
2	Appearance & Nature	Viscous liquid	14, 23
3	Colour	Reddish dark brown	24
4	pH	5.2–5.7	14, 23
5	Density (g/cm ³)	0.92–0.94	14, 23
6	Viscosity (30°C) (Poise)	47.2–59.3	14, 23
7	Moisture (%)	3.9–6.0	24
8	Ash content (%)	1.22 ± 0.3	24
9	Saponification value (mg KOH/g)	47–58	10
10	Iodine value (g/100 g)	215–235	25
11	Acid value (mg KOH/g)	12.1–15.4	23
12	Free Fatty acid (mg KOH/g)	6.1–7.8	24

moisture content is about 3.9-6.0%²¹, and the ash content is 1.22±0.3%²¹. The acid, free fatty acid and saponification value range from 12.1-15.4²¹, 6.1-7.8²¹ and 47-58⁹ (mg KOH/g), respectively. The iodine value is approximately 215-235 (gI/100 g)²¹.

The physicochemical properties that are crucial to industrial applications are shown in Table 3. These properties provide information on the suitability of CNSL for a variety of applications, including polymer synthesis, coatings, adhesives, and lubricants. This dataset plays an important role in the exploration of sustainable alternatives to petrochemical-derived compounds in industry. A comprehensive examination of the data highlights its industrial relevance. The acidic nature of CNSL with a pH range of (3.52-5.1) is crucial for its reactivity in polymerisation reactions, especially in phenolic resin production. Lower pH values are advantageous for anti-corrosive coatings and speciality chemical formulations. The density (0.86–0.98 g/cm³), which is close to that of standard petroleum-based resins, enables easy substitution without extensive formulation adjustments. Moisture Content (0.52–1.04%) was low making it ideal for long-term storage and highly pure. The viscosity (1780–3750 cP at 30°C) can be tailored to the chosen solvent, enabling industry-specific optimisation. The

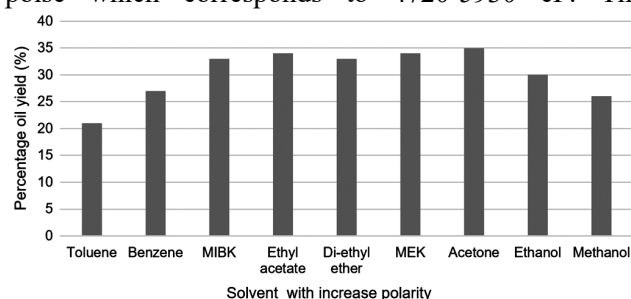


Table 3 — Physicochemical properties of extracted CNSL at 40–110°C and 1 atm

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hydroxyl value (86.55–96.11 mg NaOH/g) is a key Factor for polymer synthesis. Hydroxyl groups in CNSL play a critical role in the formation of polyurethane and epoxy resins by participating in crosslinking reactions. A higher hydroxyl value indicates strong potential for polyurethane foams and coatings. The acid value (12.52–16.01 mg NaOH/g) & free fatty acid value (6.26–8.01 mg NaOH/g) influence functional performance and determine the reactivity of CNSL in polymerisation and esterification reactions. The saponification value (46.9–60.32 mg NaOH/g) indicates the potential for CNSL-derived surfactants and emulsifiers. The iodine value (49.03–62.21 gL/100 g) reflects the degree of unsaturation and affects CNSL's drying properties in coatings and polymer applications. Additionally, these compounds have various medicinal applications. Anacardic acid has been reported to exhibit antimicrobial, antioxidant, and anticancer activities due to its salicylic acid moiety and long hydrophobic side chain, which enhances membrane interaction²²⁻²⁴. Cardol has demonstrated cytotoxic and antibacterial properties, while cardanol has shown anti-inflammatory and industrial bioactive applications²²⁻²⁴.

In the present study, MAE of CNSL was carried out using different polar and non-polar solvents, and all physicochemical properties were measured using AOAC methods. Variations in density, viscosity, and moisture content were due to the extraction technique and the operating conditions employed during the experiment. The greater variation in iodine value is attributed to the varying degrees of unsaturation in the C15 alkyl side chain at the meta position.

High performance liquid chromatography (HPLC)

In the above HPLC chromatograms, the X-axis represents the retention time, while the Y-axis shows the intensity corresponding to the concentration of compounds eluting at that time. Each peak corresponds to a different component in the CNSL mixture. The height and area of each peak correlate with the amount of that compound. In Fig. 6, peaks appearing around 10–20 minutes correspond to various phenolic compounds such as cardanol, cardol, and anacardic acid commonly present in CNSL. In Fig. 7, the standard cardanol sample shows multiple peaks, including a prominent one around 10 minutes, indicating the presence of cardanol and other related compounds. The isolated cardanol sample shows fewer and sharper peaks, with a dominant peak at the same retention time (~10 minutes), indicating higher purity.

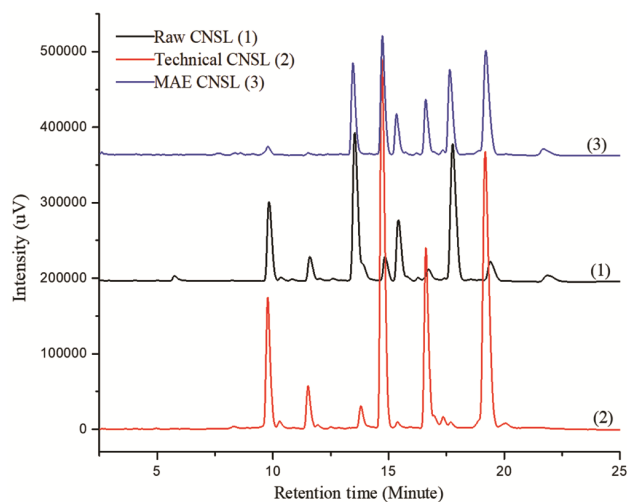


Fig. 6 — Comparison of HPLC analysis of Raw CNSL (1), Technical CNSL (2) and Microwave Extracted CNSL (3).

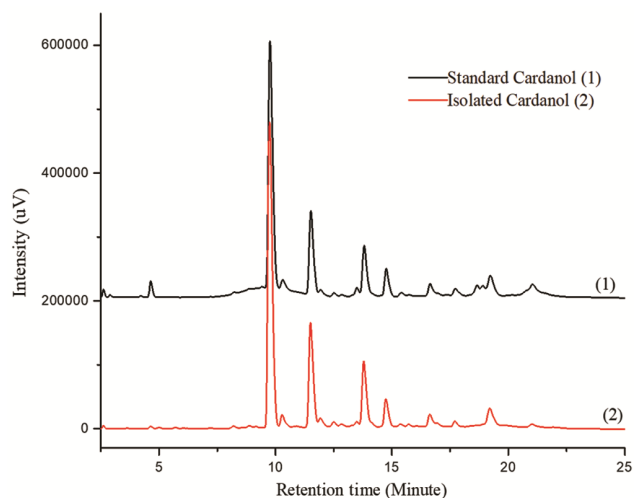


Fig. 7 — HPLC analysis of Standard Cardanol (1) and Isolated Cardanol (2).

Conclusion

This study confirms that MAE is a rapid, efficient, and sustainable method for recovering CNSL from CNS. Optimisation of solvent polarity showed that mid-polar and ketonic solvents achieved the highest extraction yield (up to 35%) within 30–40 minutes, significantly reducing extraction time and solvent consumption compared to conventional methods. The extracted CNSL exhibited suitable physicochemical properties— pH (3.52–5.1), lowest moisture Content (0.52–1.04%), acid value (12.52–16.01 mg NaOH/g), hydroxyl value (86.55–96.11 mg NaOH/g), iodine value (49.03–62.21 gL/100 g), saponification value (46.9–60.32 mg NaOH/g) and viscosity (1780–3750 cP). These characteristics demonstrate strong

potential as a bio-based feedstock for applications in polyurethane, epoxy and phenolic resins, coatings, adhesives, and speciality chemicals. Overall, MAE offers a green, scalable approach to CNSL extraction, supporting the development of sustainable materials and reducing dependence on petrochemical resources.

Conflict of interest

The authors declare that there is no conflict of interest regarding the publication of this manuscript.

AI use disclosure

The authors used ‘ChatGPT’ and ‘QuillBot’ AI tools for language assistance only for this manuscript.

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