REVIEW ON EXTRACTION AND ISOLATION OF CASHEW NUT SHELL LIQUID

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Abstract:

India is the largest producer and processor of cashews (*Anacardic occidental L*). Cashew nut shell liquid (CNSL) is a by-product from cashew nut processing. CNSL is a dark brown viscous liquid present inside a soft honey comb structure of the cashew nut shell. It contains phenolic compounds, mainly cardanol. Cardanol is a monohydroxyl phenol with a long carbon chain in the metaposition. It has the potential as a substitute for phenol in resin phenolic-base chemical products. The present review discusses various application of CNSL as well as the applications of constituents of CNSL. Various methods of extraction of CNSL are available in the literature. This review highlights various methods of extraction of CNSL and isolation of major constituents. The quantitative and qualitative analysis of CNSL is also presented in this review. Scope for future work is also discussed.

Key words: Cashew Nut Shell Liquid, Extraction, Cardanol

Introduction:

Cashew (*Anacardium occidentale*) is an important plantation crop of India and is one of the well known species of the Anacardiceae family.

India has the largest area under cashew (9.23 lakh ha) and stands as the second largest producer of cashew (7.00 lakh MT) in the world [1]. India has a comparative advantage in the production and processing of cashew nuts on account of its skilled labor force. Today, India is the largest processor and exporter of cashew in the world [2]. Maharashtra ranks first in the production (28.78 % of the country) and productivity of cashew nut in India. The fruit (cashew apple) can be used to make juice, jelly, jam,

wine and syrup and its waste has been proposed as a feedstock for protein-enriched animal feed or as a high fructose source [3,4,5,6].

The nut also represents a major source of alkenyl phenolic compounds [7]. The fruit of the tree consist of an outershell (epicarp), a tight fitting inner shell (endocarp), testa and kernel. Cashew kernels have the highest protein content among tree nuts (19.5%). This protein content matches soybean and is higher than peanut.

The cashew nut shell contains 25-30% dark reddish brown viscous phenolic liquid known as Cashew Nut Shell Liquid (CNSL)and is a by-product of the cashew industry. It is a naturally occurring substituted phenol which can take part in a variety of reactions. It is a cheap and renewable substance and can be employed for the manufacture of a multitude of useful products. It can replace phenol in many applications with equivalent or better results [8]. The CNSL destiny is either the overseas market if the price is convenient, or used internally as fuel, if the international price is low. Better extraction methods and isolation of the components make value addition.

Extraction of CNSL:

There are three main methods generally used in extracting cashew nut shell liquid from cashew nuts namely thermal, mechanical and solvent extraction. Solvent extraction in turn can be carried out by cold extraction, hot extraction by different solvent using Soxhlet extractor, Ultrasonication and super critical carbon dioxide extraction [9]. Cashew nut shell liquid was also extracted using vacuum pyrolysis. CNSL is classified into

Technical CNSL and natural CNSL depending upon the type of extraction. Technical CNSL is rich in Cardanol (also known as Decarboxylated CNSL) where as Natural CNSL is rich in Anacardic acid.

1. Thermal extraction:

a) Roasting method:

This is the traditional method of removing CNSL and it involves roasting the nut in drums or baths [10]. The roasting process not only removes the corrosive CNSL, but also makes the shell brittle, thereby aiding the cracking process. This method causes the loss of most of CNSL. In order to extract the retained CNSL, the nuts are roasted in baths at a temperature of 180–185 $^{\circ}$ C. This method recovers 85–90 % of the liquid. Scrapping the shell in a rotary apparatus with sand and steel wool, heated at 100–300 $^{\circ}$ C for 1 h and then roasted at 400–700 $^{\circ}$ C in an inert atmosphere to remove residual oil is also reported [11].

b) Hot oil bath method:

This is the most common method of commercial extraction of CNSL in practice nowadays. The technique can be different depending upon the raw material, which is either raw cashew nut shell or cashew nut. For the first, cashew nut shells were collected in the cylinder, where steam heating was applied at temperatures around 200-250 $^{\circ}$ C for 2-3 minutes. CNSL was then released from the shells and the process was repeated. This method yields CNSL of around 7-12 % by weight. For the latter, the raw nuts are passed through a bath of hot CNSL itself, when the outer part of the shell bursts open and releases CNSL. This method produces CNSL which was around 6-12 % by weight of nut.

c) Using Solar Cooker:

Cashew nut shell oil was extracted using concentrating solar cooker of 1.4 kW capacities and a diameter of 1.4 m. The focal point diameter of the cooker was 30m and was used to collect the reflected heat from reflector and achieved a temperature of 225-300 $^{\circ}$ C. Solar cooker of the following specifications was used for carrying out the extraction [12]. Specifications Type: Parabolic Solar Cooker Model: S K-14 Reflector: Aluminium sheet (reflectivity >75%) Diameter: 1.4 m Reflector area: 2.2 m² Temperature: 200–215 °C Thermal efficiency: 40 % Cooking capacity: 13 x 10-3 m³/hr

The authors could achieve CSNL to the tune of $550 \times 10^3 \text{ m}^3$ from 5 kgs of shells in 5 minutes. The composition of analysis of CNSL could not be performed. They carried out the proximate analysis of de-oiled cake. Low percentage of ash content and higher percentage of volatile matter and calorific value revealed the suitability of de-oiled cashew shell as a fuel for thermal application using the gasification route for better control and higher efficiency. Combustion of shell could be done either in a fixed bed combustion chamber which gave 50% efficiency or fluidized bed chamber which gave 85% efficiency [13].

2. Screw press method:

The raw cashew nut shells are put in the hydraulic press on screw pressing and then exert high pressure in order to release CNSL from shells. This method is rather straightforward and quick among others. Work was reported for extraction of CNSL by means of tapered compression screw, feeding rollers of transversal zigzag surface type and cylindrical casing with 2 mm diameter holes. By using screw speed of 7-13 rpm and feeding rate of 54-95 kg/h, the percentage of CNSL extracted was 20.65-21.04 percent, the percentage of CNSL purity was 85.53-87.8 wt % and the rate of extraction was 11.93-14.90 kg/h. However, the residue from this method still contained significant proportions of CNSL, around 10 to15%. Moreover, this method of extraction had higher levels of impurity, higher viscosity, lower thermo-oxidative stabilities and lower ebullition temperature. The CNSL obtained by this process contained 42% cardol, 47% anacardic acid and 3% Cardanol [14].

3. Solvent extraction:

This method gives off most of CNSL compared to other methods. The oil remains in the residue was less than 1% by weight [15]. Extraction solvents fall into two groups; those which are less dense than water and those which are denser. Commonly used extraction solvents which fall into the first group include diethyl ether (the

most common extraction solvent of all), ethyl acetate and hydrocarbons, such as light petroleum, hexane or toluene. The second group comprises chlorinated solvents, such as dichloromethane and chloroform, with dichloromethane being the preferred solvent because of its lower toxicity. However, chlorinated solvents do have a greater tendency to form emulsions than non chlorinated solvents. Emulsions are suspensions of small droplets of one immiscible liquid in another. Properties like Dielecrtic Constant, Boiling Point, Flammability and Toxicity of extracted CNSL varies with the type of solvent used for extraction [16].

Methods of extraction:

The higher percentage of anacardic acid in solvent extraction of raw cashew nut shells compared to extraction using supercritical carbon dioxide were reported [17]. Hence, if anacardic acid of high percentage is required, cold extraction using solvent extraction seems to be the possible feasible solution. Moreover, the CNSL obtained by cold solvent extraction preserves the original properties of the liquid. Anacardic acid percentage in steam roasted shells is less compared to raw cashew nut shells suggesting decarboxylation during the separation of kernel from the shells by steam roasting.

The percentage of unsaturated constituents in pentane extract of Indian raw cashew nut shells was higher as compared to all other sources. This compositional difference is reflected in the resultant technical CNSL (rich in cardanol) obtained from industrial processing. Lower percentage of % Cardols in Kenyan and Mozambique CNSL is observed. It can also be seen that there is compositional variation of CNSL obtained from different geographical locations [17].

Methods of Soxhlet extraction using different solvents were also available in the literature [18, 19, 20, 21, 22]. Extraction using vacuum pyrolysis was also reported in the literature [23]. 51. The table also suggested that there are two methods of isolation of CNSL from the shells. They are

- Cold solvent extraction
- Supercritical carbon dioxide extraction

These methods along with the other methods of extraction available in the literature are discussed in the following paragraphs.

• Cold extraction:

Pentane was used as a solvent for the extraction of phenolic lipids [17]. The broken shells were placed in Erlenmeyer flask and were covered with the pentane. After 12 hours, the extract was filtered off; the shells were again covered with solvent. Five such extracts were combined and evaporated on rotary evaporator under reduced pressure, below 300 °C. The solute to total solvent ratio was maintained at 1:10. The authors have carried out only the analysis of CNSL and no quantification of CNSL was done. In another method of cold extraction [24], diethyl ether was used as solvent. The broken shells were placed in 100 ml of diethyl ether containing 0.1% anti-oxidant. After 24 hours, the ethereal solution was decanted and the ground-up residual shell material re-extracted with another 100 ml of diethyl ether during a further 24 hours. The combined extracts were filtered and evaporated to constant weight. Solute to solvent ratio was not mentioned in their work. Experiments of cold extraction with carbon tetrachloride, light petroleum and diethyl ether were carried out by soaking the shell material (100 gm) is soaked in 400 ml of solvent for one week [19]. The authors performed extraction using Ultrasonication and concluded the solvent requirement is less compared to other methods of extraction. Cold extraction was also carried out using hexane where extraction time was reported to be 14 days. The authors did not report the solute to solvent ratio in their work [14]. In the above methods of extraction, the extraction time is very long. To reduce the time of extraction, hot extraction using Soxhlet extractor was carried out. Extraction using Soxhlet extraction apparatus was carried out using light petroleum oil at a solute to solvent ratio of 1:3.75 [19]. Work was also reported using hexane as solvent for extraction of CNSL from Shells [20]. 35 gm of CNSL was obtained for every 100 gm of Shells charged into the extractor. The CNSL obtained consisted of 10% cardol, 50% cardanol and 30% anacardic acid. The physical properties

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like pH, viscosity, refractive index and specific gravity were also determined. The solute to solvent ratio maintained was 1:17.5. In another experimentation the authors carried out work using different solvents like acetone, hexane, methanol, and toluene in solvent extraction method [21]. They have considered a solute to solvent ratio of 1:20. Their study suggested that of all the solvent used, acetone gave more amount of CNSL and the properties rely with industrial CNSL. The authors studied the effect of time on extraction of CNS and mentioned that maximum percentage recovery of 96% was obtained after one hour of extraction. The time of extraction seems to be too less, since possible mechanism for extraction is that the solvent has to diffuse into the matrix of the shell, dissolution in the oil and then diffusion back into the solution. This mechanism coupled with the shape of the shells gives long extraction times. Their studies also suggested the methanol extract to be slightly more acidic than other extracts. They reported a maximum yield of 8.75 gm of CNSL for 25 gm of shells taken. They also studied the effect of temperature on extraction performance in the range of 30 to 100 0 C and reported that the effect of temperature in the range considered was not significant. The yields of anacardic acid were reported as 45.2%, 43.7% and 44% respectively for the three solvents considered. Since, on heating Anacardic acid undergoes decarboxylation; the authors might have confined themselves to the temperature range mentioned, even though higher temperatures to the tune of boiling points of respective solvents may have given more amount of CNSL. This was the procedure adopted by some authors with their work using ethanol as a solvent for extraction of CNSL in a Soxhlet extractor [22]. The authors performed experiments at different temperature using different types of impellers at various agitation speeds. The use of 4 pitched turbine blade, the agitation speed of 400 rpm and a temperature of 70 °C for extraction of CNSL using 95% commercial ethanol were recommended by the authors. They reported an optimum solute to solvent ratio of 1:10. The CNSL was reported to contain 36.3% anacardic acid and 33.7% Cardanol. The optimum time of extraction reported by the authors was however 6 hours. Extraction was done using hexane, methanol and ethanol as solvents [23]. The authors varied the solute to solvent ratio from 1: 4 to 1:10 and mentioned that the ratio of 1: 8 was adequate for extracting anacardic acid. It can be inferred from the above that there is still some debate about the optimum solute to solvent ratios. Even though, the solvent extraction is generally employed to get higher concentration of phenol, the limitations with solvent extraction of CNSL are that they give rise to colored impurities [25]. Moreover, the solvent extraction has a serious problem of elimination of polluting organic solvent from the extract. To improve the quality of CNSL, to reduce the extraction time, to make the extraction process more environment friendly, supercritical carbon dioxide was contemplated even though earlier work using supercritical carbon dioxide reported an extraction time of 17.5 hours [19].

• Extraction using supercritical carbon dioxide:

Recently, carbon dioxide near critical and supercritical states has drawn much attention as solvent, especially in food and pharmaceutical industries. This is particularly for the interest of avoiding the use of organic solvents that are economically and environmentally unfriendly, besides the difficulties of completely eliminating organic solvents from the desired end products [26]. Literature was also available for use of supercritical fluids in the extraction (SFE) of bioactive compounds from plant matrices with focus on essential oils, phenolic compounds, carotenoids, tocopherols, and tocotrienols [27]. They mentioned that the most important advantages of SFE is the possibility of changing operational conditions of pressure and temperature to facilitate the extraction of specific compound leading to not only extraction, but fractionation. Selection of these conditions depends on the specific compound or compound family to be extracted.

The authors also mentioned that the kinetics of SFE can be represented by the overall extraction curve (OEC). Generally, OECs are characterized by three time periods: (1) a constant-extraction rate period (CER); (2) a falling-extraction rate period (FER); and (3) a diffusion controlled rate period (DCR).

Supercritical Extraction was carried out at a pressure of 250 bar and a temperature of 400 0 C for 16 hours [15]. They reported that extraction rate was maximum between 5 to 10 hours. Though the yield was only 60% of that obtainable, the product was nearly colorless. The flow rate of carbon dioxide was set at 5 kg/hr (80 kg for

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the entire extraction process). They obtained 0.7 gm of CNSL for every kg of carbon dioxide. The amount of carbon dioxide was reduced in subsequent works by Smith and co-workers [26]. They carried out extraction by two methods. First method was typical extraction method during which the yields obtained were much less than the theoretical yields. However, the yield was at 10×10^{-3} Kg/Kg of CO₂. Second method was based on pressure profile extraction method with intermediate depressurization steps. This not only reduced the amount of carbon dioxide, but also increased the yield of CNSL. However, they discussed the quality and quantity of CNSL and no attempt was made to separate the components of CNSL. Based on pressure profile method, CNSL was separated from cashew nut shell [28]. The shells were contacted with high pressure carbon dioxide at elevated pressures of 30 MPa for 1 hour and the pressure is released before separation process begun. Extraction yields of CNSL to the tune of 10 times those obtained by supercritical fluid extraction were obtained. It was found that the temperature has different effects on the solubility depending on the pressure. At low pressures (<12 MPa), increasing temperature results in a decrease in CO₂ solubility in the CNSL phase. At pressures higher than 20 MPa, increasing temperature results in an increase in CO₂ solubility in the CNSL phase. Conditions that resulted in higher CO_2 solubility in the CNSL gave high extraction yields. The extraction mechanism of pressure profile method seems to occur by (i) penetration of the CO₂ through the shell material, (ii) dissolution of the CO_2 into the CNSL, (iii) expansion and rupture of the shell matrix due to depressurization that increases mass transfer and phase contact area. The effect of time of pressurizationdepressurization (PD) on CNSL extraction was also carried out [29]. In initial stages of the trials, before a PD step was performed, the extraction temperature did not have a significant effect on the yield. This means that probably only a very small amount of CNSL was available to the solvent. After a PD step was performed, however, the results for the various temperatures changed greatly with the higher temperature giving the higher yields for a given amount of CO_2 than for yields obtained at lower temperatures. Once that the CNSL was in good contact with the CO_2 , the temperature influence on the CNSL- CO_2 phase behavior became more important. The PD step is thought to break the oil-bearing membranes and to create channels in the cashew shell honey-comb matrix. They also carried out extraction of ground shells and concluded that grinding the shell breaks the CNSL-bearing membranes and allows good contact between CO₂ and the CNSL. Further, it can be implied that the PD step plays a similar role as grinding since the PD step must promote contact between CO_2 and CNSL. In the extractions, it was found that a pressurization-depressurization step that was approximately 1 h gave extraction yields as high as 60%. Extraction of ground shell material gave extraction yields as high as 90%. Processing of Cashew nut using supercritical fluid technology was available in the literature [30]. The authors explained the two possible mechanisms for extraction. The first mechanism is the permeation and diffusion of the carbon dioxide into the matrix and subsequent diffusion of CNSL into the bulk phase. The second possible mechanism is that the carbon dioxide penetrates into the natural matrix and partially dissolves into the oil phase. This causes the oil to swell and its viscosity to become gradually reduced. This allows the oil to flow out of the honeycombed matrix and then diffuse into the bulk phase. Work was carried out for extraction using supercritical carbon dioxide and the effect of pressure, temperature and mass flow rate of carbon dioxide on yield of CNSL [31]. It was reported that total yield increased four times when the pressure was increased from 200 bar to 300 bar at 333 K and 1.0 kg/hr flow rate. The rate of extraction was reported to be high in the initial stages of extraction. With increased temperature, the yield of CNSL increased at a given mass flow rate. With increased solvent ratio, the total CNSL yield increased. However, due to lower retention time, loading of solvent was lower, thereby reducing the capacity of utilization of the solvent. The calorific value of the oil was found to be 39 MJ/Kg for all extraction conditions. The calorific value was 49 MJ/kg in case of vacuum pyrolysis. The authors also reported maximum daily profit at 145 minutes and 13% oil extraction corresponding to optimum time of extraction.

Vacuum pyrolysis: Work was also reported for extraction of oil from CNS using vacuum pyrolysis [25]. Pyrolysis is generally used to describe processes in which preferred products are liquid oils especially those

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with desirable chemical composition and physical attributes for liquid fuels, fuel supplements and chemical feedstock. The liquid pyrolysis fuels apart from being energy rich are easier to handle, store and transport in combustion application and can be upgraded to obtain light hydrocarbons for transport fuel. In the method described, CNS on removal of oil at 1500 °C is termed as Bio-oil CO1 and this is pyrolysed for study of product distribution in a packed bed vacuum pyrolysis unit. The reaction conditions are maintained at, initial reactor vacuum pressure of 5 kPa and at various maximum temperatures between 400–6000 ⁰C with an increment of 500 0 C for each experiment. The total condensable collected in the condensing train is termed as total liquid. Among the total liquid, first three fractions, which are directly combustible without any further treatment, are termed as bio-oil CO2. The total liquid percentage varies from 37% (4000 °C) to a maximum of 42% (500–550 °C) and dropping to 36% (at 6000 °C). However, the liquid to oil ratio was reported to be independent of maximum temperature of pyrolysis in the temperature range of 4000 °C to 5500 °C. The calorific value of Bio-oil CO1 was reported to be 33 MJ/kg while that of Bio-oil CO2 was reported to be 40 MJ/kg which is unusually high like petroleum fuels. Their physical properties analysis showed high miscibility with diesel and methanol in a useful factor for consideration as a fuel. They also reported the viscosity data which is even though high at 300 ^oC reduces drastically at higher temperatures. The ash content for both oils was reported at .01% while that of moisture content for CO1 was reported at 3.5% while that of CO2 was mentioned ad 3%. In addition to the possibility of some of the CNSL polymerizing at elevated temperature, thermal extraction can adversely affect the quality and color of the obtained CNSL. This oil is rich in cardanol and the liquid samples obtained from pyrolysis were reported to contain higher percentage of polymeric material and difficult to load the sample in to the extractor for isolation of phenols using supercritical carbon dioxide extractor [32]. To overcome this problem, pyrolysis oil was mixed with the inert material like saw dust (1:1 by weight) and then it was placed in the extractor. This solid inert material was essentially used as a surface support for the bio-oil. CNSL obtained after decarboxylation of CNS at 1800 ^oC and after pyrolysis was used for the studies. Extraction kinetics for CNSL at different operating pressures and operating temperature of 323 K and at a solvent mass flow rate of 1.2 kg/hr were reported. The batch time was taken as 150 minutes. The increase in yield of CNSL to the tune of 50% was observed with increase in pressure from 225 to 300 bar, keeping other operating parameters constant. It was also interesting to note from GC-MS results that the total concentration of phenols and Cardanol has also increased with increase in pressure. Extraction was also carried out in the temperature range of 303-333 K, pressure range of 120- 300 bar and mass flow rate of 0.7-1.2 kg/hr [33]. The yield of Cardanol rich oil from both pyrolysis CNSL and 1800 ^oC CNSL at 300 bar and 333 K was found to be 63% and 45% respectively. The Concentration of cardanol by this technique was found to be 86% with 5% of phenols.

Scope for future work:

Work can be focused on using different solvents and combination of solvents for extraction of CNSL from Indian cashew nut shell, both for Steam roasted shells as well raw cashew nut shells and their yields at different solute to solvent ratios. This enables optimum solute to solvent ratios for extraction of CNSL. Supercritical extraction of Cold extracted CNSL as well as CNSL obtained from Steam extracted shells can also be carried out for recovery of anacardic acid to compare the extent of anacardic acid obtained with that of chemical methods.

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