

## EXTRACTION OF RESIN FROM AGRO-INDUSTRIAL WASTES

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### ABSTRACT

In the present work, the extraction of resin from the cashew nut sludge (agro-Industrial waste) was done using three different solvents namely methanol, propanol and diethyl ether. Among the three solvents propanol shows the better results for extraction of resin and it was further confirmed with FTIR, TGA and DSC. The FT-IR (Fourier Transform Infra Red Spectroscopic analysis) was used to structural changes during the extracted resin using propanol. The maximum peak obtained using propanol as a solvent for the extraction of resin was 14-16 min intensity. All the experimental study throughout the present study indicated that the obtained resin has wide application on waste water treatment. In future, it can be used as a cheap substitute instead of commercial resin for a better environment.

**Keywords:** Cashew nut sludge, Extraction, Methanol, Propanol, Diethyl ether, FTIR, TGA and DSC

### INTRODUCTION

The largest amount of wastes is generated every year from the industrial processing of agricultural raw materials. Most of these wastes are used as animal feed or burned as an alternative for elimination. However, such wastes usually have a composition rich in sugars, minerals and proteins, and therefore, they should not be considered "wastes" but raw materials for other industrial processes. The presence of carbon sources, nutrients and moisture in these wastes provide conditions suitable for the development of microorganisms, and this opens up great possibilities for their reuse in solid-state fermentation (SSF) processes.

Agro-industrial wastes can be used as solid support, carbon and nutrient source in SSF processes for the production of a variety of value-added compounds. At present, cashew cultivation was made worldwide and researchers were going on to improve cashew crop globally. In India cashew cultivation was taken into care by Indian Council of Agricultural Research under Directorate of Cashew Research to improve the crop cultivation and processing. By their efforts cashew cultivation in India was done in different states like Andhra Pradesh, Kerala, Maharashtra, Orissa, Tamil Nadu and West Bengal. In Tamil Nadu cashew growing districts are Cuddalore, Kanchipuram, Thanjavur, Pudukottai, Thiruchrapalli, Chengalpattu, Villupuram and Ariyalur.

Cashew Nut Shell Liquid is a by-product of the cashew industry. The Cashew Nut Shell Liquid is extracted from the shell and has widely used for many applications [1]. The CNSL has got wide range of application for making raw material for Automobile Braking. The CNSL is also used for manufacturing of Paint, Varnish, Epoxy Resin [2], oil soluble resin, surface active agent, synthetic rubber, was compounding and foundry resin [3].

Resin in the most specific use of the term is a hydrocarbon secretion of many plants, particularly coniferous trees. Resins are valued for their chemical properties and associated uses, such as the production of varnishes, adhesives and food glazing agents. Some varieties of resins also contain high quantities of resin acids. Different plants produce different types of resin [4].

The resin produced by most plants is a viscous liquid, composed mainly of volatile fluid terpenes, with lesser components of dissolved non-volatile solids which make resin thick and sticky. The resin is extracted from the Cashew nut sludge and used for many applications indicated that Cashew Nut shell liquid (CNSL) represents the largest readily available bioresource of alkenyl phenolic compounds [5]. CNSL is a natural product that consists of a mixture of phenolic compounds was separated into cardanol, cardol, and 2-methylcardol using column chromatography. The separation was aimed at recovering cardanol that can be used in the synthesis of cation-exchange resin [6]. The CNSL is a drying oil and it is useful in industries for paints, varnishes and surface coatings [7].

CNSL represents the largest readily available bio resource of alkenyl phenolic compounds. The CNSL extract had a clear light brownish pink color and exhibited no evidence of polymerization or degradation [8]. The isolation of starch from defatted cashew nut shell (CNS) using wet milling was reported where the product that contains 85 wt % was recovered from the defatted CNS [9].

This process is designed to remove residual oil by contacting the deliquified sludge with hydrocarbon solvents. The solvents are intermediate or product streams produced by most refineries. Proprietary design features of the extraction vessel ensure efficient contacting, minimal solids entrainment in the extract stream, and simple and effective removal of de-oiled solids from the extraction vessel.

Design provisions have also been made which effectively handle upset or unusual conditions. The single-cycle extraction process utilizes a light hydrocarbon such as propane as the solvent. The multi-cycle extraction process utilizes a series of extractions with different solvents. The processing sequence depends on sludge characteristics, solvent availability, recycling considerations and treatment requirements. In a typical three-cycle process, the first cycle removes the bulk of the oil from the sludge with a light hydrocarbon solvent. The second cycle employs a heavier solvent to remove poly nuclear aromatics. The third cycle uses a light hydrocarbon to remove residual second cycle solvent and provide final polishing [2] Rajesh *et al.*, 2006). In this present article an attempt was made to develop an extracted resin using cashew nut sludge with different solvents like Methanol, Propanol, and Diethyl ether. The extracted resin was characterized using FTIR, TGA and DSC to study the thermal and morphological characteristics.

### MATERIALS AND METHODS

Cashew nut sludge (CNS) was obtained from the cashew nut industry in Panruti (muthandikuppam). They were grounded, sieved, and stored at 4°C to minimize the degradation of its compounds. Defatting of CNS was carried out using propanol at 50°C for 12h followed by n-hexane at 69°C for another 12 h in a Soxhlet extractor. The two solvents used for defatting CNS were of HPLC grade. n-Hexane and propanol. CNS (15 g) was soaked in water with a CNS to water ratio of 1-5(w/w) for 2 h at 30°C. The mixture was blended for 5 min and screened using a 50-mesh sieve. The residue was re-blended with 50 ml of 70% ethanol for 5 min, passed through a 60-mesh sieve and the residue was re-blended with 50 ml of 0.1 M NaOH for another 5 min, then screened using a 50-mesh sieve. The residue restrained at the filter paper was dried by using a freeze drier. The dried starch was kept at 5°C prior to analysis. All purification and analysis experiments were done at least in duplicates. Thermal analysis was performed using DSC-6 Perkin

Elmer at heating rate of 10°C/min under nitrogen whilst TGA was performed using TGA-(Model Hi. Res. 2950 TGA unit interfaced with Thermal Analyst 2100 control unit) in nitrogen atmosphere. FTIR spectrum was obtained using ABB BOMEN MB-3000, with a spectrometer in the range of 4000–400 cm<sup>-1</sup>.



Fig 1: cashew nut sludge



Fig. 2: Extracted resin from CNS

#### Pretreatment of Sludge

The components of cashew nut sludge were more accessible while pretreatment was carried out. About 5g of finely ground above raw materials was mixed with 20ml of alkali (0.1N) solution and was soaked for a period of 12 hrs. The conical flask containing these raw materials was filtered using muslin cloth. The residue was washed with water till a neutral pH was obtained. It was dried at 60°C over night. Fig 2 shows the extracted resin.

## RESULTS AND DISCUSSION

### FTIR

Fourier transform infrared (FTIR) spectroscopy is a measurement technique that allows one to record infrared spectra. Infrared light is guided through an interferometer and then through the sample. A moving mirror inside the apparatus alters the distribution of infrared light that passes through the interferometer. From Fig 2, it was concluded that the extraction of resin using propanol was found to be best when compared to resin extraction using methanol as a solvent. Since the presence of resin was confirmed with the peak (912) present in the FTIR analysis. There is no peak observed when methanol and diethyl ether was used as solvents.

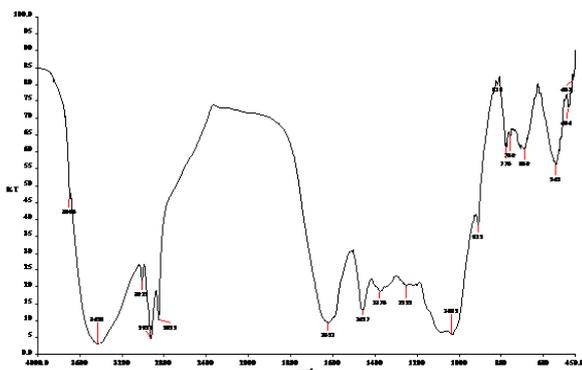


Fig.3 FT-IR-Spectroscopy analysis result using propanol

The FTIR spectra of the extracted resin showed the presence of the relevant functional groups. Alkynes bend (C–H bending) was observed at 675-1000 cm<sup>-1</sup>. The presence of hydroxyl group (O–H stretch) was characterized by the absorption peak at 3600- 3700 cm<sup>-1</sup>. The ester group (C–O stretch) was identified at 1000-1260 cm<sup>-1</sup>. The presence of aromatic amines (C–N stretch) was confirmed at 3300-3500 cm<sup>-1</sup>. The peaks corresponding to the presence of alkenes (–C=C– stretch) were found at 1400-1600 cm<sup>-1</sup>. The alkane (C–H stretch) group was identified at 2850-2960 cm<sup>-1</sup>. The presence of alkenes was characterized by the absorption peak at 3010-3080 cm<sup>-1</sup>. These peaks confirmed that the presence of cationic resin functional groups in the extraction of resin using propanol. Single-cycle extraction with propanol provides excellent bulk oil removal, but is somewhat less effective than the multi-cycle process for removal of heavy organics like poly nuclear aromatics (PNAs) (10).

### Thermo Gravimetric Analysis (TGA)

Thermo gravimetric Analysis is a technique in which the mass of a substance is monitored as a function of temperature or time as the sample specimen is subjected to a controlled temperature program in a controlled atmosphere. The percent weight loss and thermal degradation characteristics of the prepared samples were evaluated by Thermo gravimetric analyzer (TGA) of TA Instrument (Model Hi. Res. 2950 TGA unit interfaced with Thermal Analyst 2100 control unit). About 5–10mg of sample was taken in a platinum sample pan and nitrogen was purged at 60 ml/min during the dynamic runs. Most TGA experiments use an inert sample purge gas. This is done so the sample only reacts to temperature during decomposition.

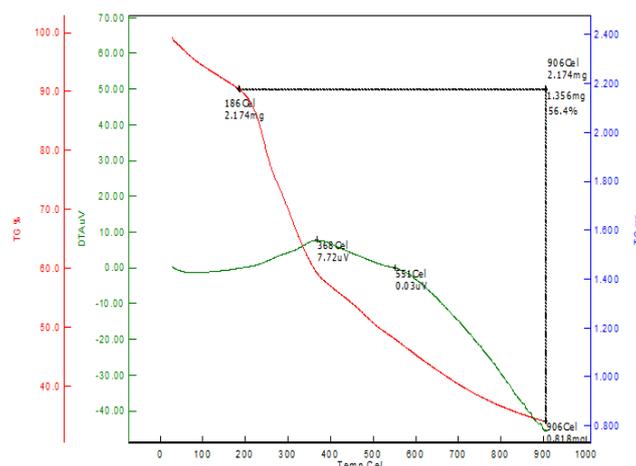


Fig. 4. Thermogravimetric Analysis for extracted resin

Figure 4 shows the Thermo gravimetric (TG) curves of the extracted resin. It can be seen from the TG curve that the sample has a little of mass loss before 100°C; this may be caused by evaporation of residual water in the resin. After 350°C, due to the resin chain breaking, the resin mass loss rapidly the sample mass loss is about 56.5% at 906°C. The results show that the extractive resin has a good thermal stability.

### Differential Scanning Calorimetric (DSC) Analysis

Cure temperatures of the prepared samples were observed by taking very little quantity of samples of Differential Scanning Calorimeter (DSC). This was placed in sample cell of the instrument. The starting temperature, programmed rate and final temperature were taken at heating rate of 10 °C/min. Dynamic scans were obtained which were used for assuming the cure temperature. In Fig 5, the cure temperature initiation was observed at 48°C. The onset Temperature of curing (Tonset), the exothermic peak temperature or the temperature of maximum cure (Tp) and the final temperature or the temperature at the end of the cure (Tstop) for the samples were determined from DSC as 98.5 °C, 354.7 °C and 474.4 °C scans. Where ΔH was -2.13mJ/mg. From the above results it was confirmed that the cationic type of resin

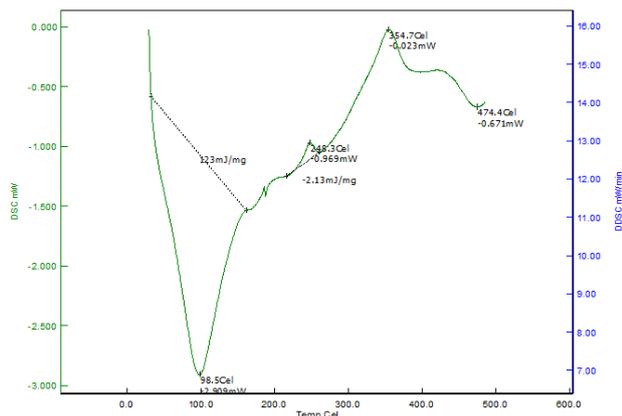


Fig.5.Differential scanning calorimetric for extracted resin

## CONCLUSION

From the above results, it was concluded that the analysis shows that propanol is the best solvent for the extraction of resin. Other solvent like methanol and diethyl ether does not show better peaks. The FTIR spectra were further confirmed as the resin which was extracted from cashew nut sludge waste. The TGA and DSC analysis indicated the type of resin and it was found in the cationic category of resin.

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