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EXTRACTION OF TARTARIC ACID FROM TAMARIND PULP AND ANALYSIS OF THE ACID COMPOSITION IN LEAVES

Gullipalli Santhosh Roopa[1] and Professor V.Kasiviswanatham[2]
[1,2] Department of Chemical Engineering, Gayatri Vidya Parishad College of Engineering, Visakhapatnam, Andhra Pradesh,India
sroopa804@gmail.com, v_kashi2001@gvpce.ac.in

Abstract

Tamarind a naturally obtained, long lived, evergreen and less expensive raw material. It comprises organic acids like high content of tartaric acid 12-18%, malic acid, citric acid and byproducts like pectin, potassium Bitartrate. The purpose of this research was to extract the tartaric acid from tamarind pulp by hot and cold extraction followed by cooling and by addition of less expensive chemicals. The obtained solid form of acid detected with NMR Spectra and its concentration analyzed with UV spectrophotometer using metavanadate. Where as the composition of acid in powdered form of leaves explored with two methods. By organic extraction using suitable solvent, the composition of acid in leaves is extracted using own making equipment percolator with small lab scale apparatus and waste material like plastic drink bottle and another method is soxhlet apparatus. The extracts obtained from process are added with another suitable solvent and were analyzed by Thin Liquid Chromatography. As a whole the process becomes economically more competitive than other process and to utilize the product in both traditional and agricultural applications.

Key words: Tamarind pulp, tartaric acid, NMR spectra, UV spectrophotometer, equipment with waste material, TLC, agriculture benefit.

1. INTRODUCTION

Tamarind fruit pulp is found in the pea-like pods (up to 15cm long by 2.5cm across) surrounding one to twelve seeds and 3 - 8 inch long, brown, irregularly curved pods. While at West India, has shorter pods containing only 3 – 6 seeds. Most tamarinds in the America are of the shorter type. Its medicinal actions are digestive, carminative, laxative and ant scorbutic. Leaves are eaten as a vegetable and are also used medicinally. Leaf juice is good for bilious fevers, urinary disorders and jaundice. A fresh leaf poultice is applied locally over swellings of ankles and joints, sprains, boils, sore eyes and scabies. A mature tree can bear about 160kg (350lb) of fruit annually. Tamarinds are long-lived, evergreen trees.

Products Obtained From Pulp:

- *Pectin* is a byproduct, naturally-occurring thickening agent that is most often used by adding it to jams, jellies and similar products to help them gel and thicken.

- *Potassium Bitartrate* is a byproduct, *it* is n the form of crystals type of salt with different shapes depending upon temperature and time.
- *Tartaric acid* is an unusual plant acid with useful properties. It can be used to enhance yield in agriculture with isopropyl.

2. EXPERIMENTAL DESCRIPTION I:

Extraction of Tartaric Acid from Tamarind Pulp:

- Tamarind pulp extracted using 1:2 volumes ratio of water: pulp at a temperature in the range of 25◦c to 100◦c on a hot plate at about 6hrs.
- Cool the extract for hrs at a temperature of 10◦c. then the *Potassium Bi-tartarate* is precipitated. The mother liquor is kept aside.
- The obtained potassium bi-tartarate is dissolved in sufficient amount of water, add carefully known grams of calcium carbonate in powder Bi-tartarate.It becomes *calcium tartarate* and neutral potassium tartarate this reaction releases CO_2.
- Known grams of calcium chloride is dissolved into the hot water and added to the above. Here, a *Double exchange reaction* takes place.
- The obtained precipitates are filtered, to the obtained precipitate add 25ml of 96% H2SO4 and after 30min calcium tartarate are decomposed to pure tartaric acid and at the bottom calcium sulfate settles.
- Filter while it is still hot. Wash many times the sulfate that contains still quantities of tartaric acid and add the washing water to the filtrate.
- Let it evaporates for 6-10 days, than we will collect crystals of tartaric acid (*see figure 1*).

Modification for recovery of tartaric acid:

- Due to more expensive of tertiary amine (trioctyl amine) in the prior art process is skipped after recovery of potassium Bitartrate.
- Used ice tubes, water and thermacol box, plastic bottle as an alternative to cool and precipitate potassium Bitartrate.

2.1 Instruments Used For Product Analysis: Pulp

a) **NMR Spectroscopy:**

b) **UV**-visible spectrophotometer:

a) NMR Spectroscopy:

Nuclear Magnetic Resonance spectroscopy is a powerful and theoretically complex analytical tool.

b) UV-visible spectrophotometer:

Step 1: Tartaric Acid Standard Solution Preparation:

0.016 M standard solution of tartaric acid L^+ is prepared .

Step2: Prepare Diluted Quantitative Standards:

Take known grams of metavanadate, calculate its molarities and prepare solution*)*.

Step 3: Blank preparation:

Take a blank in cuvette, always run a blank a blank is like a control.

Step 4: Sample preparation:

Take samples in a cuvette*)*.

Step 5: To run the unknown sample:

Prepare Diluted Quantitative Standards.

Observations and Calculations:

The concentration of unknown solutions can be determined by plotting the absorbance value. Unknowns can be compared to the standard curve to determine their concentration (*see figure 2)*.

3. RESULTS AND DISCUSSION:

⇒ **Recovery Process**

Weight Balance:

Previous process:

500 g pulp →18g potassium Bitartrate →15 g pectin →35g tartaric acid.

Estimated amount of acid by above Modified Research:

¼ times of actual grams taken for the process

125g pulp →4.5g potassium Bitartrate →3.75g pectin→8.75g tartaric acid.

Obtained amount of grams:

125g pulp→4g potassium Bitartrate → 4g tartaric acid.

⇒ **Concentration calculation :**

$$\text{Weight of tartaric acid Present in unknown sample} = \frac{\text{M of } C_4H_6O_6 \times \text{molecular wt of } C_4H_6O_6 \times 20}{1000}$$

$$= \frac{0.00638 \times 150.09 \times 20}{1000}$$

$$= 0.01915$$

$$\text{\% of tartaric acid present in initial weight} = \frac{\text{Wt of } C_4H_6O_6 \text{ present in unknown sample}}{\text{Initial wt of } C_4H_6O_6 \text{ dissolved in water}} \times 100$$

$$= \frac{0.01915}{0.6} \times 100$$

$$= 3.19 \%$$

4. EXPERIMENTAL DESCRIPTION II:

Organic Extraction of Tartaric Acid From Leaves and Analysis of Acid Composition

Organic solvent extraction is one process for separating the desired substance from plant material. Fresh plant and dried plants are used for extraction. The plants are first ground and then thoroughly mixed with a solvent.

4.1 Process:

a) Percolation (*see figure 3*).
b) Soxhlet extraction (*see figure 4)*.

4.2 Materials for percolator setup:

-Plastic bottles	-Necked flask
-Cotton	-Tripod Stand
-Funnels: small and big	-Teflon tape
-Test tube (half cut)	-Burettes stand to hold separating funnel.
-Separating funnel	-A sharp knife

4.3 How to make the setup:

Step 1: Cut off the bottom of a plastic drink bottle

Step 2: Take a decent size lump of cotton and stuff it up into the Second end of test tube (half cut).This is the filter that

will prevent Particles from coming through, second end is tied with Teflon tape to provide seating.

Step 3: Add tamarind leaves powder to extract on top of the Cotton. The leaves powdered is soaked initially in a solvent

in a percolator .Place a small funnel on flask and cover the entire set up with bottom cut plastic bottle.

Step 4: Take a separating funnel with ethanol 70% and water 30% and hold it with burette stand. Place big funnel set up

(step3) on tripod stand. The plant material allowed to percolate slowly.

Step 5: Fluid extract was separated and mixed with 50 mL of hexane and it transferred to a round bottom flask and analyzed with Thin Liquid Chromatography.

4.4 Instruments Used For Product Analysis: Thin Liquid Chromatography:

This method is a simple, quick, and inexpensive procedure to identity of a compound in a mixture when the R_f of a compound is compared with the R_f of a known compound (preferably both run on the same TLC plate).

Step 1: Dip the micro cap into the solution and then gently touch the end of it onto the proper location on the TLC plate.

Step 2: Place the prepared TLC plate in the developing jar.

Step 3: TLC plate is dipped in Potassium permanganate and dried

Step 4: If there are any colored spots, circle them lightly with a pencil. Most samples are not colored and need to be visualized with a UV lamp Hold a UV lamp over the plate and circle any spots

Calculation of R_f Value:

Length of plate taken = 2.5 cm

A – d spot = spot 1 --- 0. 2mm | spot 2 ---0.7 mm.

B – Level to which Solvent migrates. = 1.5cm

Formula:

$$R_f = \frac{d_{spot}}{d_{solvent\ front}} = \frac{0.2}{1.5} = 0.133$$

$$R_f = \frac{d_{spot}}{d_{solvent\ front}} = \frac{0.7}{1.5} = 0.466$$

b) Soxhlet Extraction:

- A solid material is placed inside a thimble, which is loaded into the main chamber of the Soxhlet extractor.
- The Soxhlet extractor is placed onto a flask containing the extraction solvent. The Soxhlet is then equipped with a condenser (*see figure 4*).

Reference to identify acid:

Organic acids of reference	R
Oxalic acid	0.00
Citric acid	0.06
Tartaric acid	0.13
Malonic acid	0.27
Malic acid	0.30
Fumaric acid	0.36
Succinic acid	0.44
Salicylic acid	0.79
Benzoic acid	0.81

- When the Soxhlet chamber is almost full, the chamber is automatically emptied by a siphon side arm, with the solvent running back down to the distillation flask (*see figure 4*).

5. CONCLUSION

When comparing with previous process it deals with costly chemicals and non available Instruments. So the modified processes developed by this research I, two commercially important constituents of the tamarind pulp are potassium Bitartrate and tartaric acid obtained. On the other hand, research II analysis the acid composition of early leaves is done with low cost chemicals to get equal amount of acid.

6. FUTURE SCOPE OF THIS RESEARCH:

The wide variety of uses for Tamarind in many of the countries has not been exploited, although in the future the area and extent of production are likely to increase as tamarind assumes greater recognition and importance. Tartaric is an unusual plant acid formed from the primary carbohydrate products of photosynthesis, and once formed, it is not metabolically used by the plant. The content of tartaric acid does not decrease during fruit ripening, suggesting it is not utilized in fruit development. This research can implement further to get more amount of acid with this modified process in large scale instead of using expensive chemicals and rare equipments.

ACKNOWLEDGEMENT:

In the modified process developed, two commercially important constituents of the tamarind pulp are potassium Bitartrate and tartaric acid. On the other hand, studied the chemical composition of tartaric acid in early leaves.

It enables recovery of two products of commercial value from tamarind pulp, a raw material abundantly and cheaply available in tropical countries and we use less expansive chemicals for the recovery. As a result, the process becomes economically more competitive compared to other process.

Tartaric acid and isopropyl alcohol each form an ester with Cr (VI), resulting in a termolecular complex that greatly enhances the reduction over that which would occur with only tartaric acid. By reducing Cr(VI) will enhance the yield in agriculture.

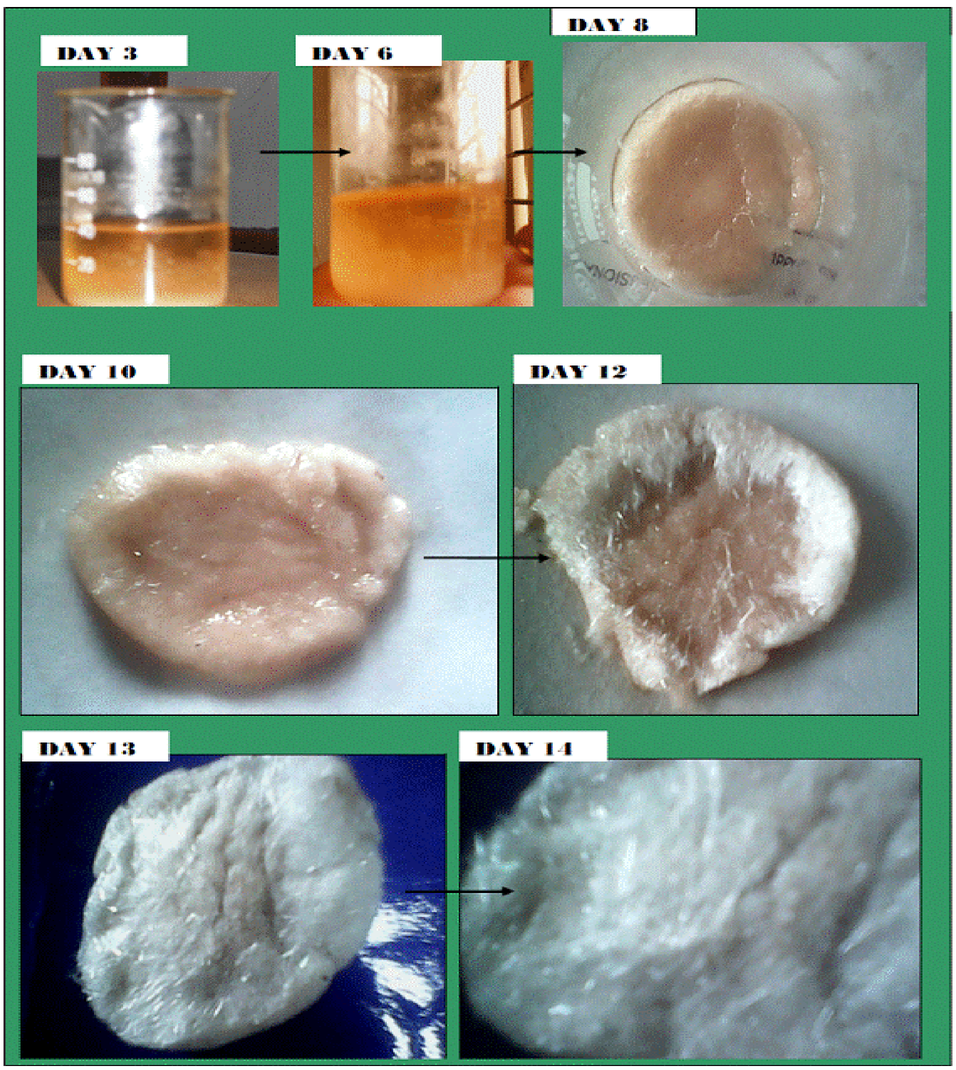

Figure 1: Recovery Process

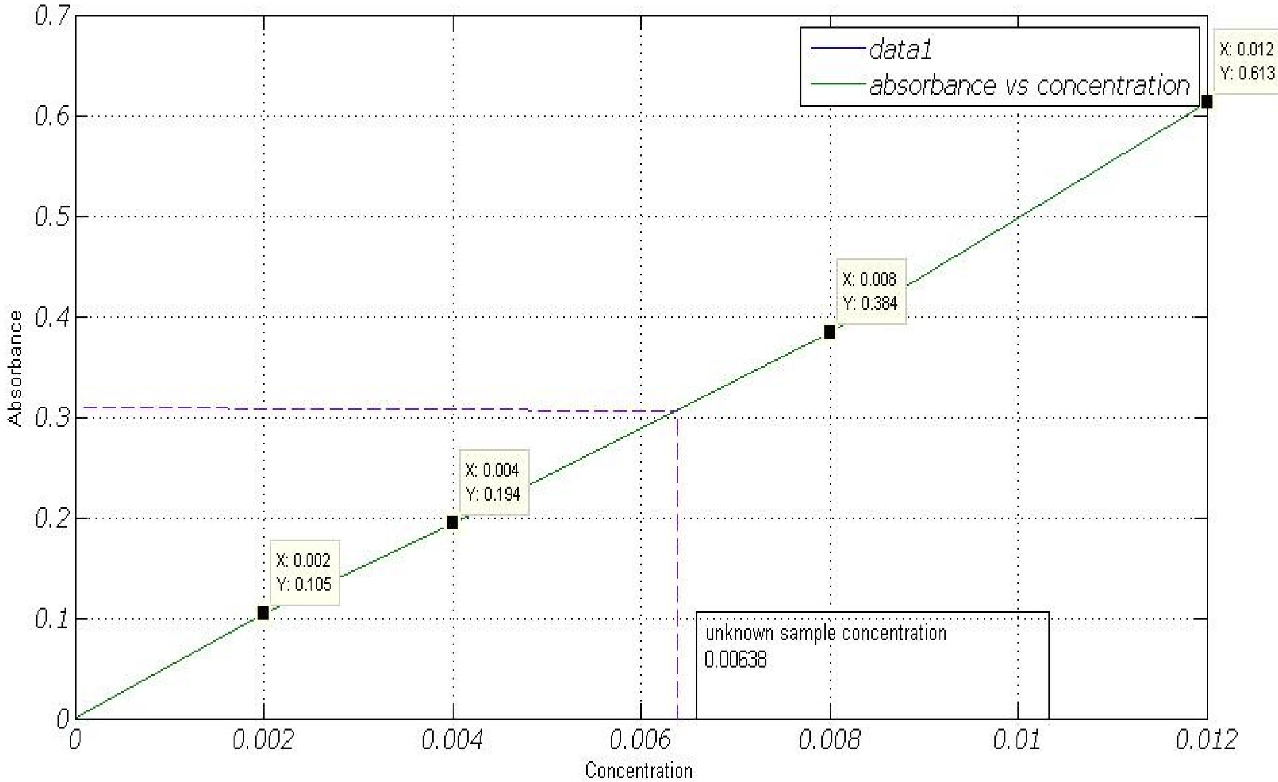

Figure 2: Calculation of Concentration.

```
function createaxes(Parent1, X1, YMatrix1)
%CREATEAXES(PARENT1,X1,YMATRIX1)
%  PARENT1:  axes parent
%  X1:  vector of x data
%  YMATRIX1:  matrix of y data
%  Auto-generated by MATLAB on 13-May-2013 12:20:44
% Create axes
axes1 = axes('Parent',Parent1,'FontSize',12,'FontName','Verdana',...
    'FontAngle','italic');
% Uncomment the following line to preserve the Z-limits of the axes
% zlim(axes1,[-1 4]);
box(axes1,'on');
grid(axes1,'on');
```

```
hold(axes1,'all');

% Create multiple lines using matrix input to plot
plot1 = plot(X1,YMatrix1,'Parent',axes1);
set(plot1(2),'Color',[0 0.5 0],'DisplayName','absorbance vs concentration');
% Create xlabel
xlabel('Concentration');
% Create ylabel
ylabel('Absorbance');
```

Figure 3: Percolator Set up.

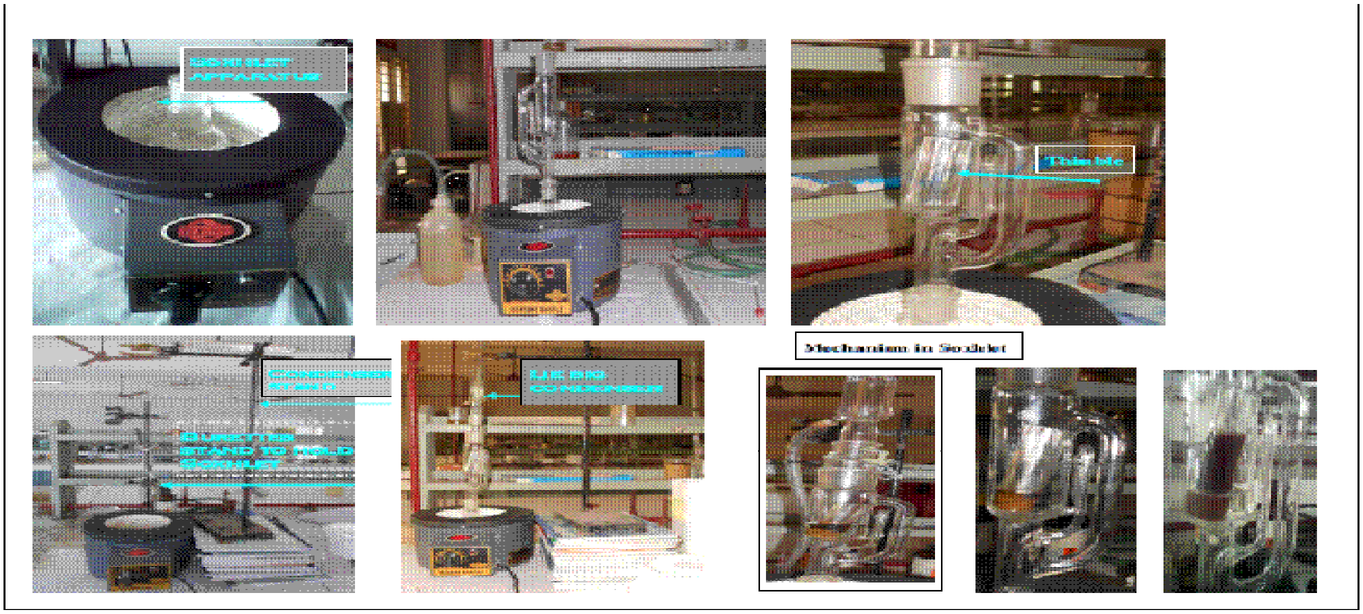

Figure4: Soxhlet Extraction

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Author Biography:

Ms.G.Santhosh Roopa has been graduated from Gayatri Vidya Parishad College of Engineering of Andhra Pradesh, India. She is author of research paper entitled *Extraction of tartaric acid from tamarind pulp and analysis of acid composition in Tamarind leaves.* She received her diploma in chemical engineering at the GICE of Andhra Pradesh. During academic of diploma she earned his spot as a trainee in Hindustan zinc ltd., and Visakhapatnam Steel Plant of Andhra Pradesh. During her under graduation she did mini project at Hindusthan Petroleum Corporation ltd., Andhra Pradesh, India. Currently she is focusing on projects of algae and eucalyptus family, seeking to acquire the sponsoring from research centers. She can be contacted at sroopa804@gmail.com .

Mr.V.Kasi Viswanatham is Associate Professor of Chemical Engineering at the Gayatri College of Engineering of Andhra Pradesh, India. He received his M.Tech in Specialization electro chemical engineering from Andhra University and eventually earned his spot as Safety Manager at the chick film hybrid micro electronics manufacturing. Later he moved to teaching profession at GVP college of engineering. He can be contacted at v_kashi2001@gvpce.ac.in.