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RESEARCH ARTICLE

Extraction of Pectin from Fruit wastes– an effective method of municipal solid waste management

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Abstract

The study was aimed at utilization of the fruit wastes collected from UzhavarSanthai, Chokkikulam, Madurai. Different fruit wastes were collected in separate baskets before they were dumped to the bin and were utilized for the extraction of useful by products viz., pectin and ethanol. Pectin was extracted from the peels and ethanol was extracted from the pulp. The ethanol extracted from the fruit pulp was used to extract pectin from the fruit peel. A considerable amount of pectin could be extracted per day from the collected fruit wastes. The extracted pectin was characterized by using spectral and thermal techniques. The food quality of the extracted pectin was compared with that of the FCC standard and it was found to be of good quality.

Introduction:

Since the beginning, human kind has been generating waste through various means. With the progress of population, industrialization and urbanization, the waste generated became more complex in nature. Modernization and progress has had its share of disadvantages and one of the main aspects of concern is the pollution (land, air, and water) which it causes to the earth. Solid waste can be classified into different types depending on their source like household waste (vegetable and fruit waste) which is generally classified as municipal waste, industrial waste as hazardous waste, biomedical waste or hospital waste as infectious waste and e-waste or electronic waste. There has been a significant increase in the generation of Municipal Solid Waste (MSW) in India over the last few decades mainly due to rapid population growth in the country. The solid waste generated in Indian cities has increased from 6 million tons in 1947 to 48 million tons in 1997 and is expected to increase to 300 million tons per annum by 2047 (Sujatha P. Janardhanam, 2012).

This waste is ultimately thrown into municipal waste collection centers from where it is collected by the area municipalities to be further thrown into the landfills and dumps. If at this stage the management and disposal is not properly done, it can cause serious health impacts and lead to the spread of infectious diseases. Unattended waste lying around attracts flies, rats, and other creatures that in turn spread diseases. Normally it is the wet waste that decomposes and releases a bad odor. This leads to unhygienic conditions and thereby to a rise in the health problems (Suryawanshi P.C. et al., 2013).

According to a statistics, domestic waste generation from households comprising vegetable waste, food waste, paper, packing material, glasses, metals etc., in Madurai is estimated as 288 tons per day which constitutes nearly 64 percent of the total waste generation.

Processing of fruits produces mainly two types of wastes, a solid waste of peel/skin and seeds, and a liquid waste of juice and wash water. In some fruits the discarded portion can be very high (eg. mango 30-50%, banana 20%, pineapple 40-50% and orange 30-50%). Therefore, there is often a serious waste disposal problem. There are a number of possibilities for use of some types of solid fruit wastes. One of the main problems in using fruit wastes is to ensure that the waste has a reasonable microbiological quality. Only waste produced during the same day should

therefore be used - it is not advisable to store-up wastes. The possible products obtained from fruit waste are candied peel, oils, pectin, reformed fruit pieces, enzymes and wine/vinegar. Among them the most useful product is pectin and this is a gelling agent used in jams and some sweets. Pectin is found to a greater extent in most fruits. Commercially, pectin is extracted from citrus peel and apple pomace. The utilization of the peels remaining after pulp removal offers possibilities for pectin extraction. Pectin can be obtained from many sources with a variation in the percentage yield.

Pectic substances are complex mixtures of polysaccharides containing units of galacturonic acid as the main chain. In this main chain, L-rhamnose units are occasionally inserted through glycosidic linkages and the carboxyl groups are partially esterified by methyl alcohol (Thomas Happi Emaga et al., 2008 & Lalitha Kumara B.et al., 2013).

Pectin is widely used in the food industry as a thickener, emulsifier, texturizer and stabilizer. It has also been used as a fat substitute in spreads, ice-cream and salad dressings (Jittra Singthong et al., 2005). Pectin is found to lower blood cholesterol levels and low-density lipoprotein cholesterol fractions, which is beneficial for human health (Liu et al. 2006). According to the FAO (1969), pectin is considered to be a safe food additive that can be taken daily without limits (Jittra Singthong et al., 2005).

Pectin can be extracted through various methods. Usually, industrial pectin is extracted in a multiple-stage physico-chemical process characterized by an extraction step with hot dilute mineral acid and recovery through alcohol precipitation (Mollea C. et al., 2008). The Physico-Chemical parameters of extracted pectin need to be compared with that of FCC in order to determine its quality as a food ingredient.

The FCC is a compendium of internationally recognized standards for determining the purity and quality of food ingredients. It is a valuable resource for authenticating a wide variety of ingredients, including processing aids, preservatives, flavorings, colorants, and nutrients (Willats W.G.T. et al., 2006). Since solid waste poses serious environmental problems, the present study is focused on conversion of waste to wealth. The study also invokes the concept of "zero waste" through complete utilization of fruit waste for the extraction of ethanol (from the pulp) which will be used to extract pectin (from the peel). The solid mass left behind will then be used as adsorbent for the treatment of Industrial effluents.

Materials and methods

Collection and pretreatment

The primary data was collected from the Fruit vendors of Uzhavar Santhai, Chokkikulam. The fruit waste consisting of banana, papaya, lemon, orange used for the study was collected every day morning during the month of December. The fruit wastes were segregated according to their type and washed with water for about three times. Then the peels and the pulps of fruits were separated. The peels were washed with plenty of water and rinsed with distilled water. The washed peels were kept in a tray and dried in shade. The peels were then powdered using a blender. 50 g of the dried peel powder was weighed and mixed with 100 ml of deionized water which was stored in refrigerator. The pulp was then used for the production of ethanol

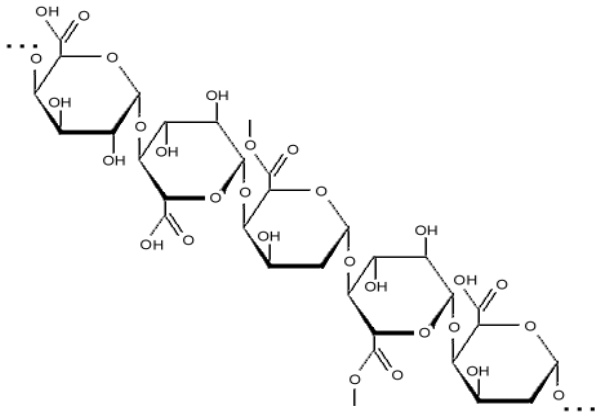

Fig. 1: Repeating segment of Pectin molecule

Extraction of pectin from peels

The pectin extraction procedure was shown as flowchart in fig 2.

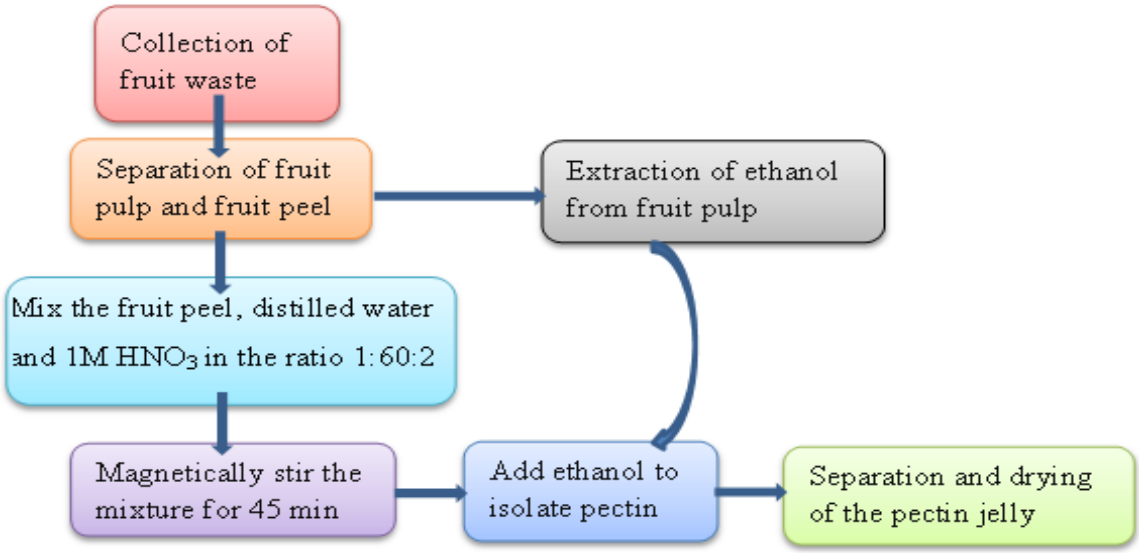

Fig. 2. Flow chart for the extraction of pectin from fruit peel

Identification and characterization

Identification of pectin by using FCC-Food Chemical Codex

The procedures from FCC-Food Chemical Codex were used to identify and characterize the extracted pectin (Willats W.G.T.et al., 2006 & Erika Kliemann, 2009). The following tests were carried out to identify the formation of pectin.

(a) To 1 in 100 solution of a sample in water, equal volume of alcohol was added. A gelatinous precipitate confirms the presence of pectin.

(b) To 5 ml of a 1 in 100 solution of the sample in water, 1ml of sodium hydroxide was added and allowed to stand at room temperature for 15min. A gel was formed which indicated the presence of pectin.

(c) The gel formed in the previous step was shaken with 1ml of HCl. A colourless, gelatinous precipitate formed, which on boiling, became white and flocculent. This shows the presence of pectinic acid.

Characterization of pectin for food quality by using FCC standard

(a) Determination of total ash

About 3g of the sample was weighed accurately in a crucible, ignited at a low temperature, not to exceed very dull redness, until free from carbon, cooled in a desiccator and weighed. Finally added the filtrate evaporated to dryness and heated the whole to a dull redness. If a carbon –free ash was still not obtained, cooled the crucible, added 15 ml of alcohol, again heated the whole to a dull redness, cooled and weighed.

(b) Determination of acid insoluble ash

Boiled the ash obtained with 25 ml of diluted hydrochloric acid for 5 min, collected the insoluble matter washed with hot water, ignited in a silica crucible and weighed. The percentage of acid –insoluble ash was calculated from the weight of sample taken.

(c) Determination of degree of esterification (DE), degree of amide substitution(DAS), total anhydrogalacturonides.

5.0g of the pectin was transferred into a beaker and stirred for 10 min with a mixture of 5ml of concentrated hydrochloric acid and 100ml of 60% isopropyl alcohol. It was filtered through a dry Buchner funnel and washed six times with 15ml of the acid –alcohol mixture followed by the 60% isopropyl alcohol until the filtrate is free from chloride. Finally, the residue was washed with 20ml of anhydrous isopropyl alcohol, dried at 105^{o}C for 2.5 h, cooled and weighed.

500.0 mg of the washed and dried sample was transferred into a 250ml round bottomed flask, and moistened with 2ml of alcohol. 100ml of water was added, stoppered and stirred occasionally until the sample was completely hydrated. Added 5 drops of phenolphthalein and titrated with 0.5N sodium hydroxide. The initial volume

V_1 (ml) was recorded and 20.0 ml of 0.5 N sodium hydroxide was added, stoppered, shook vigorously and allowed to stand for 15min. 20.0ml of 0.5N hydrochloric acid was added into it and shook well until the pink color disappeared, then 3 drops of phenolphthalein was added, and titrated with 0.1N sodium hydroxide V_2 (saponification titre value).

The contents were transferred into a 500 ml distillation flask fitted with a water cooled condenser, the delivery tube of which extends well beneath the surface of a mixture of 150ml of water and 20.0ml of 0.1N hydrochloric acid in a receiving flask. To the distillation flask added 20.0ml of sodium hydroxide, and heated carefully to avoid excess of foaming. The heating was continued until 80 to 120 ml of distillate has been collected. Added a few drops of methyl red to the receiving flask, and titrated excess acid with 0.1 N sodium hydroxide, recorded the volume required as S, in ml. Performed a blank determination on 20.0ml of 0.1N hydrochloric acid, and recorded the volume required as B in ml. Recorded the amide titre (B-S) as V_3, and the total titre ($V_1+V_2+V_3$) as V_t.

The degree of esterification was calculated by using the formula $100 \times(V_2/ V_t)$.

The degree of amide substitution was calculated by using the formula $100 \times(V_3/V_t)$.

The weight percent of total anhydrogalacturonides was calculated by using the formula

$3.52V_1 + 3.80 V_2 + 3.5 V_3$.

Qualitative tests for free alcohol, amide and acid

(a) Test for alcohol

To 0.1g of substance added 1ml of acetic anhydride and 3 drops of con.H_2SO_4. Heated it for 2-3min, cooled and poured the contents into 20ml of dilute Na_2CO_3 solution.

$$ROH + (CH_3CO)_2O \longrightarrow CH_3COOR + CH_3COOH.$$

(b) Test for Amide

To 0.1g of substance added 2ml of 10% NaOH solution and heated strongly. Smell of NH_3 indicated the formation of amide. Heated the solution until no more NH_3 was evolved. Added a few drops of conc. HCl. Absence of precipitate indicated the presence of aliphatic amide.

$$CH_3CONH_2 + NaOH \rightarrow CH_3COONa + NH_3$$
$$CH_3COONa + HCl \rightarrow CH_3COOH + NaOH$$

(c) Test for acid

To 0.1g of substance, 2ml of $NaHCO_3$ solution was added. Brisk effervescence was noted with evolution of CO_2. The evolved gas was passed into lime water and white precipitate was formed. This indicated the presence of acid.

$$RCOOH + NaHCO_3 \rightarrow RCOONa + H_2O + CO_2$$
$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O$$

Instrumental techniques

The formed pectin was characterized by using UV-Visible spectrophotometry and IR spectrometry. 1% of the extracted sample was analyzed using thermo scientific (Helious-alpha) UV-Visible spectrophotometer. The IR spectrum was recorded using Thermo scientific IR Spectrometer by using KBr pellets. The surface morphology of pectin was determined using JSM- 5610LV JEOL Scanning Electron Microscope. Thermal analysis was carried out using Pyris Thermogravimetric analyzer.

Extraction of ethanol from squeezed pulp

50 g of squeezed pulp was taken in a beaker. A small quantity of yeast was added into it and was kept 24 h for fermentation. The fermented pulp was transferred into the flask of Rotary evaporator (Supervac) and 25ml of de-ionised water was added into it. Then the temperature was adjusted to 72^oC and heated. The liquid collected was used for the extraction of pectin.

The spent liquor after the extraction of pectin was also treated by the same procedure and the unused ethanol was regenerated (Amit Kumar, 2010).

Results and discussion

Statistical analysis

There are 12 fruit vendors in Uzhavar Santhai, Chokkikulam, Madurai. Only 10 of them responded to the questionnaire. An analysis has been carried out from the primary data collected. It was found that about 50% of the respondents sell their fruits harvested from their own fields, 30% of them buy fruits from other sources and the remaining from both the sources (Fig.3). Nearly 65% of the respondents expressed that they always threw the fruit waste near the road side and the remaining respondents use the waste bin for their disposal (Fig.4).

Nearly 55% of the respondents expressed that maximum amount of fruit has gone as waste every day (12-15 kg) and 30% of them expressed that only minimum amount (below 12 kg) of fruit has gone as waste every day. On an average approximately10 kg of fruit turned out as waste per person (Fig.5.).

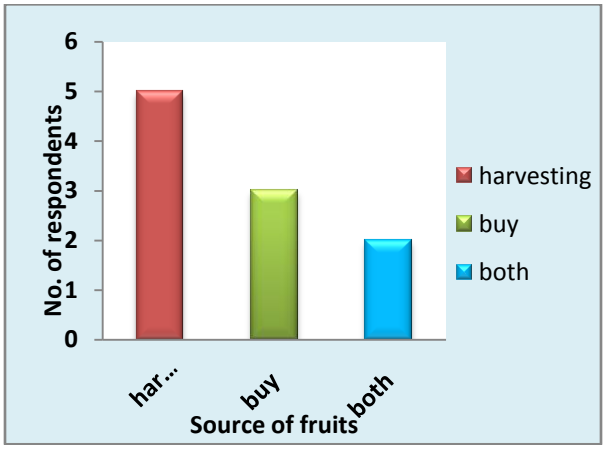

Fig.3. Source of fruits

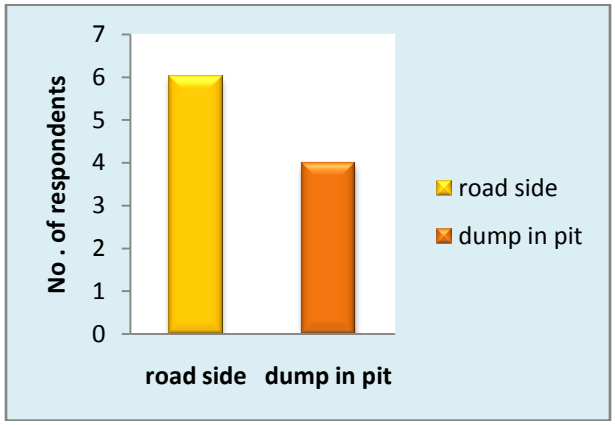

Fig. 4. Ways of disposal of fruit waste

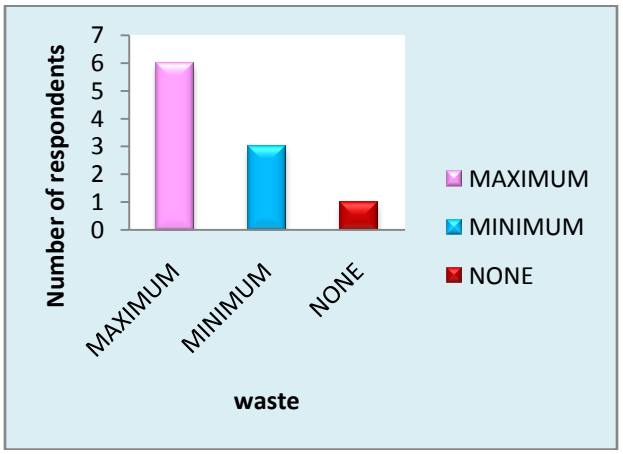

Fig.5. Fruit waste generated per day

Extraction of pectin

Pectin was extracted from the different fruit waste collected from the study site. The amount of pectin extracted was found to be high in the mixture of fruit peels.

Table1. Amount of Pectin formed

Fruit waste	Amount of pectin extracted per 50 g of fruit peel
Banana	6.4%
Citrus	6.8 %
Papaya	6.4%
Mixture of all	8.6 %

The isolation of pectin from different fruit waste was shown in Fig. 6.

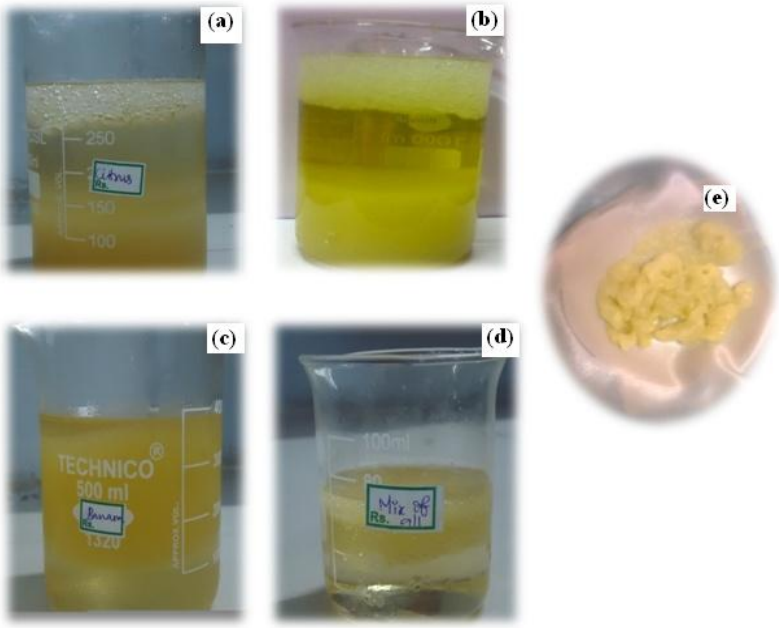

Fig.6. Extraction of pectin from the peels of the fruits (a) citrus, (b) papaya, (c) banana (d) mixture of all and (e) dried pectin

Identification of pectin

Based on the procedure given in the FCC manual, several tests were performed to identify the formation of pectin. All the results were found to be positive and hence the isolated product was confirmed to be pectin. The presence of functional groups viz., acid, amide, and alcohol were confirmed by the qualitative tests.

Characterization of pectin

By using FCC standard

The various parameters of the isolated pectin were determined and compared with the standard FCC values as shown in Table 2. Based on the data it was found that the quality of the isolated pectin lies within the standard FCC values.

Table 2. Comparison of the various parameters of the isolated pectin with that of the standard FCC values.

Physico-chemical Parameters	Fruits				
	Papaya (%)	Banana (%)	Citrus (%)	Mixture of all (%)	Standard FCC value
Acid-Insoluble Ash	0.28	0.32	0.30	0.25	Not more than 1%
Ash (total)	2.50	3.20	2.80	3.50	Not more than 10%
Degree of esterification	31.72	34.32	35.01	32.71	Not more than 50%
Degree of amide ubstitution	17.14	26.86	25.77	28.34	Not more than 40%
Total anhydrogalacturonides	101.0	132.0	128.0	167.0	Not less than 70%

Surface morphology

The morphology of pectin was observed using Scanning Electron Micrograph (SEM).

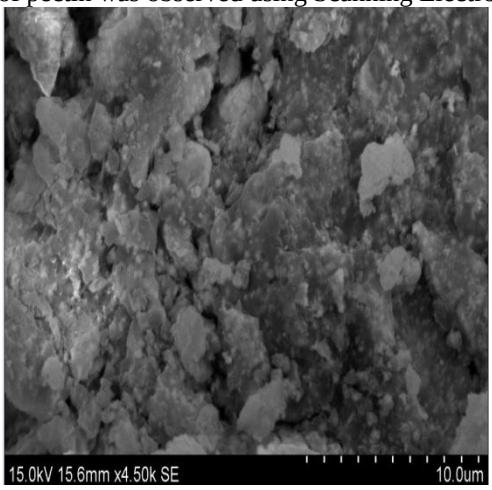

Fig. 7. SEM image of pectin.

Spectral characterization

In the UV – Visible spectra of the formed pectin the absorption peaks found at 288nm and 376 nm coincides with the standard UV- Visible spectra of commercial pectin (Zhang J, et al. 2001).UV-Visible spectra of the isolated pectin form one of the sources is shown in fig.8

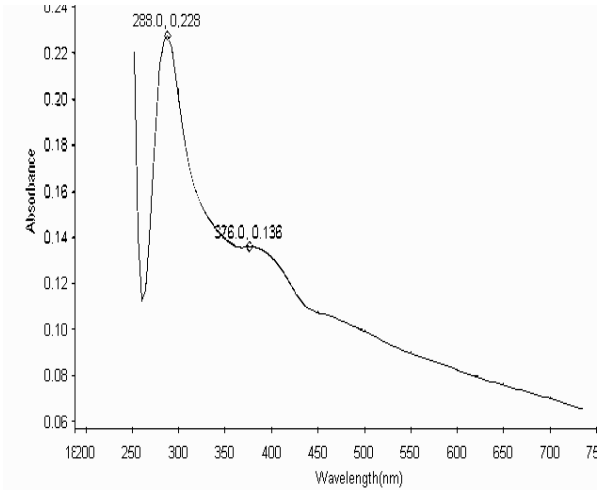

Fig. 8. UV-Visible spectrum of Pectin.

IR spectral data of the isolated pectin from different fruit wastes is shown in Fig. 9. The major functional groups in pectin usually showed characteristic peaks in the region between 1000 and 2000 cm^{-1}. The bands at 1645-1649 cm^{-1} and 1741-1745 cm^{-1} indicatedthe presence of carbonyl group in the free and esterified carboxyl groups. The absorption bands between 1100 and 1109 $cm^{-1}$was due to the presence of ether (R-O-R) and cyclic C-C bonds in the ring structure of pectin molecules. No major difference was observed in the FT- IR spectra of the pectin samples produced by various fruit sources. The broad band around 3400 cm^{-1} may be due to presence of –OH group present in the pectin samples. A doublet in the region 2350 to 2400 cm^{-1} indicated the presence of N–H stretching of some amidated compounds (Rakesh K. Mishra, et al., 2008).

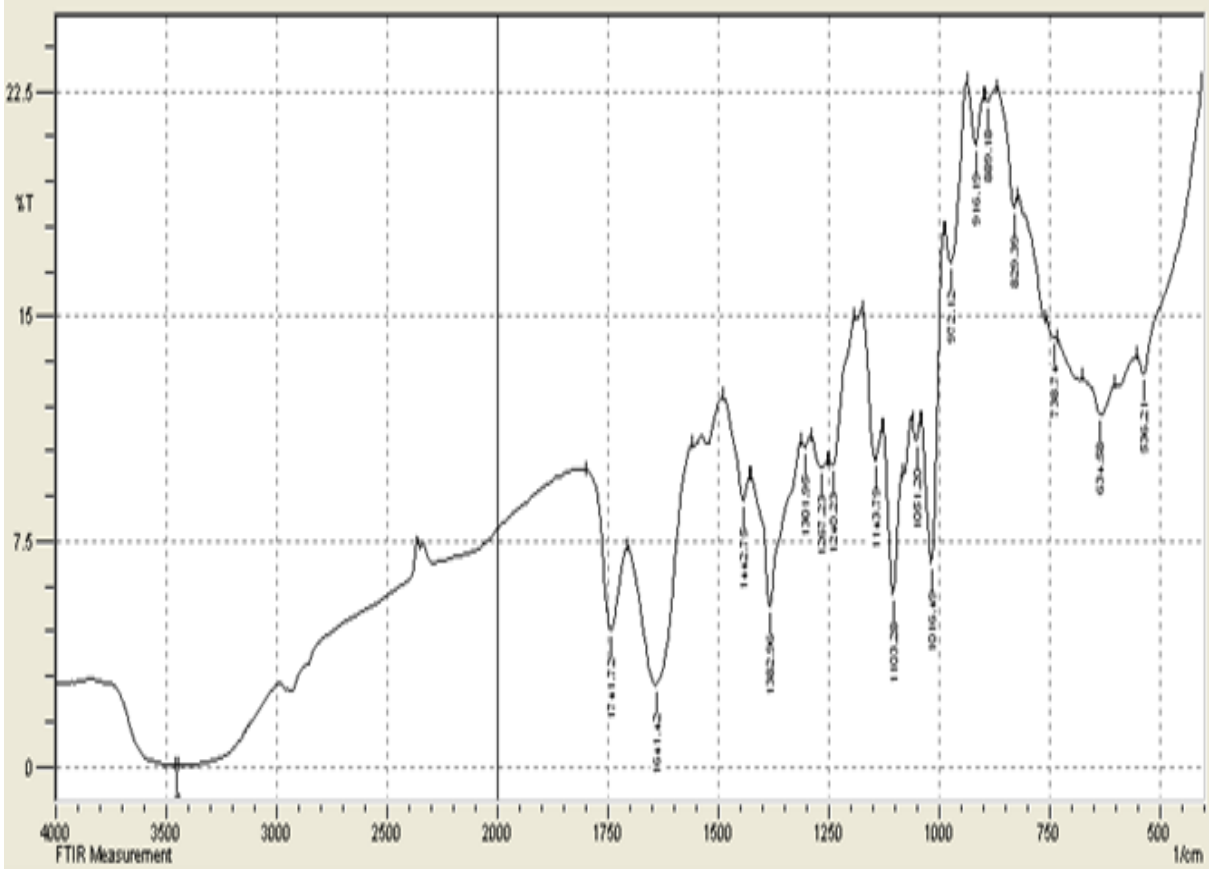

Fig. 9. F-IR Spectrum of isolated pectin

Thermal analysis

TGA and DTA analysis are identified as sensitive techniques to record the thermal properties such as melting, decomposition and weight loss of the given sample. It was observed that the decomposition occurred in two stages for pectin. The first thermal decomposition occurred between 200° and 250°C with a weight loss around 10%. The second weight loss occurred around 430 -500 °C (25%) may be due to the by-product generated by the thermal decomposition of pectin (Sivakumar M. et al., 2006). In DTA curve, an exothermic peak observed at 250°C corresponding to glass transition temperature and the intense peak observed at 350°C represented the crystallization temperature The exothermic peak at 500°C was attributed to thermal degradation of pectin.

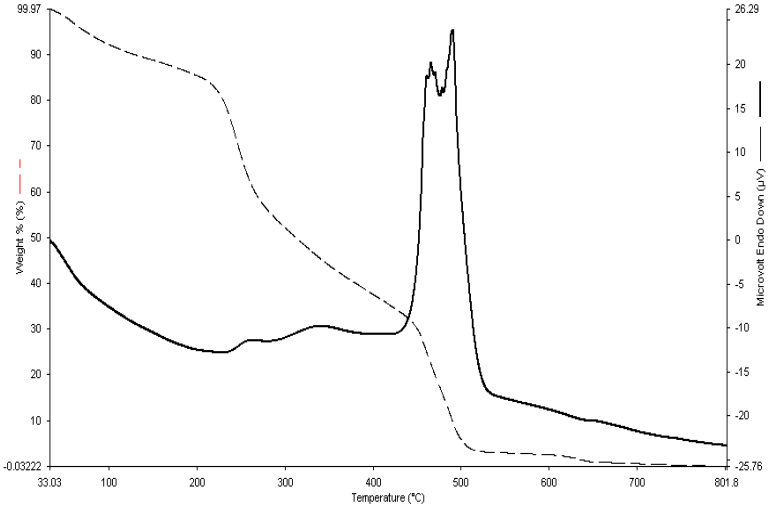

Fig. 10.TGA and DTA curves of pectin.

Conclusion

In this study, the fruit wastes generated from Uzhavar Santhai, Chokkikulam, Madurai was statistically analyzed and pectin, a commercially value-added product, was extracted from the fruit waste. The extracted pectin was tested for its food quality by using FCC standard and characterized by using spectral techniques. The ethanol used in the process was extracted from the pulp of the fruit wastes and therefore this study was considered to be the effective method of municipal solid waste management.

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