e-ISSN 2320 –7876 www.ijfans.org
Vol.11, Iss.7, 2022
 © 2012 IJFANS. All Rights Reserved, UGC CARE Listed (Group -I) Journal

Extraction And Characterization Of Pectin From Watermelon By-Products

N. Saiharini and A. Padmaja*
Dept. of Food and Nutritional Sciences,
Sri Sathya Sai Institute of Higher Learning, Anantapur, India
e-mail: ambatipadmaja@sssihl.edu.in

10.48047/IJFANS/V11/I7/52

Abstract

Watermelon *(Citrullus lanatus)* fruit, which is a seasonal fruit, is a good source of valuable components. Peel, rind and seeds which are the major by-products of watermelon fruit processing industries, are good sources of nutrients, fibre, pectin and phytochemicals. Pectin was extracted from watermelon fruit peel and rind and also from citrus peel for comparison studies. The influence of extracting medium and isolating solvent was studied by using ethanol and acetone independently to know their efficiency. Physical and chemicals properties were studied for the extracted pectin from peel and rind and compared with the citrus and commercial pectin properties. FT-IR analysis was performed to the pectin extracted from peel and rind to determine the function groups present in them. Scanning Electron Microscopy (SEM) analysis was carried out to study the morphology of extracted watermelon fruit peel and rind pectin. Results showed that citric acid extraction along with ethanol gave the highest yield of pectin when compared with acetone. The pectin content ranged between 15-20%. The extracted pectin quality was comparable with that of the commercial pectin. It was observed that the FT-IR spectra of both the pectin samples had characteristic peaks at 3420.9, 2936.8, 1739 and 1082.9 cm-1 corresponding respectively, to –OH, -CH, C=O of ester and acid, and –COC- stretching of the galacturonic acid. It could be concluded from the above investigation that good quality pectin can be extracted from the

 © 2012 IJFANS. All Rights Reserved,

peel and rind using citric acid as an extracting media and ethanol as a precipitating solvent. Recovery of valuable by product pectin from fruit peel and rind not only reduces the waste disposal and environmental pollution but also offers great scope for utilization in development of many functional foods.

Keywords: Watermelon by-products, peel, rind, pectin, extraction and waste utilization

Introduction

The most common problem in food processing is the disposal of the generated by-products. In recent times more attention has been focused to recover valuable components from discarded food parts (food "losses", "wastes", "by-products" or "wasted by-products") and recover and process them in an economic and sustainable way for value addition in foods[1] (Amatullah Quadri and Avanti Rao). *Citrullus lanatus* or watermelon is a tropical fruit that is widely cultivated around the world. The outer part of the fruit is known as the peel which is green with dark green stripes and the inner part of this is rind which is white in colour. The juice or pulp from watermelon is considered as the edible portion but rind, peel and seeds are discarded as major solid wastes. As watermelon rind represents about one third of the total fruit mass, researchers have been trying to discover new ways to utilize it. Although some studies have shown that watermelon rind can be used as an adsorbent for the removal of heavy metals from aqueous solution, it is considered as waste and has no commercial value. The rind contains mineral salts, fat, protein, carbohydrates, vitamins, phytochemicals and citrulline. Watermelon is mainly used by local juice vendors, which generate large amounts of waste without a proper disposal treatment. These watermelon residues have great potential as a source of pectin and other value-added products. Watermelon peels account for roughly 30% of the total mass of the fruit. As a high-emission agricultural waste, watermelon peel has become a serious environmental concern, making it necessary to understand the sustainable

 © 2012 IJFANS. All Rights Reserved,

value of the peel from both ecological and economic perspectives. Pectin is a polysaccharide that contains mainly galacturonic acid joined by α-(1,4) linkages. It is also a multifunctional component found in the intercellular space and middle lamella of higher plants. In food and pharmaceutical industries pectin, which is a biopolymer is widely used due to its hydrocolloid properties. By-products of the apple and citrus fruit industries are the major pectin sources. In food industry, pectin is widely used as a food additive. It can function as a stabilizer in beverages such as yogurt drinks and milk, a thickener in bread, frozen dough and yogurt, an emulsifier for cream, milk and ice cream and a gelling agent for jam and jellies[2] [Khye Yeong Lee and Wee Sim Choo). The quality parameters of pectin include extraction yield and chemical properties such as ash content, free acidity, methoxyl content, degree of esterification, anhydrouronic acid content and Fourier transform infrared spectroscopy (FTIR) analysis. Pectin can be classified based on degree of esterification as high methoxyl pectin (> 50%) and low methoxyl pectin (< 50%) which define its gelling properties. High methoxyl pectin (DE > 50%), characterized by forming gels in high soluble solids and acidic systems. In low methoxyl pectins (DE < 50%), the gelation occurs on a widespread pH range than high methoxyl pectin, and require the presence of divalent cations. However, pectin from agro-industrial processes wastes is a growing interest currently, as their usage can reduce environmental impacts and could add value to the agro-industrial production chain. Hence, watermelon peel and rind can be used to produce pectin, while adding value to the watermelon's agribusiness. Several works have been reported on the extraction of pectin from watermelon wastes by using different methods such as acid hydrolysis and assisted microwave extraction[3] (Jose Perez). However, the acid hydrolysis is mostly employed method for the pectin extraction from food waste. Therefore, the objectives of the present study are to standardise the pectin extraction method from watermelon fruit peel and rind using citric acid as an extracting media and to determine optimal conditions for the highest

 © 2012 IJFANS. All Rights Reserved,

pectin yield and to characterize and analyse the physicochemical properties of pectin which determines the quality of the extracted pectin.

Methods and Material

Procurement and preparation of raw materials

Watermelon fruit peel and rind were procured from small scale juice processing units in Anantapur. Analytical grade chemicals were procured from SRL, SD Fine and Sigma companies.

Watermelon peels and rind were washed thoroughly with deionized water. They were sliced further with a stainless-steel knife. Any adhering pulp was scooped out from the rind with the spoon.

Preparation of peel and rind powder

The fruit peel was grated and rind was cut into small pieces with the stainless-steel knife and they were kept for drying in the tray drier at 40ºC. After drying, they were powdered using the laboratory mixer. It was stored in an airtight container for further analysis.

Variables of extracting pectin

Extracting media

Citric acid and Sodium hexa metaphosphate (SHMP) were used to extract pectin from watermelon fruit peel and rind in different concentrations. Extracting media at different pH i.e., 1.0, 1.5 and 2.0 were prepared and were used for extraction of pectin from the dried watermelon fruit peel and rind powder.

Extraction time

Extraction of pectin was varied for 30, 60 and 90 minutes to determine the time of extraction required for maximum yield of pectin from the dried peel and rind powder.

 © 2012 IJFANS. All Rights Reserved, UGC CARE Listed (Group -I) Journal

Extraction temperature

Peel and rind powder were weighed and extractant solution was added to the samples and transferred into a 250ml conical flask. They were kept on a thermostatically controlled water bath at the required temperature. The temperature of the water bath during extraction was maintained at 70, 80, 90 and 100˚C.

Pectin extraction using SHMP

Pectin from peel and rind was extracted individually using the extractant SHMP solution corresponding to different pH by refluxing for 2hrs. The mixture was centrifuged at 2000 rpm for 15min. To the supernatant equal amounts of ethanol was added with continuous stirring and kept aside for incubation. It was filtered through whatmann paper and the precipitate was washed with ethanol to remove the impurities. The pectin was dried and stored in an air tight pouch under refrigerated conditions.

Pectin extraction using citric acid

Peel and rind powder were treated with citric acid solution at different pH and pectin was precipitated with ethanol and then filtered. The pectin thus obtained was further dried and stored in air tight conditions for further analysis and application studies.

Physico-chemical properties of pectin

Physical properties of pectin

The extracted pectin from peel and rind was analyzed for its quality aspects like color and solubility. Color was measured using a colour reader (Konica MINOLTA CR -10), using the Hunter L, a and b units, where L indicates luminosity or brightness, a'corresponds to greenness (-) / redness (+) and b' corresponds to blueness (-) / yellowness (+). Solubility in hot and cold water was determined by following the procedure described[4] and solubility in hot and cold alkali were determined by following the procedure[5].

Chemical properties of pectin

The extracted peel and rind pectin was estimated for its ash content, equivalent weight and methoxyl content by using the methods[6]. Anhydrouronic acid content and degree of esterification was estimated by following[7].

Characterization of pectin

Scanning electron microscopy (SEM)

Watermelon peel and rind pectin was subjected to SEM analysis to determine the morphology. It was carried out using Environmental scanning electron microscope of Joel Company Japan, JSM – IT – 300 model. SEM analysis is a near surface technique, as it measures the electrons produced by an electron gun which strikes the specimen being irradiated. Pectin samples were mounted on platinum studs after sputtering and were observed under SEM[8] (Shan Qin et.al. 2014).

Fourier Transform Infrared Spectroscopy (FTIR) analysis

The FTIR spectra of both the pectin samples were obtained at a resolution of 4 cm–1. Spectra were recorded using a Nicolet iS10 spectrometer (Thermo Fisher Scientific, USA) with KBr method[9] in the wavenumber range of 4000–500 cm^{-1} and resolution of 4 cm^{-1}.

Statistical analysis

Analysis of samples was carried out in triplicates. Values were expressed as means of three independent samples analysed in triplicate ± standard error of mean (SEM). The data analysis was done using Microsoft Excel software 2019.

Results and Discussion

Standardization of pectin extraction methods from watermelon fruit peel and rind

The influence of extracting medium and isolating solvent was studied. Different extracting mediums such as Citric acid and Sodium hexameta phosphate and solvents like ethanol and

e-ISSN 2320 –7876 www.ijfans.org
Vol.11, Iss.7, 2022
 © 2012 IJFANS. All Rights Reserved, UGC CARE Listed (Group -I) Journal

acetone were used independently as precipitating solvents to standardize the extraction method. Each showed different results with different extracting mediums.

Results showed that the citric acid along with the ethanol gave the highest yield among the other chemicals and solvents as shown in Figure.No.2. The average pectin content was 15-17% which was in the range of citrus peel pectin content (15-30%).

Citrus peel (*Citrus limetta*) commonly known as mausambi was taken for comparative study on pectin. Citrus peel was dried, made into powder and stored in an air tight pouch until for further analysis. Pectin was extracted from the citrus peel following the same procedure as it was for watermelon fruit peel and rind. The extracted citrus peel pectin was also analysed for its chemical characteristics. Commercial pectin was used as a standard and analysed for the same parameters as it was for citrus pectin.

Effect of pH and extracting media

Figure.No.2. shows the Citric acid at 2.0 pH was efficient in extracting and isolating pectin from peel and rind. pH is one of the important parameters which affects the quantity and properties of extracted pectin.

Effect of extraction time

The yield of peel and rind pectin at different extraction times is shown in Figure.No.3. It shows that extraction time of 90 minutes gave a maximum yield of pectin. The extraction time was maintained at 30, 60 and 90 minutes, among which 90 minutes proved to be the best extraction time.

Effect of temperature

Effect of extraction temperature on the yield of pectin is represented in Figure.No.4. Extraction of pectin was carried out at different temperatures keeping pH of extractant at 2.0

and extraction period of 90 minutes. The extraction was carried out at 70, 80, 90 and 100℃. With the increase in temperature of extraction from 70-100℃ the yield of pectin significantly increased. The total yield of pectin was significantly less at 70-80℃ and as the temperature increased the yield of pectin was also increased being maximum at 100℃ and further increase in the temperature resulted in less yield which could be due to the partial degradation of pectin. Hence, the optimum temperature for both the pectin extraction was found to be 100℃.

Effect of precipitating solvents

Ethanol and acetone were used independently as precipitating solvents in different volumes to know their efficiency. Ethanol in equal volumes added to the filtrate gave the highest yield of pectin when compared with that of the acetone which was used double the volume of filtrate. In case of ethanol precipitation, highest yield and best quality pectin was obtained from peel and rind.

Colour of pectin

The colour of the peel and rind pectin obtained was brown in colour and values was L 33, a=7.0 and b= 4.7. Colour of the pectin is one of the crucial parameters which could affect the appearance of the products. Different extraction conditions had different effects on both the pectin colour. Pectin extracted at high temperature (100℃) was highly coloured than those extracted at low temperatures (70 and 80℃).

Physical properties of pectin

Solubility of pectin in water

 © 2012 IJFANS. All Rights Reserved,

The dried pectin was tested for its solubility in hot and cold water. The dried peel and rind pectin was insoluble in cold water but it was soluble in hot water at 85-90℃ as given in **Table.No.1.**

Solubility of pectin in alkali

In cold alkali (NaOH) the peel pectin formed pale yellow precipitate and rind pectin formed very light yellow where as in hot alkali at 85-90℃ both the peel and rind pectin were soluble and formed pale brown solution as given in Table.No.1. Solubility tests were done to citrus peel pectin whereas the result was almost same to that of the watermelon fruit peel and rind pectin. Both pectins were insoluble in cold water and cold alkali, but were soluble in hot water and hot alkali. Watermelon fruit pectin formed pale yellow precipitate whereas citrus pectin formed yellow precipitate.

Chemical properties of pectin

Ash

The ash content of the extracted watermelon fruit peel and rind pectin, citrus pectin and commercial pectin was 1.5 and 1.1%, 1.6% and 1.28% as given in Table.No.2. The inorganic impurities in pectin are indicated by the ash content and lower ash content indicates good quality of pectin. The upper limit of ash content is considered to be as 10% for good quality pectin. The results of the present study showed that the extracted pectins were within the range. The difference between the ash content of fruit peels pectin and commercial pectin could be due to presence of natural substances present in peels.

Methoxyl content

Suitability of pectin for different purposes would be determined by their methoxyl content. Methoxyl content is an important factor for controlling the setting time of pectin and the

 © 2012 IJFANS. All Rights Reserved,

ability to form gels. If 50% of carboxyl groups are methylated it is said to be high methoxy pectin and less than 50% is called low methoxy pectin. The methoxyl content of watermelon fruit peel and rind pectin in the present study was 5.59 and 5.52% as shown in Table.No.2. Methoxyl content was checked in the citrus pectin and commercial pectin. Citrus pectin had 9.3 and commercial pectin had 15.3% of methoxyl content which was more than the watermelon fruit peel and rind pectin.

Equivalent weight

The equivalent weight of the extracted pectin from peel and rind was 508.43 and 417.65 g/ml as given in Table.No.2. It was estimated in both the citrus and commercial pectin. Citrus pectin had 500.66 and commercial pectin had 417.05 g/ml which was lesser than the watermelon fruit peel and rind pectin.

Anhydrouronic acid content

The anhydrouronic acid was estimated to be 65 and 60% as shown in Table.No.2. for watermelon peel and rind pectin which indicates its purity. Estimation of anhydrouronic acid is important to determine its purity and degree of esterification and also to evaluate the physical properties of pectin. Anhydrouronic acid content was estimated in citrus and commercial pectin. Citrus pectin had 87% that is higher than the watermelon fruit peel and rind pectin. Commercial pectin had 130.74% which was more than the citrus and watermelon fruit peel and rind pectin.

Degree of esterification

The degree of esterification in watermelon fruit peel and rind pectin was 55 and 52% as given in Table.No.2. The degree of esterification is an important factor to classify pectins which is expressed as a percentage of the esterified carboxyl groups. Pectin can be classified based on degree of estarification as high methoxyl pectin (> 50%) and low methoxyl pectin (< 50%). Hence this study indicates that watermelon fruit peel and rind pectin can be categorized as high methoxyl pectin. Degree of esterification was analysed in both the citrus and commercial pectin and was found that citrus pectin had 60% and commercial pectin had 67.71%.

Characterization of pectin

Scanning Electron Microscope (SEM)

Morphology of watermelon fruit peel and rind pectin samples which were extracted under acidic condition was characterized by using SEM and the micrographs are shown in Figure No.5. Both the peel and rind pectin were subjected to SEM analysis to study the morphological structure of the pectin. Figure No.5. shows watermelon fruit peel pectin continuous nanostructure and flaky in shape with mild wrinkles on surface. Watermelon rind pectin had a block shape and a wrinkled surface with a compact texture as it was shown in Figure.No.5.

Fourier Transform Infrared Spectroscopy (FTIR) analysis

Validation of the pectin from watermelon rind and peel was done using FTIR as shown in Figure.No.6 &7. The peaks at approximately 3,429 and 2,928 cm^{-1} corresponded, respectively, to stretching vibrations of hydroxyl groups (OH) and C–H of CH, CH_2, and CH_3 groups. The characteristic chemical shifts of 3360, 2920 and 1201 cm^{-1} were due to inter and intramolecular hydrogen of O-H, C-H of CH_2 and CH_3, and C-O-C of glycoside compounds. The chemical shifts of 1646 and 1734 cm^{-1}, as the proprietary signals of pectin structure, are attributed to the free and esterified carboxyl groups, respectively. It was observed that the FT-IR spectra of both the peel and rind pectin samples had characteristic peaks at 3420.9, 2936.8, 1739 and 1082.9 cm^{-1} corresponding respectively, to –OH, -CH, C=O of ester and acid, and –COC- stretching of the galacturonic acid.

Conclusion

It could be concluded from the current study that good quality pectin can be extracted from the watermelon fruit peel and rind using citric acid as an extracting media and ethanol as a precipitating solvent. The extracted pectin quality was comparable with that of the commercial pectin. FT-IR spectra showed the functional characteristic groups present in the extracted peel and rind pectin. Recovery of valuable by product pectin from fruit peel not only reduces the waste disposal and environmental pollution but also offers great scope for utilization in development of many functional foods. The techniques developed for extraction of pectin, will add value and reduce environmental impact of inappropriate disposal of food processing waste.

References

1. Amatullah Quadri and Avanti Rao. Development, Organoleptic Evaluation and Acceptability of Products Developed from Agro Based Wastes-Seeds and Rind of Watermelon. Advances in Recycling and Waste Management. 2018 August; 3:2.
2. Khye Yeong Lee and Wee Sim Choo. Extraction Optimization and Physicochemical Properties of Pectin from Watermelon (Citrullus lanatus) Rind: Comparison of Hydrochloric and Citric acid Extraction. Journal of Nutraceuticals and Food Science. 2020 January; Vol.5 No.1:1.
3. Jose Perez1, Karina Gomez & Lorena Vega. Optimization and Physicochemical Characterization of Pectin Extraction from Watermelon Rind (Citrullus lanatus) with Citric Acid. Research Square. 2021 March.

4. Fishman, M.L., H.K. Chau, D.R. Coffin and A.T. Hotah kiss. A comparison of Lime and orange pectin which were Rapidly Extracted from Albedo. Advances in pectin and pectinase Research. 2003; 6: 107-122.

5. Joslyn, M.N. Methods of Food Analysis. Physical, Chemical and Instrumentation Method of Analysis. 1980; second edn. Academic Press, New York, pp: 67-70.

6. Ranganna, Handbook of analysis and quality control for Fruit and Vegetable, 2nd edition, 1986, M.C Grawhill publishing Co.Ltd. New Delhi.

7. ErmiasGirma and Mr. TeshomeWorku. Extraction and characterization of pectin from selected fruit peel waste. International journal of scientific and research publications 2016; Volume 6, Issue 2, 2250-3153.

8. Shan qin Liew, Nyuk Ling Chin, Yus Aniza Yusof. Extraction and characterization of pectin from passion fruit peels. Agriculture and Agricultural Science Procedia. 2014; 2:231-236.

9. Seyed Saeid Hosseini, Faramarz Khodaiyan, Milad Kazemi, Zahra Najari. Optimization and characterization of pectin extracted from sour orange peel by ultrasound assisted method. International Journal of Biological Macromolecules. 2019; 125: 621–629

 © 2012 IJFANS. All Rights Reserved,

Table.No.1. Physical properties of pectin

Parameter	Watermelon peel pectin	Watermelon rind pectin	Citrus pectin
Colour	Light yellow	Very light yellow	Yellow
Solubility in cold water	Insoluble	Insoluble	Insoluble
Solubility in hot water (85-90°C)	Mixture dissolves	Mixture dissolves	Mixture dissolves
Solubility in cold alkali	Pale brown precipitate	Pale brown precipitate	Yellow precipitate
Solubility in hot alkali (85-90°C)	Soluble, formation of pale-yellow solution	Soluble, formation of pale-yellow solution	Soluble, formation of yellow solution

Table.No.2. Chemical properties of pectin

Parameter	Watermelon peel pectin	Watermelon rind pectin	Citrus pectin	Commercial pectin
Ash (%)	1.5 ± 0.04	1.1 ± 0.03	1.6 ± 0.04	1.28 ± 0.02
Methoxyl content (%)	5.59 ± 0.02	5.52 ± 0.01	9.3 ± 0.01	15.3 ± 0.02
Equivalent weight (g/ml)	508.43 ± 0.03	417.65 ± 0.02	500.6 ± 0.02	417.05 ± 0.05
Anhydrouronic acid content (%)	65 ± 0.01	60 ± 0.01	87 ± 0.01	130.7 ± 0.02
Degree of esterification (%)	55 ± 0.01	52 ± 0.01	60 ± 0.01	67.71 ± 0.02

 © 2012 IJFANS. All Rights Reserved,

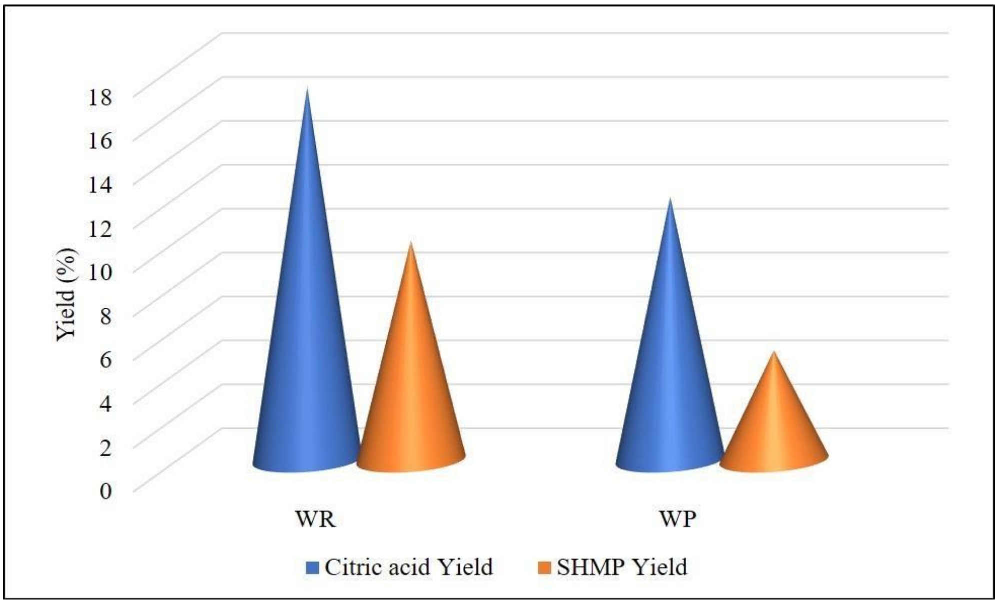

Figure.No.1. Effect of extractant on extraction of pectin

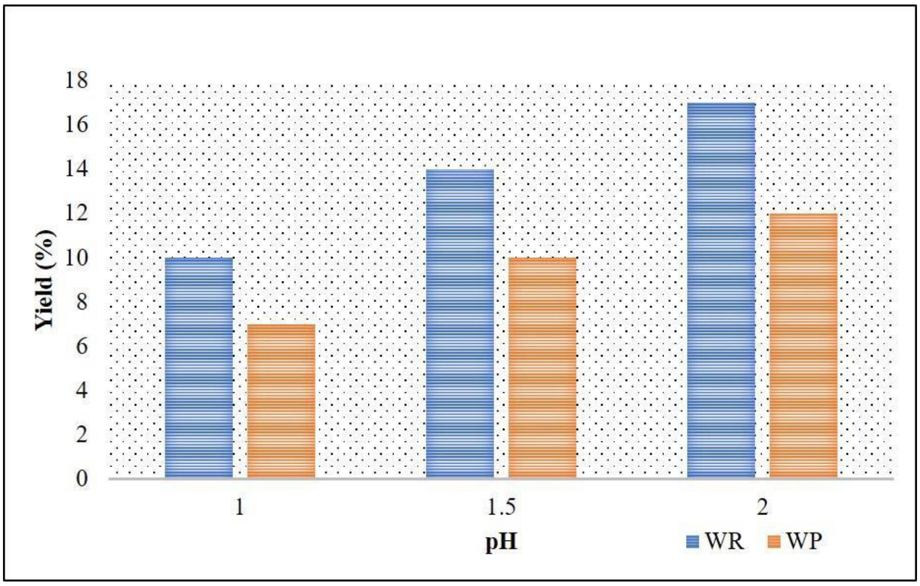

Figure.No.2. Effect of pH on extraction of pectin

 © 2012 IJFANS. All Rights Reserved,

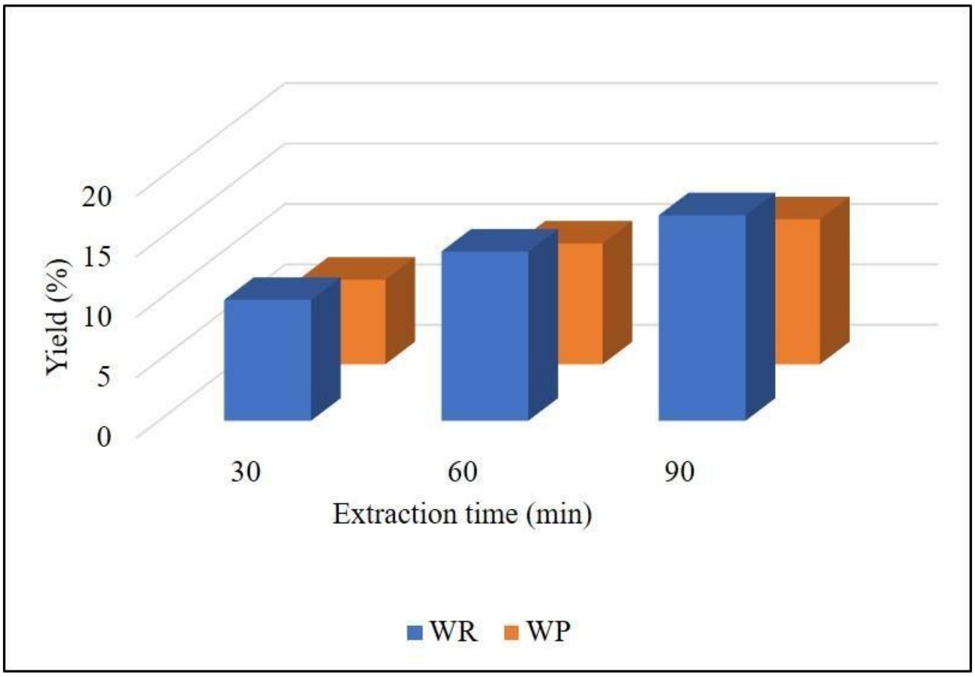

Figure.No.3. Effect of time on extraction of pectin

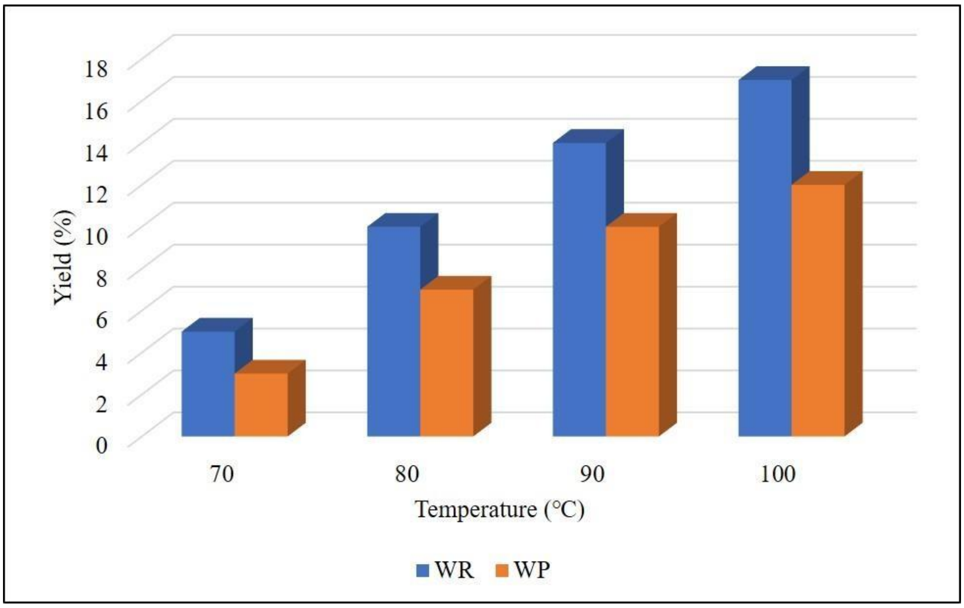

Figure.No.4. Effect of temperature on extraction of pectin

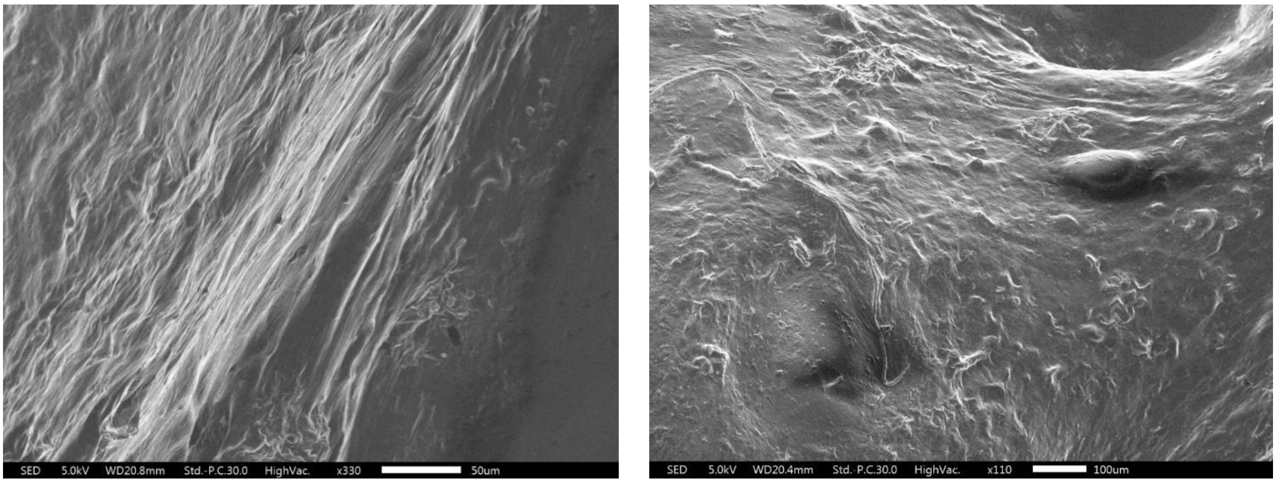

Figure.No.5. Micrograph of watermelon fruit peel and rind pectin

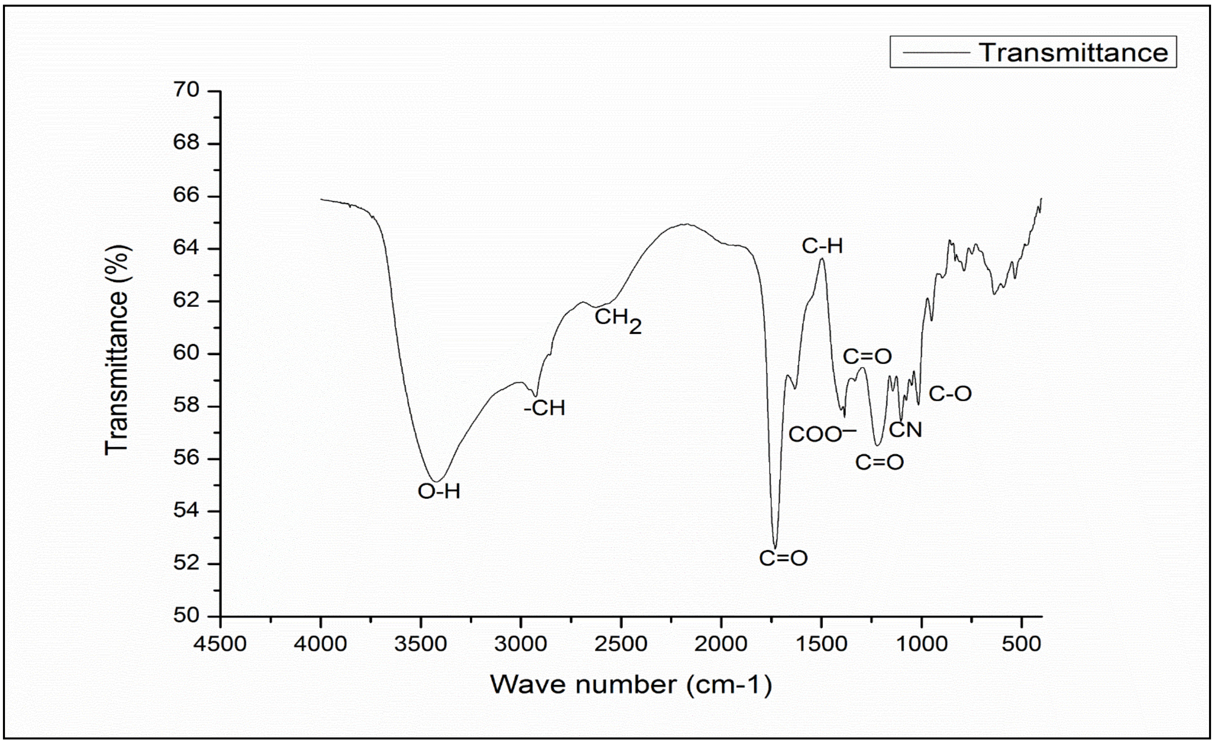

Figure.No.6. Fourier Transform Infrared Spectra (FTIR) of watermelon peel pectin

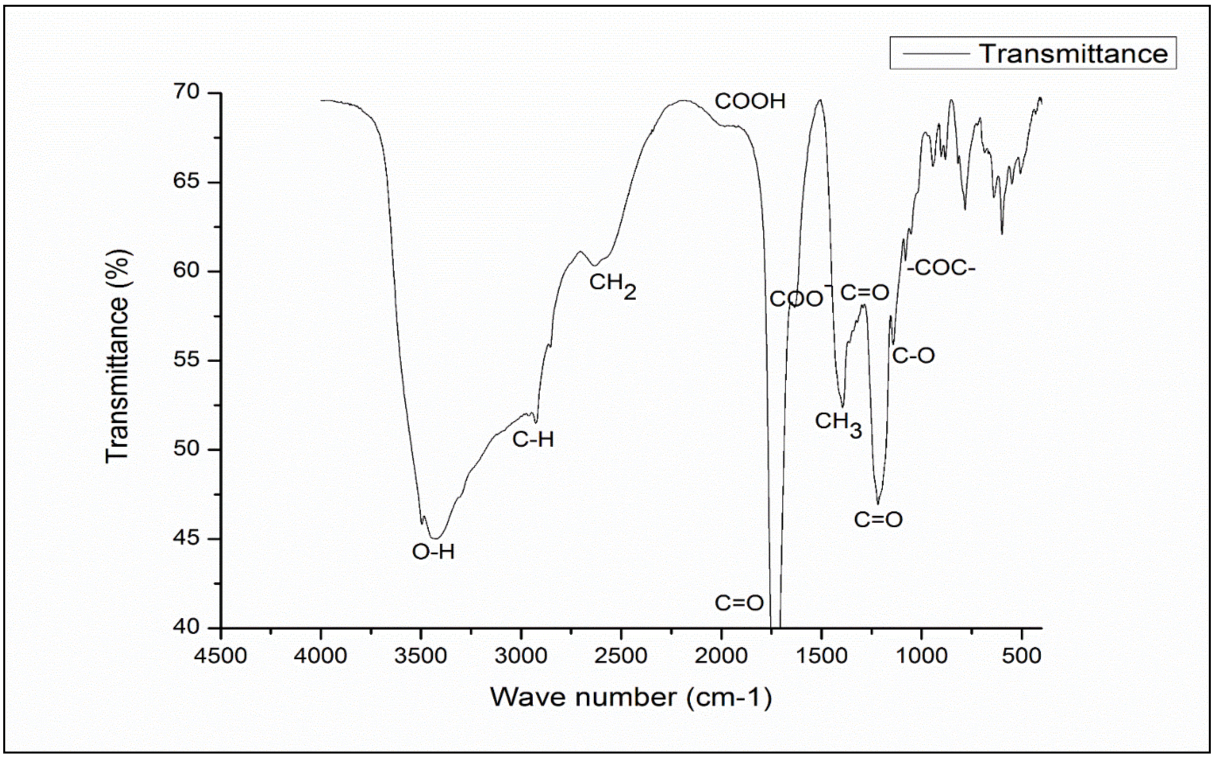

Figure.No.7. Fourier Transform Infrared Spectra (FTIR) of watermelon rind pectin