

## COMPARATIVE STUDIES ON CONVENTIONAL (WATER-HOT ACID) AND NON-CONVENTIONAL (ULTRASONICATION) PROCEDURES FOR EXTRACTION AND CHEMICAL CHARACTERIZATION OF PECTIN FROM PEEL WASTE OF MANGO CULTIVAR *CHAUNSA*

SHABANA KAUSER<sup>1</sup>, ASMA SAEED<sup>1</sup> AND MUHAMMAD IQBAL<sup>1,2,\*</sup>

<sup>1</sup>Food and Biotechnology Research Centre, PCSIR Laboratories Complex, Ferozpur Road Lahore-54600, Pakistan

<sup>2</sup>Centre for Applied Molecular Biology, 87-West Canal Bank Road, Thokar Niaz Baig, Lahore-53700, Pakistan

\*Corresponding author e-mail: iqbalmdr@brain.net.pk ; Tel.: +92 42 99230688; Fax: +92 42 99230705

### Abstract

Pectin, a naturally occurring heteropolysaccharide, is widely used as a functional ingredient in food and pharmaceutical industries due to its gelling and stabilizing properties. During the present study pectin was extracted from peel of mango (cultivar *Chaunsa*) using conventional (water-hot acid) and non-conventional (ultrasonication) methods. In conventional method, HNO<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub>, or HCl was used under variable conditions of pH (2.0, 2.5, 3.0), temperature (70, 80, 90, 100°C), duration of extraction (30, 60, 90, 120 min), and solvents (ethanol, methanol, acetone, isopropyl alcohol). Maximum yield of 16.6 g pectin 100 g<sup>-1</sup> peel was obtained with HNO<sub>3</sub> at pH 2.5, 90°C, 90 min extraction, and ethanol. Whereas in non-conventional method, ultrasonication was used for different time intervals (10, 20, 40 min) using HNO<sub>3</sub> at pH 2.5 and 90°C. Maximum yield of 15.8 g pectin 100 g<sup>-1</sup> peel was obtained by this method in 20 min. Pectin extracted by the above two methods was found to be of high quality as was determined in respect of methoxyl and galacturonic acid contents, degree of esterification, equivalent weight, and FTIR spectra. Extraction of pectin from mango peel by employing non-conventional method (ultrasonication) was observed to be an energy efficient method due to its less extraction time (20 min as compared to 90 min in conventional method) suggesting its suitability on commercial scale for the extraction of pectin from mango and other available fruit peel wastes.

**Key words:** Water-Hot acid, Ultrasonication, Pectin, Peel waste, Mango

### Introduction

Mango (*Mangifera indica* L.) is a delicious tropical fruit, with worldwide annual harvest of >35 million tons, to which Pakistan contributed 1.8 million tons as the fourth biggest producer in 2011 (Anon., 2011; Anon., 2011). However, postharvest losses and diseases cause huge damages to mango crop (Ullah *et al.*, 2012; Khan *et al.*, 2013; Raza *et al.*, 2013; Nafees *et al.*, 2013). Besides its consumption as fresh fruit, about 20% of the produce is processed into several kinds of commercial products (Mahayothee, 2005). During the processing huge quantities of mango waste are generated, mainly peel and stone-kernel, respectively, accounting for 7-24% and 9-40% of the whole mango fruit, both being good source of valuable phytochemicals (Ashoush & Gadallah, 2011). Stone-kernel has been studied for the extraction of edible oil, polyphenolic antioxidants, antimicrobial activity, as animal-feed constituent, and production of activated carbon (Teguia, 1995; Abdalla *et al.*, 2007a, 2007b; Koubala *et al.*, 2008). Peel, the other principal waste component, is a source of high quality dietary fibre, pectin, and antioxidants (Koubala *et al.*, 2008; Berardini *et al.*, 2005). Among these, pectin has significant commercial potential, as its annual global market is estimated in excess of 30,000 tons with 4-5% growth per annum (Yeoh *et al.*, 2008).

Pectins are complex polysaccharides, located mainly in primary cell walls and middle lamellae of plant tissues (Bagherian *et al.*, 2011). It contains a backbone of (1→4) α-D-galacturonic acid (GalA) residues, esterified with methyl alcohol at the carboxylic acid end (Wang *et al.*, 2007). Pectins are characterized by high GalA content, which FAO and EU regulations require to be at least 65% (Willats *et al.*, 2006). Pectins are classified by their GalA content, as high methoxyl (HM) and low methoxyl (LM), with respective

degree of esterification of >50% and <50% (Lima *et al.*, 2010). The gels in HM pectins are formed in the presence of high concentrations of soluble solids (>55%) at low pH (2.5-3.8), whereas LM pectins form gels in the presence of divalent cations such as Ca<sup>2+</sup> and low (≈30%) soluble solids (Willats *et al.*, 2006; Koubala *et al.*, 2008). Pectins are widely used in food, cosmetics and pharmaceutical products as thickening, stabilizing, emulsifying, and gelling agent.

Industrial production of pectin is done by water-hot acid extraction of citrus peel and apple pomace yielding homogalacturonan polymers with low (≈50%) or high (≈70%) degree of methylation (Basanta *et al.*, 2012). Other sources of pectin are also known (Gan *et al.*, 2010; Kulkarni & Vijayanand, 2010; Basanta *et al.*, 2012). Procedures for the extraction of pectin from mango peel have been reported (Berardini *et al.*, 2005; Koubala *et al.*, 2008), which have however not evaluated the comparative efficiency of different mineral acids and solvents under variable conditions. The content and degree of esterification of pectin in different mango cultivars, furthermore, have been reported to range considerably (Berardini *et al.*, 2005). The present comparative study was done, therefore, to extract mango peel pectin from a popular Pakistani cultivar *Chaunsa*, not investigated before for the purpose, using several mineral acids and recovery solvents. Efficiency of pectin recovery by the water-hot acid method was further compared with ultrasonication method, which no study has been reported earlier for pectin recovery from mango peel. Ultrasonication has been reported to reduce the extraction period, thus preventing pectin degradation and improving both quantity and quality of the recovered pectin (Bagherian *et al.*, 2011). This study also reports the qualitative and quantitative characteristics of mango peel pectin, extracted using acid and ultrasonication methods.

## Materials and Methods

**Sample preparation:** Fresh mango peel of the local *Chaunsa* (vern., Urdu; origin, Pakistan) cultivar, collected from the pilot plant of Food and Biotechnology Research Centre, PCSIR Laboratories, Lahore, Pakistan, was washed thoroughly under running tap water to remove the adhering pulp and fibre, blanched at  $90\pm 1^\circ\text{C}$  for 2-3 min, sun-dried, milled to the U.S. 80 mesh size powder, packaged in airtight polyethylene bags, and stored in desiccators till further processing.

**Water-hot acid extraction of pectin (conventional method):** Mango peel powder (100 g) was added to 1000 ml of deionized water (1:10 w/v) and well-shaken into a uniform slurry. Three mineral acids, viz., HCl,  $\text{H}_2\text{SO}_4$ , or  $\text{HNO}_3$ , were separately added to the peel slurry to achieve pH 2.0, 2.5 and 3.0. The pH-adjusted peel slurries were incubated at different temperatures (70, 80, 90,  $100^\circ\text{C}$ ) for various intervals of time (30, 60, 90, 120 min) with constant stirring. After completion of the respective incubation periods, the acid-treated slurries were filtered through cheesecloth and cooled to  $4^\circ\text{C}$  for 60 min to obtain pectin extract. Pectin was precipitated with commercial grade ethanol (1:1; ethanol to pectin extract) during investigation on all variables, after 90% ethanol was noted to be a better precipitating solvent than 75% methanol, 75% acetone, or 65% isopropyl alcohol (Fig. 1). The precipitated pectin was filtered, washed with ethanol, and dried at  $40\pm 1^\circ\text{C}$  in hot air oven (Mitchell Dryer, UK) to constant weight. Pectin yield was calculated as follows:

$$\text{Pectin (g/100g)} = \frac{\text{Weight (g) of dried pectin}}{\text{Weight (g) of dried powdered peel}} \times 100 \quad (\text{Eq. 1})$$

The extracted pectin was ground to U.S. 60 mesh size, stored in airtight containers at  $10^\circ\text{C}$  for further studies on chemical characterization.

**Ultrasonication extraction of pectin (non-conventional method):** Ultrasonic waves were applied as a non-conventional method for the extraction of mango peel pectin. Peel powder (100 g) was added to 1000 ml of deionized water (1:10 w/v) and well-shaken into a uniform slurry. pH of the slurry was adjusted to 2.5 with three mineral acids, namely, HCl,  $\text{H}_2\text{SO}_4$ , or  $\text{HNO}_3$ , and placed in an ultrasonication cycle apparatus (180 W, Au-60C, Eyela, Japan). The ultrasonicator was set to emit intermittent cycles of ultrasound waves (waves emitted for 30s, no waves emitted for 30s), automatically repeated for the set time of 10, 20 and 40 min. Ultrasonication of the extraction slurry was done in a waterbath at  $90^\circ\text{C}$ . Temperature was monitored throughout the ultrasonication procedure and as soon as a change in the set temperature occurred, it was adjusted by manipulating the water bath temperature control. It was noted that ultrasound waves had no instant effect on the temperature of extraction slurry, which remained around  $2\pm 1^\circ\text{C}$  during the time periods studied. The extraction slurry was removed from the ultrasonicator, filtered through cheesecloth, cooled to  $4^\circ\text{C}$  for 60 min, followed by pectin

precipitation with ethanol and subsequent procedure as described in above section. The yield of pectin was calculated according to Eq. 1.

## Chemical analysis and characterization of extracted pectin

**Moisture and ash content:** Both moisture and ash contents were determined in accordance with AOAC methods (Anon., 2005). Briefly, a known weight of peel sample was placed in a pre-weighed crucible overnight at  $105\pm 2^\circ\text{C}$ , and the weight loss was used to calculate percent moisture content. For ash determination, 1.0 g of peel was ignited in a pre-weighed porcelain crucible in a muffle furnace at  $600\pm 5^\circ\text{C}$  for 3 h, and weighed after ignition to determine percent ash.

**Equivalent weight determination:** Equivalent weight was determined according to the method of Owens *et al.*, (1952). Briefly, to 0.5 g pectin was added 5 ml ethanol in Erlenmeyer flask, followed by the addition of 1.0 g NaCl and 100 ml of  $\text{CO}_2$ -free distilled water, and titrated against 0.1 N NaOH using phenol red as indicator to pink end-point. Equivalent weight was determined as below:

$$\text{Equivalent wt.} = \frac{\text{Weight (g) of dried pectin}}{\text{Vol. of NaOH} \times \text{N of NaOH}} \times 100 \quad (\text{Eq. 2})$$

**Methoxyl content:** To the neutral solution as obtained in equivalent weight determination, 25 ml of 0.25 N NaOH was added, mixed thoroughly, stoppered, allowed to stand at room temperature for 30 min, and 25 ml of 0.25 N HCl was added to titrate with 0.1 N NaOH using phenol red as indicator to pink end-point, and methoxyl (MeO) content was determined as below:

$$\% \text{ MeO} = \frac{\text{Volume of NaOH} \times 31}{\text{wt. of pectin (mg)}} \times 100 \quad (\text{Eq. 3})$$

where 31 = molecular weight of  $\text{OCH}_3$

**Uronic acid determination:** Galacturonic acid (GalA) was spectrophotometrically (UV-1800 spectrophotometer, Shimadzu, Japan) determined with carbazole reagent (McComb & McCready, 1952). Briefly, 0.5 ml of pectin solution (0.5% w/v) was ice-cooled, followed by the addition of 2.5 ml 98%  $\text{H}_2\text{SO}_4$ . The reaction mixture was incubated in boiling waterbath for 10 min. Colour was developed with 0.15% (w/v) carbazole solution, absorbance was measured at 530 nm, and uronic acid content was determined from GalA standard graph.

**Degree of esterification:** Degree of esterification (DE) of pectin was determined by potentiometric titration with NaOH before saponification and back titration with HCl after saponification as reported by Bocek *et al.*, (2001). Briefly, 0.2 g oven-dried pectin, moistened with few drops of ethanol, was dissolved in 20 ml warm water ( $40^\circ\text{C}$ ), stirred well for 2 h, titrated with 0.1 N NaOH using phenolphthalein as indicator to neutralize polygalacturonic acid groups, and the NaOH volume used

in the titration was designated as initial volume ( $I_v$ ). Excess (10 ml) 0.1 N NaOH was then added to the neutralized polygalacturonic acid groups. The mixture was stirred for 2 h at room temperature to saponify the esterified carboxyl groups, followed by the addition of 10 ml 0.1 N HCl. Excess of 0.1 N HCl was titrated with 0.1 N NaOH to determine the number of esterified carboxyl group, and the volume of 0.1 N NaOH used in titration was designated as the final volume ( $F_v$ ). DE was determined as below:

$$\% \text{ DE} = \left( \frac{F_v}{I_v + F_v} \right) \times 100 \quad (\text{Eq. 4})$$

**Fourier transform infrared (FTIR) spectrometry:** FTIR analysis of mango peel pectin was done using 3 mg fine pectin powder well-mixed with 300 mg KBr, made into discs with a laboratory hand press. IR absorbance data were obtained spectrometrically at wavenumbers in the range of 400 to 4000  $\text{cm}^{-1}$  (Perkin Elmer 1600 Spectrometer, USA).

## Results and Discussion

### Water-hot acid extraction of pectin (conventional method)

#### Selection of pectin extraction medium and optimum pH:

Determination of suitable and optimum conditions, such as the medium of extraction, pH, period and temperature of extraction, and recovery solvents is necessary to obtain maximum yield and the desired quality of pectin (Rolin, 1993; Yeoh *et al.*, 2008). For selection of best medium for the extraction of pectin from mango peel waste of *Chaunsa* cultivar, water-hot HCl,  $\text{HNO}_3$ , or  $\text{H}_2\text{SO}_4$  were used under different experimental conditions of pH, temperature and extraction period. pH of the extraction medium for the studied acids was adjusted at 2.0, 2.5, and 3.0. Best extraction was achieved at pH 2.5, when extraction was done at 90°C during the extraction period of 90 min with all the three mineral acids used (Fig. 2). At the same temperature and duration of extraction using  $\text{HNO}_3$ , the yield of pectin at pH 2.5 was 34% and 25% higher, respectively, than the higher and lesser acidic conditions of 2.0 and 3.0. Comparative efficiency of the three acids was noted to be in the order of  $\text{HNO}_3 > \text{H}_2\text{SO}_4 > \text{HCl}$  at all the three pH values of 2.0, 2.5, and 3.0, with the highest respective pectin extraction of 16.6 > 13.7 > 10.7 g 100 g<sup>-1</sup> dried mango peel powder at pH 2.5. It was further observed that differences in pectin extraction using HCl at the three pH values were insignificant, whereas extraction using  $\text{H}_2\text{SO}_4$  recorded a jump of 23.9% from pH 2.0 to 2.5, nevertheless showing similar levels of extraction at pH 2.0 and 3.0, and also not much different compared with the efficiency of HCl at these pH values (Fig. 2). Extraction of 17.8 g pectin 100 g<sup>-1</sup> peach pomace using  $\text{HNO}_3$  has been reported (Pagán *et al.*, 2001). Other studies on pectin yield from different mango cultivars have reported: 101 mg g<sup>-1</sup> and 153 mg g<sup>-1</sup> respectively from mango cultivars *Améliorée* and *Mango* using HCl at pH 1.5 (Koubala *et al.*, 2008); 17.6% from cultivar *Tommy Atkins* using  $\text{H}_2\text{SO}_4$  at pH 1.5 (Berardini *et al.*, 2005); and 170 mg g<sup>-1</sup> from cultivar *Ceni* using HCl at pH 1.5 (Kratchanova *et al.*, 1991). Total pectin extraction was 14.8 g 100 g<sup>-1</sup> from passion fruit using HCl at pH 2.0 and 99°C during two extractions of 60 min each done on the same material

(Kulkarni & Vijayanand, 2010), and 9.0% from cocoa husk using HCl at pH 2.5 and 95°C during 60 min of extraction (Mollea *et al.*, 2008).

#### Optimization of extraction conditions (temperature and duration of extraction, recovery solvent):

In order to determine the optimum temperature for hot acid extraction, mango peel powder slurry in water at 1:10 (w/v) ratio was treated with  $\text{HNO}_3$ ,  $\text{H}_2\text{SO}_4$ , or HCl at pH 2.5 for the duration of 90 min at different temperatures between 70 and 100°C. It was observed that as the temperature was increased from 70 to 90°C, all acids showed significant increase in the yield of pectin (Fig. 3). However, extraction efficiency of the three acids was appreciably different in the order of  $\text{HNO}_3 > \text{H}_2\text{SO}_4 > \text{HCl}$ , whereas best extraction using  $\text{HNO}_3$  at all the different temperatures was in the order of 16.6 > 12.9 > 9.9 g pectin 100 g<sup>-1</sup> mango peel powder respectively at the temperatures of 90 > 100 > 80 > 70 °C. The increase in pectin yield on raising temperature of water-hot  $\text{HNO}_3$  medium from 70 to 90°C was 40.2%. However, further increase in temperature from 90 to 100°C resulted in a drop in the yield of pectin by 22.3%, which may be attributed to the suggested  $\beta$ -elimination reaction at the higher temperature leading to disaggregation of pectin networks (Fishman *et al.*, 2000; Kurita *et al.*, 2008). It has been further observed that higher the extraction temperature, higher the yield of pectin, but methoxyl content and equivalent weight of pectin decrease, which may be due to the partial degradation of pectin (Kulkarni & Vijayanand, 2010).

For the determination of optimum time of extraction, mango peel powder slurry in water in the ratio of 1:10 (w/v) was incubated for different time periods between 30 and 120 min with  $\text{HNO}_3$ ,  $\text{H}_2\text{SO}_4$ , or HCl at pH 2.5 and 90°C. All the three acids showed maximum pectin recovery during extraction period of 90 min in the order of  $\text{HNO}_3 > \text{H}_2\text{SO}_4 > \text{HCl}$  (Fig. 4), which with  $\text{HNO}_3$  followed the order of 90 > 120 > 60 > 30 min of extraction respectively to yield 16.6 > 13.6 > 10.9 > 10.2 g pectin 100 g<sup>-1</sup> mango peel powder. The best recovery of 16.6 mg 100 g<sup>-1</sup> using  $\text{HNO}_3$  was respectively 17.5% or 37.1% higher than the pectin yield using  $\text{H}_2\text{SO}_4$  or HCl. It may be noted that as the extraction time was increased, extraction of pectin also increased to the optimum time of 90 min., however, further increase in time from 90 to 120 min resulted in the decrease of pectin yield. It has been suggested that beyond a certain time period of extraction, degradation of the extracted pectin sets-in resulting in the production of other products and thus a decrease in the yield of pectin (Pagán *et al.*, 2001). It has been further observed that higher the extraction time, higher the yield of pectin, but methoxyl content and equivalent weight of pectin decrease due to partial degradation of pectin (Kulkarni & Vijayanand, 2010).

It has been already mentioned that 90% ethanol was noted to be a better precipitating solvent than 75% methanol, 75% acetone, or 65% isopropyl alcohol, which respectively yielded 16.6%, 13.6%, 15.6%, or 14.6% pectin on water-hot  $\text{HNO}_3$  extraction at pH 2.5 and 90°C during 90 min extraction, when the ratio of solvents to pectin extract was 1:1 (Fig. 1).

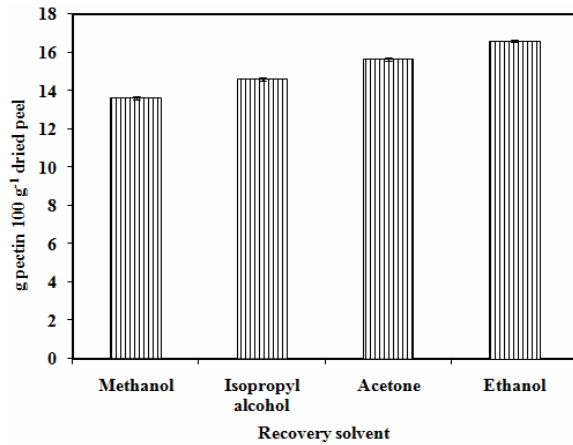


Fig. 1. Comparative recovery of pectin from mango peel waste by different solvents (90% ethanol, 75% methanol, 75% acetone, or 65% isopropyl alcohol) on extraction with HNO<sub>3</sub>, pH 2.5 at 90°C, during extraction period of 90 min.

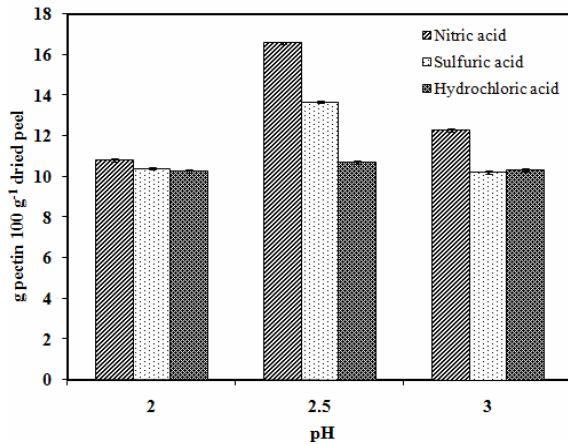


Fig. 2. Comparative recovery of pectin from mango peel waste on extraction with HNO<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub>, or HCl, at different pH values and 90°C, during extraction period of 90 min, with 90% commercial ethanol.

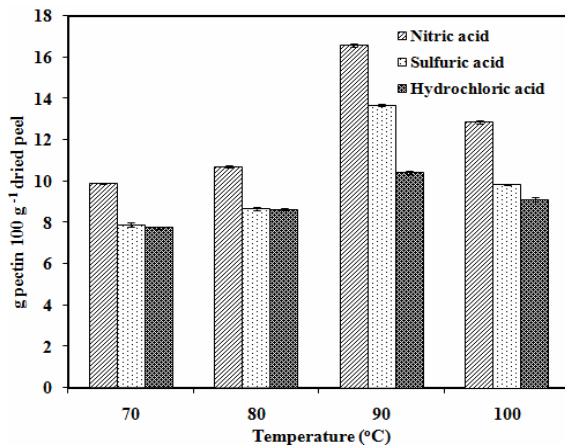


Fig. 3. Comparative recovery of pectin from mango peel waste on extraction with HNO<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub>, or HCl, at different temperatures during extraction period of 90 min., at pH 2.5, with 90% commercial ethanol.

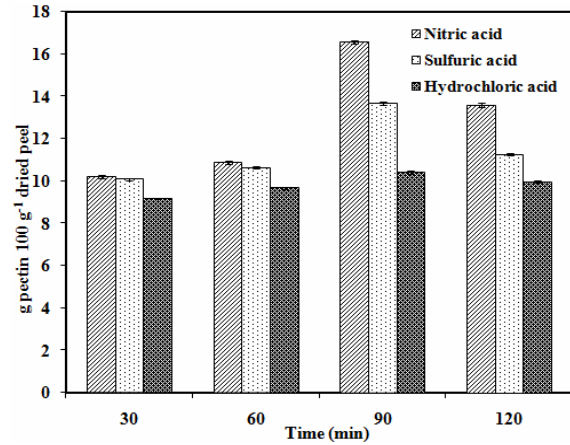


Fig. 4. Comparative recovery of pectin from mango peel waste on extraction with HNO<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub>, or HCl, during different extraction periods of 90 min., at pH 2.5 and 90°C, with 90% commercial ethanol.

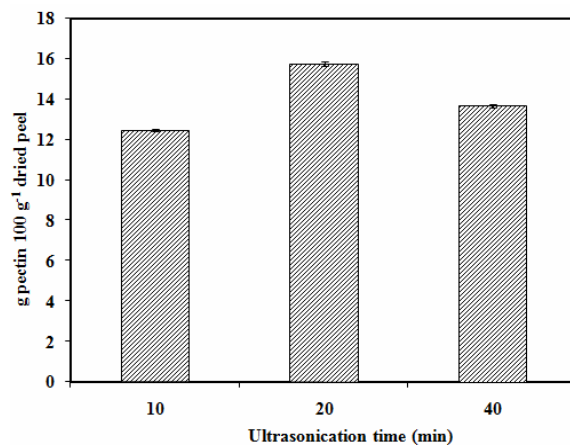


Fig. 5. Comparative recovery of pectin from mango peel waste on extraction with HNO<sub>3</sub>, during different periods of ultrasonication at pH 2.5 and 90°C, with 90% commercial ethanol.

#### Ultrasonication extraction of pectin (non-conventional method):

Following on the data obtained during studies on the conventional procedure that HNO<sub>3</sub> was the best acidic medium at pH 2.5 and 90°C, an attempt was made to reduce the period of pectin extraction by the non-conventional procedure of ultrasonication. For this purpose, pectin extraction from mango peel powder under these conditions was done by ultrasonication for 10, 20, or 40 min. Best extraction of 15.8% was obtained on ultrasonication for 20 min, which was 20.9% and 13.3% respectively higher than pectin obtained on ultrasonication for 10 and 40 min., (Fig. 5). It has been reported that there is always an optimum ultrasonication time to obtain the best yield (Panchev *et al.*, 1988), and that pectin yield increases first, reaches a maximum, and decreases as the time of extraction is further increased. Decrease in pectin yield has been suggested to be due to thermal degradation on increasing the period of extraction beyond the optimum time (Bagherian *et al.*, 2011). Maximum pectin recovery of 15.8% on ultrasonication for 20 min was only 4.9% less than 16.6% during extraction period of 90 min, and 37.1%

greater than 9.9% during extraction period of 30 min by the conventional procedure (Table 1). These observations are in agreement with the previously reported studies that ultrasonication increased yield, rate of extraction and reduction in extraction time (Bagherian *et al.*, 2011), and improvement in pectin yield by 28% along with reduction in the time of extraction and minimized degradation (Panchev *et al.*, 1988). Faster pectin extraction on ultrasonication has been attributed to cell disruption and enhanced mass transfer in the boundary layers surrounding the solid matrix (Bhaskaracharya *et al.*, 2009), and also to the disruption of cell walls due to the energy generated from collapsing cavitation bubbles thus releasing cellular materials (Patist & Bates, 2008). The non-conventional ultrasonication procedure has indicated several advantages, principally, greater cost-effective operation at less energy input, better quality pectin due to less degradation, and improved yield at lesser processing time.

**Chemical characteristics of mango pectin:** Comparative characteristics of pectin extracted by the conventional and non-conventional methods during different periods of extraction are reported in Table 1. There was a gradual decrease in MeO content and equivalent weight of the extracted pectin as the time of extraction was increased in both the conventional and non-conventional procedures. Although the decrease in the two parameters was not significant, it was consistent and comparable in the two procedures, and in agreement with the reported decreasing trend on increase in the time of pectin extraction from passion fruit peel, due likely to partial degradation of pectin (Kulkarni & Vijayanand, 2010). The reduction in MeO content and molecular weight on prolonged heating has been suggested to be due to chain cleavage into smaller fragments of pectin molecule (Bagherian *et al.*, 2011). The degree of esterification (DE) of the extracted pectin was also noted to change with the change in duration of extraction time; increasing from the lesser periods of extraction to a maximum as the time of extraction was increased, and then decreasing as the time period was increased further. Maximum DE values in both the conventional and non-conventional procedures corresponded with the optimum time of extraction.

Longer time of extraction has been suggested to cause degradation of methyl ester groups in pectin into carboxylic acid (Ramli & Asmawati, 2011). Therefore, fall in DE content of mango pectin extracted in the present study using both the conventional and non-conventional procedures, on increasing the time of extraction beyond the optimum time, may be attributed to this kind of degradation. The pectin extracted from mango peel waste, with DE values between 72.81–73.81%, may be characterized as commercial food grade high methoxy ester pectin (Bagherian *et al.*, 2011). Increase in GalA values also followed the same trend as noted with DE content, reaching the maximum at optimum period of extraction then reducing when the period of extraction was increased further. This increase in GalA content has been attributed to the hydrolysis of protopectin (Ramli & Asmawati, 2011).

FTIR spectra of both water-hot HNO<sub>3</sub> and water-hot HNO<sub>3</sub> ultrasonication extracted pectin were compared with standard citrus pectin (Fig. 6). All the three spectra have much resemblance in the appearance of characteristic broad bands between 3000 and 3600 cm<sup>-1</sup> due to the presence of –OH stretching of inter/intra-molecular hydrogen bonding of galacturonic acid backbone (Singthong *et al.*, 2004). An O-CH<sub>3</sub> stretching band between 2950 and 2750 cm<sup>-1</sup> usually refers to C-H adsorption, which includes C-H, C-H<sub>2</sub> and C-H<sub>3</sub> stretching, and vibration bands; and specifically referring to the band at 2939 corresponding to the presence of methyl ester group of galacturonic acid (Singthong *et al.*, 2004; Kumar & Ghanshyam, 2010). Strong mixed peaks of ester and carboxylic C=O stretching vibrations, respectively, at 1733 and 1628 indicated methyl esterified carboxyl groups (Kurita *et al.*, 2008; Gan *et al.*, 2010). Based on the peaks of functional moieties with specific band stretching in the three spectra, it can be concluded that the quality of pectin extracted from mango peel waste was comparable to the standard commercial citrus pectin. It is also evident that water-hot HNO<sub>3</sub> ultrasonication procedure did not affect the quality of extracted pectin, facilitating to reduce the operational time by a factor of 4.5, at lesser energy input.

**Table 1. Chemical characterization of mango peel waste pectin extracted by conventional (water-hot HNO<sub>3</sub> at pH 2.5 and 90°C) and non-conventional (ultrasonication) procedures, recovered with 90% ethanol.**

Component	Conventional procedure (extraction time, min)				Non-conventional procedure (ultrasonication time, min)		
	30	60	90	120	10	20	40
Pectin yield (%)	9.92 ± 0.09	10.71 ± 0.07	16.59 ± 0.06	12.89 ± 0.05	12.47 ± 0.08	15.77 ± 0.04	13.67 ± 0.04
Moisture (%)	6.87 ± 0.01	6.77 ± 0.01	6.98 ± 0.02	6.71 ± 0.01	6.51 ± 0.03	6.37 ± 0.07	6.32 ± 0.01
Ash (%)	3.90 ± 0.02	3.89 ± 0.01	3.88 ± 0.01	3.85 ± 0.02	3.51 ± 0.07	3.34 ± 0.03	3.31 ± 0.05
<sup>a</sup> MeO content (%)	9.71 ± 0.17	9.69 ± 0.13	9.64 ± 0.15	9.61 ± 0.12	9.67 ± 0.11	9.65 ± 0.14	9.61 ± 0.17
<sup>b</sup> DE (%)	65.77 ± 0.06	70.27 ± 0.02	72.81 ± 0.05	69.17 ± 0.03	72.12 ± 0.07	73.81 ± 0.03	71.27 ± 0.02
<sup>c</sup> GalA content (%)	62.72 ± 0.07	68.67 ± 0.05	70.19 ± 0.08	65.12 ± 0.02	69.91 ± 0.04	70.87 ± 0.07	68.21 ± 0.04
Equivalent wt.	825.71 ± 1.20	820.52 ± 1.17	815.21 ± 1.15	807.74 ± 1.19	827.07 ± 1.27	825.23 ± 1.15	803.51 ± 1.13

<sup>a</sup>MeO = Methoxyl; <sup>b</sup>DE = Degree of esterification; <sup>c</sup>GalA = Galacturonic acid

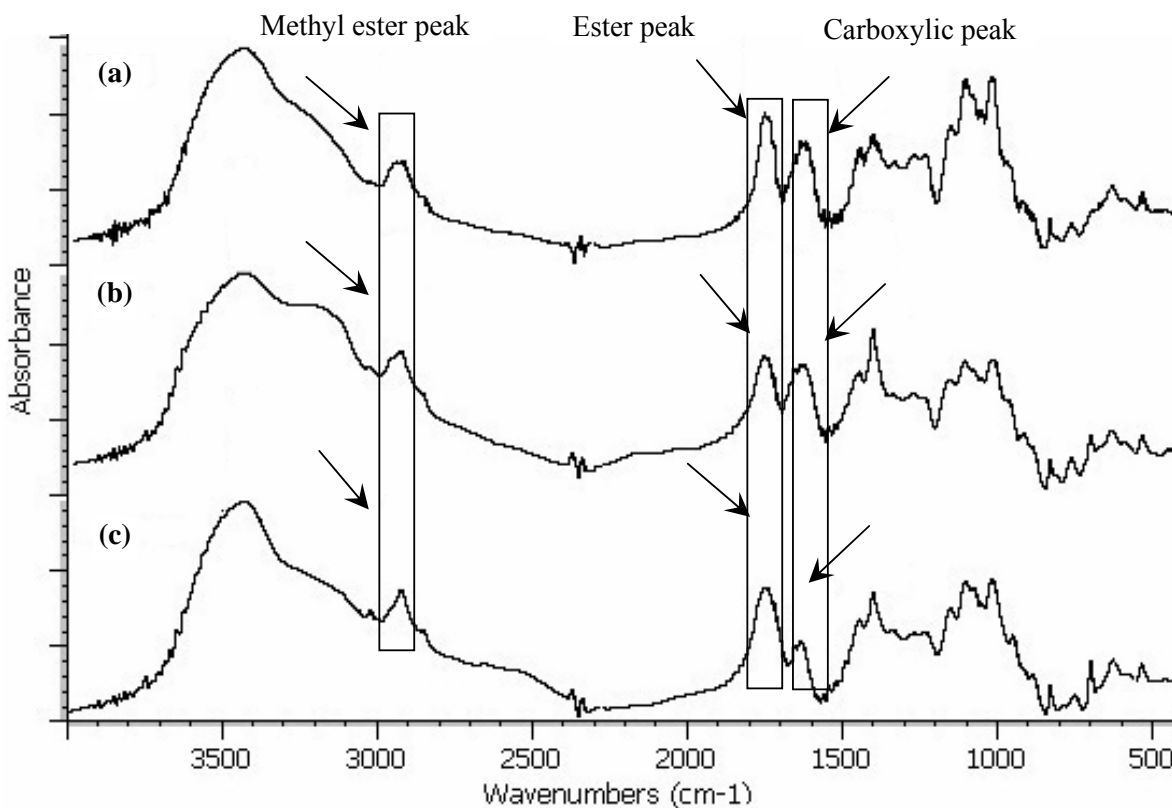


Fig. 6. FTIR spectra of (a) standard citrus pectin; and pectin recovered from mango peel waste on extraction with (b) conventional water-hot  $\text{HNO}_3$  procedure, and (c) non-conventional ultrasonication procedures.

## Conclusion

Comparative studies on pectin extraction from fruit peel of mango cultivar *Chaunsa*, under variable conditions in three different mineral acids, showed maximum yield of  $16.6 \text{ g } 100 \text{ g}^{-1}$  in  $\text{HNO}_3$ , pH 2.5, at  $90^\circ\text{C}$ , 90 min period of extraction, and ethanol as the recovery solvent. However, almost similar yield of  $15.8 \text{ g } 100 \text{ g}^{-1}$  was achieved in 20 min when the extraction medium was ultrasonicated under the same conditions, which was faster by a factor of 4.5. This advantage can be used to save the major cost of energy input to maintain temperature at  $90^\circ\text{C}$  for additional 70 min. Furthermore, by heating for lesser period, thermal degradation of pectin can be minimized and its quality appreciated. Data presented on the comparative efficiency of different recovery solvents may be useful for cost-effective selection of a solvent depending on its market price in the local area of the processing unit. The pectin obtained from mango peel waste was of high methoxyl category containing +70% galacturonic acid, thus meeting the minimum value of FAO and EU regulations. Other chemical characteristics of methoxyl content and degree of esterification also showed that mango pectin meets the criteria of quality pectin. FTIR spectra of pectin, extracted using the conventional and the non-conventional ultrasonication procedures, compared well with citrus pectin as the reference standard.

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