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EXTRACTION AND CHEMICAL QUALITY CHARACTERISTICS EVALUATION OF ORANGE PEEL PECTIN

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Optimum conditions for the extraction and precipitation of pectin from orange peels were investigated. Changes in pH, temperature and extraction time, significantly, affected the extraction of pectin. Maximum pectin yield was 17.7%, which was obtained on soaking finely ground orange peels in sulphuric acid solution of pH 2.5 at 80°C for 120 min. Maximum pectin was precipitated from the extract by adding 95% ethanol at the rate of 200ml/l. Anhydrogalacturonic acid and methoxyl contents of pectin obtained under these optimum extraction conditions were 72.80% and 9.77% respectively, while equivalent weight value was 943. These chemical characteristics values were within the accepted limit of good quality pectin.

***Key words*:** Orange peel, Pectin, Extraction yield, Quality.

Introduction

Pectin is a complex heteropolysaccharide which mainly consists of long unbranched chains of polygalacturonic acid with carboxylic group partially esterified with methyl alcohol (Awan 1993). It is extensively utilized by the food processors especially for the conversion of low grade fruits into good quality products like jam, jelly, marmalade and candies. Low methoxyl pectin is used to increase firmness of canned fruits while high methoxyl pectin helps to prepare stable fruit beverages.

There are various methods for the extraction of pectin from fruit and vegetable wastes but the literature regarding optimum conditions for extraction of pectin is scanty. The quality of pectin depends mostly upon the source as well as the method employed for its extraction. Pectin is usually extracted by suspending chopped fruit and vegetable wastes in different mineral acids and salts solutions (Baltaga 1962; Srirangarajan and Shrikhande 1979; Jain *et al* 1984). Pectin from grape fruit orange and lemon peels was extracted with acidic solution of nitric acid and precipitated with ethanol (Rouse and Crandall 1976). Pectin extracted by soaking different plant materials in aqueous sodium nitrate solution containing amino acids (Golubev *et al* 1991). Pectin extract from soybean okara with sodium hexametaphosphate solution is reported by Yamoguchi *et al* (1996) and pectin from potato waste using solution of aluminium sulphate as precipitating agent obtained by Chen *et al* (1999). Muralikrishna and Tharanatha (1994) obtained only 1.43 - 5.37% pectin by soaking pulse husks in hydrochloric acid and EDTA solution at 70°C, whereas, Karpovich *et al* (1990) and Turquios *et al* (1999) were reported the yield 9-10% of good quality pectin from potato waste using solution of aluminium sulphate as precipitating agent. Microbial enzymes were also used for the production of pectin and pigment from orange peels (Elian *et al* 1984). Industrial pectin of good gelling property was prepared from peach pomace by Pagan *et al* (1999). Fresh Kiwi fruit peels were also used for the extraction of pectin by Wu Hui-Fang *et al* (1999). These methods for extraction of pectin are lengthy, complicated and laborious. Moreover, yield of pectin by most of the previous methods was very low. Therefore, present work was undertaken to optimize the conditions (pH, temperature, time period of extraction) for the development of a simple and efficient pectin extraction method from orange peels. Besides yield, chemical characteristics of pectin were also studied.

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Materials and Methods

Fresh orange peels were collected from a local commercial fruit processing plant. The peels were passed through shredding machine to separate albedo (Pectin rich) and flavedo (oil + pigment) portions. The albedo portion was then minced mechanically and washed with cold water to remove any adhering juice. The washed and minced albedo was then dried in a cabinet dryer at 65°C to reduce moisture content to 5-6%. The dried material was finally ground to 80 mesh size before extraction of pectin.

Extraction of pectin. Ground material was mixed well with water of different pH (1.5, 2.5, 3.5) keeping substrate to water ratio 1:40 (w/v). The desired pH of the mixture was adjusted

Table 1
Effect of pH, temperature and time period of extraction on yield (% age) of pectin from orange peels

Extraction temp. (°C)	pH - 1.5 Extraction time period (min) 30	60	120	pH - 2.5 Extraction time period (min) 30	60	120	pH - 3.5 Extraction time period (min) 30	60	120
60	7.20^{a}±0.2	7.80^{a}±0.2	8.50^{a}±0.4	9.80^{a}±0.4	11.20^{a}±0.5	14.00^{a}±0.6	8.20^{a}±0.2	8.30^{a}±0.3	8.90^{a}±0.5
70	8.00^{b}±0.4	9.30^{b}±0.6	10.80^{b}±0.5	12.00^{b}±0.7	14.20^{b}±0.3	15.90^{b}±0.4	10.00^{b}±0.5	10.60^{b}±0.1	11.00^{b}±0.5
80	9.20^{c}±0.6	10.70^{c}±0.6	11.60^{c}±0.3	14.00^{c}±0.5	15.50^{c}±0.5	17.70^{c}±0.7	11.20^{c}±0.7	11.60^{c}±0.4	12.00^{c}±0.3
90	8.10^{b}±0.5	8.00^{a}±0.5	8.70^{a}±0.4	11.50^{b}±0.3	11.00^{a}±0.3	12.70^{a}±0.2	10.00^{b}±0.6	10.20^{b}±0.2	10.40^{b}±0.4

Mean value ± S.D. triplicate determination
Mean values within a column with different superscripts are significantly different at $p < 0.05$

with 0.1N sulphuric acid on pH meter (PYE UNICAM model-1292) and then incubated at temperatures (60, 70, 80 & 90°C) for various time intervals (30, 60, 120 min) with frequent stirring. After incubation, the contents were filtered through cheese cloth and pectin from the filtrate was precipitated with 95% ethanol. The obtained pectin was dried in a vacuum oven at 40°C to obtain constant weight and ground finally to study chemical quality characteristics.

Yield was calculated as dried pectin g/100g dried orange peels. Anhydrogalacturonic acid content, equivalent weight and methoxyl contents were determined as quality characteristics of orange peel pectin by the standard methods of Owens *et al* (1952). Triplicate determinations were performed for all parameters studied and standard deviations (SD) were calculated according to the method of the Steel and Torrie (1980).

Results and Discussion

Optimum conditions for extraction of pectin. Data presented in Table 1 showed that pH extraction, temperature and time distinctly affected the extraction of pectin from finely ground orange peels. pH of the solution played a significant role in the extraction of pectin. The % yield of pectin was 11.60, 17.70 and 12.00 at pH 1.5, 2.5 and 3.5 respectively, when orange peel (albedo) powder was suspended at 80°C for 120 min (Table 1). Results revealed that maximum yield of pectin was obtained by soaking the raw material in a solution of pH 2.5 which was significantly, ($p < 0.05$) higher than the pectin yield obtained at pH 1.5 and 3.5. However, decline in pectin yield was observed at pH 3.5 at all studied temperatures and extraction time.

Variable amounts of pectin was obtained from orange peels at temperatures ranging from 60-90°C (Table 1). Maximum amount of pectin was obtained by soaking albedo portion of orange peels in acidic solution at 80°C. However, significant reduction in pectin yield was observed when temperature was raised from 80 to 90°C. At 80°C, pectin yield was 15.50% which was reduced to 11.00% at 90°C on soaking orange peels

Table 2
Effect of different mineral acids solution on the yield of pectin

Extractants	Extraction conditions pH	Temp.(°C)	Time (min)	Yield %
Hydrochloric acid	2.5	80	120	13.45^{a}±0.7
Sulphuric acid	2.5	80	120	17.80^{c}±0.5
Nitric acid	2.5	80	120	15.11^{b}±0.5

Mean value ± S.D. triplicate determination
Mean values within a column with different superscripts are significantly different at $p < 0.05$

Table 3
Effect of different precipitation agent on the yield of pectin

Precipitating agent	Extraction conditions Amount added ml/l	pH	Temp (°C)	Time (min)	Yield %
Ethanol	50	2.5	80	120	7.20^{a}±0.5
	100	2.5	80	120	11.00^{b}±0.6
	200	2.5	80	120	17.80^{d}±0.8
Acetone	50	2.5	80	120	5.40^{a}±0.1
	100	2.5	80	120	9.00^{a}±0.3
	200	2.5	80	120	14.44^{c}±0.5

Mean value ± S.D. triplicate determination
Mean values within a column with different superscripts are significantly different at $p < 0.05$

Table 4
Chemical quality characteristics of orange peels pectin extracted at 80°C

Chemical characteristics	pH - 1.5 Extraction time period (min)			pH - 2.5 Extraction time period (min)			pH - 3.5 Extraction time period (min)		
	30	60	120	30	60	120	30	60	120
Anhydrogalacturonic acid (%)	$66.73^{b}\pm1.6$	$67.45^{b}\pm1.4$	$68.72^{b}\pm1.4$	$70.88^{c}\pm1.4$	$71.45^{c}\pm1.3$	$72.80^{c}\pm1.3$	$8.20^{a}\pm0.2$	$8.30^{a}\pm0.3$	$8.90^{a}\pm0.5$
Methoxyl content (%)	$8.11^{a}\pm0.10$	$8.15^{a}\pm0.10$	$8.33^{a}\pm0.11$	$9.20^{c}\pm0.10$	$9.67^{c}\pm0.12$	$9.77^{c}\pm0.12$	$8.03^{a}\pm0.14$	$8.24^{a}\pm0.15$	$8.72^{b}\pm0.11$
Equivalent weight	$815^{b}\pm2.0$	$820^{b}\pm2.5$	$835^{b}\pm2.5$	$932^{c}\pm2.6$	$939^{c}\pm2.6$	$943^{c}\pm2.1$	$798^{a}\pm2.4$	$801^{a}\pm2.3$	$803^{a}\pm2.0$

Mean value ± S.D. triplicate determination
Mean values within a row with different superscripts are significantly different at $p < 0.05$

in acids solution of pH 2.5 for 60 min. Similar results were also obtained at other two pH values i.e. 1.5 and 3.5. Decrease in pectin yield at higher temperature 90°C could be attributed to the break down of pectin molecules as already observed by Chang *et al* (1994).

Extractability of pectin was also affected by extraction time ranging from 30 to 120 min. At pH 2.5, pectin yield was 14.0, 15.50 and 17.7% respectively at 80°C after 30, 60 and 120 min extraction time respectively, Table 1. It is also apparent from Table 1, that maximum pectin yield was also obtained at pH 1.5 and 2.5 after 120 min. These results are consistent with the findings of other workers who reported that prolonged extraction and higher temperatures adversely affected the yield of pectin from different sources (Turmucin *et al* 1983; Chang *et al* 1994). Rouse and Crandall (1976) extracted 11.00, 8.15 and 6.35% pectin from lemon, orange and grape fruit peels at pH 1.6 respectively, while 20% pectin yield was obtained from orange peels by precipitation with ferric salt (Zhao 1995). The differences between our results and reported in literature may be due to variations in raw material, particle size and extraction methods.

Extraction of pectin using different mineral acids. It is evident from Table 2 that maximum amount of pectin 17.80% was obtained with sulphuric acid solution of pH 2.5 at 80°C after 120 min extraction time while minimum amount of pectin (13.45%) was obtained with hydrochloric acid solution under the same extraction conditions. However, nitric acid solution extracted 15.11% pectin from orange peels, which is comparatively more than hydrochloric acid solution of pH 2.5. Earlier workers also obtained variable amounts of pectin from citrus peels using different mineral acids (Snyder 1970; Huang 1973; Braddok *et al* 1976). Better extraction of pectin with sulphuric acid might be due to the presence of sulphate ions in soaking solution.

Precipitation of pectin. Pectin was precipitated from the filtrate by the addition of variable amount of ethanol and acetone. Maximum pectin yield was 17.80% when ethanol at the rate of 200 ml/l was added slowly with stirring in the filtrate, while 14.44% pectin was recovered with acetone 200 ml/l (Table 3). Therefore, ethanol was found to be better precipitating agent as compared to acetone.

Chemical quality characteristics of pectin. Table 4 summarizes the chemical characteristics of orange peel pectin. It is apparent from these results that pH, temperature and extraction time affected the chemical quality characteristics of orange peel pectin. However, chemical quality of pectin obtained at pH 2.5 was comparatively better than the pectin obtained at pH 1.5 and 3.5. Anhydrogalacturonic acid and methoxyl contents were 72.80% and 9.77% respectively, while equivalent weight value was 943 for pectin extracted at pH 2.5, 80°C after 120 min. These results were within the range of reported values for anhydrogalacturonic acid (68.5 - 75.0%) and methoxyl contents (8.4 - 9.7%) of good quality orange peel pectin (Zafiris and Oreopoulou 1992).

Therefore, it is concluded that good quality pectin with maximum yield can be obtained by soaking finely ground albedo portion of orange peels in sulphuric acid solution of pH 2.5 at 80°C for 120 min. Ethanol can be successfully used as precipitating agent for maximum recovery of pectin from the extracted filtrate.

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