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# Extraction and Characterization of Mango Peel Pectin as Pharmaceutical Excipient

## Ekstrakcja i charakterystyka pektyny ze skórki mango jako farmaceutycznej substancji pomocniczej

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## Summary

**Aim of study**. Present study includes extraction and characterization the mango peel derived pectin as a pharmaceutical excipients.
**Material and methods.** Pectin was obtained using acidified water based extraction in soxhlet apparatus. To characterize the extracted pectin phytochemical screening was done and micromeritic properties, flow behavior, surface tension, viscosity and swelling index were calculated.
**Results.** Using water based extraction method 25.26% yield of pectin was obtained. The result revealed the fact that extracted mango peel pectin exhibited good flow properties (angle of repose 28.01), 41.90±2.62 dynes/cm$^2$ surface tension, 0.46 % w/w total ash, 0.76% loss on drying and pH was found 4.15, showed that this can be used in dosage form, without any irritation. Extracted pectin was soluble in warm water while insoluble in organic solvents.
**Conclusions.** Results of evaluated parameters showed that mango peel derived pectin can be used as pharmaceutical excipient to prepare solid oral dosage form **(Polim. Med. 2012, 42, 185–190).**

**Key words:** mango peel pectin, pectin, extraction, characterization, pharmaceutical excipient, natural polymer

## Streszczenie

**Cel pracy**. Celem prezentowanej pracy było przedstawienie zagadnienia ekstrakcji i charakterystyka pektyny ze skórki mango, jako farmaceutycznej substancji pomocniczej.
**Materiał i metody.** Skórka mango była używana jako surowiec do uzyskania pektyny. Badania obejmoway: fitochemiczne badania przesiewowe, właściwości mikromerytyczne, zachowanie sypkości, ocenę napięcia powierzchniowego i lepkości, wskaźnik pęcznienia pektyny ze skórki mango jako składnika farmaceutycznego. Pektyna ze skórki mango była uzyskiwana w drodze ekstrakcji z użyciem zakwaszonej wody w aparacie soxhlet. Wykorzystano farmakopealne procedury do badania: właściwości mikromerytycznych, rozpuszczalności, właściwości organoleptycznych, pH, napięcia powierzchniowego, lepkości, wskaźnika pęcznienia oraz charakterystyki powierzchni pektyny ze skórki mango.
**Wyniki.** Po zastosowaniu ekstrakcji z użyciem wody uzyskano wydajność otrzymywania pektyny na poziomie 25,26%. Wyniki badań wykazały, że ekstrahowana ze skórki mango pektyna posiada dobrą sypkość (kąt zsypu 28,01), napięcie powierzchniowe 41,90 ± 2,62 dyn/cm$^2$, popiół całkowity 0,46%, stratę przy suszeniu 0,76% i pH 4,15. Badania te wykazały, że pektyna może być stosowana w postaci leku bez wywoływania podrażnień. Wyekstrahowana pektyna była rozpuszczalna w gorącej wodzie, a nierozpuszczalna w rozpuszczalnikach organicznych.
**Wniosek.** Wyniki ocenianych parametrów wykazały, że pektyna ze skórki mango może być używana jako farmaceutyczna substancja pomocnicza w przygotowaniu stałej doustnej postaci leku **(Polim. Med. 2012, 42, 185–190).**

**Słowa kluczowe.** Pektyna ze skórki mango, pektyna, ekstrakcja, charakterystyka, farmaceutyczna substancja pomocnicza, polimer naturalny

# Introduction

Pectin, a multifunctional constituent of cell wall is a high value functional food ingredient. It is produced commercially as a white to light brown powder, mainly extracted from fruits. Pectin is a linear chain of α-(1-4)-linked D-galacturonic acid that forms the pectin-backbone [1]. Pilnik *et al* and Schols *et al* described that "Pectins are mainly used as gelling agents, but can also act as thickener, water binder and stabilizer. Low methoxyl pectins (< 50% esterified) form thermoreversible gels in the presence of calcium ions and at low pH (3–4.5) whereas high methoxyl pectins rapidly form thermally irreversible gels in the presence of sufficient (for example, 65% by weight) sugars such as sucrose and at low pH (< 3.5); the lower the methoxyl content, the slower the set" [2, 3].

The study was designed to extract the pectin from mango peels, to study the physiochemical characteristics and to study its micromeritic properties as a pharmaceutical excipient. Micromeritic studies includes different parameters such as particle size analysis, bulk density, tapped density, true density, angle of repose, cars index, bulkiness and to find out the surface tension, viscosity and swelling index of mango pectin.

# Material and Methods

## Extraction Procedure

Mango peel was obtained as a waste material from local fruit shop selling mango juice. Collected peel was carefully washed and dried under shade for 24 h, further dried at 30–40°C until constant weight was obtained. Dried fruit peel was cut into pieces and powdered in to electric grater. Powdered peel was further passed from sieve # 20 and stored in air tight container until used. Extraction of pectin includes two steps.

### Step1: Extraction of Pectin

As the authors described elsewhere, pectin was extracted under reflux in a condensation system using water acidified with citric acid to pH 2. Temperature of extraction media was maintained at 70°C and duration of extraction was adjusted about 6 h. The extractor thimble was a Whatman cellulose thimble with 33 mm internal diameter and 80 mm external length. Dried powdered mango peel was taken in soxhlet and reflux was continued for 6 h [4, 5].

### Step2: Isolation of Pectin

As shown by authors in a previous publication, hot acid extract was pressed in cheese cloth bag and the concentrated juice was cooled to 4°C. Pectin was precipitated by alcohol-juice treatment 2:1 (v/v) followed by continuous stirring for 15 min and mixture was further allowed to stand for 2 h for better pectin precipitation. This allow to filter pectic substances because pectin remains float on the surface of alcohol–water mixture. Floating pectin coagulate was filtered through cheesecloth, washed with alcohol (95%) and pressed. Pressed pectin was further dried to constant weight at 35–45°C in hot air oven. Hard pectin cake was ground and sieved through sieve # 20, stored in desicator for further used [4, 5].

## Physicochemical Characterization of Mango Pectin

### Identification tests for carbohydrates

As the authors described in previous publication, aqueous extract was mixed Molish's reagent followed by addition of sulfuric acid. The violet color ring appeared at junction, showing presence of carbohydrates [5, 6].

### Determination of purity of mango pectin

To determine purity of extracted pectin tests for alkaloids, proteins, mucilage, fats, tannins and amino acids were performed as already described by authors in previous publication [5, 6].

### Organoleptic evaluation of isolated pectin

As authors described elsewhere, isolated pectin was characterized for organoleptic properties such as color, odor, taste, fracture and texture [5].

### Ash values

As discussed by authors in previous publication ash values such as total ash, acid insoluble ash and water-soluble ash were determined [7].

### Solubility behavior

As already described by authors one part of dry pectin powder was shaken with different solvents and further solubility was determined [5].

### pH of pectin

The pectin was weighed and dissolved in water separately to get a 1%w/v solution. The pH of solution was determined using digital pH meter as described by authors in previous publication [5].

### Swelling index

As described by authors in previous publication "swelling index is the volume (in ml) taken up by the swelling of 1 g of test material under specified conditions. The swelling indices of the selected pectin were determined by accurately weighing 1 g of pectin, which was further introduced into a 25 ml glass-Stoppard measuring cylinder. 25 ml of water was added and mixture was shaken thoroughly every 10 min for 1 h. It was then allowed to stand for 3 h at room temperature. Then the volume occupied by pectin, was measured". The procedure was repeated thrice and the mean value was calculated [8].

### Surface tension

The surface tension of the selected polysaccharides was determined by drop count method, using a stalagmometer [5, 9].

### Loss on drying

The test was carried out according to the procedure described by authors elsewhere. One gram of hydrogel powder was weighed accurately in a tared glass stoppered bottle and was dried in a hot air oven at 105°C and the weight was checked at intervals of 1 h, until a constant weight was obtained. The percentage of weight lost by the powder was calculated [5, 6].

### Bulk density and bulkiness

It has been described by authors that inverse of bulk density is called as bulkiness. As per previous study accurately weighed quantity of (50 g) was introduced into a graduated measuring cylinder. The cylinder was fixed on the bulk density apparatus and the volume occupied by the powder was noted. Then, the powder was subjected to tapping in a bulk density apparatus until constant volume was obtained. The final volume (bulk volume) was noted [5, 10, 11].

### True density

Among various methods available for the determination of true density, liquid displacement method is the simplest method and was used in the present study. Acetone was selected as the liquid for displacement, because, pectin is insoluble and heavy in acetone. This method has been used by authors [5, 10, 11].

### Total porosity

Total porosity of a substance is expressed as percentage, and is denoted by 'ε'. The total porosity values for pectin were calculated using bulk and true density values [5, 10, 11].

### Powder flow property

Flow characteristics were measured by angle of repose as previous publication of authors. Same study was repeated here. Using the readings and the formula, the angle of repose was calculated [5, 10, 11].

### Powder Compressibility (Carr's Consolidation Index)

This property is also known as compressibility. As described in previous publication finely powdered pectin (5 g) was transferred into a measuring cylinder and calculations were done using bulk density apparatus [5, 10, 11].

### Surface characteristics of mango pectin using scanning electron microscopy (SEM)

As described by authors in previous study powder was evaporated with carbon and then sputtered with gold to make the samples electrically connected. The SEM was taken in Hitachi S-2400 electron microscope [5].

## Infrared spectra of the isolated pectin

Hundred milligrams of the powdered pectin was mixed with potassium bromide (400 mg) and was compressed in a hydraulic press to form a pellet at 15 tons pressure. The pellets were scanned from 4000 to 400 $cm^{-1}$ in a Perkin Elmer FTIR spectrophotometer as described by authors elsewhere [5].

# Results and Discussion

After acidified hot water extraction and further precipitation by ethyl alcohol the yield of pectin was 25.26% w/w obtained. The isolated sample was subjected to identification. This showed presence of carbohydrates in sample powder. Confirmation of pectin was done when it gave negative test for mucilages, gums, tannins, alkaloids and proteins. Other phytoconstituents were absent in the isolated powder. This can be considered as proof for purity of the isolated pectin as depicted in table 1.

Isolated pectin was found brownish white in colour with characteristic taste, organoleptic properties of pectin was shown in table 2.

pH of 1% solution was found to be 4.15, which indicated that it should be non-irritating for mucous membrane. Solubility behavior of pectin was shown in table 3.

Ash values were also calculated to characterize pectin; total ash, acid insoluble ash and water soluble ash

**Table 1.** Determination of purity of isolated pectin

**Tabela 1.** Określanie czystości izolowanej pektyny

| Tests | Present/Absent |
|---|---|
| Carbohydrates | + |
| Hexose Sugar | + |
| Monosaccharides | – |
| Proteins | – |
| Fats and oils | – |
| Tannins and Phenolic Compounds | – |
| Alkaloides | – |
| Amino acids | – |
| Mucilage | – |
| Gums | – |

+ Present; – Absent.

**Table 3.** Solubility profile of isolated pectin

**Tabela 3.** Profil rozpuszczalności pojedynczej pektyny

| Solvent | Solubility |
|---|---|
| Cold water | Swells to form gel |
| Warm water | Soluble |
| Benzene | Insoluble |
| Ether | Insoluble |
| Chloroform | Insoluble |
| n-Butanol | Insoluble |
| Ethanol | Insoluble |

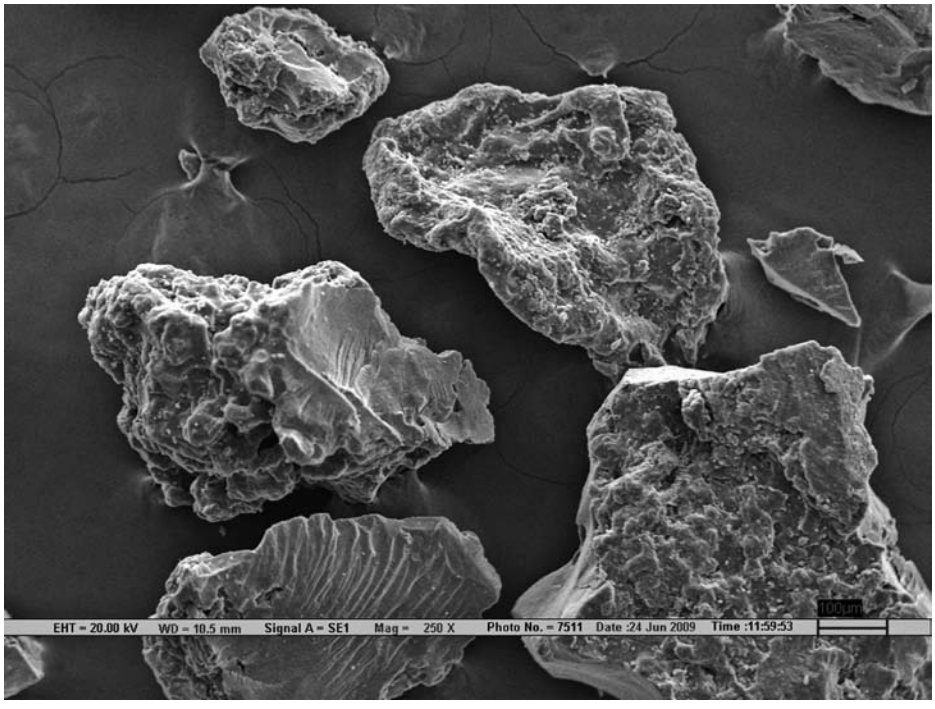


**Fig. 1.** Scanning Electron Micrograph of Mango peel pectin

**Ryc. 1.** Skórka pektyny mango (skaningowy mikroskop elektronowy)

were found 0.46%, 0.185% and 0.103% respectively. Surface tension of 0.01% w/v solutions of pectin was found to be 41.90±2.62 dynes/cm$^2$. The surface tension of the polymer has been reported to influence the binding quality of the polymer in tablets. The effect of surface properties such as wetting and spreading of binder over substrates, binder-substrate adhesion and binder cohesion in determining the optimum granulation with polymer binders has been reported. Lower surface tension promotes better penetration and spreading of polymer solution over the drug during wet granulation and hence leads to formation of better granules.

The results for loss of drying showed value of 0.76%. Alternatively, the moisture content of the sample was found to be 24.53%. This indicated that pectin is hygroscopic in nature and need to be stored in air-tight containers.

**Table 2.** Organoleptic properties of isolated pectin

**Tabela 2.** Właściwości organoleptyczne pojedynczej pektyny

| Color | Odor | Taste | Texture | Fracture |
|---|---|---|---|---|
| Brownish white | Odorless | Characteristic | Rough and Irregular | Rough |

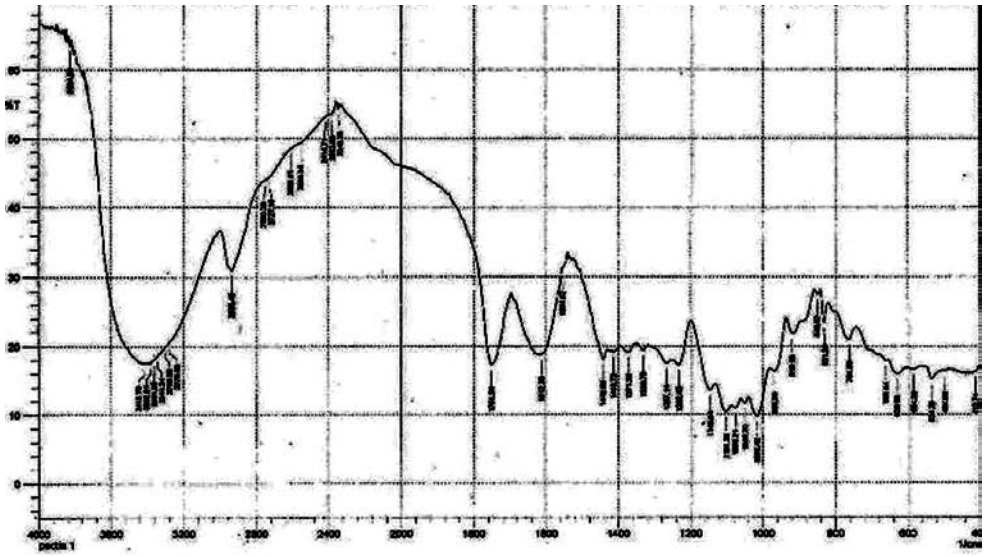

**Fig. 2.** Infrared Spectroscopic graph of extracted mango peel pectin

**Ryc. 2.** Podczerwień Spectroscopic. Wykres pektyny skórki mango

Physical characterization of pectin was carried out for bulk density and bulkiness, true density, total porosity, powder flow behavior (Table 4). The bulkiness value indicated that powder is 'heavy' in nature. The total porosity has been correlated with dissolution rate. It was also found that higher the porosity, faster the rate of dissolution. Pectin exhibited good flow characteristics.

Scanning Electron Micrograph of the pectin showed that extracted pectin had rough surface as shown in Figure 1.

IR spectroscopic study revealed the presence of characteristics group in the extracted pectin. Peak of carboxylic acid group easily identified in the mango peel derived pectin as shown in Figure 2.

**Table 4.** Micromeritic study data of pectin

**Tabela 4.** Mikrometryczne dane studyjne pektyny

| Parameters | Values |
|---|---|
| Angle of repose | 28.01 |
| Carr's index | 14.71 |
| True density (gm/ml) | 0.49 |
| Bulk density (gm/ml) | 0.81 |
| Bulkiness | 1.23 |
| % Porosity | 39.06 |
| Mean particle size (microns) | 3.44 |

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