Literature Cited

DETERMINING THE YIELD AND QUALITY OF PECTIN FROM FRESH PEEL AND PECTIN POMACE

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Abstract. A method for determining the yield and jelly grade of pectin in freshly extracted citrus peel and dry pomace is outlined. A flow diagram shows the necessary steps. The importance of the steps as well as critical control points are discussed. This method may be used to determine the amount of pectin in the peel so that the citrus processor can decide either how much peel to make into pectin pomace or how much to make into cattle feed.

The estimated annual worldwide production of pectin is approximately 7,250 metric tons, approximately 60% of which is produced from citrus. California produces approximately 30% of the world's supply. The citrus processing industry in Florida annually produces over 779,000 metric tons of dried citrus peel, mainly orange and grapefruit, for use as cattle feed. Currently, none of this peel is being made into a more valuable product, pectin pomace, which would yield 20-30% pectin. However, some of Florida's lemon and lime peel is being leached and dried into pectin pomace which is then transported to Europe for manufacture into pectin. This is an expensive way to make pectin because of the high cost of transportation and handling.

A pectin plant operating in Florida, extracting pectin from fresh rather than dried pomace, could be expected to produce approximately 15% more 150 grade pectin. Today, there is not sufficient demand for all of Florida's citrus peel to be made into pectin but a significant amount could be profitably made into pectin, and there are prospects for an increasing demand for pectin. Researchers are investigating the physiological benefits of consuming larger amounts of pectin in the diet. If additional research substantiates that pectin significantly lowers serum cholesterol in humans, then there may be a large increase in the demand for citrus pectin.

In addition, there were many people in the citrus industry who felt cattle feed would have no market if Florida ever produced more than a million short tons. However, we have

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Experimental Method

Materials. Fresh citrus peel from the juice extraction process, taken as quickly as possible, or leached and dried peel (called pectin pomace) were used for the experimental analyses. Chemicals were reagent grade and the isopropanol was checked for metal contamination. Tap water was used in the leaching step and distilled water in the extraction step. The apparatus used to test the jellies is described (5).

The word 'peel' collectively refers to both the fresh peel and pectin pomace.

Method. The procedure for evaluating the yield and quality of pectin from citrus peel is outlined in Fig. 1. This figure shows an overview of the steps involved and the approximate amounts of peel needed for each step. A detailed description of this method including the necessary apparatus, reagents, and step by step instructions, as well as example calculations and typical data sheets is available from the authors (3).

Discussion

Total solids in fresh peel. It is important to start the analysis with a known quantity of fresh peel because this will be used in calculating the yield of 150 grade pectin on a fresh peel basis. The percentage of total solids found in the fresh peel indicates how much soluble solids, pectin, and 'other' components besides water are in the peel (Fig. 1). After the fresh peel has been leached, pressed, and total solids determined, then the amount of soluble solids removed by leaching can be calculated as a difference.

The more soluble solids that are leached from the peel, the greater will be the yield of pectin from a given weight of peel. However, the larger amounts of soluble solids leached from the peel will place a greater load on the waste treatment facility. See reference (1) for a more complete discussion of leaching and utilization of the leach water.

In evaluating pectin pomace, the leaching step has already been done during manufacture, so it is necessary only to determine the percentage of total solids in the dry pomace.
PECTIN YIELD EVALUATION

Fresh Peel (500 g)
1. Det. % total solids
2. Leach with 1250 g H₂O
3. Press
4. Repeat 1-3

Leach Water
1. Waste water analysis
2. Discard

Dry to Pectin Pomace
1. Grind in blender
2. Det. amt. of acid for extraction
3. Use approx 28 g

Pressed Peel
1. Det. % total solids
2. Det. amt. of acid for extraction

\[ \text{Combined Pectin Solution 1 & 2 (1500 ml)} \]
1. Filter
2. Stir into 3 l of isopropanol
3. Let ppt stand for minimum 4 hr
4. Wash ppt 3 times with 70 % and 2 times with 99% isopropanol
5. Dry clean ppt 55° C under vacuum
6. Grind to pass 20 mesh screen

PECTIN QUALITY EVALUATION
1. To 5 g of ground pectin add dist. H₂O to make 1% solution
2. Estimate the jelly grade by consistency
3. Prepare and evaluate jellies by procedure in reference (5)

Acid extraction of pectin. A number of mineral acids can be used to extract the pectin from the other cellular material (9, 10). To determine how much acid will be necessary, a small amount of peel, water, and acid are heated together. The pH is then measured and the mixture is titrated with acid until the pH is below 1.6. Fig. 2 shows the relationship between the amount of acid added and the pH of the mixture. Preparing the curve is necessary to be able to calculate the amount of acid necessary to lower a larger amount of peel to pH 1.6 during the pectin extraction.

For the pectin extraction, 25 g of dry solids from leach peel or pomace is normally extracted at one time. Approximately 70 times as much water as peel is heated together with the acid. By using the curve (Fig. 2), the amount of acid can be calculated that is necessary to lower the pH of 25 g of dry wt of peel to pH 1.6. The hot water and acid are added to the peel to extract the pectin. The time, temperature, and pH of the extraction are critical to achieving the maximum jelly units and change depending on the cultivar and the time of the year (3, 6, 9).

Pectin recovery. The pH should be 1.6 ± 0.05 after the completion of the extraction. The pectin solution is then centrifuged away from the insoluble cellular materials and a second water wash is used to insure a more complete pectin removal. The pectin solution is dehydrated and precipitated with isopropanol. Water and isopropanol washes are used to remove any acid trapped in the pectin precipitate. The pectin is dried under vacuum and then quickly ground. The weight of this material is the yield of pectin.

Pectin quality evaluation. Some changes and modifications have been made in the previously published evaluation procedure (5). Basically, this method involves making a 1% pectin solution then estimating the jelly grade by a consistency determination (2, 3). The pectin solution is heated, sugar is added, and water boiled off to achieve a 65°Brix solution.

The hot solution is poured into jelly glasses containing acid and allowed to jell. The amount of sag in these standard jellies is measured by a Ridgelimeter (3, 5) and through the use of a graph the true jelly grade calculated.

Calculations. The jelly units can be calculated by multiplying the jelly grade times the yield. By knowing the total solids in the fresh peel, leached peel, and pomace the percentage yield can be calculated on these bases, respectively. By dividing the actual jelly grade by 150 and multiplying it times the percentage yield, the percentage yield of 150 grade pectin from fresh peel can be calculated.

Fig. 1. Flow diagram for determining the yield and quality of pectin from fresh peel and pomace.
Fig. 2. An example pH curve for 10 g of lime peel showing the change in pH per 0.5 ml of nitric acid addition.

Conclusions

Total solids in fresh peel. This information is needed in order to calculate the yield of pectin on a fresh peel basis. This is an indication whether or not this peel can be economically made into pectin. The total solids are also used to determine the efficiency of the leaching procedure and evaluating the pectin yield vs. pollution treatment costs.

Acid extraction of pectin. The citrus processor who is considering selling fresh peel or pomace needs to agree with the pectin manufacturer on the type of acid and conditions used in the extraction. This will insure an equitable basis for evaluating the peel. The time and temperature of the extraction needed to get the maximum jelly units will change depending on the cultivar of fruit and time of the year (9).

Pectin recovery. The method outlined in a laboratory method that gives an estimation of the maximum yield and quality available from the peel. In a commercial operation, the pectin solution will probably be concentrated before precipitating the pectin in alcohol or aluminum chloride.

Pectin quality evaluation. The jelly grade of an ‘uncut’ pectin determines how much sugar must be added to the pectin to make a standard 150 grade pectin. There is normally some trade off between getting a high yield of pectin and a lower quality.

Calculations. Example calculations are given in the detailed method (8). Through the use of this laboratory method, a processor can calculate the yield and quality of pectin in fresh peel or pomace. This information will be valuable for accurate assessment of the costs incurred in leaching the peel, making pomace, or manufacturing pectin.

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