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GLYCOSIDE AND PECTIN SUCCESSIVELY EXTRACTED FROM CITRUS[1,2]

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Abstract. **Orange albedo and whole lemon peel were subjected to a hot water leach to remove the soluble solids from the peel prior to pectin extraction. Neither room temp nor 90°C water leached appreciable quantities of hesperidin from the peel. Jelly units of recovered pectin ranged from 75 to 86 for orange and lemon peel leached at 90°C. For a control on the hesperidin yield, a commercial type of caustic leach was used. Yields of hesperidin ranged from 1.2 g to 4.3 g per kg of peel for the single strength press liquor and between 2.3 g and 5.5 g per kg of peel for a threefold concn.**

A pectic enzyme was tested as a substitute for $Ca(OH)_2$ to degrade the water soluble pectin from the hot leach water during the extraction of naringin and pectin from grapefruit peel. By using 100 ppm of enzyme and treating for an hr at 35°C, we recovered an average of 70% of the 4 g of naringin per kg of whole peel recovered by the original hot water leach method.

Glycosides are recovered in most commercial procedures by treating the peel with $Ca(OH)_2$, pressing and then crystalizing them from the press liquor after pH adjustment. This process renders the pectin in the peel nonrecoverable because it has been precipitated as calcium pectate.

In commercial operations, pectin is normally extracted by heating in a mineral acid solution after first leaching the peel with room temp water to remove the soluble solids (mostly sugars). If the glycosides are not leached from the peel, they will be destroyed by the heat and mineral acids used to extract the pectin (4). Thus, from either of the commercial methods only the glycoside or pectin, not both, are recovered from the same raw material.

Earlier, we found naringin could be efficiently leached from grapefruit peel followed by pectin extraction (2). In this procedure, calcium hydroxide was used to precipitate the water soluble pectin from the hot leach water.

The purpose of this research is to extend a previously reported method used to recover both naringin and pectin in an attempt to recover both hesperidin and pectin consecutively. Additionally, yields of naringin are compared using the reported hot water leach which uses $Ca(OH)_2$ to ppt. the water soluble pectin in the leach water and a new method using an enzyme to degrade the water soluble pectin.

Materials and Methods

Each of the commercially shaved albedo samples of Pineapple and 'Valencia' oranges (*Citrus sinensis* [L.] Osbeck) together with whole peel samples of 'Sicilian' lemon (*Citrus limon* [L.] Burm. f.) was divided into 2 equal batches. One half was processed using a caustic leach (Fig. 1) which served as a control and the other half using the hot water leach (Fig. 2). A room temp leach was also tested on 1 sample. The leaching procedures and subsequent extraction of pectin from the press cake (Fig. 3) were similar to those reported earlier (2). The $Ca(OH)_2$ leach procedure in Fig. 1 was modified for hesperidin extraction by using additional $Ca(OH)_2$ to reach an initial pH of 11.1 to 11.3 for 1 hr, instead of pH 8.8 to 9.0, pressed, then lowered to pH 4. In this paper, the term 'peel' refers to both the whole peel and shaved albedo samples collectively.

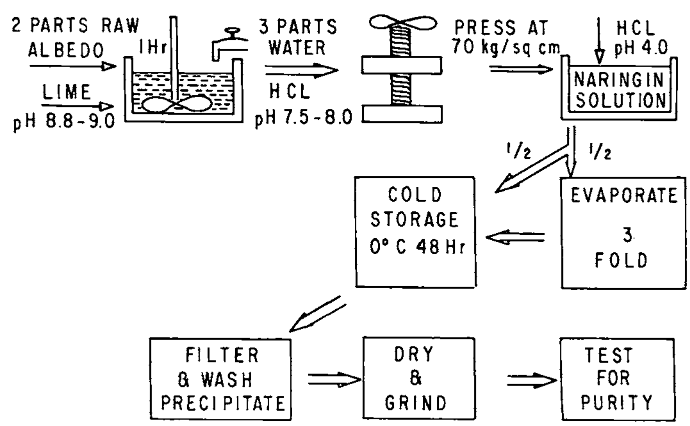

Fig. 1. Naringin extraction using $Ca(OH)_2$.

Whole peel samples of 'Marsh' grapefruit (*Citrus paradisi* Macf.) were also divided into 2 batches. One half was processed by the hot leach (Fig. 2). One half was processed by a modification of the hot leach procedure in which Irgazyme 100 (Ciba-Geigy) was used instead of $Ca(OH)_2$ (upper right corner in Fig. 2) to degrade the water soluble pectin prior to evaporation. Enzyme (100 ppm by wt of solution) was added to the sample and held 1 hr at approx 35°C. Then the sample was handled as in the hot leach procedure.

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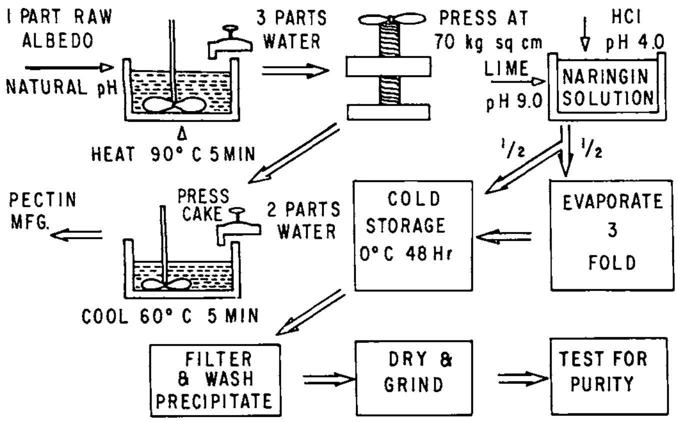

Fig. 2. Naringin extraction using hot water.

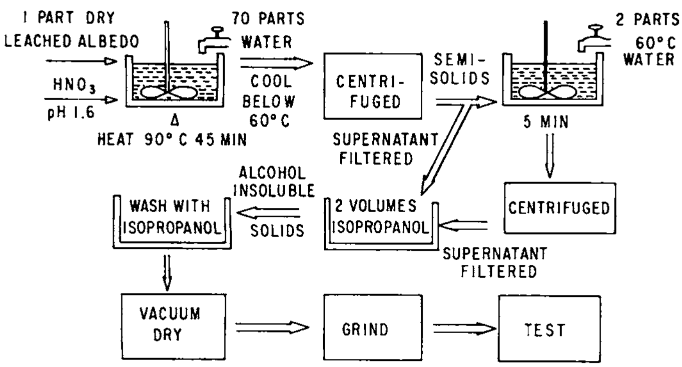

Fig. 3. Pectin extracted from the press cake after a hot water leach.

Results and Discussion

Previous work has shown hot water leaching of grapefruit albedo to be a more efficient method of recovering naringin than the traditional caustic leach (2). In this study, we found neither a room temp nor hot water leach yielded substantial quantities of hesperidin. The traditional caustic leach procedure recovered an average of 6.3 g per 2 kg wet peel (Table 1). Recovery of hesperidin was increased by approx 20% using a threefold concn of the leach water before crystallization.

Table 1. Yield of hesperidin from the caustic extraction of 'Pineapple' and 'Valencia' oranges and 'Sicilian' lemon.

Date	Single strength[z]	Concentrated[z]
	Pineapple orange	
Jan.	7.8[y]	8.9
	Valencia orange	
March	8.6	11.0
May	6.5	7.7
	Sicilian lemon	
July	2.4	4.6

[z]*Single strength* denotes the hesperidin solution after pressing while concd denotes a threefold concn of this solution.
[y]Values are expressed as g of pure hesperidin isolated per 2 kg of shaved albedo (wet wt).

Values of the pectins made from the 90°C water leached peel are shown in Table 2. Jelly units, found by multiplying percent yield x grade, were highest for lemon; however, this value was high compared to our earlier work (7). The jelly units for the grapefruit averaged 73.6 with a SD of 3.6 while orange averaged 76.1 with a SD of 2.4. The yield of 150 grade pectin expressed on an original fresh peel basis was lower for the grapefruit largely because of a lower total solids.

Table 2. Yield of pectin extracted with nitric acid (pH 1.6) following a hot water leach to remove glycosides (calculated on a dry wt basis).

Sampling month	% yield	Jelly grade	Jelly units	Yield of 150 grade[z]
		Marsh grapefruit		
Feb.	30.6	254	77.7	4.20
March	33.4	217	72.4	3.86
April	32.8	216	70.9	3.73
		Valencia orange		
March	35.2	212	74.8	7.19
May	34.4	229	78.9	8.35
		Pineapple orange		
Jan.	32.6	229	74.7	7.20
		Sicilian lemon		
July	33.2	259	86.0	11.3[y]

[z]Yield of 150 grade pectin calculated on the original fresh peel basis.
[y]Our later work on lemon shows this value to be twice that of lemons later in the season.

Previous work (2) has shown that naringin and pectin could be successively recovered from grapefruit peel leach water using $Ca(OH)_2$ to remove the water soluble pectin prior to evaporation. Several preliminary experiments were tried using enzymes to degrade the water soluble pectin (1). The enzyme treatment reduced the time needed to filter the naringin solution by half and also reduced the time needed for evaporation. Recoveries with the enzyme treatment averaged 70% of that recovered by using the $Ca(OH)_2$ treatment. The average yield for the enzyme treatment was 5.8 g per 2 kg of whole peel with a SD of 4.2 while the $Ca(OH)_2$ treatment had an average yield of 8.6 g with a SD of 2.4 g per 2 kg of whole peel. This yield from the hot water leach with caustic treatment is less than we found earlier (2) because in this experiment, we used the whole peel rather than the shaved albedo and in this experiment, we used more mature fruit. Maturity and using whole peel have been shown to decrease yields of naringin (3). Naringinase in many commercial pectic enzyme preparations (9) could also have an influence on yield.

Energy considerations

An important consideration in any new process is the amount of energy required. We have shown that a room temp water leach of citrus peel will not yield appreciable quantities of glycosides—only pectin. Using a $Ca(OH)_2$ leach of citrus peel will allow only glycosides not pectin to be recovered and the hot water leach yields both naringin and pectin. So, we should consider whether the extra energy required for the hot water leach is compensated for by increased yield of naringin and pectin. The pectin manufacturing operation is complex and has been dealth with by Snyder (8) and Rouse (6).

Comparing the naringin recovery from the $Ca(OH)_2$ leach vs. the hot leach preceding the pectin operation (Figs. 1 and 2) one can see the differences in the 2 processes. The basic differences lie in the ratios of peel to water 2:3 for the caustic and 1:3 for the hot water process. The hot water leach requires that the peel and water be heated to 90°C; however, the heat remaining in the press liquor can be utilized during evaporation, an energy savings. The additional energy consumption in the hot water leach process can be estimated assuming we start with *1 metric ton* of albedo in both processes and assuming negligible loss of heat to the environment. We are also assuming the water from the second leach (Fig. 2) may not be needed because there are cheaper cooling methods. By using experimentally determined values, we find that an additional 1865 kg of H_2O will need to be heated to 90°C from 25°C which requires 5.1 x 10^8 joules. Additionally, 570 kg of H_2O at 90°C will have to be evaporated. The joules required for evaporation will vary depending on the type of evaporator used. Assuming 1.09 kg of water can be evaporated with 1.02 x 10^6 joules (2.4 lb. per 970 BTU), then the 570 kg will require 5.3 x 10^8 joules for evaporation. This would give a total of 1.0 x 10^9 joules of extra energy consumed per metric ton of albedo processed. This amount of energy can be supplied by 454 kg (1000 lb.) of steam with a current cost of about $3.50 (5). However, the hot water leach process would yield approx 47 kg of 150 grade pectin in addition to increased naringin yields (2). These additional yields from the hot water leach process should more than adequately compensate for the small increase in the cost of energy consumed.

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EVALUATION OF HUMIDITY CONTROL PANELS FOR REFRIGERATED STORAGE OF CITRUS[1,2]

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Abstract. **Panels of a natural clay mineral have been used in meat storage and associated industries for desorption and adsorption of water to reduce relative humidity (RH) fluctuation and refrigeration operating time. Such panels were installed and tested for fresh citrus storage at the AREC, Lake Alfred at temp of 10 and 21°C. In all cases, average RH differential was less with the panels installed. However, the panels displayed high adsorption capacity above 80% RH. Refrigeration operating time was reduced by ca. 25% at 21°C. Equilibrium moisture contents were obtained to determine actual adsorption capacities. When the mineral directly contacted the fruit, specifically 'Marsh' grapefruit, no detrimental effects were noticed in subsequent 3 weeks storage.**

In general, citrus fruit are stored for shorter durations than deciduous fruit. However, environmental storage conditions are important as losses do occur from decay, desiccation and physiological disorders. Recommended storage conditions for citrus vary considerably depending upon the variety, geographic location, time of year and seasonal variations. ASHRAE (1) presents recommended temp and relative humidity (RH) conditions for various citrus varieties. For all varieties, RH above 85% is suggested. Such high moisture conditions are usually maintained through direct humidification or air-water counterflow refrigeration system as described by Deason and Grierson (2).

The more conventional direct humidification system results in additional costs since moisture is condensed on the evaporator coil. Usually, the coil is oversized, 0.5 to 1.5°C temp split, to reduce such problems by keeping temp above the dew point. An alternative to the increased cost caused by high humidity would be the incorporation of a material that would adsorb and desorb moisture, therefore, maintaining, a more uniform RH and partially reducing water condensation on the evaporator coils. According to Sereg (personal communication), such systems have been used for meat storage on an experimental basis. The research reported in this paper is an initial evaluation of this humidity control material (HCM) for fresh citrus storage.

Most adsorbent materials are used for extraction of vapor or gases brought into contact with them. The desorption phenomena is usually not encountered since the ambient partial vapor pressure is above that of the adsorbed vapor. Hence, their primary use has been in dehumidifying applications. The aggregate material used for humidity control in this study was a combination of calcite (45%), allophane (25%) and quartz (30%). Data on characterizing the material for adsorption water capacity is also included in this paper.

Materials and Methods

To establish design information on the moisture adsorbing characteristics of the HCM, a series of tests were conducted to establish equilibrium moisture content at various RH levels. Two temp were chosen, 4 and 21°C, which represent temp in the low and high range of citrus storage. Various RH conditions were established in sealed desiccators by using different concn of sulfuric acid and distilled water as described by Hall (3). Three oven-dried HCM samples, approx 50 g each, were placed in separate flat dishes and placed in each desiccator. Measurements of

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