PASSION FRUIT BY-PRODUCT: PROCESS DESIGN OF PECTIN PRODUCTION

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ABSTRACT

This work aimed to dimension a processing line for the extraction of pectin from passion fruit peels to the scale-up of an industrial pulp and juice processing plant. Taking into account that a medium-sized industry in Brazil processes 3,000 tons of passion fruit annually, the production of pectin was 23,934.24 kg·year^{-1}, under the extraction conditions of 80 °C, 52.5 min, and 0.0002 mol·L^{-1} of citric acid. The process includes unit operations such as solid-liquid extraction, filtration, drying, and grinding of the pectin. The calculations of mass balance, energy balance, and the dimensioning of the equipment were made from data provided by the literature and by equipment suppliers. The results achieved help in the implementation of this process on an industrial scale.

1. INTRODUCTION

Currently, there is an increase in the consumption of fruit derivatives, such as pulps and juices, for reasons of practicality and search for quality of life [1]. However, the industrialization of fruit can generate a large volume of by-products and waste. An example is the processing of passion fruit (Passiflora edulis flavicarpa) for the production of juice, in which peels and seeds are generated. These parts have chemical compositions that allow their transformation into new materials, such as polysaccharides and oils [2], [3].

During the processing of passion fruit in the food industry, high amounts of peel and seed are removed from the fruit and are not used properly, which can lead to significant economic losses. The peels correspond to 51 % w/w of the total weight of the fruit [2]. According to [4], the peel of the passion fruit has a significant amount of fibers (63.98-72.62 % bs), especially pectin (6.98-19.6 % bs). The pectin is a polysaccharide galacturonic acid, used as a thickening, stabilizing, and gelling agent due to its technological and functional properties [5], [6], [7], [8], [9].

The world production of passion fruit was 23,874,632 tons in 2018. Brazil is among the ten largest worldwide producers of passion fruit, with an output of 602,652 tons [5], which is destined to the fresh fruit market (10 %) and industrial processing (90 %). In that year, the average shell waste in Brazilian processing industries was around 276,616.81 tons.

Studies have been described the extraction of pectin from passion fruit peel at a laboratory scale [2], [4], [5], [10], [11], [12], [13], [14]. However, few studies address the process design and scale-up, with little data on handling highly viscous solutions, calculations for scaling up and equipment sizing. In this context, the objective of this work is to establish data for the industrial production of pectin, based on the observed data of yield of its extraction from the passion fruit peel, by using a solution acidified with citric acid. Relevant technical aspects influencing the scale-up have been presented as a useful tool for determining of the industrial process.

2. MATERIALS AND METHODS

2.1. METHODOLOGICAL APPROACH

For the pectin extraction, the methodology of the pectin extraction followed [2], for which the optimized extraction condition (80 °C, the citric acid concentration of 0.0002 mol·L^{-1} and extraction time of 52.5 min) presented the yield of 13.44 %. The process flowchart is shown in Figure 1.
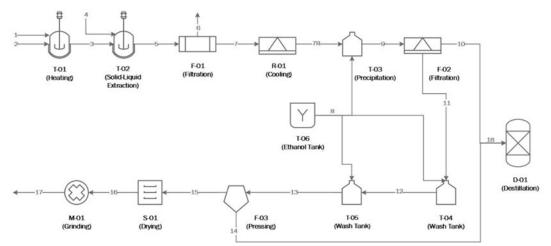

Figure 1: Process diagram for pectin production from passion fruit peel: (1) citric acid 99.5%; (2) distilled water (25 ºC); (3) 0.0002 mol·L^{-1} acidified solution ; (4) fresh passion fruit peel; (5) suspension; (6) solid material retained in the filter; (7) solution containing the extracted pectin; (7R) cooled solution containing the extracted pectin; (8) absolute ethanol; (9) suspension containing the precipitated pectin; (10 and 14) filtered solution without pectin; (11, 12 and 13) pectin gel; (15) solid and moist pectin; (16) dry pectin; (17) dry and ground pectin.

Initially, in a mixing tank under heating (T-01), citric acid (stream 1) and distilled water (stream 2) were mixed to produce an acidified solution at a concentration of 0.0002 mol·L^{-1}. The acidified solution was heated to 80 °C and then added to the passion fruit peel (stream 4), in the proportion of 1:30 m/v (peel: solution), in T-02. After extraction for 52.5 min, stream 5 was filtered F-01 to remove suspended solids. A fraction of 15 % of the total volume of stream 5 was retained in the filter (stream 6), and the other 85 % of the suspension volume was separated as a filtered liquid containing pectin (stream 7). Stream 7 was cooled in a tank (R-01) until it reached 4 ºC, and pectin was precipitated in T-03, after the addition of absolute ethanol in the ratio of 2:1 v/v (stream 8: stream 7R), remaining at rest for one hour. The suspension containing precipitated pectin (stream 9) was filtered through F-02, with 85% efficiency. The pectin gel formed (stream 11) was washed twice with absolute ethanol, from stream 8, in

tanks T-04 and T-05, to remove soluble impurities, such as salts and sugars. Then, the gel (stream 13) was filtered (F-03) to remove the liquid (stream 14, which represents 85 % of the suspension). The solid and moist pectin (stream 15) was allowed to dry in S-01 at 45 ºC until it reached a constant mass. Finally, the dry pectin (stream 16) was ground in a hammer mill (M-01), obtaining stream 17. The effluents containing ethanol (streams 10 and 14) were collected and fed (stream 18) to the column distillation system D-01, to recover ethanol and reduce process costs.

2.1.1. PHYSICAL PROPERTIES OF LIQUID FLOWS

The physical properties of the liquid resulting from the hydrolysis, precipitation, and filtration steps were determined. The viscosities and specific masses of currents 3, 7, 10, and 14 were measured using a viscometer, pycnometer, and analytical balance (model FA 2204C, accuracy 0.0001 g). The alcohol levels of currents 10 and 14 were determined using a portable refractometer for alcohol (Contec, ATC, China).

2.1.2. PECTIN GEL DRYING CURVE

The drying tests of the pectin gel were conducted to obtain data to solve the mass and energy balances and to scale-up the drying unit operation. The drying operation was carried out in an oven (Nova Ética, 400-4ND, Brazil) with forced air circulation at 45 °C. During drying, the samples were weighed using an analytical balance (model FA 2204C, accuracy 0.0001 g). Sample masses were recorded every 5 min in the first hour and then every hour after that. The dry matter mass of the sample was determined and used to calculate the moisture on a dry basis (XBS). The tests were done in duplicate. The mathematical models used to describe the drying curves are Henderson and Pabis [15], Henderson and Pabis Modified [15], Logarithmic [18], Two terms [19], Verna [20] and Midilli-Kucuk [21].

2.2. MASS BALANCE

To the mass balance calculation, the process was outlined in a block diagram, representing the unit operations involved and the streams between the operations. Equation 1 was used in the calculations of the general mass balance:

$$Input + Generation - Output - Consumption = Accumulation \qquad (1)$$

A medium-sized passion fruit company in Brazil annually processes 3,000 tons taking into account 12 months of work and 22 days per month. Based on experimental data, the peel represents 51 % of the total mass of the fruit. Therefore, the amount of peel available for pectin production is 1,530 t·year[-1]. According to the time required in the laboratory experiments, one day was enough to carry out two batch processing. The knowledge of the process conditions in batch mode allows the mass balance calculation and, consequently, the dimension of the process equipment.

2.3. ENERGY BALANCE

Equation 2 was used to solve the overall energy balance:

$$\Delta H + \Delta E_c + \Delta E_p = Q - W \qquad (2)$$

Where ΔH is the variation of enthalpy (J), ΔEc is the variation of kinetic energy (J), ΔEp is the variation of potential energy (J), Q is the energy associated with heat that is removed or supplied (J), W is the energy associated with work (J).

The working pressure of the boiler, 8 bar, is the same as that of the medium-sized agribusiness. Heat capacity (Cp) was defined as reported by [20].

2.4. STIRRED AND HEATED TANK

The energy balance in the tank with agitation and heating was solved, taking into account some considerations, such as there is no variation in kinetic energies and potential energy, and the system is adiabatic. The currents used to perform the calculation are shown in Appendice A1.

2.5. DRYING

The assumptions to solve the energy balance in the dryer were (1) pectin inlet and outlet temperatures of 25 °C and 30 °C respectively; (2) air inlet and outlet temperatures of 90 °C and 45 °C respectively; (3) inlet air humidity of 0.010 $kg_{water} \cdot kg_{dry}^{-1}$; and (4) the material to be dried comes from two batches, that is, the result of the production of one day. Appendice A2 represents the flow diagram used to calculate the energy balance.

2.6. EQUIPMENT SIZING

The equipment used in the process was defined based on laboratory experiments. The dimension of the equipment, the size of the lots and the number of batch cycles were determined from the mass balance calculations considering the design data available in the literature [20], [21], [22], [23], [25].

2.6.1. TANKS DESIGN

Cylindrical tanks were used for extraction, precipitation, and washing operations. Two tanks in series were used for washing, and its dimensions were calculated as suggested in the literature [20], [21], [22], [25]. The relationship between the height of the liquid and the diameter of the tank was adjusted to 1.0. The total volume of the tank was calculated to fill, on average, 75 % of the final amount.

2.6.2. AGITATION AND HEAT TRANSFER

The internal measures of the extraction tank were dimensioned according to the literature [20], [21], [25], and the dimensions are presented in Appendice A3.
The heating time in the tank was determined using Equation 3.

$$T_2 = T_v - (T_v - T_1) \cdot \exp\left\{\left[(UA)/(mC_p)\right] \cdot t\right\} \tag{3}$$

Where T_2 is the final heating temperature (°C), Tv is the steam temperature (°C), T_1 is the initial temperature (°C), U is the global heat transfer coefficient ($W \cdot m^{-2} \cdot K^{-1}$), A is the area of the tank (m²), m is the mass of the fluid (kg), C_p is the heat capacity of the fluid ($kJ \cdot kg^{-1} \cdot K^{-1}$), t is the time (s).
The heat flux depends on the overall heat transfer coefficient (Equation 4).

$$U = \left\{1/\left[(1/h_{TK}) + (1/h_C) + (R_F)\right]\right\} \tag{4}$$

Where, h_{TK} is the heat transfer coefficient of the fluid in the tank ($W \cdot m^{-2} \cdot K^{-1}$), h_C is the vapor heat transfer coefficient ($W \cdot m^{-2} \cdot K^{-1}$), R_F is the fouling resistance.
The values of RF and h_C were adopted, as suggested by the literature [26], [27]. The h_{TK} value (Equation 6) was estimated as reported by [28]. The values of k_S and n (11.5 and 0.5, respectively) were adopted, as suggested by [28].
The power required to drive the stirrer was determined using empirical equations [20], [21]. Energy consumption was calculated using equation 5.

$$N_P = P/\left(\rho \cdot N^3 \cdot Da^5\right) \tag{5}$$

Where, NP is the power number, P is the power (W), ρ is the specific gravity of the fluid (kg·m^{-3}), and Da is the diameter of the agitator (m).

2.6.3. FILTRATION

The plate and frame filter press were dimensioned by using data collected experimentally. For that, tests were carried out at operating pressure adjusted to 1 atm. The filtrate volume was measured over the filtration time, generating graphs with observed data, making it possible to determine the parameters α and β for the filter design. Equations 6 and 7 were used to determine the values of the mean resistivity <α> and the resistance of the filter medium (R$_m$) [21].

$$\alpha = \left[s_p / \left(2 \cdot A^2 \right) \right] \cdot \left[\left(\rho \cdot \mu \right) / \Delta P \right] \cdot <\alpha> \tag{6}$$

$$\beta = \left[\mu / \left(A \cdot \Delta P \right) \right] \cdot R_m \tag{7}$$

Where, s$_p$ is the absolute mass fraction of solid (kg$_{solid}$·kgl$_{iquid}$$^{-1}$), A is the filtration area (m^2), ρ is the specific mass of the filtrate (kg·m^{-3}), μ is the dynamic viscosity of the fluid (kg·m^{-1}·s^{-1}), ΔP is the pressure variation (kg·m^{-1}·s^{-2}).

The filtration area was calculated using equation 8; γ was equal to 1 [21], because the filtrate volume was higher than the amount of the pie. For to determine the number of frames (nq) and plates (np) needed for each filtration operation, the face area (AF) of the filter was adopted according to the standard sizes of industrial filters, as reported by [21].

$$\left(t_F / V \right) = \left[\mu / \left(A \cdot \Delta P \right) \right] \cdot R_m + \left[s_p / \left(2 \cdot A^2 \right) \right] \cdot \left[\left(\rho \cdot \mu \right) / \left(\Delta \cdot P \right) \right] \cdot \gamma \cdot <\alpha> \cdot V \tag{8}$$

Where t$_F$ is the filtration time (min) and V is the filtrate volume (m^3).

3. RESULTS AND DISCUSSIONS

3.1. PHYSICAL PROPERTIES

The physical-chemical properties of some streams are shown in Table 1. The stream 3 has specific mass and viscosity values close to that of pure water, which can be justified by the low concentration of citric acid. Note that the data of specific mass and viscosity of stream 7 are inversely proportional to the temperature. Stream 10 and 14 showed lower specific mass values when compared to other currents, which can be justified by the presence of ethanol.

Table 1: Experimental parameters of the process net flow

Stream	T (°C)	ρ (kg·m^{-3})	μ (kg·m^{-1}·s^{-1})	Alcohol content (%)
3	25 °C	1000	0.94·10^{-3}	-
7	25 °C	1040	1.50·10^{-3}	-
7	80 °C	985	0.85·10^{-3}	-
10	25 °C	850	1.11·10^{-3}	64.50
14	25 °C	890	1.14·10^{-3}	73.35

Stream 3: solution acidified with citric acid; Stream 7: liquid containing pectin (1st filtration); Stream 10: solution without pectin (2nd filtration); Stream 14: liquid without pectin (3rd filtration).

3.2. DRYING CURVE OF PECTIN GEL

The drying curve of pectin can be seen in Figure 2. After 6h of drying (360 min), the moisture of the pectin gel on a dry basis (XBS, %) does not present significant changes. However, it took 8 h (480 min) to reach equilibrium humidity (XBS = 5.72 %).
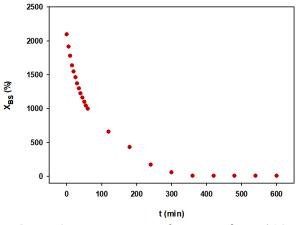

Figure 2: Drying curve of pectin gel at 45 °C.

The fit of the models to the drying curve data, and the results of the adjustments are represented in the Figure 3 and in Table 2, respectively. The evaluation of the model adjustments was based on the highest values of determination coefficients (R^2) and lowest values of relative average error (EMR) and root of the mean square of the residue (RQMD). Thus, it was observed that the "Two terms" model proved to be more suitable for describing the process, with R^2 = 0.9978, EMR = 3.60 %, and RQMD = 30.35.
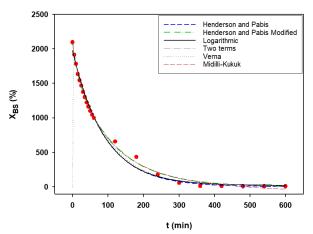

Figure 3: Mathematical models applied to the drying curve of pectin gel.

Table 2: Adjustment parameters of the models applied to the experimental data of the pectin gel drying

Model	R^2	EMR (%)	RQME
Henderson and Pabis	0.9928	6.21	60.56
Henderson and Pabis Modified	0.9978	3.82	33.35
Logarithmic	0.9929	6.29	59.89
Two terms	0.9978	3.60	30.35
Verna	0.5827	48.36	160.03
Midilli-Kucuk	0.9975	3.83	35.42

R2: Determination coefficient; EMR: relative average error; RQME: root of the average square of the residue.

3.3. MASS BALANCE

The quantities of the reagents used to carry out the processing steps are shown in Table 3.

Table 3: Fixed reagent amount to carry out the process

Operation	Description	Value	Unity
Extraction	Concentration of citric acid in the acidified solution	0.038	$kg_{acid} \cdot m^{-3}_{water}$
Extraction	Peel quantity per volume of an acidified solution	284.42	$kg_{acid} \cdot m^{-3}_{water}$
Precipitation	Volume of absolute ethanol (99.5 %) for liquid containing pectin	2	$kg_{acid} \cdot m^{-3}_{water}$
Wash	Volume of absolute ethanol (99.5 %) per amount of water in T-02	1/3	$kg_{acid} \cdot m^{-3}_{water}$

The block diagram of the studied process, with the control volumes, is presented in Figure 4. The volume control was not dashed in the cooling step because no addition or loss of mass was observed in this stage. The mass balance results are shown in Table 4.
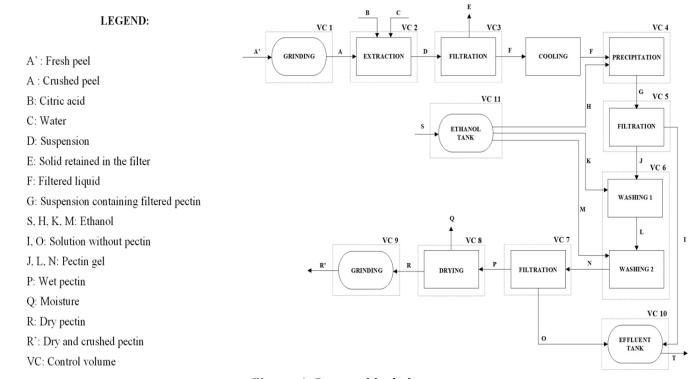

Figure 4: Process block diagram.

Table 4: Batch mass balance

Item	Stream										
	A'	A	B	C	D	E	F	G	H	I	J
Mass (kg)	2,897.73	2,883.24	0.39	7,579.55	10,463.18	1,569.48	8,893.71	22,405.30	13,521.59	19,044.51	3,360.79
Water (%)	88.26	88.26	0.00	100.00	96.76	81.16	99.51	39.80	0.50	35.49	64.19
Solid (%)	10.25	10.25	0.00	0.00	2.82	18.83	0.00	0.00	0.00	0.00	0.00
Pectin (%)	1.49	1.49	0.00	0.00	0.41	0	0.48	0.19	0.00	0.00	1.28

Citric acid (%)	0.00	0.00	100.00	0.00	0.003	0.003	0.003	0.002	0.00	0.001	0.002
Ethanol (%)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	60.00	99.50	64.50	34.53
	K	M	N	O	P	Q	R	R'	S	T	
Mass (kg)	1,337.04	1,337.04	6,034.88	5,129.65	905.23	859.68	45.56	45.33	16,185.68	24,174.16	
Water (%)	0.50	0.50	35.97	26.65	88.80	*	*	*	0.50	33.62	
Solid (%)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	
Pectin (%)	0.00	0.00	0.71	0.00	4.74	0.00	94.15	94.15	0.00	0.00	
Citric acid (%)	0.00	0.00	0.001	0.001	0.003	*	*	*	0.00	0.001	
Ethanol (%)	99.50	99.50	63.31	73.35	6.46	*	*	*	99.50	66.38	

*Fraction of the aqueous solution (water, acid, and ethanol).

3.4. ENERGY BALANCE

The masses of steam produced in the boiler and used to extract and dry the pectin were 986.77 kg and 2,955.91 kg, respectively. The annual steam demand was 1,301,374.80 kg for 528 batches per year (for extraction and drying). The drying operation was carried out in 2 batches.

3.5. EQUIPMENT SIZING

3.5.1. TANKS

The diameters calculated for the precipitation and wash tanks correspond to 3.37 m and 1.79 m, respectively. The calculated values are close to those defined by [29], who performed the equipment sizing calculations of a scale-up project of industrial pectin extraction plant from orange peel. The internal dimensions of the stirring and mixing tank are shown in Table 5. The global heat exchange coefficient calculated was 435.24 W·m^{-2}·K^{-1}, which implies that the time required for heating the acidified solution was 26.46 minutes.

Table 5: Internal dimensions of the extraction tank

Dimension	Size (m)	Dimension	Size (m)
D_{TK}	2.61	D_A	1.31
D_A	1.31	D_D	0.87
L	0.33	J	0.22
C	0.87	D_T	2.17
W	0.26	D_C	0.08

3.5.2. FILTER PRESS

The graphs elaborated with the experimental data of filtration at constant pressure are shown in Figure 5. Initially, for each test, a linear behavior was verified. Table 6 presented the obtaining parameters.
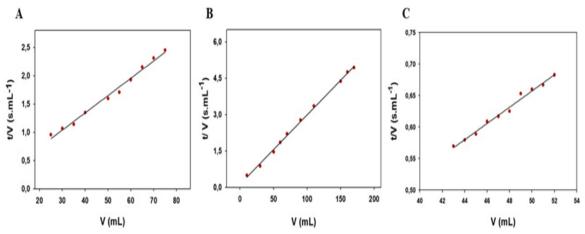
Figure 5: Experimental data performed for filtration at constant pressure: (A) Filtration 1 (F-1); (B) Filtration 2 (F-02); (C) Filtration 3 (F-03).

Table 6: Parameters of the filtration tests

Parameters	F-01	F-02	F-03
R^2	0.9898	0.9976	0.9878
A	0.0306	0.0286	0.0129
B	0.1234	0.1337	0.0140

The calculated values for the sizing of the filters are shown in Table 7. Two filters were selected for the second filtration (F-02), since this configuration reduces the number of plates, getting closer when compared to the other filtrations (F-01 and F-03). The calculated values are close to those defined by [27], for a scale-up project of industrial pectin extraction plant from orange peel.

Table 7: Sized filtration units

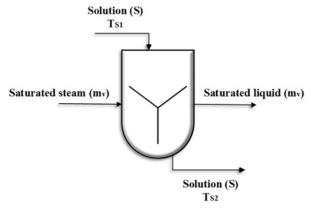| Data	F-01	F-02	F-03
s_p ($kg_{sólido} \cdot kg_{líquido}^{-1}$)	0.029	0.002	0.007
V (m^3)	8.55	21.39	6.03
$\langle \alpha \rangle$ ($m \cdot kg^{-1}$)	$8.27 \cdot 10^{12}$	$1.89 \cdot 10^{14}$	$2.29 \cdot 10^{13}$
R_m (m^{-1})	$7.19 \cdot 10^{10}$	$1.05 \cdot 10^{12}$	$1.07 \cdot 10^{10}$
A (m^2)	437	1213	282
n_q	55	76	35
n_p	56	77	36

4. CONCLUSIONS

According to the data obtained, it can be seen that the fresh passion fruit peel represents a large amount of by-product (51 % of the total fruit mass) generated in the juice industry, can be used as a raw material for pectin extraction in an acidic environment, using an organic acid.

Regarding the mass balance data, it was established that 45.33 kg of pectin are produced per batch, totaling 23,934.24 kg·year^{-1}. Due to the extensive volume of ethanol used in the precipitation and washing steps (16,185.68 kg·bacth^{-1}) it is recommended to reuse it, in order to make the process viable.
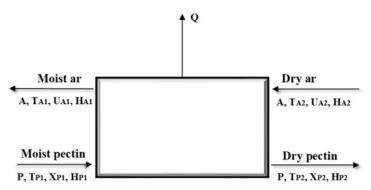
As a suggestion for future work, it is considered essential to perform computer simulation of the reuse of ethanol and economic analysis of the process, in order to determine its applicability on an industrial scale.
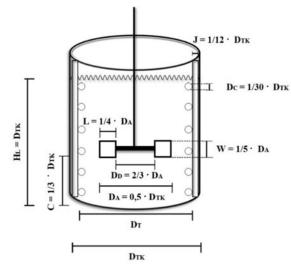
APPENDICE

Appendice A – Equipment sizing

Appendice A1: Representation of the energy balance in the thank.

Appendice A2: Flow diagram for the dryer.

Appendice A3: Representation of the internal measures of the tank.

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CONFLICT OF INTEREST

The author have declared that no competing interests exist.

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