Tutor
Dr. José María Gutiérrez González
Departament D'Enginyeria Química

Grau en Enginyeria Química

Treball Final de Grau

Design of a plant for extraction and purification of pectin.
Diseño de una planta para la extracción y purificación de pectina.

Saray Martínez Pizarro

June 2016

Universitat de Barcelona
Dos campus d'excel·lència internacional
B:KC Barcelona Knowledge Campus
HUB Health Universitat de Barcelona Campus

Lo que sabemos es una gota de agua; lo que ignoramos es el océano..

Isaac Newton

Quiero agradecer este trabajo a mi tutor, el Dr. José Gutiérrez, por su apoyo, ayuda y consejos en la elaboración de este proyecto.

También, a mi compañera y sobretodo amiga Eli, con la que he compartido estos años de carrera y sin ella no hubiese sido lo mismo. A mis amigas Carmen y Marta por todo el ánimo y apoyo. Agradezco también a mi Aaron, por estar conmigo en todo momento, por toda su paciencia y cariño.

Gracias a mi familia, por lo importante que son para mí y por apoyarme y ayudarme siempre. A mi tía y mi abuela, por ser los pilares fundamentales de mi vida, y a mi hermano, por lo indispensable que es para mí.

.

REPORT

CONTENTS

SUMMARY

Pectin is a natural substance found in the tissue of fruits and vegetables. Because of its great water absorbing capabilities, it presents gelling, stabilizing and thickening properties. Pectin is widely used in the food, pharmaceutical and cosmetic industries.

The purpose of this project is to design a plant for the production of pectin, operating in batch. Generally, the raw materials used are vegetable residues of juice factories, and, for this work waste of apple, peach, orange and lemon have been selected as raw materials. The plant to be designed should have a production capability of 6 t/year of pectin.

The production process of pectin consists basically in a prior selection and washing of raw material; solid-liquid extraction (at 70°C and in acid medium) to solubilize pectin; and filtering of solid waste; precipitation of pectin by addition of ethanol; solid-liquid separation of the precipitated pectin; and, finally, drying and milling and proceeds to final conditioning.

The units that are designed for the process are: opened stirred and jacketed tank for extraction, filter press to separate solid waste, closed stirred and jacketed tank for precipitation of pectin, basket centrifuge for solid pectin separation, and tray dryer to remove the humidity (alcohol and water).

Keywords: Pectin, production, plant, batch, process, design, waste.

RESUM

La pectina es una sustancia natural que se encuentra en el tejido de frutas y vegetales. Por la gran capacidad de absorber agua que tiene, presenta propiedades espesantes, gelificantes y estabilizantes. La pectina se utiliza ampliamente en la industria alimentaria, farmacéutica y cosmética.

El propósito de este trabajo es diseñar una planta para la producción de pectina en discontinuo. Generalmente, las materias primas que se utilizan son residuos vegetales de fábricas de zumos, y, para este trabajo se han seleccionado como materias primas residuos de manzana, melocotón, naranja y limón. La planta dimensionada permite una producción de pectina de 6 t/año.

El proceso productivo de pectina consiste básicamente en una selección y lavado previo de las materias primes; una extracción sólido-líquido (a 70°C y en medio ácido), para solubilizar la pectina; filtrado de los residuos sólidos; la precipitación de la pectina mediante la adición de etanol; una separación sólido-líquido para separar la pectina precipitad; y, finalmente, se seca, se muele y se procede a su acondicionamiento final.

Los equipos que se han dimensionado para el proceso son: tanque agitado abierto y encamisado para la extracción, filtro prensa para separar los residuos vegetales, tanque agitado cerrado y encamisado para la precipitación de la pectina, centrífuga de canasta para la separación de la pectina sólida y secador de bandejas para retirar la humedad (alcohol y agua).

Paraules clau: Pectina, producción, planta, discontinuo, proceso, diseño, residuo.

1. INTRODUCTION

In Spain, the fruit is placed in the third place of the food consumed in greater quantity, below meat and fish, and much of it is used for making juice, nectars, canned, etc. (Carbajal, 2012).

Industrial processing of fruit (canned, frozen, juice, concentrates and nectars) generates large volumes of vegetable residues and by-products, such as citrus peels, apple pomace, among others. If these agro-industrial waste are not given proper use, they will favour the proliferation of insects, fungi, bacteria and odours of decomposition, i.e., causing pollution problems. However, they could generate economic and environmental benefits if they were properly managed. It is therefore important to find other uses for leverage these wastes such as use them as raw materials for get high value-added products such as pectins, which have excellent emulsifying and stabilizing properties (Li -ping Qiu et al., 2010). Therefore, it is interesting to extract pectin from vegetable residues because is achieved that the pectin, which has no value being in the residue, pass to have great value once produced.

This work aims to design a plant which use waste fruit for extraction and purification of pectin. Pectin is a natural substance that is formed mainly in the primary cell wall and the middle lamella of tissue of fruits and vegetables, and acts as intercellular adhesive. It is a biopolymer that due to its thickening, gelling and absorption properties is used in the food industry, cosmetics and pharmaceutical (Chasquibol et al., 2008). Currently, pectins are very important ingredients in the food industry, for the manufacture of gelatins, jams, jellies, ice cream, sauces, etc. In the pharmaceutical industry they are used to modify the viscosity of their products (Devia, 2002). In addition, pectin reduces glucose intolerance in diabetic patients and even lower blood cholesterol levels (Chacin et al., 2010). In the cosmetics industry, they are used for toothpastes, oils, creams, deodorants, shampoos and bath gels by its stabilizing and softening properties (Chasquibol et al., 2008).

Pectins are heteropolysaccharides whose main component is polygalacturonic acid, which exists partially esterified with methanol (Herbstreith, 2001). It is known as degree of esterification, or methoxyl content, the number of carboxyl groups esterified with methanol.

Depending on the percentage of esterified galacturonic acid residues, pectins are classified as high methoxyl pectins when the percentage is above 50% (typical range 55-75%) and low methoxyl when less than 50% (typical range 20-40%) (Ullmann, 2011). High methoxyl pectin is the most common and allows gelled foods with a sugar content exceeding 55% and a pH of 2.2 to 3.3. Fruit pectins vary in methoxyl content and the gelling power, according to the type of fruit and maturity.

2. OBJECTIVES

The overall objective of this work is to design a plant that allows the extraction and purification of pectin from vegetable waste and operating in batch.

To get this objective will be necessary to address specific objectives:

- Select the appropriate and necessary raw materials, considering the pectin content and the availability at place in which the plant will be located.
- Know the parameters that affect the extraction of pectin, such as time, temperature, pH, and select operating conditions.
- Describe the pectin extraction process and the steps that involves, selecting and designing the necessary equipment for each.

Selection of raw materials to be used for the extraction of pectin, depend, among other things, on the products which containing this substance are consumed in the place where it is intended to place the plant. This study on raw materials will be addressed in "3.1. Determination of raw materials". A study to decide where the plant will be placed is performed, which will be explained in the section "3.2. Location of the plant". Production to be chosen, depends on various factors (production capacity, raw materials available, time that is producing the plant, etc.), this will be explained in the section "3.3. Determination of production".

Once known production and raw materials needed, the process to be used must be selecte. For this purpose, all the information found on the subject, either in encyclopaedias, magazines, patents, literature is taken into account. The necessary equipment and operations that performed each of tasks will be established and an outline of the process will be developed. This topic will be held in the section "4. Synthesis of process".

Then, the sizing of equipment will be held, which will be reflected in the section "5. Design of equipment".

Finally, the operation of the plant is explained in the section "6. Plant operation".

3. RAW MATERIALS, LOCATION OF THE PLANT AND PRODUCTION

3.1. DETERMINATION OF RAW MATERIALS

Pectin is a natural soluble fiber found in the cell walls of most plants, and is obtained from vegetable raw materials, mainly fruit because it reaches a high concentration of pectin in their skins (Devia, 2002).

The products that contain greater or lesser pectin amount are:

- Fruits: Some foods with greater amount of pectin are citrus fruits. The peels of citrus fruits such as oranges, lemons and grapefruits contain high levels of pectin. They also contain pectin apples, tangerines, cranberries, currants, grapes, quince, peaches, figs, bananas, pears, plums, apricots, pineapple, etc. Other fruits such as cherries, strawberries and raspberries do not contain much pectin.
- Vegetables: vegetables with the highest levels of pectin are carrots. They also contain pectin peas, cucumbers, celery and tomatoes.

The most common raw materials used for manufacturing of pectin are citrus peel such as lemon and lime, and apple pomace. Pectin is also produced from other less preferred citrus such as orange and grapefruit (Ullmann 's Encyclopedia of Industrial Chemistry).

When fruits that are to be used for the production of pectin have to be chosen it is important to know what are the most generally used, but it is also important that typical fruits of the place where the plant will be located are selected, as it would not be economical to use a product widely used to extract pectin if it is not available in large quantities and need to transport over long distances.

To select which fruits used as raw materials will be taken into account production and consumption in Spain, the country in which the plant will be located, as it is important to have high availability and volume of waste. In Spain the most produced sweet fruits are apple and peach, and the production of citrus, orange and lemon represents practically two-thirds (MAGRAMA, 2013). A large percentage of these productions is intended for domestic consumption and inside this consumption a large percentage is for industry to manufacture juices, preserves, etc.

Due to the large production and consumption in Spain of apple, peach, orange and lemon, plus the high generation of waste involved, these fruits are selected as raw materials for the plant will be designed.

For extracting pectin are used residues from industries which process these foods as are the industries of juices, nectars and cider, whose processes involve a great waste generation as peels, pulp, seeds, skins, etc. In this way, taking advantage of these residues to obtain a recoverable product as is pectin, and simultaneously, the volume of waste and environmental impact that they can cause decreases.

The following table lists the contents of pectin that have the fruit residues chosen based on dry matter.

BY-PRODUCT	% PECTIN
APPLE	**15 – 17,5**
ORANGE	**16 - 17**
LEMON	**25 - 32**
PEACH	**7,5 - 10**

Table 1.Pectin content of raw materials.

The vegetable waste that have higher content of pectin are residues of apple, orange and lemon. Using them a higher yield will be achieved. Furthermore, although it has a low content of pectin can be profitable the use of peach residues as obtained in large quantities, as in Spain large amounts of peach juice are produced. In this way, the volume of waste is reduced and it will get with them a recoverable product like is pectin, that in the waste has no value, but once produced is an expensive product.

The following table shows, approximately and depending on the weather, times of collection of each fruit, as a preamble to a subsequent process of selection, extraction, pasteurization and packaging (MAGRAMA). Therefore, waste derived from the production of juices, nectars and cider will be obtained during these periods collection, once have carried out all selection processes and manufacturing. It is necessary to take care of the times when each residue will be available to properly choose the different fruits of which pectin is extracted, and thus, keep the plant in operation for a reasonable period throughout the year.

Fruits	JAN	FEB	MAR	APR	MAY	JUN	JUL	AUG	SET	OCT	NOV	DEC
Orange												
Lemon												
Peach												
Apple												

Table 2. Each fruit season.

Given this seasonality, the plant to be designed could simultaneously process waste of oranges and lemons, and consecutively, peaches and apples residues. The table shows the period of collecting fruit so it will take a margin of time for that fruit to being processed and for the residue to being generated. In all the cases, the vegetable waste processed for the extraction of pectin will have to begin immediately after the manufacture of juices, nectars or cider to prevent microbial degradation.

Furthermore, it could keep the plant running for all seasons of the year: From December to May using waste of orange and lemon; From June to October, waste of peach; and from September to February, waste of apple. It has to keep in mind that it is going to work with vegetable waste which undergo decomposition with the passage of time, so it will has to work for campaigns since the waste cannot be saved. Furthermore, as the content of pectin varies depending on which fruit is the residue used, the amount to be produced in each period will be variable and may be period that do not produce due to lack of residues.

3.2. LOCATION OF THE PLANT

The location of the plant depends on the location of raw materials used for the extraction. These residues come largely of juice manufacturing industries, which use fruits as raw materials and require large volumes of them. Therefore, the manufacturing of juices is usually placed near the production areas of the fruits achieving a lower cost in the transport of raw materials. The study will be focused on Spain, the country where the plant will be located.

Firstly, the production areas of selected fruits for extracting pectin have to be localized. It is also important to know the intensity of production, as it will be more convenient to locate the plant near areas of high production than low.

- Apple: apple production is mainly in the Ebro Valley, particularly Catalonia and Aragon (Casals and Iglesias, 2013).

- Orange: the main producing area is Valencia. Followed by Alicante, Seville, and others with lower production volume as Huelva, Murcia, ... (MAGRAMA, 2006).
- Peach: the main producing areas are the Ebro Valley, Lerida and Murcia. Also the areas of Andalusia, Extremadura and Valencia, but in smaller volume (MAGRAMA, 2006).
- Lemon: the main producing area is the east coast, the most important Valencia, Alicante and especially Murcia, and in Andalusia almost all production is collected in Malaga (MAGRAMA, 2006).

The distribution of the production of each fruit in Spain is illustrated in maps. The regions marked with thicker lines are those that produce in a larger volume.

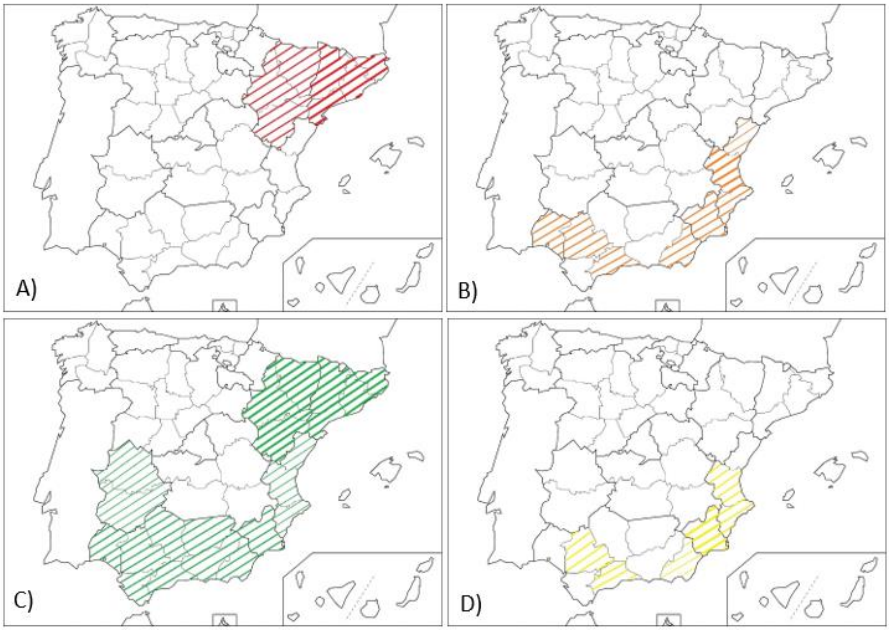


Figure 1. Production of fruits in Spain by regions. A) Apple, B) Orange C) Peach D) Lemon.

It is noted that the production of apple, orange, peach and lemon is spread across the country, but must take care of the differences between the production of the different types of fruit and their amount. Consequently, fruits that occur in larger volume will be more crucial to

decide the location of the plant pectin. The dates found about the Spanish production of fruits that are being considered are shown in the following table.

Spanish productions in 2011 [Mt]	
Apple	**579**
Orange	**853**
Peach	**2801**
Lemon	**720,5**
Source: FAO, EUROSTAT Y MAGRAMA	

Table 3. Pectin content of raw materials.

Using this table, we can see that the increased production would undoubtedly be the orange, but the peach and apple, as we have seen in previous maps, share a region that occur in larger quantities, Catalonia.

Another aspect that we must know to locate the plant is where are the most important juice factories, and therefore the ones which will generate more volume of waste. By searching for information, a business organization that integrates many juice producers in the country was found, which is the Spanish Association of Manufacturers of Juices (Asozumos). Moreover, other major manufacturers that are not within the organization have been found too. It is useful to make a selection by choosing the most prominent manufacturers and using the selected fruits. Juice manufacturers found are:

- Agrozumos, S.A. (Lekunberri, Navarra).
- Frusa: Frutos y Zumos, S.A. (Albal, Valencia).
- Go Fruselva, S.L.: (La Selva del Camp, Tarragona).
- Indulleida, S.A. (Alguaire, Lleida).
- Infrusesa: Industrialización de Frutas de Segre, S.A. (Soses, Lleida).
- Juver Alimentación, S.L. (Churra, Muria).
- Zuvamesa: Zumos Valencianos del Mediterráneo, S.A. (Puerto de Sagunto, Valencia).

The vast majority of juice factories are in Catalonia and Valencia. The comparation of all the aspects that have been studied is represented on the same map about the productions of each fruit and location of industries.

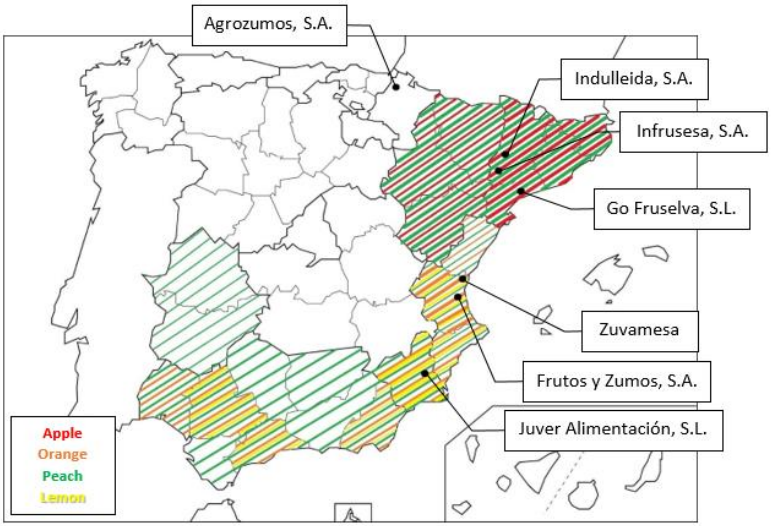


Figure 2. Fruit productions and industries.

The region of higher production of oranges is Valencia, of lemon is Murcia, of apple is Catalonia, and of peach is Catalonia and Aragon. Andalusia and Extremadura are discarded for the location of the plant, because the largest volumes of fruits are not produced and there are not coming any juice manufacturing industries. The plant would not be located in the area of higher production of lemon, Murcia, because as we are studying, peach and orange are more productive and that is why it is better to situate them close to these two fruits.

Although apple production in Spain has the lowest volume compared to other fruits (579 Mt), the fact that the greater production of apples and peaches agree with the zone (Catalonia), makes it interesting to consider this area for determining the location of the plant. If only the amount produced was decisive, knowing that the orange is the fruit with increased production, the plant would be located in the most intensive production thereof, i.e., Valencia. However, it is also important to put the plant near areas where there are several manufacturing industries juices, as it will be used as raw materials plant residues, which are by-products of these industries. In Catalonia, specifically between Lleida and Tarragona is where we find the most

industries of juice, so it would be desirable for the extraction plant pectin place himself in this area. Finally, it is decided that the location of the plant is in the south of Lleida, near the Indulleida and Infrusesa industries as well there are two providers of plant residues very close, and if it is not enough, Go Fruselva will be able too.

3.3. DETERMINATION OF PRODUCTION

In the bibliographic research have not found data on the production of pectin in Spain, but if of the world-wide production of pectin that is roughly of some 35.000 t/year. The main producing countries are Denmark, Holland, United States, Canada, Mexico, Switzerland and Germany, producing more than 8 one thousand annual tons (Chasquibol, 2008). Since no concrete data found for Spain, a study based on the information available is made and so it determines as the form of focus the determination of the production of the plant that is going to design.

The production depends directly on the amount of waste available. Waste available depends on the quantity of fruit produced and which percent of this is for the fruit juice industry. Waste required to produce pectin must not involve the use of all available waste, since it is something that is not feasible for a single plant due to the large volume of waste. To establish a production there are different criteria, such as preselect an annual production from an estimated amount of demand, etc., and on the other hand, set a charge and from it determine which production is obtained.

In this case, production of the plant is determined by a design study in which a reasonable load of solid waste. With this load, the amount of pectin to produce ir determined. To determine the annual production of pectin it has to be determine how many cycles can be made every day, taking into account the time required for each process, and also is necessary to determine how many days a year the plant works.

The cycles that can be made in one day are determined in later sections, once known the process and timing of each operation. The days that the plant should work are about 220, considering that not working on Saturdays and Sundays, there are 14 public holidays and one-month vacation. But these days can see reduced by the fact that the plant operate on campaigns, as indicated in paragraph of raw materials, it is possible that at some times not available residue and the plant cannot produce.

4. SYNTHESIS OF PROCESS

4.1. PECTIN EXTRACTION METHODS

Several patented methods of pectin extraction from vegetal tissue exist. Different pectin qualities are obtained from each one of these, since both pectin quality as its possible applications greatly depend on the obtention process (Devia, 2002). This fact is justified by these polysaccharides' structural complexity and natural variations, which depend, among other things, on the fruit species and ripening conditions. Since pectins are mostly used in food, it is necessary that reactives, dissolvents and equipment used in the extraction process do not leave any toxics in the final product (Sánchez et al., 2011). These different techniques use physic-chemical, microbiologic or enzymatic procedures (Zapata et al., 2009).

Most used extraction methods are: extraction on an acid medium, assisted extraction through microwaves and enzyme extraction.

4.1.1. Extraction on an acid medium

Consists on hydrolyzing, extracting and separating pectin from the rest of different fruits through acidification. In other words, pectin is extracted treating raw matter with hot mineral acid diluted on low pH. These steps are reproduced under the influence of different factors, such as temperature, pH, and treating time. The pH can be adjusted either using mineral acids; as chlorhydric acid, sulfuric acid, nitric acid or phosphoric acid, organic acids as citric acid, oxalic acid, and acetic acid. Lastly, it may also be adjusted with an alkali metal hydroxide of a divalent cation, such as sodium or calcium hydroxide, as well as a carbonate or bicarbonate of an alkali metal or a divalent cation (Nakamura, 2015). The acid's presence in a water environment, at relatively high temperatures quickly makes the fruits' peel cellular walls break sharply, producing hydrolysis of protopectin. Usually, extraction through acids produces pectins with a high esterification grade, while otherwise, using salts produces pectins with a low esterification grade (Sánchez et al., 2011). Afterwards, pectin is precipitated through mixing the extract with composts as aluminium salts, copper salts, calcium pectate or adient alcohols, as: ethanol, methanol or 2-propanol. These substances are used because of their capacity of coagulating or gelling pectin in their presence (Cerón & Cardona, 2011). Afterwards, it is dried, granulated and finally sieved. This method is known as the conventional method of pectin extraction. (Sánchez et al., 2011).

On the other hand, a method exists in which pectin is extracted without adding acid. Recently it has been discovered that pectin can be hydrolyzed and extracted of vegetal issue using fruits' and tissue's natural pH, which is enough to break links that tie pectin to tissues. Through this method, pectins high in methoxy (higher than 85) are obtained. But this method has a huge inconvenience: the time necessary to achieve results is too long to allow industrial scale production. Furthermore, problems may arise to obtain necessary pH (Devia, 2002).

4.1.2. Microwave-assisted extraction

This type of extraction uses energy dissipated by the electromagnetic camp to increase surface porosity. Extraction conditions used in the conventional method produce a thermic degradation of proteins, generating losses in quantity and quality of extracted pectin. That why new methods have been established in which pectin can be extracted in less time and with better quality and efficiency, as for example, extraction with previous microwave application.

This method consists in pre-treating vegetal residue with delimited heating through microwaves. This rises temperature and pressure in vegetal tissue; through a rise in pressure, partial disintegration of vegetal tissue is achieved, and intracellular substances are freed; moreover, through a rise of temperature, enzymes which degrade pectin are inactivated. This microwave pre-treatment increases pectin solubility and higher resistance gel is achieved. Also, this method reduces extraction time, taking from 15 to 20 min, but has the inconvenience of high operating costs. (Sánchez et al.2011; Durán & Villa, 2014).

4.1.3. Extraction with enzymes

There exist few works on this type of extraction. These enzymatic techniques for pectin extraction are based on using enzymes generated by microorganisms, which convert high methoxylated pectins in low methoxylated pectins without depolymerizing the pectin molecule. (Sánchez et al., 2011). This microbial enzymes solubilize pectin from protopectin and are called protopectinases (Zapata et al., 2009).

4.2. SELECTION AND DESCRIPTION OF THE PROCESS

The chosen extraction method, and therefore, the one this work on designing a pectin extraction and purification plant is based on, is the method of extraction on an acid medium or conventional method, since it is the method most commonly used industrially, it doesn't

represent such a high operation cost as in extraction assisted through microwaves, and the process is relatively simple. Moreover, when consulting encyclopedias (Ullmann, 2011), pectin extraction is described through this method, and is also found in the patent which describes process of pectin obtention (Nakamura, 2015), and several other related articles. So it is understood that it is the most applied method, and thus another reason why to base this work on it. Therefore, everything which is explained from now on in this work is based on the industrial process of extraction and purification of pectin through acid medium extraction.

Through the investigation carried out on the matter it has been found that the general obtention process of pectin may be divided in three blocks, shown in the following figure:

Figure 3. General diagram of process.

Below, each block will be explained, and through a study of the process, stages will be subdivided.

- **Pretreatment**

This block spans all the processes to which prime matter is usually subjected to, with the purpose of making the extraction process easier. Generally, it consists of the following sages: selection, peeling, cutting up (milling), washing (inactivation) and drying.

First of all, it is important to select residues in good shape, or in other words, throw away those residues which present damages in the peel (because of a humidity loss, meaning wrinkling, or because of microorganism attack, meaning debilitation), which have fungi or decomposing parts (Cuesta & Muñoz, 2010). Once residues which can be considered in a good state have been selected, selection of which aspect is sought in the final pectin follows, as the stage of peeling depends on this decision. If a purer pectin, meaning of a whiter colour, is sought, the mesocarp must be separated from the exocarp (see figure 7), on the other hand, if a pectin with less purity and a bit of color is acceptable, we may omit the peeling stage. Afterwards, we must carry out the cutting up or milling, which consists in setting the residue in small pieces, as this increases superficial contact area, which eases the extraction process (Devia, 2002). The next treatment is washing or inactivation of enzymes, which consists in soaking the residues in water and heating it up to 80-100ºC, for 10-15 minutes. With this

process we inactivate enzymes which may degrade the pectin, we eliminate impurities, dirt, microorganisms, and organic acids, sugars and pigments are leached. That's why it is considered as a process of separation in the purification of pectin. The last process of this block consists in drying up the residues to enhance its storage, but this process isn't always carried out, for example, in pectin production plants neighboring juice factories, where residues are used without previous drying (Ullman, 2011).

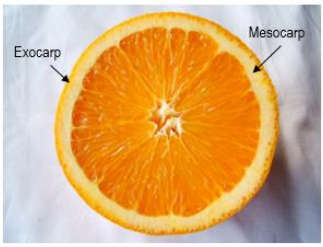


Figure 4. Parts of Orange peel.
(Image from Besana Portal Agrario)

Previous treatment stages for the plant which is going to be designed have been decided, which will be: selection, cutting and washing or enzyme inactivation. In this case it has been decided that the peeling process will be omitted to save time, and also because obtaining a less pure pectin is compensating if through this we may redirect time into other process stages to achieve higher performance. No specific data has been found on suitable cutting sizes, therefore, it has been decided that pieces will be 1 x 1 cm approximately. This size is considered fitting for dispersion and it not too big to hinder contact. It has also been decided to omit the drying stage as it is a unitary operation that, generally, requires a long operation time and does not compensate to use so much time on drying raw materials for storage if they can be used directly, as the plant will be situated next to juice industries, as it has been pointed out in point 3.2, so residue processing will be able to start immediately after juice production.

- **Extraction process**

The process of extraction is mainly based in a solid-liquid extraction between vegetal residues and a water dissolution in an acid environment, through which protopectin, which is insoluble form of pectin, is hydrolyzed to pectin, which is soluble. Most used acids are chlorhydric acid, nitric acid and sulfuric acid (Devia, 2002). On the information search about the process it has been found that typical conditions to carry out the extraction are: pH from 1 to 3,

temperature from 50 to 90ºC and operation time from 1 to 12 hours. Moreover, it has to be carried out with stirring to favour contact and dispersion, preventing solids from settling. Through this treatment we can eliminate the chemical block of galacturonic carboxyls which partially insolubilize pectins (Vian, 2006; Ullmann, 2011). Afterwards, a solid-liquid separation is done to separate the extract from exhausted residues. On the studies about the matter, it has been found that this separation is generally done through filtering, although in very few cases centrifugation or sedimentation may be used.

Filtering has been chosen the operation that will carry out this separation on the plant which is going to be designed. Since big solids are going to be used, and sedimentation does not compensate for mass production as it requires a longer time. Therefore, in this case the extraction process will span two stages: extraction in an acid medium with stirring and filtering.

- **Final conditioning**

After filtering, we may optionally concentrate the extract to prepare the solution for the next step, which is a precipitation with alcohol. Evaporation on a moderate temperature and in low pressure or void is the method used to concentrate the solution in order to avoid degrading pectin. The aim of this concentration is to reduce the quantity of alcohol needed to carry out precipitation (Devia, 2002; Ullmann, 2011). Afterwards, a fitting alcohol is added to the extract to precipitate pectin, producing formation of gelatinous coagulum, in other words, a gel is formed (Mueckay, 2006). Precipitated pectin is separated from the alcohol solution, and to cleanse impurities, it is washed with more alcohol. Next, it is pressed to drain as much liquid as possible. To purify the pectin even more, it is dried to reduce humidity content. The drying process is carried out in low temperatures to degrade pectin as less as possible, usually at around 40-50ºC with a hot gas stream, or also out in the open for several days (Devia, 2002). Afterwards, pectin is milled to reduce and homogenize particle size and enhance its appearance. Properties and quality of obtained pectin are influenced by many factors, as for characteristics of used the raw matter. Used vegetal residue characteristics depend on various factors, as for example, origin fruit, time of harvest, climate conditions or ripening in fruit production. Because of these variations, the resulting powder is mixed with other pectin lots and/or sucrose to guarantee its uniformity and provide a product with a constant degree of gelification, process known as standardization (Ullmann, 2011). Finally, pectin is packed in containers which protect it from

humidity and contamination, to avoid alterations in quality and appearance. It must be stored at room temperature and in a dry place.

Final conditioning of the designed plant will span the following steps: Precipitation, separation, washing, drying, milling, standardization and packaging. It has been decided that concentration will not be conducted, a decision that will be argued in a subsequent point (4.2.4), which will delve into precipitation and used alcohol.

With this study we have come to know about the steps that can belong to the process of pectin extraction and purification. Industrial extraction methodology is varied, as not all industries carry out the procedure following the same steps, in other words, each industry has its own methodology. But main processes, as enzyme inactivation, acid extraction and precipitation, are invariable, as they are essential to the process. By the considerations and decisions taken, the designed plant's pectin extraction process will span the steps shown in the following figure:

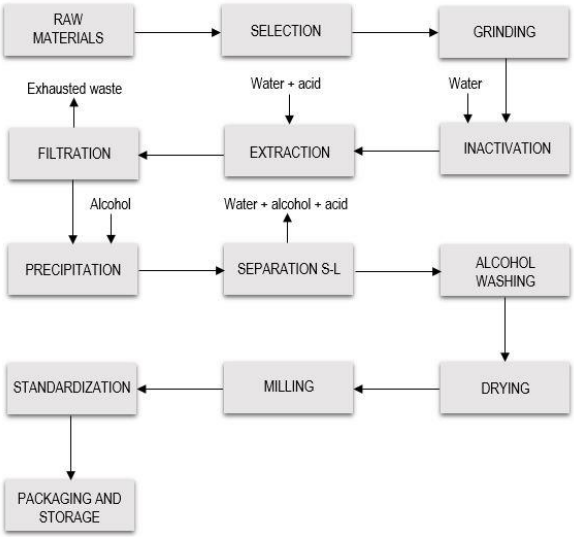


Figure 5. Block diagram of process operations.

It has been determined that the limit of battery for the design that is going to be realized will comprise the steps of: inactivation, extraction, filtering, precipitation, separation solid-liquid and drying. So these are the steps that we are going to take a closer look at, and from which the

corresponding design will be realized. Below we are going to detail and determine the characteristics of each step, deciding in which conditions are going to be carried out.

4.2.1. Inactivation of enzymes

As explained in the pre-treatment point above, the enzyme inactivation process consists of soaking the residues in water and heating up to 80-100ºC, during 10 to 15 minutes approximately with stirring. The aim of this washing is to make the extraction process more efficient, as dirt and microorganisms are eliminated. The water to be used for this wash is water at 25°C, as it is cheap and easy to obtain. In this step, temperature, time and proportion of residue-water must be determined.

10 minutes have been decided as operation time, because mass scale production makes time saving desirable, and furthermore, a 5-minute difference won't proportionally deliver increased results to such an extent as to compensate time loss.

Regarding temperature, it has been decided to use an average, i.e. 90ºC. Since operation time will be the minimum, it is desirable that operation temperature is not the minimum to compensate. Maximum temperature of 100ºC hasn't been chosen because it implies reaching boiling point, which means higher costs of time and energy.

Through information searches it has been found that residue-water proportions used are 1:4 (Guidi and Arandia, 2010) or 1:3 (Devia, 2002). As more water is used, more diluted will be dirt and impurities that can be solubilized, but as this water is contaminated with impurities, organic matter, microorganisms, organic acid, etc., it must be discarded and subsequently treated. (A topic addressed in the "4.4 Out of reach aspects of this project" section) it is desirable that the minimum possible is produced. That is why, use of a minimal proportion of residue-water has been decided, i.e., approximately 1:3. Since we are dealing with a mix of solid-liquid and solids are big, the proportion cannot be less, as stirring would be hindered and too much energy would be needed to keep solids suspended.

4.2.2. Extraction (acid hydrolysis)

The process of extraction consists in adding an aqueous acidulated solution to the vegetal waste already free of impurities, heat and keep in agitation during a determinate time. A lot of investigations show that the performance and the quality of the pectin obtained depend of the

pH, temperature, time of extraction, agitation and solid proportion-liquid (Qior et al., 2010). As it has explained previously, the acids that are used to use are hydrochloric acid, nitric acid or sulfuric acid (Devia, 2002), and the typical conditions to make the extraction are: pH of 1 to 3, temperature of 50 to 90°C and time of operation of 1 to 12 hours.

In the search for information it has been found that the proportion used for waste - water extraction is varied, have found proportions and concentrations of residue-water of 1:3, 300 g/L, 5:95, 40 g/L, etc., this difference is because it is not the same from wet residue that dry residue. That is, if part of dry residue, the amount of water needed is bigger for no finding viscosity problems later. However, if from of wet residue, as it contains a certain amount of water itself, the amount of water needed is less. Since this work has not been raised as an experimental work, does not have the ability to assess what is the right proportion. Therefore, one of those found will be selected. In this case it was decided from wet waste, because with it the quantity of water needed to add is lesser. It has been decided to use the concentration of 300g / L because it is considered to be a reasonable relationship, and is used in an article that studies this process (Devia, 2002). Furthermore, this ratio affects the amount of alcohol needed in the precipitation step, the more water added, the amount of alcohol is needed later. Therefore, it is preferable to use a lower proportion requiring the addition of less water, because later need a lower amount of alcohol.

The acid to be used in the plant to design is hydrochloric acid at 37% since it is the one who use the majority of sources consulted (Chacín, 2010; Cuthis and Muñoz, 2010; Devia, 2002; Nakamura et al., 2015; Ullmann, 2011) and indicate that is the one that better performance is obtained in the process. The suitable acid conditions favor the hydrolysis of the protopectin, the insoluble form of the pectin, by what is important to work to a suitable pH. A low pH favors the dissociation of the links that keep the pectin in the vegetal tissue, by what the performance of the extraction of pectin increases with the acidity. But in contrast, when the pH is lower, the product loses too degree of esterification. In the investigation on the subject, it has been found that the optimum pH range for extracting pectin is from 1 to 3 (Ullmann, 2011). Thus, the pH chosen for the extraction of the plant to design has to be within this range for the yield of pectin is favorable. It has been decided that the pectin to be manufactured is high methoxyl, whose degree of esterification is high, since it is the most common. Therefore, the pH that goes to use is 2 to 3, to be inside of the optimum pH range and have a good yield of pectin, and also obtain

pectins high degree of esterification. That is, the plant is adapted to obtain pectins high degree of esterification, but if the market demand increases pectins low degree of esterification, the pH of the process to obtain them would be lowered.

As it has said, the temperature also affects performance in the extraction of pectin. High temperatures enhance the performance of the pectin extraction and increasing its solubility, but cause the degradation of the molecules of the chain of pectin. In contrast, low temperatures favor the production of pectin with a relatively low degree of esterification, but tend to lengthen the time of extraction and to hinder the solubility of pectin (Masmoudi, 2008; Qiu, 2010). The optimum temperature range is 50 to 90°C (Ullmann, 2011). To determine the temperature at which extraction is made, a compromise between the aspects that detail just set. Since it has been decided that the plant to designed is to be adapted for the production of pectin, high degree of esterification, the temperature must not be low since it would encourage the production of pectin with low-esterification grade. In addition, too high a temperature cannot be used to not degrade too the pectin extracted. Accordingly, it has been decided that the operating temperature is 70°C, with which is achieving the desired pectin, within the range that provides acceptable performance without much degradation. As with the pH, if subsequently, by the needs of the market wished to obtain a pectin of low degree of esterification would modify the process going down the temperature of extraction to be able to fulfil the demand.

The extraction time can vary from 1 to 12 hours (Nakamura, 2015; Ullmann, 2011). A longer operation leads to a higher extraction yield of pectin, and in general decreases the degree of methylation, which favors obtaining pectins low degree of esterification. In addition, a longer extraction time tends to increase production costs. Conversely, short operation time tends to decrease the extraction efficiency. Due the fact we want to carry out a large-scale production it is preferred save on time everything possible, therefore, is chosen an operating time of 2 hours, which within the range that is available is small. With this operating time the extraction yield will be somewhat lower, but as pectin is a product that has no value being in the residue and acquires great value once produced, compensated, but less is produced, therefore there is no reason to speed in this regard. Moreover, not being a long time to obtain pectins of the high degree of esterification is favored. Compensates choose this time operation because, although somewhat less for performance, it is saved in time. In the same way that the temperature and

pH, if it were desired at a later time to obtain pectins low degree of esterification, would increase the operating time to facilitate removal.

4.2.3. Filtration

After extraction, the extract containing the dissolved pectin be separated from the solid residue. This separation is not easy because the solids phase is plant residues soft and exhausted, and the liquid phase is a viscous liquid which containing the dissolved pectin, which is a thickener. So that the greater the amount of dissolved pectin, higher viscosity. The process used by most of the sources of information for this solid-liquid separation is filtration. Since there were no reasons for changing the method they use to sources, it has been decided to be used filtration. This filtration requires low viscosity, and therefore, the pectin concentration should be between 0.6 to 1 %. Separate plant residues are commonly used as cattle feed.

4.2.4. Precipitation

As explained above, precipitation comprises adding an appropriate alcohol, in which pectin is insoluble to precipitate the pectin. Also, it could precipitate using salts, but it is preferred to use alcohols as pectins are mostly used in the food industry and it has to avoid waste, while salts a careful washing is needed to remove waste. It has been decided that the alcohol used is ethanol, for lower cost compared to other and ease of purchase on the market. In addition, two sources that are considered very reliable, such as encyclopedias (Ullmann, 2011) and patent (Nakamura, 2015), this alcohol used to carry out the precipitation, another reason that justifies its use. The amount of alcohol to be added must be a volume equivalent to 60 or 90 % of the solution to be precipitated (Cerón and Cardona, 2011; Devia, 2002; Nakamura, 2015). Since it goes to produce a large scale, it involves a lot of ethanol, by what has decided to recover the alcohol in the same plant for reuse and reduce costs. Since the alcohol is going to be recuperated, it has been decided that the amount to be used is a volume equivalent to 60 % of the solution, i.e. the solution to precipitate alcohol must be at a concentration 60 % to reduce costs. The purity of the ethanol used is normally 96%, but as will recover in situ, purity to be obtained is not so high, therefore be used ethanol of 94%.

The concentration process prior to precipitation, explained above, which consisting of evaporating water from the solution containing pectin to use less amount of alcohol in the precipitation, it was decided to omit. By not evaporate water must be used more alcohol, but as

it has decided to recover it will have to evaporate the alcohol. The reason for omitting the process prior concentration is that is preferred to evaporate alcohol, since it is easier to evaporate than the water, because alcohol has a latent heat of evaporation and a boiling point lower.

Substance	Latent heat of vaporization, λ [kJ/kg]	Boiling point, Tb [°C]
Water	2500	100
Ethanol	841	78

Table 4. Latent heat of vaporization and boiling point of water and ethanol.

The precipitation process has to be performed at a low temperature to favor the pectin precipitate and disfavoring the solubility. When the system by which this stage will be cooled is explained, in subsequent sections, will be studied until what temperature it is convenient to cool.

With this precipitation process is achieved precipitate pectin, which precipitates as threads, fibers, creating a gelatinous mass (Mueckay, 2006).

4.2.5. Solid-liquid separation

After precipitation, it has to remove the precipitated pectin from the liquid solution. Because the pectin content in the wastes is low, the amount of solids in this separation will be much less than in prior separation to precipitation. Therefore, in this case it is decided to opt for a centrifuge. In addition, after performing the separation, the separated solid has to be washed with ethanol, so when choosing the equipment with which the separation is carried out, it must take into account if you can make him wash. The separated liquid not be discarded because it contains ethanol and has to be recovered.

4.2.6. Drying

To remove moisture of pectin obtained was subjected to a process of drying with a gas stream. Since evaporation was a mixture of alcohol - water, with a large proportion of alcohol, it has been decided that the gas used for drying is nitrogen, for safety, because alcohol is combustible. In this drying process it has to be careful of the temperature at which it is performed as it could degrade pectin and darken too. The temperature used for drying must be between 40 to 60°C (Chasquibol, 2008; Cuesta, 2010; Devia, 2002). For the plant to be designing it has been decided that the drying temperature will be 45°C, for not degrade much

pectin and not too long drying time. The final moisture has been decided to pectin is 8 %, because in literature sources has been found that final moisture is adequate.

It has to take into account that the liquid containing pectin before drying is not just water, is practically water and ethanol, since hydrochloric acid lot has been removed by washing the solid cake with ethanol.

4.3. SELECTION OF EQUIPMENT

Once determined operating conditions of each design stage has to make a selection of teams to be held each.

4.3.1. Equipment for inactivation of enzymes

This stage consists basically to put in contact solid waste with water. Wants to keep the solids in suspension in the liquid for what is necessary some type of stirring.

The most recommended unit by bibliographic sources to carry out a suspension of solids is a stirred tank (Sinnott, 2005; Treybal, 1994). The stirred tank can be opened or enclosed (without exchange of matter with the outside). Choose one or another depends of the fluid that handled and of the operation performed. In this case, to make the inactivation it goes to use a tank agitated open, since there are not products that evaporate that they can interest us or that they are harmful. Besides, a opened stirred tank facilitates us the load of the solids. For the geometry of the tank has decided that the bottom goes to be ellipsoidal, with the end to favor the flow, avoiding zones in which they do not penetrate the currents of flow that can do that they remain deposited the solids.

There are different types of agitators and each is suitable for operation characteristics. In this case, in which working with solids that must be kept in suspension, the most suitable type of agitator is a turbine, flat sheet, with or without disk, Rushton turbine 6-blade in particular, because they are used for operations mass transfer and solids suspension (Sinnott, 2005; Treybal, 1994).

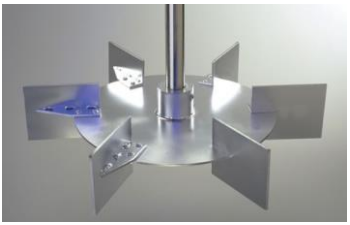


Figure 6. Example of flat-sheet impeller
(Image from Direct Industry web)

Since the process of inactivation of enzymes must be done hot, the tank must have a heating system. The heating system to be used is an outer jacket, since it is the most widely used in the chemical industry and use the tank is easier to clean (Cunill, 2010). The heater fluid that will pass on the jacket will be steaming hot water because it is relatively easy to obtain and has a high heating value. Because be heated to 90°C and a solution of waste-water, which is initially about 25°C, be used steam at 110°C. No steam is used at a higher temperature because, having a bulky system with suspended solids, will not have good transfer coefficient, and a too high temperature in the walls that can deteriorate the mixture would be obtained. That is, the higher was the steam temperature heating, less time required to heat, but for safety of product should use steam at 110°C.

4.3.2. Equipment for extraction

This stage has the same mechanism as the previous stage, put in contact a solid with a liquid. In this case, is contacted waste with acidified water to extract the pectin and solubilized in the remaining liquid. Since the operating characteristics are very similar to the inactivation step (constant stirring is required to prevent solids sink to the bottom of the tank, is to be heated and maintained in suspension), it has been decided to use the same jacketed tank open from the previous step to perform the hydrolysis. Using the same tank is accomplished save costs and facilitate the production process. To perform the two operations in the same tank you will have to add a discharge outlet at the bottom to remove the dirty water of the inactivation process

In this case, it has to heat to 70°C, using the same steam that in the previous stage, with the difference that at this stage the time required to reach operating temperature will be lower.

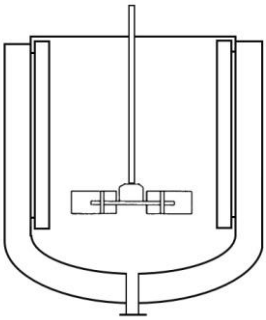


Figure 7. Illustration of opened jacketed stirred tank for inactivattion and extraction

4.3.3. Equipment for filtration

After extraction of pectin has to be done a solid-liquid separation, and, as explained in previous sections, this separation is to be performed by filtration. It is important that the concentration of pectin in the suspension to be filtered be between 0.6-1%. Due to the fact that, as a thickener, if it be in higher concentrations cause problems when filtering by its high viscosity (Ullmann, 2011). In this section it has to select the equipment of filtered more adapted to carry out the separation.

It is a suspension with high solids, at a temperature of 70°C, of which the phase that we are interested in is the liquid phase containing the dissolved pectin. The process is to be performed discontinuously, so that a filter that can operate in batch is required. Filtration is essentially a batch process because the equipments have to stop to download the cake and clean. Most batch filters operating under pressure instead of vacuum (Perry, 1999).

For the selection of filtration equipment must take into account various factors, such as (Sinnott, 2005):

- The nature of the slurry and the cake formed.
- The solids concentration in the feed.
- Throughput required.
- The nature and physical properties of the liquid (viscosity, toxicity, flammability, corrosiveness).
- The cake dryness required.
- Whether the valuable product is the solid or the liquid, or both.

The most important factor is the filtration characteristics of the cake, i.e., if it is a quick filtering (cake has a low specific resistance) or a slow filtering (cake with high specific resistance). In this case it is a fast filtering and the solids are relatively large pieces of waste that do not have a very high resistance. In addition, since the phase of interest is the liquid that contains the product to be produced, the pectin, it is important to get the solid separate as dry as possible.

Given the characteristics of the suspension to be treated and comparing between different filters that would fulfil the requirements, is selected as equipment for this separation a filter press. It has taken this decision because, with this filter, operating properly, a drier and dense compared with most other filter cake is obtained. In addition, the filter is indicated by a reference article for performed this stage (Devia, 2002). The advantages of this filter are (Coulson et al., 1981):

- Simplicity.
- Low maintenance cost.
- Large filter area in a small space.
- Most seals are external, so leaks are easily detected.
- Are achieved high pressures with ease.
- It is suitable whether the product you want is the liquid or cake.

This filter consists of a series of plates and frames arranged and supported by rails. The hollow frame is separated of the plate with the filter cloth. The suspension is introduced through an orifice having the frames and the filtrate passes through the cloth, forming the cake in each chamber (Coulson et al., 1981).

Figure 8. Example of filtre press.
(Image from Direct Industry web)

4.3.4. Equipment for precipitation

At this stage wants to contact the liquid that comes from filtration, which contains the pectin dissolved, with ethanol. As it explained, with the addition of alcohol is achieved precipitate pectin, which precipitates as fibers and creates a gelatinous mass by its viscosity. In this operation will be in contact liquid and solid in suspension. As in the inactivation step and extraction, the equipment most recommended in the literature to perform this operation is a stirred tank (Devia, 2002; Sinnott, 2005; Treybal, 1994). The solids concentration at this stage will be lower than in the extraction step. The type of agitator used is the same as for the hydrolysis tank, Rushton turbine 6 blades. Ethanol is a flammable alcohol tends to evaporate, therefore it must work with caution. Alcohol should be in a concentration of 60% in the solution to precipitate so large quantities are needed. For these reasons, it is to use a stirred tank, but at this stage, will be closed to prevent evaporation of ethanol.

To assist the precipitation of the pectin low temperatures are needed, therefore the stirred tank will be provided with a jacketed whereby cooling is done. The liquid stream that arrives to the tank, from the filtrate, have a temperature of about 60°C. It is not convenient to add ethanol to a solution which is 60°C, so, for safety, be cooled the current of the filter about 40°C, and once that this is at 40°C is to be add ethanol, which is at 25°C, and continue cooling to a certain temperature. The cooling fluid is to be used for cooling will be water at 25°C, and the solution was cooled to about 30°C. It has been decided to cool with water at 25°C as easily obtainable, however, use water at 10-15°C requires having a refrigeration compressor, i.e. great cost. Therefore, it is cooled to 30°C, because down to about 20°C the cost would skyrocket. The time it will take to be higher using cool water at 25°C, but this is offset by the cost savings involved. The following figure shows an outline of substances currents and temperatures.

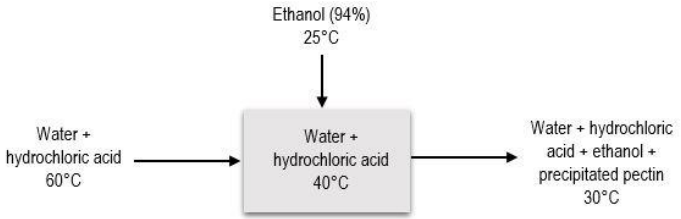


Figure 9. Scheme of substances and temperatures in the precipitation step.

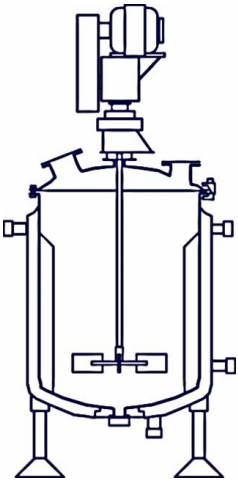


Figure 10. Illustration of closed jacketed stirred tank for precipitation.
(Image from ingenieriaquimica.org)

4.3.5. Equipment for solid-liquid separation

Once precipitated the pectin has to separate from the mother liquor, for this operation solid-liquid separation is necessary. The amount of suspended solids will be rather less than the amount of solids had after extraction. Furthermore, in the extraction, the solids were pieces of waste which were solid relatively large, however, after precipitation the solid is pectin, which precipitates as fibers, gelatinous clots, with a creamy coloration (Mueckay, 2006), which are solid smaller. It has been decided that, since the concentration of solids will be relatively small, the separation will be accomplished by centrifugation.

Centrifuge types are classified according to the mechanism used for solids separation (Sinnott, 2005):

- Sedimentation centrifuges: in this kind of centrifugal the separation occurs by difference in density between the liquid and solid phases, the solid being the heavy phase.
- Filtration centrifuges: which separate the phases by filtration. The walls of the centrifuge are porous baskets in which the solid accumulates, and the liquid is filtered through the deposited cake of solids and is removed.

The type of centrifuge selected depends on the nature of the feed and product requirements. As a general rule, sedimentation centrifuges are used when the phase of interest is the clarified liquid, and filtration centrifuges are used to produce a dry solid (Sinnott, 2005). Because in this case the phase of interest is the solid, and it is not a heavy solid, it is to use a filtration centrifuge. Although the phase of interest is the solid, the liquid phase will not be neglected because it contains a large amount of ethanol and for reduce costs, it was decided to recovery this alcohol. Therefore, the liquid removed by centrifugation, will be directed to the appropriate unit for recovering ethanol (subject to be discussed in the section 4.4). Filtration centrifuges are divided into two types depending on how the solids are removed: fixed bed (the solids cake remaining on the walls of the centrifuge until they are automatically or manually removed) or moving bed (cake solids are moved along of the cuvette by the action of a displacement). The filtration centrifuge chosed for this process is of fixed bed, because after making the separation has to do the washing of solids cake, and is has decided that washing is to be performed in the centrifuge. For that reason, it is necessary that solids remain in the centrifuge to add the washing alcohol solution and centrifuging again. This washing is carried out with a solution of water-ethanol, 80% ethanol, and aims to remove the hydrochloric acid may be impregnated in the precipitated pectin.

For the above reasons, and taking into account the classification found in the literature (Sinnott, 2005, page 421, fig. 10.20), it is selected basket centrifuge fixed bed for the separation of this stage.

Figure 11. Example of centrifuge Basket..
(Image from Direct Industry Web)

In addition, knowing that the particle diameter of pectin is approximately 250μm, with the following table, elaborated from data found in the literature (Sinnott, 2005, page 416, fig. 10.16), it can be seen that the particle size of the solid to be treated is within the permissible for the different types of centrifuge basket, so that is a suitable kind of centrifuge to perform this separation.

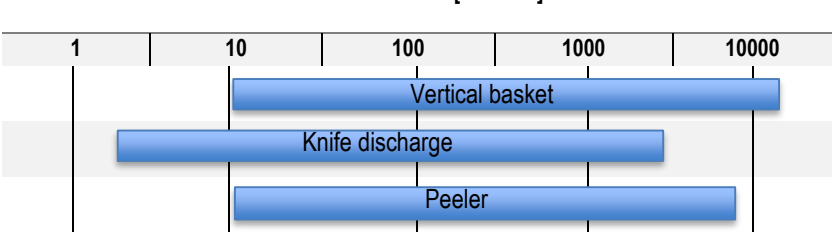


Table 5. Particle size of solid for diferent types of basket centrifuges.

4.3.6. Equipment for drying

As explained in previous sections, drying is to be carried out with a stream of nitrogen gas at a temperature of 45°C, since otherwise could degrade the pectin. The equipment selected for this process should operate batch, since the plant is being designed is required to operate discontinuously. The choice of drying has to be noted that the solid to be dried is pasty, by high viscosity of the pectin.

The type of heating chosen to carry out the process is direct, i.e., the heat transfer is achieved by direct contact between the hot gas and the moist solid. This type of heating has advantages that make it suitable for the process, as it is possible to precisely control the gas temperature, and also is relatively easy to ensure that the solid material is not heated above a set temperature.

Given the solid to be treated, it has selected a tray dryer to perform the operation, since this type of dryer is suitable for pasty or solid materials to be fastened on plates. In addition, various information sources that talk about the production process of pectin (Devia, 2002; Guidi, 2010) use this type of dryer, and has not found reasons to be inadequate. This type of dryer is a rectangular chamber containing a sheet metal racks, which contain trays or dishes mobiles. These trays, in which load the solid to be dried, usually are square and shallow. Between trays air is circulated tangentially. Once the desired degree of dryness is reached, the dryer is opened

and the plates are replaced with a new batch. To save time between drying cycles can be changed simply by placing the dryer trays on carts, which can be removed and put into the camera with ease and can be charged and discharged out of the dryer.

Figure 12. Example of tray dryer.
(Image from slides of biotechnology subject)

4.4. ASPECTS OUTSIDE THE SCOPE OF THIS PROJECT

There are several issues on which no delves into this work since they have been outside the scope of the project. But still it is important to explain because they are important issues that cannot be overlooked.

- **Water of inactivation of enzymes**

The water used to clean impurities from waste goes to decant, but this water is water polluted because it will have lots of microorganisms, organic acids, sugars, and especially organic matter: COD (chemical oxygen demand) and BOD (biological oxygen demand). The treatment of this polluted water is beyond the limit of this project, but it would be necessary to apply the wastewater treatment consisting of a pre-treatment, primary treatment, secondary and tertiary treatment. This process is done in wastewater treatment plants (WWTP or EDAR).

- **Current of waste separated in the filter press**

This exhausted waste stream that has been extracted pectin and they have been separated by the filter press is often used as cattle feed (Ullmann, 2011).

- **Inerting of precipitation tank**

In the precipitation tank in which ethanol is added, it is anticipated inerting between loads to avoid problems with alcohol as it is a highly volatile and flammable substance. Inerting is, once

the tank is filled with liquid precipitation coming from the filter press, passing high purity nitrogen, and then adding ethanol. Of this way, substitutes the humidity of the empty space of the tank by inert nitrogen and entirely dry. This does that there is a protective layer of nitrogen over the ethanol. Through a control system tank valve it would ensure that when the tank is full or empty, the nitrogen content is compensated and the protective layer is maintained.

- **Alcohol recovery**

By a matter of time, designing equipment for the recovery of ethanol it has been left outside the scope of this project, but the recovery process ethanol is indispensable for the functioning of the plant due to the large amount required and the high cost implying not reuse.

The process used to recover alcohol is a simple distillation, with which can be obtained high purity ethanol. With distillation column a mixture of about azeotropic point (96%) is achieved (Pacheco, 2012). As it goes to recover the alcohol on the same plant, it is not logical to think that a recovery of 100% will be achieved, so ethanol is derived with a slightly lower purity. It has considered that the purity that can be obtained is 94%, therefore, to make the productive process of pectin is used ethanol to 94%.

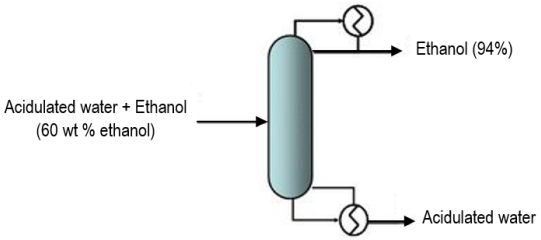


Figure 13. Distillation sequence of ethanol recovery.

- **Waste water in distillation of ethanol**

The acidulated water remaining after distillation will contain all soluble polysaccharides that have not precipitated with alcohol, lots of organic matter, COD and BOD. Therefore, this contaminated water must be treated as contaminated water from enzyme inactivation in wastewater treatment plants.

- **Treatment of the gas stream dryer outlet**

Pectin treated in the dryer contains water and ethanol, so what goes to evaporate is not only water, but also nitrogen drag ethanol. Therefore, when performing evaporation, the gas cannot be released to the atmosphere with alcohol. To solve this problem, there will be a system of gas

purification by adsorption would allow recovery of ethanol, but this system is beyond the scope of the project.

5. DESIGN OF EQUIPMENT

5.1. OPENED STIRRED AND JACKETED TANK

Firstly, to make the design of the units, the load of waste to treat must be determinate. This load depends on the capacity of the tank of extraction, which comes limited by the agitation, since it is necessary to keep the solids in suspension. Solids to treat are relatively large. If the amount of solids is too big, they can cause problems in stirring, warming or cooling of the suspension. Therefore, to avoid these problems, and following the article *Process to produce Citrus Pectins* (Devia, 2002), the load established is 300kg of humid solid waste. As it has indicated in the selection of the process, the concentration to be used in the extraction is 300g/L. Accordingly, 300 kg of solid waste require 1000L of water. This load is considered adequate to allow the agitation of solids and keep them in suspension. Considering an average of the fruits' content of pectin (20% wt. of pectin in base on dry matter. See table 1), from 300kg of solid waste should be obtained 12kg of pectin. For a greater production would have to put units in parallel.

In the opened stirred tank are made two operations: the inactivation and the extraction. The design of the tank depends on the operation that require greater volume, because it guarantees that the tank have sufficient space for the task of lower volume. By the conditions established in the sections 4.2.1 and 4.2.2, the volume of suspension of each operation, known like volume of reaction, is determinate. The sizing from the unit is made considering the volume of extraction suspension.

	Inactivation	**Extraction**
Vreaction [L]	1283	1291

Table 6. Volume of each suspensions.

The volume of reaction has to be 80% of the volume of the reactor, by heuristic. By means of the following relations, recommended by the literature for solid extraction-liquid with solids in suspension (Treybal, 1994), determine the dimensions of the tank: H height, T diameter. Besides, they determine the width of the baffles (b), and the distance of these to the wall of the tank (p).

- H/T =1,2 (Higher than wider tank to promote mass transfer)
- Width of baffle, b: T/12 (Prevent formation of vortexes)
- Distance baffle-wall tank, p: b/2 Avoids stagnation of liquids and solids.

Impeller parameters are also determined, by means of the following relations, recommended for impellers for transfer of mass and suspension of solids (Treybal, 1994).

- Diameter of the impeller, d_i: di=T/2
- Width of the impeller, w: w=T/5
- Blade length, L: L=T/4
- Distance impeller-bottom tank, C: C=T/3

PARAMETER	VALUE
$V_{REACTOR}$ [m^3]	1.62
T [m]	1.20
H [m]	1.44
b [m]	0.10
p [m]	0.05
d_i [m]	0.60
w [m]	0.24
L [m]	0.30
C [m]	0.40

Table 7. Opened stirred tank dimensions.

The impeller power is determinate by the following expression (Treybal, 1994).

$$N_p = \frac{P}{\rho_L \cdot N^3 \cdot d_i^5}$$

The parameter N is determinate by:

$$Re = \frac{{d_i}^2 \cdot N \cdot \rho_L}{\mu_L}$$

For the suspension of solids, the flow has to be turbulent by what the Reynolds has to be high (Re ≥ 10000). By a graph (Perry, 1999, page 665) that relation Re with power number, Np, depending on the type of impeller used (in this case Rushton turbine), the necessary parameters are calculated and the results are:

Parameter	Result
N [rpm]	50.5
P [W]	454

Table 8. Power and sped of the impeller.

- **Jacketed**

To determine the time required for heating the suspension has been to solve the following energy balance in nonstationary state.

$$m \cdot \hat{C}_p \cdot \frac{dT}{dt} = U \cdot A \cdot (T_v - T)$$

Rearranging and integrating:

$$t = \frac{ln\frac{T_v - T}{T_v - T_0}}{\frac{U \cdot A}{m \cdot \hat{C}_p}}$$

Temperatures T, T_0 and T_v are indicated in the section 4.3.1 and 4.3.2. The value of overall heat transfer coefficient, U, is selected with of the following table that has elaborated from data obtained in the bibliography (Couper et al., 2012).

Equipment	**Process**	**U [**Btu/(hr)(sqft)(°F)**]**
Stirred tank, jacketed	Liquid-condensing steam	90-260
	Boiling liquid-condensing steam	120-300
	Water-liquid	25-6

Table 9. Range of Overall Heat Transfer Coefficient, U. (1 Btu/(hr)(sqft)(°F) = 5.6745 W/ (m2·°C).

The value of U chosen is of the small range as it is a bulky solids suspension and is not expected a good transfer. The parameters used for each stage are:

Parameter	**Inactivation**	**Extraction**
m [kg]	1300	1310
A [m^2]	6.52	6.52
T_v [°C]	110	110
T_0 [°C]	25	38
T [°C]	90	70
C_p [J/(kg·°C)]	4180	4180
U [W/(m^2·°C)]	500	500

Table 10. Parameters for calculation of heating time.

	Inactivation	**Extraction**
Heating time [min]	40	17
Operation time [min]	10	120
Total time [min]	50	137

Table 11. Heating time and total operation time results

5.2. FILTER PRESS

To make the design of the filter press is important to know the volume to be filtered and the amount of solids containing, as they are parameters dependent filtering. There are two ways to

perform batch filtration: maintaining constant pressure and decreasing the flow, or maintaining constant flow and gradually increasing the pressure. In this case, in which a load is filtered, the rate is to be fixed and pressure progressively increase given the accumulation of solids. The equation used for filtration is as follows (Couper, 2012):

$$Q = \frac{A\Delta P}{\mu(R_f + \alpha c \frac{V}{A})}$$

The filtrate volume and solids concentration are known parameters, since the suspension from Tank hydrolysis is filtered. In the suspension to be filtered are solubilized waste part and pectin, so that higher viscosity than water is assumed. As indicated in paragraph 4.3.3, the percentage of pectin for filtering can be performed properly must be between 0.6-1%. In this case, the percentage of pectin in the slurry is 0.90%, which is within the range set.

A heuristic indicate that the production capacity of a filter press is between 1.5 to 10 kg of solids per m^2 of surface filtration, it has chosen a filtration capacity of 4 kg/m^2, taking into account the solid filter. With the filtering capacity and the amount of solids to filter, total filtrate area is determined.

The solid cake formed by filtering accumulates and exerts resistance. The specific cake resistance, α, is an experimental parameter so it would be appropriate to perform an experiment to determine. In this case, in the absence of experimentation, an assumption is made considering the values that can be α, which are found in the literature (Couper, 2012). Because the solids are pieces relatively large that do not have a high resistance, a low range α is chosen.

The cake is divided between the number of available plates and accumulate the solids, so, the pressure increasing with time. It is important that this pressure is within appropriate values, and not excessive. In the literature it has been found that the filters operate at a pressure of between 3 and 10 atm (300kPa to 1000kPa) (McCabe, 1991; Perry, 1999). Assuming a time for filtration, a rate flow that may be considered appropriate is estimated. With this rate and all other data, the pressure is determined and checked whether it is within the limits.

Parameter	Value
Filtration volume [L]	1291
Volume of solids [L]	277
Q [L/h]	2582
A [m^2]	72
ΔP [kPa]	500

Table 12. Parameters for filtration

Consults in the bibliography, is found that filters have plates, usually square, from 0.15 by 0.15 m (6 by 6 in) to 2 by 2 m (78 by 78 in). Plate thickness ranges from 0,6 to 5 cm (0.25 to 2 in) (McCabe, 1991; Perry, 1999). Relating the parameters obtained with these values decides that the plates of the filter go to be about 1 by 1 m. Considering the area of filtered that it requires has decided that they go to use 70 plates.

Consulting different providers, and comparing with the requirements for the filter, has chosen a filter press.

Technical data	Value of provider (VLS Technologies)
Plate size [m]	1x1
Average cake thickness [mm]	32
Number of plates	50-70
Total cake volume [L]	764.5-1233
Total filtering surface area [m^2]	53.5-87
Aprox. Dimensions (LxWxH) [m]	7x1.4x2.0

Table 13. Filter press dimensions.

5.3. CLOSED STIRRED AND JACKETED TANK

The procedure for the sizing of this unit is the same that in the open tank

Given the proportions indicated in paragraph 4.3.4, the reaction volume is calculated, that is 2820L. The tank dimensions are determined using the same relationships and considerations in section 5.1, as it is a suspension of similar characteristics. In this case, the solid is precipitated pectin, and the solid concentration is less than in the case of extraction.

Parameter	Result
N [rpm]	77
P [W]	1037

Table 14. Power and speed of the impeller

PARAMETER	VALUE
$V_{REACTOR}$ [m3]	3.53
T [m]	1.55
H [m]	1.86
b [m]	0.13
p [m]	0.07
d_i [m]	0.52
w [m]	0.31
L [m]	0.39
C [m]	0.52

Table 15. Closed stirred tank dimensions

- **Jacketed**

The process to design jacketed is the same that for the section 5.1. In this case, the fluid is refrigerator (water to 25°C), and his temperature is Tr. In the section 4.3.4 it has indicated that, firstly, the solution is cooled until 40°C. Afterwards, it adds the ethanol and cools until 30°C. Therefore, get two times of refrigeration.

Parameter	Value (before ethanol)	Value (after ethanol)
m [kg]	1022	2462
A [m^2]	11	11
T_r [°C]	25	25
T_0 [°C]	60	33.2
T [°C]	40	30
C_p [J/(kg·°C)]	4180	3146
U [W/(m^2·°C)]	1000	1000

Table 16. Parameters of precipitation jacketed.

Parameter	Value
Cooling time [min] (Before addition of ethanol)	5
Cooling time [min] (After addition of ethanol)	5.35
Operation time [min]	60
Total time [min]	70.35

Table 17. Cooling time and total operation time results.

5.4. CENTRIFUGE

The centrifuge is to be selected based on the requirements. It will select the most appropriate, taking into account the volume of suspension to centrifuge, the amount of solid and volume. In addition, the centrifugal chosen should allow washing solid separated. Knowing the densities of each substance and the amounts of each one in the suspension, the parameters necessary for the selection of the centrifuge are determined.

Parameter	Value
Total volume of suspension [L]	2820
Total weight of suspension [kg]	2462
Volume of solids [L/h]	16.20

Table 18. Parameters for the selection of the centrifuge.

For these characteristics, the most suitable centrifuge has been found is a Peeler type. This type of centrifuge has several profits in front of other types, like flexibility, low humidity in the product and good washing results. The selected centrifuge provides a single sealed environment.

Technical specifications	
Cake volume [L]	15-1920
Maximum load [kg]	19-2400
Speed [rpm]	2700-950
Centrifugal force [G]	2037-1000
Weight (without motor) [kg]	650-33000

Table 19. Specifications of centrifuge.

Since the centrifuge is too small for the volume of liquid it has been decided that centrifugation is performed in two loads.

5.4. TRAY DRYER

For the design of the dryer, the basic parameter is the time that requires to dry the humid pectin until the humidity wished. The sizing of the dryer of tray bases in determining the time of drying, the area and dimensions of the trays and the number of necessary trays.

Firstly, the initial and final humidity of the pectin is defined. The content of humidity can express in humid base (kg humidity/humid solid kg) or in dry base (kg humidity/dry solid kg). To make the calculations uses the humidity in dry base.

Parameter	Initial	Final
Humidity [%]	35	8
Humidity (dry basis), X [kg/kg]	0.54	0.087

Table 20. Humidities of solid

To determine the drying time is necessary to know the rate of drying, n_c. The drying process have two periods: period of constant rate (in which free moisture is evaporating) and decreasing rate period (in which the free moisture content is lower and the velocity decreases). Then they have to determine the time required for each period. The model determines each of the periods shown below (Treybal, 1994).

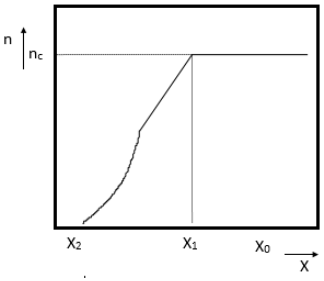


Figure 14. Typical curve of drying velocity

$$n_c = \frac{dX}{dt} = \frac{Ah(T_g - T_b)}{\lambda}$$

- Constant rate drying

$$\theta = \frac{m_S}{A}\int_{X_1}^{X_0}\frac{dX}{n_c} = \frac{m_S(X_0 - X_1)}{An_c}$$

- Decreasing rate drying

The decreasing rate period is difficult to determine, but in order to estimate, it is considered that the velocity decreases linearly from X_1 to X_0. In this particular case we can estimate the drying speed on this stretch linking it with the slope: n = mX + b.

$$\theta = \frac{m_S}{A}\int_{X_2}^{X_1}\frac{dX}{mX + b} = \frac{m_S}{mA}ln\frac{mX_1 + b}{mX_2 + b}$$

$$\theta = \frac{m_S(X_1 - X_2)}{A(n_1 - n_2)}ln\frac{n_1}{n_2} = \frac{m_S(X_1 - X_2)}{An_{ml}}$$

The drying area depends on the size and number of trays. The literature provides a heuristic that indicate that the trays are usually square and 0.5 by 0.5m to 1 by 1m and depth, 2 to 4 cm (McCabe, 1991; Perry, 1999). Given these characteristics and quantity of solid obtained in the precipitation, the dryer would be required for a load would be too small. Therefore, it was decided that several loads are stored and dried together. Based on this, a suitable size and number of trays for a number of loads to be stored is determined reasonable. Also important is the distance between trays, as determined, along with the width of the tray, the air passage section. In the literature a separation between trays of not more than 4 cm (Perry, 1999) recommend. The area outputting these trays influences the drying rate time and therefore also has to take into account that provide time reasonable drying. The characteristics of the dryer trays are shown in the following table:

Parameter	Value
Width tray [m]	0.5
Tray area, A [m^2]	0.25
Number of trays [-]	10
Depth tray [cm]	3
Distance between trays [cm]	4
Total capacity of trays [kg]	79
Number of charges [-]	5

Table 21. Parameters for dryer.

To calculate rate of drying, gas temperature (45°C), humidity (50%) and the wet bulb temperature, which is determined by the psychometric diagram (34°C) are needed. In the tray dryer, gas is recirculated to save energy and purged to maintain moisture.

The value of X_1, humidity to which the drying rate is constant, is unknown and experimentation is required to determine it. Therefore, a value that is considered appropriate is estimated. It has been considered constant speed until $X_1 = 0.3$.

In the drying it is evaporating a solution of ethanol and water, but it is considered that ethanol will evaporate faster than water because, among other things, the latent heat of alcohol is lower. Then, it is considered that at first mostly evaporates the alcohol. Since making the estimate for sizing drying for alcohol-water mixture is more complex, it is based on the water. By humidities and the amount of pectin to produce, the amount of water-alcohol mixture is determined in two sections removing drying. The results are shown below.

Parameter	Initial (X_0)	Intermediate (X_1)	Final (X_2)
Humidity [%]	35	23	8
Humidity (dry basis), X [kg/kg]	0.54	0.30	0.09
Mass of pectin [kg]	17	14.4	12

Table 22. Humidities and mass of pectin.

Parameter	Value
Humidity to remove (X_0 to X_1) [kg] (constant drying)	2.6
Humidity to remove (X_0 to X_1) [kg] (decreasing drying)	2.4
Total humidity to remove	5

Table 23. Humidities to remove.

The drying time is calculated with all the considerations and the values shown. The results obtained are:

Parameter	Result
Constant drying time [h]	2.20
Decreasing drying time [h]	3.40
Total drying time [h]	5.60

Table 24. Results of drying time.

6. OPERATION OF PLANT

6.1. PRODUCTION

Through the studying and designed carried out, it has been determined that from a load of 300 kg of residue are obtained 12 kg of pectin. A complete production cycle corresponds to about 11 hours. For drying is going to accumulate 5 charges, so that drying not be performed in each cycle. Then, the cycle time regardless drying is reduced to 5.30 hours. Considering working time of a worker 8 hours, the plant will work in two shifts, i.e., 16 hours a day. Since the operating cycle is 5.30 hours, it can be made 3 cycles per day. Then, drying is performed on alternate days: the first day, the 3 loads of wet pectin produced in the 3-daily operating cycles are stored; the second two loads of wet pectin is stored and proceed to drying of the 5 charges together (the two produced the same day and 3 of the previous day). Consequently, every other day will produce 60 kg of pectin. The plant will be operational 200 days a year (given that no work: Saturdays and Sundays, holidays, vacations, and possible periods in which they cannot produce for lack of raw material). Consequently, pectin production plant will be 6000 kg per year, or what is the same 6 t/year.

Figure 15. Pectin

6.2. OPERATION

This section make reference to equipment by the code indicated of each one on the PFD that has been developed, which is in Annex I.

Initially, 300 kg of vegetable wastes are charged into the stirred tank (E-4), then add 1000L of water in the same tank, and stirring and heating to a temperature of 90 ° C is started. The suspension is kept at this temperature 10 minutes, with stirring, and then the dirty water is discharged. After, is added to the same tank (E-4) 1000L of water and 8L of HCl, the suspension is heated to 70 ° C and is kept stirred 2 hours. At this stage the pectin is

dissolved in the liquid. The suspension is conveyed to the filter press (E-6), separating the exhausted waste of liquid. Filtering finished, the liquid extract is sent to closed stirred tank (E-8). It proceeds to cool the solution to a temperature of 40°C, and once this temperature is reached is added to 1800L ethanol at 94%. the tank solution is cooled to 30°C and maintained with stirring 1 hour. At this stage is has been precipitated pectin. Then the suspension to the centrifuge (E-9) is carried and the precipitated pectin is separated from the liquid. The liquid containing ethanol is sent to the recovery process (E-14). A solution of ethanol-water (80% of ethanol) is added on the centrifuge to wash the pectin and centrifuged again. After the centrifugation, the wet pectin was removed from the centrifuge and stored for later drying. When 5 charges have accumulated wet pectin, proceed to place them in trays of tray dryer (E-11) to reduce the moisture content to 8%. The drying process requires a time of 5.6 hours. After the drying, dry pectin is passed by a ball mill to reduce and standardize their size, and improve their appearance. Finally, we proceed to packaging, storage and distribution.

Given the characteristics of the plant, it is not very automated so that has not been expressed the control.

7. CONCLUSIONS

Several conclusions have been obtained from the development of this project.

Through the development of this project, the plant for pectin production in batch has been designed. An analysis on which applicable plant residues has been made, which concludes that the most suitable residues for the designed plant are apple, peach, orange and lemon waste. The designed plant allows 6 t/year of pectin production.

Analysing the pectin extraction process, it has been concluded that the most important stages of the process are: selection and washing plant residues; solid-liquid extraction, in acid medium, to pectin solubilisation; filtration of solid waste; pectin precipitation with ethanol; solid-liquid separation to separate the precipitated pectin; drying and milling. For this plant, it has been determined that the extraction conditions of pectin should be: 70°C, an operating time of 2 hours and pH of 2 to 3.

Moreover, thanks to the study carried out, it has been considered that the most suitable equipment of pectin extraction for the plant, and therefore those which have been dimensioned, are: opened stirred and jacketed tank, for washing and extracting pectin; filter press to separate the exhausted waste; closed stirred and jacketed tank, for precipitating the pectin by addition of ethanol; basket centrifuge for the separation of the precipitated pectin solution; and tray dryer to remove moisture (alcohol and water) and obtain the dry pectin.

Lastly, pectin production would not be economic if the process of alcohol recovery wasn't carried out, since costs would be raised excessively. Treatment of wastewater produced in the process must also be considered, due to organic load it contains. Although this fact lies outside the scope of this project, it is an important aspect and has to be kept in mind.

.

8. REFERENCES AND NOTES

Carbajal Azcona, A. (2012). Manual de Nutrición y Dietética. Universidad Complutense de Madrid. Facultad de Farmacia.

Casals Pelegrí, E.; Iglesias Castellarnau, I. (2013). Situación actual de la producción de manzana en España y análisis de la campaña actual. Revista Vida Rural.

Cerón, I & Cardona, C. (2011). Integral evaluation process for obtaining pectin and essential oil from orange peel. Ingeniería y Ciéncia, Vol. 7, nº 13, 65-86.

Chacín, J.; Marín, M.; D'Addosio, R. (2010). Evaluation of pectin content in different genotypes of guava from the south of Lake Maracaibo. Universidad del Zuli, Venezuela. Multiciencias, vol. 10, nº 1, 7-12.

Chasquibol Silva, N.; Arroyo Benites, E.; Morales Gomero, J. C. (2008). Extracción y caracterización de pectinas obtenidas a partir de frutos de la biodiversidad peruana. Universidad de Lima, 26, 175-199.

Coulson, J. M.; Richardson, J. F.; Backhurst, J. R.; Harker, J. H. (1981). Ingeniería Química. Tomo II. Versión española de la 3ª edición original.

Couper, J. R.; Penney, W. R.; Fair, J. R.; Walas, S. M. (2012). Chemical Process Equipment: Selection and Design. Third Edition.

Cuesta, M. & Muñoz, R. (2010). Extracción de pecina a partir de la corteza de maracuyá (Passiflora edulis var. Flavicarpa degener). Revista Politécnica, Vol. 31, nº1, 91-96.

Cunill, F.; Iborra, M.; Tejero, J. (2010). Reactores químicos. Universidad de Barcelona.

Devia Pineda, J. E. (2002). Proceso para producir pectinas cítricas. Revista Universidad EAFIT, nº129.

Durán, R. & Villa, A. L. (2014). Microwave assisted extraction of essential oil and pectin from orange peel in different stages of maturity. Revista de la Facultad de Agronomía, 31, 145-158.

Ferreira, S. (2007). Pectinas: Aislamiento, caracterización y producción a partir de frutas tropicales y de los residuos de su procesamiento industrial. Universidad Nacional de Colombia. Facultad de Ciencias, 20.

Guidi, A. and Arandia, M. Z. (2010). Obtención de pectina a partir de la cáscara de maracuyá mediante hidrólisis ácida. Universidad del Valle, Journal Boliviano de Ciencias, vol. 7, nº 21.

Herbstreith & Fox. (2001). "The Specialists for Pectins". http://www.herbstreith-fox.de/produkte/ (26 marzo 2016)

Li-ping Qiu, Guang-lei Zhao, Hui Wu, Lu Jiang, Xiao-feng Li, Jun-juan Liu, (2010). Investigation of combined effects of independent variables on extraction of pectin from banana peel using surface methodology. Carbohidrate Polymers. Elsevier, 80, 326-331.

MAGRAMA Ministerio de Agricultura, Alimentación y Medio Ambiente.

Masmoudi, M.; Besbes, S.; Chaabouni, M.; Robert, C.; Paquot, M.; Blecker, C.; Attia, H. (2008). Optimization of pectin extraction from lemon by-product with acidified date juice using response surface methodology. Carbohydrate Polymers, Elsevier, 74, 185-192.

McCabe, W. L.; Smith, J. C.; Harriott, P. (1991). Operaciones unitarias en Ingenieria Química. McGraw-Hill, 4ª Edición.

Mueckay, M. A. (2006). Obtención de la pectina a partir de desechos industriales de maracuyá. Universidad Agraria del Ecuador, Facultad de Ingeniería Agrícola.

Nakamura, A. and Tobe, J. (2015). Method for producing carrot pectin. Fuji Oil Company Limited.

Pacheco, J. A.; Hernández, D.; Barroso, F. O.; Hernández, S.; Segovia, J. G.; Castro, A. J.; Bonilla, A. (2012). Purification of bioetanol using extractive batch distillation: Simulation and experimental studies. Chemical Engineering and Processing: Process Intensification, Elsevier, 61, 30-35.

Perry, R. H. and Green, D. W. (1999). Perry's Chemical Engineers Handbook. Seventh Edition.

Sánchez Aldana-Villarruel, D.; Aguilar González, C. N.; Contreras Esquivel, J. C.; Nevárez Moorillón, G. V. (2011). Pectic molecules: extraction and its packing potencial application. Revista Tecnociencia Universidad Autónoma de Coahuila, Vol. V, nº 2, 76-82.

Sinnott, R. K. (2005). Chemical Engineering Design. Coulson & Richardson's Chemical Engineering, Vol. 6, Fourth edition.

Treyball, R. E. (1994). Operaciones de transferencia de masa, 2ª Edición.

ULLMANN'S Encyclopedia of Industrial Chemistry, 7th ed. (2011). Polyethylene to Potassium Compounds, Vol. 29, 420-432.

Vian O., Á. (2006). Introducción a la Química Industrial. Biosfera II: Materias Extractivas Vegetales. Editorial Reverté, 2ª edición, 486-487.

Zapata Z., A.; Escobar G., C; Cavalitto, S; Hours, R. (2009). Evaluation of pectin solubilization capability from lemon peel using protopectinase-SE. Universidad Nacional de Colombia Sede Medellín. Centro de Investigación y Desarrollo en Fermentaciones Industriales, CINDEFI.

9. ACRONYMS

d_i: Diameter of the impeller, m.

P: Impeller power, W.

N_p: Power number

N: Revolutions per second, s^{-1}.

μ: Viscosity. Pa.s

T_v: Temperature of the heating fluid, °C.

T_r: Temperature of cooling fluid, °C.

C_p: Heat capacity, J/(kg·°C).

U: Global coefficient of heat transfer, W/(m^2·°C).

α: specific resistance of the cake (m/kg).

c: wt of solids/volume of liquid (kg/m^3).

ΔP: pressure difference (N/m^2).

A: filtering surface (m^2).

V: Volume of filtrate (m^3).

Q: rate of filtrate (m^3/sec).

n_c: drying rate, kg/(m^2 s).

h: heat transfer coefficient, W/(m^2 °C).

T_g: gas temperature, °C.

T_b: wet bulb temperature, °C.

λ: latent heat of vaporization, kJ/kg.

θ: drying time, s.

m_s: dry solid mass, kg.

X: humidity, dry basis, kg/kg.

n_1: constant speed drying, kg/(m^2 s).

n_2: decreasing speed drying, kg/(m^2 s).

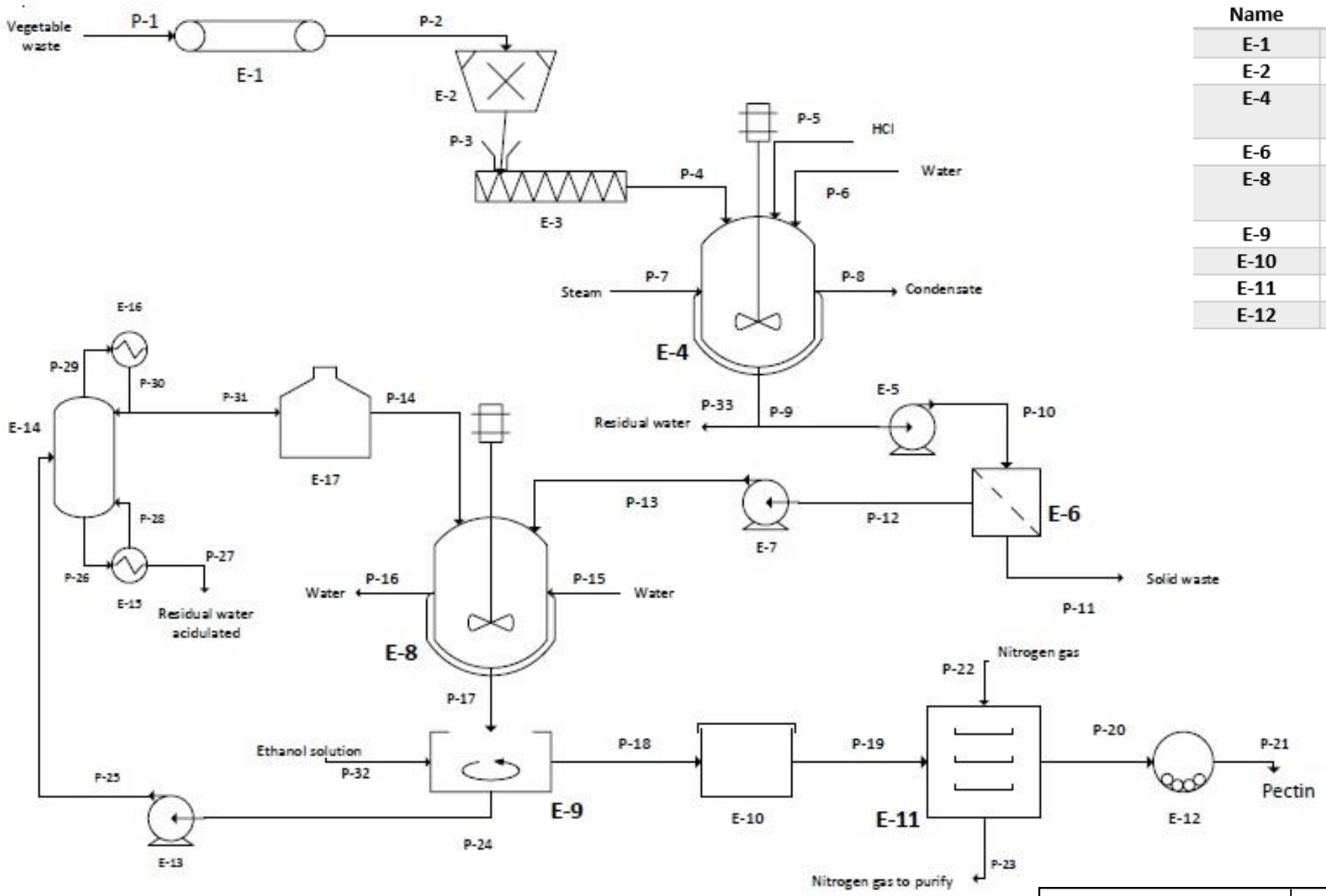
G: Gas flow, kg/s.

S: Step seccion of the gas, m^2.

d_e: Equivalent diameter, (d_e=4S/p)

APPENDICES

APPENDIX 1: PROCESS FLOW DIAGRAM



Name	Equipment
E-1	Conveyer belt
E-2	Mill
E-4	Opened stirred tank
E-6	Filter press
E-8	Closed stirred tank
E-9	Centrifuge
E-10	Storage tank
E-11	Tray dryer
E-12	Ball mill

Saray Martínez Pizarro	Treball Final de Grau
Design of a plant for extraction and purification of pectin.	
Process Flow Diagram	

UNIVERSITAT DE BARCELONA