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The viability of pectin extraction from potato fibre

Industrial Engineering and Management Bachelor Thesis

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Abstract

Valorisation of by-product streams is gaining more attention in recent years. Often these by-product streams contain valuable chemical compounds that are currently under utilized. This report has examined the valorisation of potato fibres. Potato fibres are a by-product from the potato starch industry. Pectin appeared to be having the most potential to be valorised. A preliminary design was made, based on a carefully selected extraction method. This design was techno-economic evaluated based on the cost of the necessary equipment. The outcome of the report is that it is viable to extract pectin from potato fibres. However, many assumptions that were made in the report were not specific. Further research to specify these assumptions is necessary before conclusions can be made whether or not pectin can be extracted in a viable way.

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Introduction

The production of food waste is enormous. Approximately 40% of the food waste is produced by the food manufacturing industry (Mirabella et al., 2014). Industrial ecology concepts such as circular economies considered leading principles for eco-innovation, aiming at a zero-waste economy in which waste are used as raw material for new products and applications (Mirabella et al., 2014). From both an ecological and economical point of view these are interesting concepts.

On a global basis, the potato is the fourth most important world food crop, because potato production can be conducted in a variety of different conditions, which makes it a commonly cultivated crop across the world. It follows three cereal grains, rice, wheat, and corn, in importance (Ahokas et al., 2014; Stearns et al., 1994). Processed potato products are a significant source of carbohydrates, in the form of starch (Fritsch et al., 2017; Furrer Amber et al., 2018). In developed countries up to 69.5% (in 2012) of total produced potatoes are processed (Sepelev & Galoburda, 2015). A certain percentage of these potatoes are being processed to obtain starch, by companies such as AVEBE and Agrana Stärke. During this process, a large amount of by-products are created, such as potato fibres (350k ton/year by AVEBE), potato juice (1.6 million ton/year by AVEBE) (Erik Koops Program Energy Efficiency Manager, AVEBE, 2021) and potato pulp (Lesiecki et al., 2012). These by-products have little to no value. However, they consist of valuable components such as cellulose, hemicellulose, pectic substances and pentosan (Meyer et al., 2009).

Potato cultivation and processing creates significant amounts of side streams that are not included in the main products (Ahokas et al., 2014). These side streams can be considered as waste. Potato waste after industrial potato processing can range from 15 to 40% of initial product mass, depending on the peeling method (Elo et al., 2006; Sepelev & Galoburda, 2015). Potato waste causes much impact on environmental pollution and unwanted revenue loss for potato processing industries (Gebrechistos & Chen, 2018). Potato waste consist of potato fibres, potato juice and potato pulp. Potato fibres contain chemical compounds such as lignocellulose, which comprises; hemicellulose, cellulose and lignin. These fractions have the potential to produce high-value chemicals. For example, hemicellulose and cellulose have been used for a long time in the textile and paper industry (Emmanuel et al., 2018). Potato fibre is also rich in pectin (Yang et al., 2018). Pectin are of interest because they are an attractive hydrocolloid for various food applications (Vincken et al., 2000). Potato fibre contains potential high-value compounds and therefore this report will focus on the valorization of potato fibres.

Potato starch extraction process

In order to determine how potato fibres can be valorized, it is important to discover how potato fibres are extracted. Therefore, in the following section, the potato starch extraction, from which the potato fibre originate, will be described.

The potato starch extraction process, which can be seen in Figure 2, that is performed by potato starch producers, like AVEBE, can be broken down into eight stages: quality control, washing, grinding, potato juice extraction, fibre extraction, starch classification, starch refinery, starch drying and storage (Berghaller, Witt, & Goldau, 1999; Grommers & van der Krogt, Do A, 2009). Each stage up to the fibre extraction will be described in greater detail in their respective paragraph. Potato fibres are the scope of this research therefore further description of the starch extraction process is not necessary. A complete overview of the process can be seen in Figure 2, where the red arrows indicate unused by-products and the blue arrows the main products that are being used further in the process. Potato fibres are the scope of this research and therefore are enclosed by the green rectangle.

Quality control

When potatoes arrive at the factory, they are weighed while samples are taken for quality control. The amount of starch content and the amount of dirt and other impurities, such as stones, coal, wood, are determined. Besides this, the internal quality of the potato is determined. This can be done by measuring the amount of rotting, hollow heart and greening (Bergthaller et al., 1999).

Washing

After quality control has been completed, the potatoes move to a washing station. At the washing station, stones, foliage and sand will be removed from the potatoes (Bergthaller et al., 1999; Grommers & van der Krogt, Do A, 2009). Rotary drum washers or trough washers with separated compartments are used for the washing process (Bergthaller et al., 1999).

Grinding

After the potatoes are washed, they are shredded. In that way, the cells of the potato are broken and the starch granules are liberated (Grommers & van der Krogt, Do A, 2009). Excessive grinding must be prevented; otherwise, heavily destroyed cell wall material can cause problems in successive sieving procedures (Bergthaller et al., 1999). During grinding, 98% of the starch granules are freed from cells. To prevent undesirable colouring of the potato slurry, an antioxidant is added (Grommers & van der Krogt, Do A, 2009).

Potato juice extraction

During this step, the potato juice is separated from the starch granules and the potato fibres. The separation of potato juice is performed with a decanter centrifuge. The input is the potato slurry, obtained from the grinding process. The outputs of the centrifuge are a starch-fibre cake of about 40% dry matter and potato juice, which is free of solids (Grommers & van der Krogt, Do A, 2009). The potato juice will not be further processed and is a by-product.

Fibre extraction

Starch-fibre cake consists of fibres and starch. According to (Grommers & van der Krogt, Do A, 2009), the fibres can be separated from the starch with a 125 μm sieve. This is possible because starch granules and fibre particles have different diameters. There is some overlap in the diameter ranges of starch granules and fibre particles, starch granules range between 1 and 120 μm in diameter, while fibre particles have diameters between 80 and 500 μm , see Figure 1, therefore a certain amount (2-3%) of starch does not pass the sieve and a certain amount of fibre does pass the sieve (1.2%). The rough starch milk moves on to the classification step. The fibres, that are separated, are dehydrated to 17% dry matter (Grommers & van der Krogt, Do A, 2009).

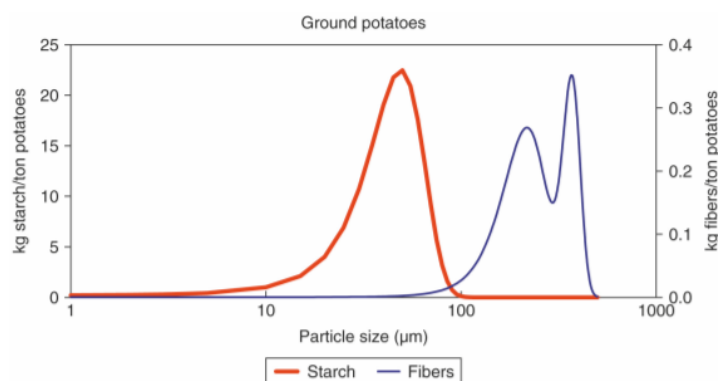


Figure 1: Particle size (diameter) of ground potatoes (Grommers & van der Krogt, Do A, 2009).

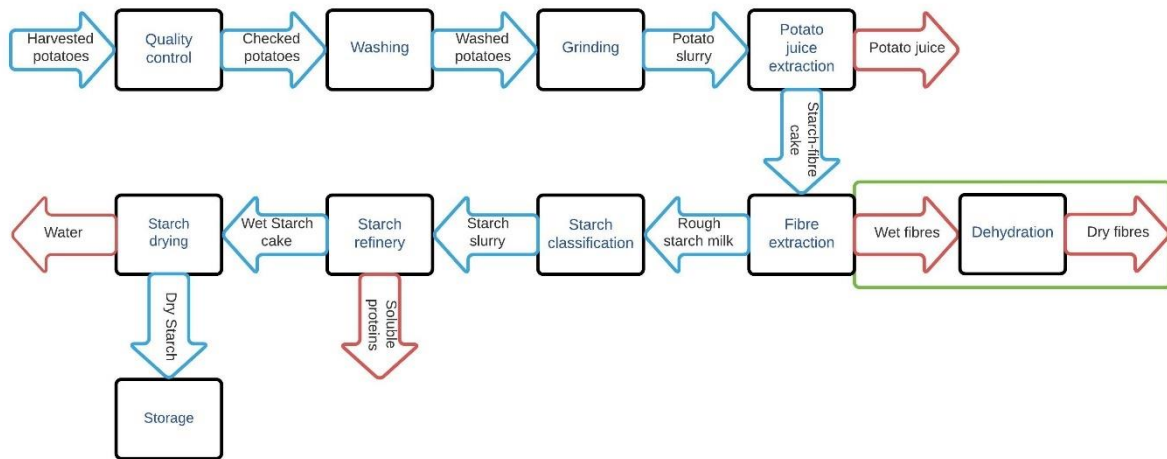


Figure 2 : Starch production process – Red arrows indicate by-products, blue arrows indicate main products, the green rectangle specifies the scope of the research.

Valorization potato fibres by AVEBE

Currently AVEBE valorizes its potato fibres by selling it as cattle feed (Grommers & van der Krogt, Do A, 2009; Nelson, 2010). Potato fibre is sold for approximately 35 euro per ton (Erik Koops, Program Energy Efficiency Manager AVEBE). It is chosen to sell potato fibres as cattle feed because it requires no additional energy and equipment to prepare the fibres. The wet fibres that are obtained from the fibre extraction can be directly sold. A small percentage of the wet fibres are dried and are sold as water binder and used in products such as ketchup (Erik Koops, Program Energy Efficiency Manager AVEBE). However, the drying process is expensive and energy intensive. Therefore, almost all obtained potato fibre is sold as cattle feed and still has potential to be valorized to a high-value product.

Problem definition

The problem that has been described before can be summarized in the following problem statement:

“With the production of potato starch, lots of potato fibers are created, which are underutilized and undervalued. However, important knowledge about what the possible outlets for these fibers are and whether or not these outlets are profitable, is missing.”

Stakeholder analysis

Two main stakeholders can be found, each with their own interest and power in the research project. A visual representation of the stakeholders and their respective power and interest can be seen in Figure 3.

The first stakeholder is the problem owner. AVEBE is a potato processing company, who is currently facing this problem. The R&D department is particularly interested in the outcome of this project. Their aim is to give the potato as much value as possible. By valorizing potato fibres, the potato's value could increase significantly. Therefore, the R&D department of AVEBE has high interest in the outcome of this research. However, they do not have any influence on the direction of this research and therefore have a low level of power.

The second stakeholder is Erik Heeres. Heeres is the supervisor of this bachelor project and therefore has a high interest in the outcome of the research. Heeres is the one who determines the boundaries and the direction of the research and has therefore a lot of power regarding this project.

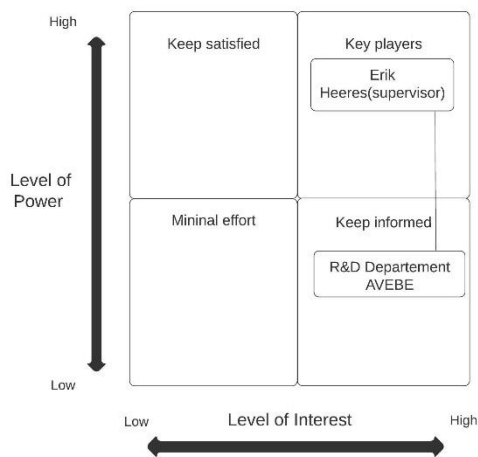


Figure 3: Stakeholder analysis, with the level of power on the y-axis and the level of interest on the x-axis.

Report goal

The problem statement has now been formulated and the stakeholders identified. Therefore, the report goal can be defined. The report goal can provide guidance during the project. The goal sets the boundaries of the research in terms of depth of the project. The problem statement and the report goal are tightly connected, because the report goal needs to be a solution to the earlier mentioned problem statement. The goal is set to be the following:

“The goal is to discover if potato fibers, one of the by-products of potato starch production, can be more valorised, while remaining financially viable.”

Research questions

To define the boundaries of this report, research questions will be used. Research questions give structure to research activities and provide a steering function, such that the desired outcome of the research is accomplished (Verschuren et al., 2010). Based on the problem statement and the report goal the following questions were constructed:

- 1) Which compound is most viable to extract from potato fibres?
 - a. What compounds do potato fibres contain?
- 2) Which extraction method has the most potential?
 - a. Which extraction methods are available for that compound?
- 3) Is the selected extraction method viable?
 - a. What are the revenues by using the extraction method?
 - b. What are the costs to realise the extraction method?

Materials and Methods

This chapter will describe how every research question will be answered. Sub-questions 1.a and 2.a will be answered with help of a literature study. Information will be gathered from papers, articles and web sources. Based on the gathered material for both the chemical compounds and the extraction methods a ranking system will be used to determine which chemical compound/extraction method has the most potential. This ranking system will be based on carefully selected parameters. The outcome of these ranking systems will answer research questions 1 and 2.

With help of a mass balance will the size of the final product stream be determined. The size of the stream together with the selling price of the product determines the revenues of the extraction, which ultimately answers sub-question 3.a. a techno-economic evaluation of the selected extraction method will form the basis of the answer of 3.b.

The conclusion of this report will answer all three research questions and its respective sub-questions. The discussion section will comment on the conclusion and give further recommendations for future research on this subject.

Results

Chemical composition potato fibre

The chemical composition of pressed potato fibre (PPF) includes cell wall polysaccharides (CWPs), such as hemicelluloses, cellulose, and pectin, and non-fibre components, such as oligopeptides, ash and starch (Ahokas et al., 2014; Al-Weshahy & Rao, 2012; Mayer, 1998; Storey, 2007). The starch in the fibre comes from potato cells which remained unbroken during the grinding process (~2% of the total starch) (Grommers & van der Krogt, Do A, 2009). Table 1 gives an overview of the composition of PPF based on dry matter.

For potato CWPs (Table 2), the content of pectin has been shown to be the highest, followed by cellulose and hemicellulose (Harris, 2009; Vincken et al., 2000). The most abundant pectic polysaccharides in potato cell walls are galactan linked rhamnogalacturonan-I (RG-I) and homogalacturonan (HG) (Caffall & Mohnen, 2009; Ramasamy, 2014). Xylogalacturonan (XG) and rhamnogalacturonan type II (RG-II) are also pectic polysaccharides that are present in plant cell walls (Carpita & Gibeaut, 1993). They have been found in potatoes, but are not yet quantified and therefore they will not be considered for valorization in this report (Harris, 2009; Ishii, 1997; Ramasamy, 2014). Potato hemicelluloses predominantly include xyloglucans and mannans, of which xyloglucans are the most abundant (Harris, 2009).

Table 1 : Composition of PPF based on dry matter (Ramasamy, 2014)

Component :	%(w/w)
Total organic matter	96
Starch	20-40
Ash	4
Proteins/ Amino acids	2-6
CWPs total	40-65
Pectin	19-31
Hemicellulose	7-11
Cellulose	14-23

Table 2 : Composition of CWPs in potato (Vincken et al., 2000)

Component :	%(w/w)
Pectin	56
Rhamnogalacturonan-I	50
Backbone	14
Side chains ((Arabino)Galactan)	36
Homogalacturonan	6
Hemicellulose	14
Xyloglucan	11
Mannan	3
Cellulose	30

Ranking system potato fibre compounds

The possible high-value chemical compounds that PPF contain are pectin, hemicellulose, cellulose. First, a decision needs to be made where the focus of this report will. The chemical compounds of PPF will be ranked by certain parameters. This ranking will decide what the focus of this report will be. The following parameters will be taken into consideration: the selling price of the compound and the weight percentage of the chemical compound with respect to the total amount of cell wall polysaccharides.

Selling price of the compound

The selling price of the specific compounds is a key factor for the determination of the research's scope. For instance, if compound A could be sold for 15 USD/kg and compound B for only 5 USD/kg, while having the same production cost, then from a financial perspective a company will always choose to produce compound A rather than compound B. For every compound, that is considered, the average price/kg is determined.

The global pectin market size reached a sales value of 1.04 Billion USD (Ahuja & Rawat, 2019). The global sales volume was estimated to be 60 thousand metric tons in 2018 (Industry Experts, 2019). The average price per kilo pectin is estimated to be 17.33 USD.

However, pectin consist of three chemical compounds: homogalacturonan (HG), rhamnogalacturonan I(RG-I), and rhamnogalacturonan II (RG-II) (Carpita & Gibeaut, 1993; Stephen, 1995; Yang et al., 2018).

HG-rich pectin is known as “smooth” pectin (Arrutia et al., 2020). “smooth” pectin is widely used and sold because of its uses in the food and pharmaceutical industries because of its excellent hydrocolloidal properties (Arrutia et al., 2020). RG-I and II rich pectin is referred to as “hairy” pectin (Willats et al., 2001). Potato pectin consists of a high proportion of RG-I (72%) and a smaller amount of homogalacturonan (HG) (Oomen et al., 2002). Therefore, the selling price of RG-I is more representative. Reports on the total value and volume of the global market of RG-I could not be found. However, RG-I extracted from potato fiber with a purity of >90% is sold for ~150 USD/2 gram, which equals 75 thousand USD per kilo (Megazyme, 2021a).

The global cellulose market reached a sales value of 1980.85 million USD in 2019. The global sales volume was 1646.67 thousand metric tons in 2019 (Azoth Analytics, 2020). The average price per kilo of cellulose with a purity of ~95% is estimated to be 1.20 USD.

Hemicellulose obtained from potato consists for approximately 80% of xyloglucan (Vincken et al., 2000). Therefore, the selling price of xyloglucan will form the basis of this factor. Xyloglucan retrieved from potato could not be found. However, xyloglucan with a purity of ~95 %, retrieved from tamarind is sold for ~150 USD/3 gram, which equals 50 thousand USD per kilo (Megazyme, 2021b).

Weight percentage with respect to CWPs

Weight percentage with respect to CWPs is also an important factor for the determination of the research's scope. A specific compound could have a high value, but if potato fibres only contain a small amount of that compound, it might not be profitable to extract and purify that component from the potato fibres.

As mentioned before in the chemical compounds composition section, potato fibres contain cell wall polysaccharides, which consist, for the most part, out of pectin in the form of RG-I (50%), followed by cellulose (30%) and hemicellulose in the form of xyloglucan (11%), see Table 2.

Conclusion ranking

The value of the two chosen parameters are determined for pectin, cellulose and hemicellulose and are summarized in Table 3. It is chosen to focus on the extraction of pectin from potato fibres. Pectin (RG-I) has the highest selling price and weight percentage. Therefore, pectin (RG-I) extraction will be the focus of this report.

Table 3 : parameter values; pectin, cellulose and hemicellulose

Ranking Summary	Selling price(USD/kg)	Weight percentage in CWP
Pectin (RG-I)	75 thousand	50%
Cellulose	1.20	30%
Hemicellulose (xyloglucan)	50 thousand	11%

Pectin

Pectin is a complex acidic macromolecular polysaccharide found in primary cell walls and the middle lamella (Yang et al., 2018). Pectic polysaccharides comprise between 30 and 50% of the cell walls of dicotyledonous plants, such as potatoes (Carpita & Gibeaut, 1993). Galacturonic acid (GalA) is the base component of pectin (Arrutia et al., 2020). GalA is present in three forms: homogalacturonan (HG), rhamnogalacturonan I(RG-I), and rhamnogalacturonan II(RG-II) (Carpita & Gibeaut, 1993; Stephen, 1995; Yang et al., 2018). Potato pectin consists of a high percentage of RG-I (72%) and a smaller amount of homogalacturonan (HG) (Oomen et al., 2002).

Pectin can be used as a gelling, thickening, stabilizing or emulsifying agent in the food, cosmetic and pharmaceutical industries. Besides, it has been reported to have various biological and physiological functions in human nutrition and health (Yamada, 1996). Physicochemical and functional properties of pectin are highly related to their structures including the molecular weight (MW), degree of esterification (DE), GalA content, and other sugar components which depend on the plant source and the extraction method (Hrabovska et al., 2018; Wandee et al., 2019).

RG-I and II rich regions are referred to as “hairy” pectin (Willats et al., 2001), while HG-rich regions are known as “smooth” pectin (Arrutia et al., 2020). Commercially available pectin is rich in smooth pectin and is used in the food and pharmaceutical industries because of its superb hydrocolloidal properties (Arrutia et al., 2020). “Smooth” pectin is mainly produced from apple pomace, sugar beet and citrus peels (Ciriminna et al., 2016). “Smooth” pectin has a high degree of methylation (DM), molecular weight, and a high proportion of the HG region. In comparison, potato pectin is richer in acetyl groups and neutral sugar side chains, but the HG domain is shorter than commercial citrus and apple pectin, and thus, potato pectin does not have good gelling ability (Yang et al., 2018).

RG-I -rich pectin, such as potato pectin, can substantially hinder the in vitro growth of numerous pancreatic cancer cell lines (Schols & Voragen, 1996). Therefore, RG-I domain has been considered as an important region in pectin because of its enrichment of pectin’s immune-modulating and anti-tumor bioactivities (Zhi et al., 2017). RG-I extraction will also be the focus of this report because of those properties.

Extraction methods pectin

Alkaline, enzymatic, and acid methods are the most common ways to extract pectin from sources such as potato, apple, citrus fruits and sugar beets (Yang et al., 2018). Methods such as microwave heating and disodium phosphate extraction are not yet used by industry but show promising results and will therefore be discussed. Every extraction method is described briefly in their respective paragraphs.

Alkaline/Acid extraction with enzymatic hydrolysis of starch (ALE/ACE)

Alkaline/acid extraction methods can retain the neutral sugar side chains in pectin. However, the methyl ester and acetyl groups of pectin are hydrolysed by the β -elimination reaction, which negatively influences the properties of pectin (Rombouts & Thibault, 1986). The emission of waste acid/alkali solution is a disadvantage of these methods.

Yield and structure and emulsifying properties of pectin are affected by alkaline/acid and depend on several factors, such as the source of pectin, type and concentration of alkaline/acid, extraction method such as heating technique, heating time and temperature (Ma et al., 2013; Wandee et al., 2019; Yang et al., 2018). The yield of acid extracted pectin varies between 4.08-14.34% (ratio obtained pectin and available pectin) (Yang et al., 2018). The yield of alkaline extracted pectin varies between 22-56% (ratio obtained pectin and available pectin) (Khodaei & Karboune, 2013).

In general, pectin extraction by alkaline/acid is performed by the following steps: (1) potato pulp is dried, grounded and then sieved. (2) Residual starch is enzymatically hydrolysed using thermostable α -amylase and then separated by centrifugation. (3) The precipitate is washed with ethanol, recentrifuged, dried overnight, grounded and finally sieved. (4) the enzymatic treated potato residue is dissolved in water and adjusted to a certain pH (> 7 and <7 for alkaline and acid extraction respectively). (5) Resulting solutions are thermally treated and afterwards centrifuged to remove hemicellulose and cellulose. (6) The supernatants are precipitated by ethanol and recentrifuged. (7) The obtained pectin is dispersed in water and freeze-dried (Yang et al., 2018).

Enzymatic Extraction (EE)

Pectin extracted by enzymes is done in several steps: (1) enzyme extract and a sodium citrate buffer are added to the pectin source and agitated for a certain amount of time under specific conditions (i.e. temperature and pH) such that the pectin substances dissolve. (2) Subsequently, the mixture is filtered and centrifuged, to separate the non-dissolved compounds (i.e. hemicellulose and cellulose) from the dissolved pectin. As final step, the dissolved pectin is precipitated with ethanol and filtered again (Vasco-Correa & Zapata, 2017).

Extraction via the use of enzymes can decrease the emission of waste acid and alkali solution. The pectin obtained via this method has a higher molecular weight and degree of esterification than the acid method, i.e., the quality of the pectin is to a lesser extent influenced. However, the processing time is longer than the acid method (Wikiera et al., 2016).

This process generated a GalA yield of up to 26 g/100 g of dry passion fruit peel, which was 40% higher than that of the traditional chemical method that uses high temperature and a strong acid (Vasco-Correa & Zapata, 2017).

(Hrabovska et al., 2018) extracted pectin from potato pulp with enzymes. The highest pectin yield of 5.11% (relative to the raw material mass) was observed while using the highest concentration enzyme preparation per gram of raw material, without using acid and any other chemical reagents. This equals a yield of ~16.5-26.9% (ratio obtained pectin and available pectin) when the composition of potato pulp is taken into account. However, despite the high yield of pectin substances in enzymatic hydrolysis, its coagulation structure is weak, thus the properties of the obtained pectin were negatively influenced.

Microwave heating extraction (MHE)

(Arrutia et al., 2020) designed a continuous-flow microwave system on a lab-scale to demonstrate the feasibility of using a scalable continuous-flow microwave-assisted thermal treatment to extract “hairy” pectin (RG-I) from industrial potato pulp, using only water as the extraction medium.

The full system is depicted in Figure 4. A feed tank that contains potato pulp is being stirred by an overhead stirrer to ensure homogeneity throughout the mixture (Arrutia et al., 2020). They added water to the potato pulp such that the mixture consists of 75% water(w/w).

From the feed tank, the potato pulp mixture is pumped to the cavity (heating region) and remains there for 0.81 seconds, where its temperature is increased to 85°C. After all the pulp has gone through the cavity, the treated pulp is collected in a beaker where it was allowed to cool down under stirring for 20 minutes, reaching a temperature of approximately 70°C. After that, it was cooled down further to 50°C in cold water. During this thermal treatment dissolves the pectin.

The thermally treated pulp was filtered with a filter paper using a partial vacuum, to separate the solids from the dissolved pectin. Next, the liquid extract with the dissolved pectin was treated with isopropanol at a 1:1 ratio (v/v) and preserved at 4 °C overnight, to precipitate the pectin oligosaccharides. Finally, the pectin-containing extract was recovered by centrifugation and was freeze-dried (Arrutia et al., 2020). The GalA content was measured as an approach to quantify the yield of the pectin extraction. A pectin yield of ~40-45% was achieved.

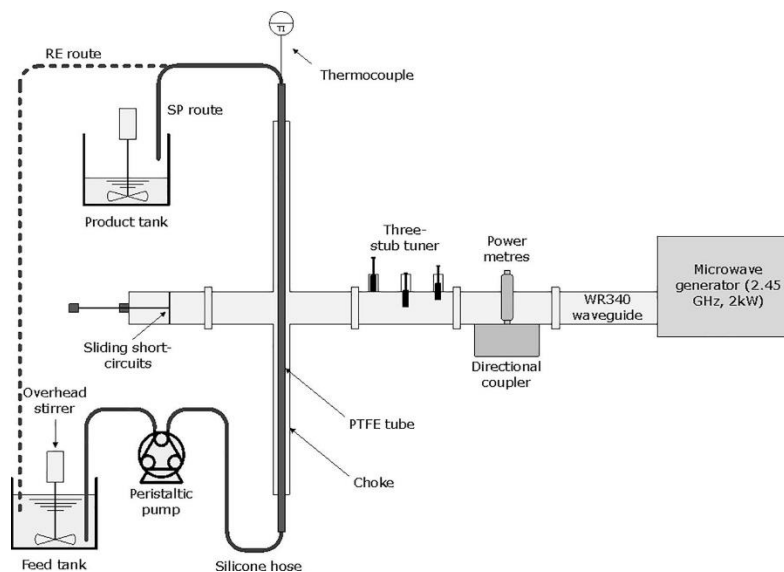


Figure 4: Continuous-flow microwave processing rig assembly and components (Arrutia et al., 2020).

Disodium phosphate extraction (DPE)

Pectin extraction from sweet potato dregs with help of disodium phosphate was performed by (Taihua et al., 2008). (1) the dregs were suspended in water and then sieved, such that a large part of the present starch is removed. (2) Amylase is used to carry out enzymatic hydrolysis of the residual small amount of starch. The mixture is then centrifuged, the supernatant is removed, and the sediments collected. (3) The sediments are prepared into a suspension and Na_2HPO_4 solution is added to the suspension to extract the pectin. As a next step, the mixture is centrifuged for 30 min thereafter the supernatant is concentrated and dried to obtain pectin powder. Pectin yield is ~95% (ratio obtained pectin and available pectin), and the pectin purity is $91.28 \pm 0.58\%$. (Taihua et al., 2008) suggests that this method is applicable to starch-containing higher raw material such as potato residues. Therefore, this method will be considered for this report.

Comparison extraction methods

As has been described earlier, potato pectin consists of a large part out of RG-I (72%) (Oomen et al., 2002). Native RG-I can, however, not be isolated from pectin obtained by the traditional industrial HG-extraction procedure as this is performed at low pH causing degradation of the arabinan side chains and inevitably causing degradation of the native RG-I molecule (Guillon & Thibault, 1990).

The type and concentration of extraction medium, heating time and heating temperature are all factors that influence the extraction yield and quality of pectin (Ma et al., 2013; Wandee et al., 2019; Yang et al., 2018). Therefore, all five extraction methods will be compared based on these factors and the yield.

Table 4 gives an overview of the data regarding the four factors of all five extraction methods. In comparison with the other extraction methods, MHE has superior extraction medium and heating times. The heating temperature for MHE is relatively high. However, in combination with the low heating times, the influence on the pectin structure (degradation) is within acceptable boundaries. Compared with ALE, ACE and DPE, MHE has no contamination products in the waste streams, such as acid and alkali. MHE has the second highest yield. Considering all of the above, MHE will be the method, the preliminary design to extract RG-I from potato fibre, is based upon.

Table 4 : overview data ALE, ACE, EE, MHE and DPE (+) indicated the best option in the respective column. (Arrutia et al., 2020; Hrabovska et al., 2018; Khodaei & Karboune, 2013; Taihua et al., 2008; Yang et al., 2018)

	Extraction medium	Heating times (in minutes)	Heating temperature (in °C)	Yield
ALE	Alkaline	1440	60(+)	22-56%
ACE	Acid	60	90	4.08-14.34%
EE	Enzyme	250	50-75	16.5-26.9%
MHE	Water (+)	20(+)	85	40-45%
DPE	Na ₂ HPO ₄	120-600	70-90	95%(+)

Preliminary design

The microwave heating extraction (MHE) method developed by (Arrutia et al., 2020) will serve as the basis of this system. MHE consists out of several stages; (1) decrease of solids concentration by adding water (2) starch reduction (3) microwave-assisted extraction (4) filtration (5) precipitation (6) overnight preservation (7) centrifugation (8) freeze-drying of solids. A visual representation of the process is given in Figure 5.

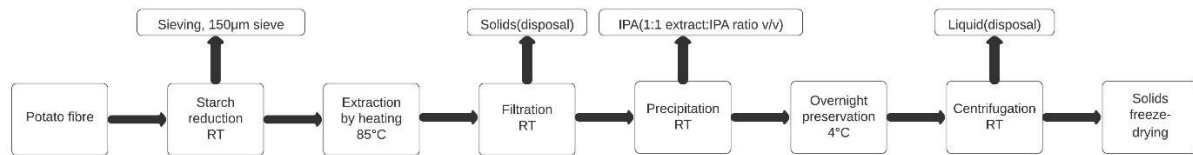


Figure 5: Overview of pectin extraction and recovery process. RT means room temperature. Adapted from (Arrutia et al., 2020).

Plant type

When a process is designed, the first question is whether the process will be a batch or a continuous process. This will be decided based on two aspects; feed stream size and production period. Plants having a capacity of greater than 4.5×10^6 kg/year are usually continuous, whereas plants having a capacity of less than 4.5×10^5 kg/year are normally batch type (Douglas, 1988). 350 thousand tons of potato fibre (17% dry matter) is produced per year by AVEBE (Erik Koops Program Energy Efficiency Manager, AVEBE, 2021). The production of potato starch is season bounded, so only a certain period per year (autumn until spring) potato fibres need to be processed. Batch plants are often preferred for products with seasonal boundaries (Douglas, 1988). The potato fibre production is season bounded. The feed stream size is relatively large. However, this will be a preliminary design for a pilot plant with a feed stream of 350 ton/year, which is relatively small. Therefore, the extraction system will be in the form of a batch plant.

Challenges during the extraction

(Arrutia et al., 2020) faced two challenges during the design of their continuous-flow microwave system; (1) problems during downstream processes (filtration and stirring) occurred when the solids concentration values were higher than 10% w/w. (2) A high starch content in the potato fibres caused problems during the heating phase. The solutions to these problems are discussed in their respective paragraphs.

Adjustment of the solids concentration

(Arrutia et al., 2020) discovered that for a continuous process the feed concentration is required to be lower than 10% w/w, to secure the flow of the feed stream. Subsequently, (Arrutia et al., 2020) adjusted the feed concentrations in a batch process to determine the effect on the yield. Feed concentration of 25% w/w and 50% w/w were tested and resulted in a limited reduction of yield. In general low solvent:solid ratios can reduce extraction yield through limitations in solubility, viscosity and a reduction in osmotic potential between the plant matrix and the solvent. However, low solids concentration and high solvent content negatively affect the economics of the process and mean higher wastewater disposal or drying costs (Adetunji et al., 2017). A batch process is selected for the preliminary design therefore the problems with the flow do not occur. As mentioned before, a limited reduction of yield was observed when the feed stream concentration was adjusted. Therefore, the initial feed stream concentration of 17% w/w is not adjusted.

Starch content in potato fibre

As mentioned before, 2-3% of starch does not pass the sieve during the potato fibre extraction phase in the starch extraction process, due to the overlap between the diameter of starch granules and fibre particles and is part of the potato fibre (Grommers & van der Krogt, Do A, 2009). This high fraction creates two problems: (1) as a result of the temperature increase during microwave-assisted extraction, gel-formation takes place (Arrutia et al., 2020). A feed in gel form does not flow across the continuous system due to the high viscosity, preventing the system from working properly (Li et al., 2018). (2) During the analytical characterisation of the end-product, high contents of starch in the form of glucose interfered with the identification of the pectin-derived sugars, affecting the results (Arrutia et al., 2020). Therefore, the starch fraction in potato fibre needs to be decreased.

(Arrutia et al., 2020) tried to solve this problem with two strategies: enzymatic hydrolysis of the starch using α -amylase and removing the starch by introducing a sieving procedure using a 150 μm mesh size. The degree of success of the de-starching procedure was tested by analysing the ionic chromatography (IC) glucose peak in the enzyme-treated extracts, the sieved extracts and the sieved permeate. The permeate showed only a glucose peak and no other sugar peaks (see Figure 7 in the Appendix). Therefore, can be concluded that that the sieving only removed the starch and not the hairy pectin regions. The enzyme-treated extracts showed a high glucose content representing starch. Hence, can be concluded that the de-starching performance of α -amylase was poor. The reason for this is that resistant starch, which can not be hydrolysed with α -amylase, is present in potatoes (Hoover & Zhou, 2003).

Considering all of the above and the fact that the sieving process allows the starch to be isolated and collected as a co-product of the process, rather than being reduced to glucose in the enzyme process, which offers the advantage of being more economic compared with the enzymatic procedure, and it would be straightforward to integrate it as a continuous upstream process to pectin extraction (Arrutia et al., 2020), a sieving extraction process was chosen to decrease the starch content in the potato fibre. The starch amount in the MHE extract in the form of glucose yielded ~36% (ratio glucose present in potato fibre and glucose present in the extract). Hence, it can be concluded that sieving extracted ~64% of the starch content from the potato fibre.

Mass balance process

As mentioned before, potato fibres obtained from AVEBE have a dry matter percentage of 17%, with the rest being water. 350 tons potato fibre 17% dry matter equals 59.5 tons dry matter. The starch fraction accounts for 20-40% (30% is used in the mass balance) of the potato fibre, based on dry matter (Ramasamy, 2014). Hence, by sieving, 64% of 30% of the dry matter in the form of starch is separated, which equals ~11.4 tons. Therefore, 338.6 tons of de-starched potato fibre is further processed. The next step is the thermal treatment at 85°C, followed by a filtration step where the solids are separated. (Arrutia et al., 2020) is not clear about the composition of the separated solids, therefore assumptions on the composition need to be made.

Table 1, displays the content of the de-starched potato fibres, based on that data, can be determined what the composition of the solids is. When proteins are heated some of the bonds that hold the molecule into shape are disrupted, as a consequence proteins clump together and solidify (Sci Bytes, 2013). Hemicelluloses and celluloses are insoluble in water (Huffman, 2003) and are part of the solids. It is assumed that a temperature increase does not affect the starch granules. Ashes are the solid remnants of fires and are assumed to be part of the separated solids. Based on the information above, it is assumed that the solids, that are separated during filtration, consist of proteins, hemicellulose, cellulose and ashes. A total of 42% solids (4% ash, 11% hemicellulose, 23% cellulose and 4% protein, based on the dry matter) is separated in the filtration step, equalling ~25 tons.

The remaining 313.6 tons of supernatant, which consists of pectin, starch and water is treated with isopropanol at a 1:1 ratio (v/v) and preserved at 4 °C overnight. The density of pectin and starch are both close to 1500 kg/m₃ (1157.8-1500.4 kg/m₃ (Coma, 2013) and 1500 kg/m₃ (Isleib, 1958), respectively). The density of water is ~1000 kg/m₃. The density of 313.6 tons of supernatant consists out of 23.1 ton dry matter (15.7 ton pectin + 6.4 ton starch) and 290.5 ton of water, which equals ~305.9 m₃ potato fibre, which is treated with 305.9 m₃ isopropanol. The preservation is followed by centrifugation and as a final step, freeze-drying. The batch experiments (Arrutia et al., 2020) achieved a pectin yield of ~40%. (Arrutia et al., 2020) analyzed the content of the dry extract with the help of ionic chromatography, the chromatogram can be seen in Figure 8 (Appendix). The purity was not determined by (Arrutia et al., 2020). However, based on the chromatogram the purity is assumed to be around 50%. Hence, in total ~12.6 ton dry extract is produced. The by-product stream contains 290.5 ton of water, 305.9 m₃ isopropanol and 10.5 ton of pectin/starch. As a final step the extract is sieved and an additional 6.1 ton starch is separated. The mass balance is visualised in Figure 6.

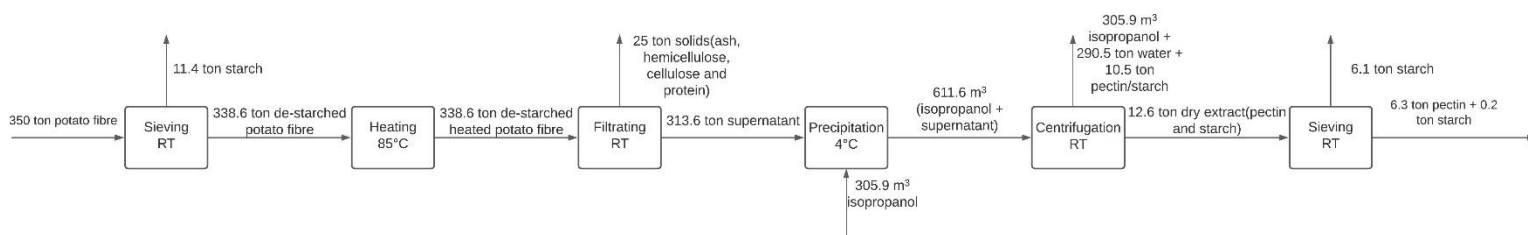


Figure 6: Mass balance pectin extraction. RT means room temperature.

Usage of output streams

In the mass balance in Figure 6 five outgoing streams can be observed; (1) 11.4 ton starch. (2) 25 tons of ash, hemicellulose, cellulose and protein. (3) 305.9 m₃ isopropanol mixed with 290.5 ton of water and 10.5 ton of pectin/starch. (4) 6.1 ton starch (5) 12.6 ton pectin/starch mixture (Rhamnose, Arabinose, Galactose, Glucose and Xylose).

Stream 1 & 4 consist purely of starch in the form of glucose, as can be seen in Figure 7 (Appendix). These streams can therefore be added to the wet starch cake that is obtained from the starch refinery process, see Figure 2.

Stream 2 consists of ash, hemicellulose, cellulose and protein. Compared to proteins from other vegetable and cereal sources, potato proteins are considered higher quality as they contain a high proportion of lysine, which is often lacking in such crops (Waglay et al., 2014). Stream 2 can be the feed stream of one of the potato protein extraction methods that (Waglay et al., 2014) compared. However, protein extraction is not the scope of this research.

Stream 3 contains isopropanol, water, starch and pectin. Since isopropanol is necessary for the precipitation of pectin it would be convenient to separate the isopropanol from stream 3 such that it can be reused. Isopropanol is known to form a homogeneous minimum boiling azeotrope with water at atmospheric pressure (Boli et al., 2018). Therefore, a high-purity isopropanol product over its azeotropic composition cannot be obtained through conventional atmospheric distillation. To obtain isopropanol from an isopropanol/water mixture azeotropic distillation can be used (Van Hoof et al., 2004). However, stream 3 also contains CWP and the influence of that on the azeotropic distillation is unknown. If it is economical and ecological viable to separate water from the isopropanol and CWP then could the water be reused in the washing station, see Figure 2.

Stream 5 contains xylose, rhamnose, arabinose, galactose and starch in the form of glucose. Extracts containing galactose, arabinose and also rhamnose have been shown to increase beneficial bacteria populations in the colon, confirming their potential as prebiotic ingredients (Gómez et al., 2016). The purity of the pectin is ~96%.

Techno-economic evaluation

The cost of the main equipment needs to be determined to perform a techno-economic evaluation. The main equipment for the pectin heating extraction consists of: a feed and product tank both with a mixer, a heat exchanger, a filter, a pump, a centrifuge and a freeze dryer. All the equipment cost, except the freeze dryer, will be based on the “Purchased Equipment Cost for Common Plant Equipment” table and the “Installation factors” table by (Sinnott & Towler, 2019). The freeze dryer will be represented by a direct contact rotary dryer. The assumption is made that for every piece of equipment the size is minimal because the size of the feed stream is relatively small. An overview of the equipment costs can be seen in Table 5. The total equipment cost was calculated to be ~1 million USD.

The average lifetime of machinery is ~26 years (Erumban, 2008). The average capital cost is ~38.5 thousand USD/year. 6.3 ton pectin (RG-I) extract with a 96% purity can be sold for ~470 million USD/year. With the extraction of pectin an additional 17.5 ton/year starch was extracted, which can be sold. The average potato starch export price stood at ~ 780 USD per ton in 2018 (IndexBox, 2019). Hence, the additional extracted starch can be sold for ~13.6 thousand USD/year. In total, a revenue of 470 million USD/year can be obtained with the extraction of pectin, in case that the obtained extract can be sold for the average pectin (RG-I) price.

Table 5 : Equipment costs

Equipment	Size	Equipment cost	Installation factor	Total cost
2x Floating roof tank	Capacity : 100.0 m ³	110.000	2,5	275.000
2x propeller mixer	Driver power : 5.0 kW	10.000	4	40.000
Double pipe heat exchanger	Area : 1.0 m ²	1600	2,5	4000
Plate and frame filter	Capacity : 0.4 m ³	76.000	4	304.000
Single-stage centrifugal pump	Flow : 0.2 L/s	3.300	4	13.200
High-speed disk centrifuge	Diameter : 0.26 m	70.000	4	280.000
Direct contact rotary dryer	Area : 11.0 m ²	9.000	4	36.000

Conclusion

This work theoretically describes the feasibility of extracting pectin (RG-I) from industrial potato fibres using thermal treatment. Potato fibres consist of proteins, residual starch, ash and cell wall polysaccharides such as pectin, cellulose and hemicellulose. Based on the ranking parameters (i.e., selling price, weight percentage of the chemical compound and potential market size), it was concluded that the extraction of pectin (RG-I) had the most potential to valorise potato fibres. Different extraction methods were compared based on extraction medium, heating time and heating temperature, factors that influence the yield and the quality of pectin. Of all compared extraction methods (i.e., ALE, ACE, EE, MHE and DPE), MHE proved to have the most potential, according to the before-mentioned factors, to form the basis of a preliminary design for pectin extraction.

During the preliminary design, it was decided that a batch process is the most suitable option for the extraction of pectin. Via a mass balance, based on data which was retrieved from (Arrutia et al., 2020), it was concluded that 350 ton potato fibre converts into ~6.3 ton dry pectin extract with a purity of ~96% and an additional 17.5 ton starch were separated. The total capital cost of the extraction was determined to be ~1 million USD, based on the techno-economic evaluation. Together with the average lifetime of machinery, the average annual capital cost was calculated to be ~38.5 thousand USD. Based on the mass balance, it was determined that the extraction of pectin (RG-I) could increase the revenue by 470 million USD/year. Hence, the extraction of pectin turns out to be viable and it would lead to a net profit of 470 million USD/year. However, the average annual capital cost and average annual revenue were based on many assumptions (e.g. the mass balance). In addition, the utility cost were not taken into consideration. In other words, the outcome of this research is that the extraction of pectin (RG-I) from potato fibre is viable but to improve the accuracy of this report, future research is necessary.

Discussion and recommendations

The outcome of the research is that the extraction of potato fibre is viable. Several assumptions influenced this result. These assumptions will be discussed whether or not they were accurate and what future research needs to be done to obtain more accurate assumptions.

Ranking chemical compounds

The ranking of the chemical compounds for the selection of the one with the most potential to be viable was based on three parameters (i.e., selling price and weight percentage). However, the yield of extraction is also an important factor for this selection. The yield of extraction and the weight percentage of a specific compound is closely related. For instance, compound A could have a weight percentage of 50%, but only a yield of 2% and compound B could have a weight percentage of 15% and a yield of 30%. So effectively, only 1 gram compound A can be retrieved per 100 gram potato fibre, while 4.5 gram compound B per 100 gram potato fibre could be extracted.

The yields were not taken into account in the ranking of the chemical compounds because there was lack of time to create an extensive detailed overview of the yields of the extraction of every specific compound.

Potential market size could also be an important parameter to rank the chemical compounds. It could be the case that the market could be overflowed by the production, which has a negative influence on the selling price. However, to determine the market size of every compound, market reports should be studied. Nevertheless, these reports can not be accessed without purchasing them. Assumptions of the market size are very hard to make. Therefore, potential market size is not considered as a parameter for the ranking of the chemical compounds.

To obtain a better overview of which compound has the most potential, an extensive study must be done on every compound and its best extraction method and what the potential market size is. The ranking can then be determined based on four parameters instead of two, which gives a more representative selection.

Techno-economic evaluation

For the techno-economic evaluation, it was assumed that for every piece of equipment the minimal size suffices for the execution of the process. Proper calculations on equipment sizes should be made to obtain a more accurate total equipment cost. Other materials that are necessary to build the preliminary design, such as pipes and control devices, were not taken into consideration for the evaluation. Finally, the utility costs were also not taken into consideration, so by including all those additional costs, the total annual cost could be determined more accurately.

Yield percentage

The pectin yield percentage was assumed to be 40-45% based on the batch experiments performed by (Arrutia et al., 2020). However, these experiments were based on a solids concentration of 25 and 50% w/w. (Arrutia et al., 2020) discovered that the value of the solids concentration had no major influence on the yield. However, in this report it is assumed that it had no influence on the yield. Further research on the effect of the value of the solids concentration on the yield should be done to obtain a more specific percentage for the yield.

Appendix

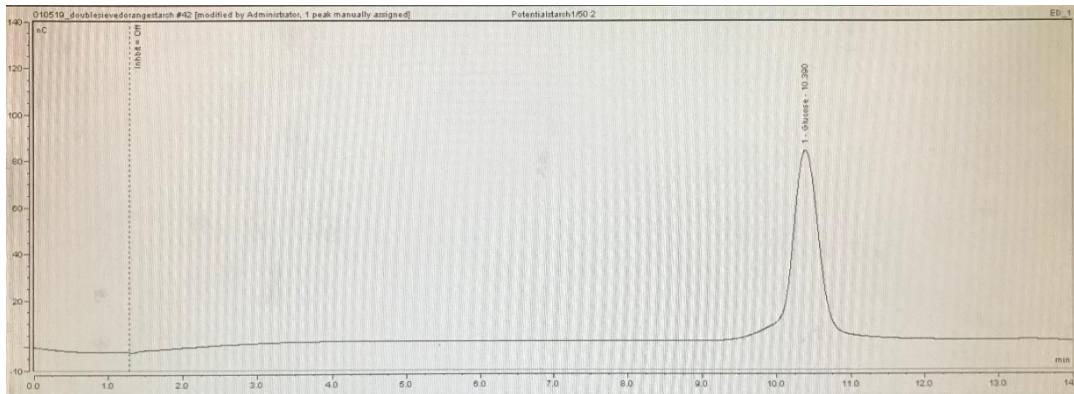


Figure 7: Ionic chromatogram of the sieved starch (Arrutia et al., 2020).

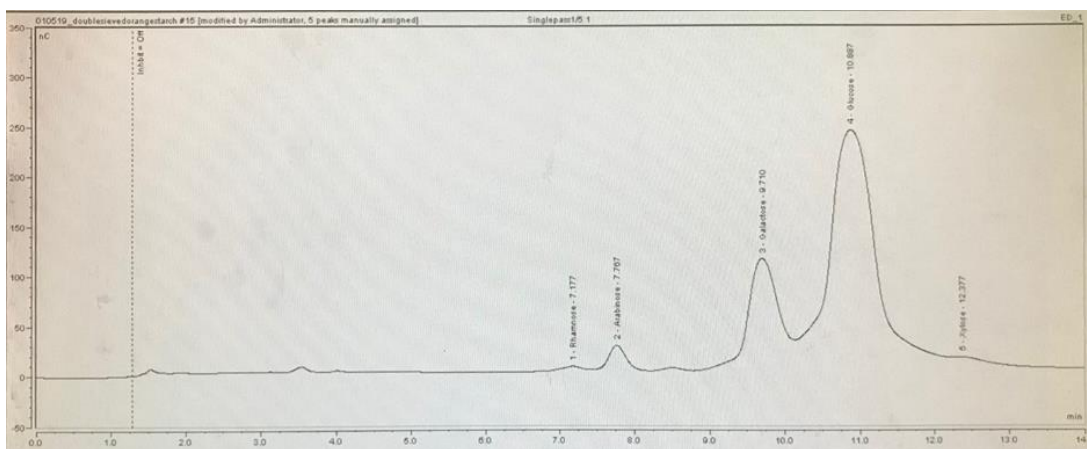


Figure 8: Ionic chromatogram of the dry extract (Arrutia et al., 2020).

Interview Erik Koops

1. How many potatoes are processed by AVEBE per year?

2 million potatoes are processed per year by AVEBE.

2. How are the potatoes being processed into starch?

They are washed and shredded. After that the potato juice is separated with help of a refining process. Subsequently the fibres are extracted (high water percentage). Then the proteins are separated from the starch and the starch is refined to a high-quality product.

3. Which by-product are created during the process and how much? What do you do with these compounds?

350k ton potato fibre (16% dry mass) is created. The fibres are sold as cattle feed for 35 euro/ton. A small percentage is dried and used as a water binder in for example ketchup. However, drying is very energy intensive and expensive.

1.6 million potato juice is obtained, from which the proteins are extracted and used for human consumption. Currently water extracted from the juice is reused for the washing treatment of the potatoes. Potato juice contains sugars, amino acids and minerals.

4. Which variety of potato are being used for the starch production?

Many varieties are collected from different places. They are not processed separately because there is only a small difference in composition between the varieties.

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