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Bachelor Thesis

Effects of steviol glycosides and maltitol in combination with different extrusion parameters on properties of liquorice

Metropolia University of Applied Sciences

Bachelor of Engineering

Biotechnology and Food Engineering

Bachelor Thesis

Date: 15.06.2016

Author(s) Title Number of Pages Date	Fabian Anzmann Effects of Steviol Glycosides and Maltitol in combination with different extrusion parameters on properties of liquorice 64 pages + 1 appendices 15.06.2016
Degree	Bachelor of Engineering
Degree Programme	Biotechnology and Food Engineering
Instructor(s)	Kirsi Jouppila, University Lecturer Satu Kirjoranta, Doctoral Student Pia-Tuulia Laine, Senior Lecturer

Liquorice confectionary are well known all over the world. Liquorice are typically produced in steam heated pots or by an extrusion process. The main ingredients for liquorice are sugar (typically sucrose, invert sugar or glucose syrup), wheat flour, molasses and the liquorice extract, which is giving those confectioneries their special taste. Besides providing the sweetness, sugar also affects other properties like glossiness or moisture stabilization. Due to the adverse health effects of sucrose, there are reasons to replace sucrose with sweeteners such as aspartame, steviol glycosides or maltitol. The aim of this research was to determine whether the different sweetener (maltitol syrup and steviol glycosides) in combination with changing extrusion parameters (mass flow) have an influence on the final product.

In the present study, two different recipes, one with liquid sugar 77 and one with steviol glycosides and maltitol syrup, were used. The calculated amounts of steviol glycosides and maltitol syrup offer the same theoretical sweetness than the liquid sugar 77. The liquorices were produced with two different water contents and one centre point (21%, 23.5%, 26%). During the production the mass flow was changed (70g/min, 100g/min, 130g/min). The variables which had been constant were the temperatures in the different sections and the screw speed (55rpm). Afterwards the water content, the water activity and the diameter of the liquorices were measured. An extension test was done to determine the maximum load with the corresponding extension. Additionally, a consumer test had been done with 40 test persons, who had to evaluate the texture, the sweetness and the overall liking of four liquorice samples, which had been chosen beforehand.

The research showed that by using maltitol syrup and steviol glycosides a higher water content has to be used to get similar diameters and textures in comparison to the liquorice produced with liquid sugar. More water was necessary because maltitol syrup has better water binding properties than sucrose. Therefore, less water was available for the starch to gelatinize. However, a particular influence of steviol glycosides on physical properties could not be detected because it was only used in very small amounts.

According to the results of the consumer test, the liquorices produced with maltitol syrup and steviol glycosides did not differ much from the liquorice with liquid sugar 77.
Further research is needed to study if steviol glycosides support the effect of maltitol by using different proportions of steviol glycosides and maltitol syrup.

Keywords	liquorice, steviol glycosides, maltitol, sweets, sweetener

Table of Content

1 Introduction

Liquorice confectionery, especially the Allsorts type, are very popular all around the world. The most important ingredients for this type of confectionary are sweeteners, wheat flour and liquorice extract [Minifie, 1997]. The liquorice extract, which is most responsible for the strong and persistent flavour of those liquorice confectionery is obtained from the liquorice shrub [Batke, 2011]. This plant (*Glycyrrhiza glabra* L.), has been also announced as the “Medicinal Plant” of the year 2012 because the extract of this shrub offers not only sweetness but also various pharmacological effects [Bielenberg, 2012].

Liquorice enjoys great popularity especially in the northern countries of Europe, for example, in Scandinavia. The Dutch are the worldwide leaders of liquorice consumption. The annual consumption of those people from the Netherlands is about 2kg per capita. In contrast, the consumption of liquorice in Germany is rather small, about 200g per capita [Batke, 2011]. Nevertheless even in Germany, there is a small tendency of an increasing liquorice consumption in the last few years, which can be seen in Table 1 and Figure 1.

Table 1. Liquorice consumption in Germany from 2010 till 2015 in millions (translated and revised) [VuMA, 08.02.2016]

Years	Several times a week	Once a week	Several times a month	Once a month	Never
2010	1.62	2.33	5.43	4.65	41.11
2011	1.76	2.04	5.2	4.92	40.51
2012	2.26	2.32	5.76	4.91	39.25
2013	2.32	2.53	6.33	4.99	37.91
2014	2.26	2.68	6.28	5.36	37.1
2015	2.01	2.7	6.02	5.54	36.91

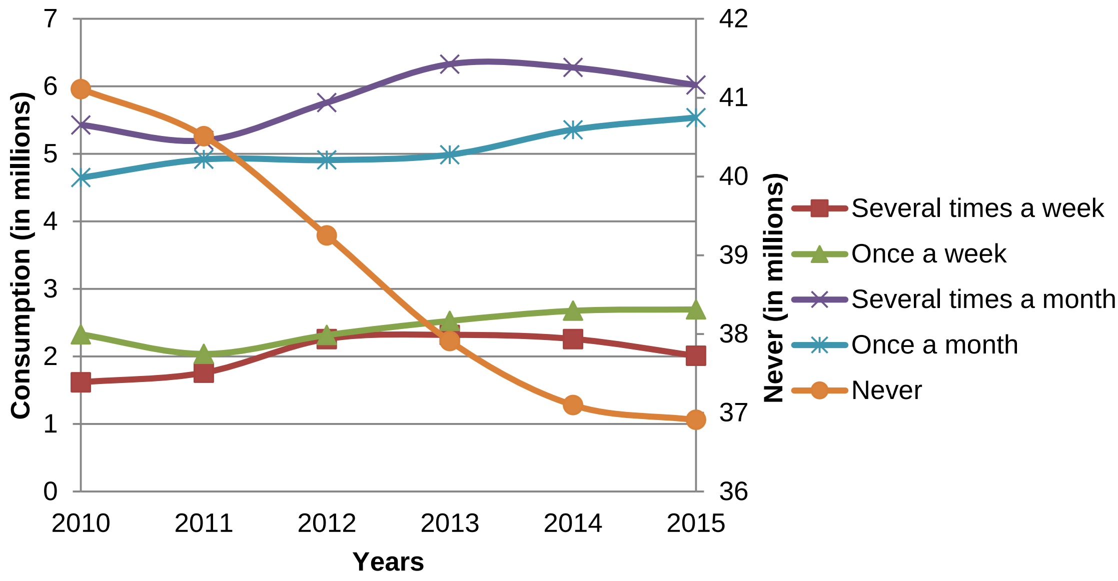

Figure1. Liquorice consumption in Germany from 2010 till 2015 in millions (translated and revised) [VuMA, 08.02.2016]

Due to the adverse health effects of sucrose like obesity, dental caries or hypertension, the replacement of sugar with other sweet compounds such as aspartame, saccharine or steviol glycoside is no longer just a small economic trend. The usage of sweeteners in the area of product development, especially for sweets, is one of the most important driving forces [Mitchell, 2006].

Steviol glycosides belong to the natural high-potency sweeteners which are gaining more and more importance [Mitchell, 2006]. This can also be seen from a survey conducted in Germany about the healthiness of sweeteners, in which steviol glycosides from the stevia plant were ranked third after honey and maple syrup [Lebensmittelzeitung, 2016].
These steviol glycosides are very often used in combination with maltitol syrup, as steviol glycosides are only allowed in confectioneries with no added sugar. That makes them good partners for confectioneries, like liquorice. However, the replacement of sucrose is challenging because sucrose produces not only sweetness but also various physical properties like texture, shelf life, colour or moisture retention [Mitchell, 2006].

The aim of this research was to use steviol glycosides in combination with maltitol instead of sucrose for the production of liquorice with a co-rotating twin screw extruder. With the help of practical tests, the research should find out how the use of steviol glycosides and maltitol affect the texture, the sweetness and the consumer's acceptance of the final product in comparison to the conventional use of sucrose.

2 Liquorice

2.1 Ingredients

2.1.1 Liquorice Extract

Liquorice confectionaries get their taste from the liquorice extract. This kind of extract is obtained by the liquorice shrub that belongs to the *Faboideae,* which is a subfamily of the *Papilionaceae*. The root of this plant contains a glycoside, which is called Glycyrrhizin (Figure 2). This glycoside has a sweetness, which is 50 times stronger than sucrose [Batke, 2011]. This compound is utilized for the production of liquorice, and it gives those candies the special liquorice taste which they are famous for. According to Belitz, [Belitz, 2009] confectionary have to have at least 5% liquorice extract so that they can be indicated as liquorice confectionary. Nevertheless higher quality liquorice products have normally an extract content of at least 30%.

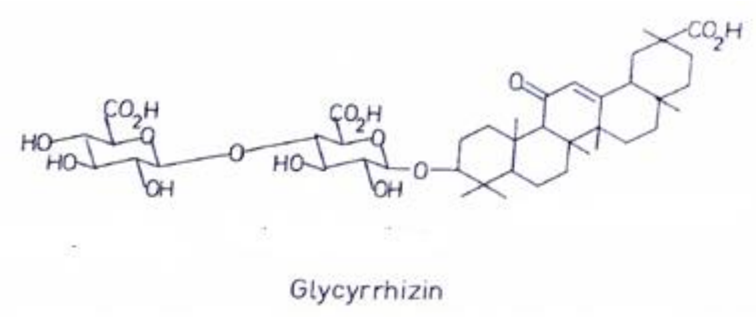

Figure 2. Structure of Glycyrrhizin [Hoffmann, et al, 2002].

For the production of the liquorice extract, the roots of the liquorice shrub have to be peeled, dried and grounded [Batke, 2011]. After that, there is an extraction with hot water, in which the glycoside glycyrrhizin and other substances are separated from the root. The extract is vaporized, and the concentrate is getting tried [Hoffman, et al., 2002].

2.1.2 Sugar

Normally sucrose, invert sugar and/or glucose syrup are used as sugar in the liquorice production. In liquorice confectioneries, the normal amount those sugars is around 50-60% [Minifie, 1997]. Sucrose is a non-reducing disaccharide (Figure 3), which is composed of two reducing monosaccharides fructose and glucose.

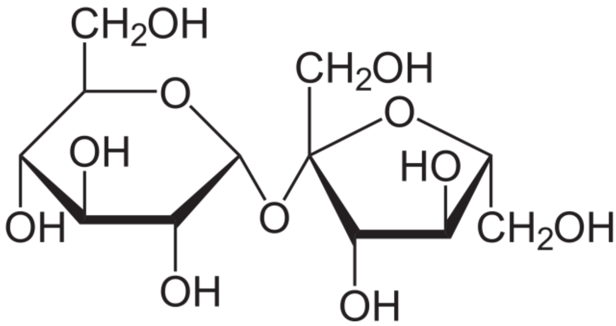

Figure 3. Structure of Sucrose

Invert sugar syrup, in contrast, is a mixture of fructose and glucose. The big advantage of that mixture is that the fructose does not crystalize because the occurrence of crystallization would generate glucose. Thus, it is possible to make an even solution out of invert sugar with a concentration of 80% without the danger of crystallization, which is not possible with sucrose [Edwards, 2000]. However, glucose syrup can be made from almost any source of carbohydrates by hydrolyzing it in the presence of acid. Hence, it is replacing invert sugar because the production cost are much cheaper, and the properties of glucose syrup and invert sugar syrup are pretty similar [Edwards, 2000].
Worth mentioning is that with an increasing amount of invert sugar, the equilibrium moisture content will decrease at the same time. That makes the product more hygroscopic, which means that the final product will absorb more water during the storage.
The same also goes for glucose syrup with a high DE-Value, which means that there are mainly low-molecular sugars instead of polysaccharides because a high amount of soluble saccharides also increases the gelatinization temperature of the starch [Hoffmann, et al., 2002].

2.1.3 Wheat flour

The gelatinization of the starch of the wheat flour is very important for the texture and also for the water binding ability of the liquorice. In liquorice, the amount of flour varies from 30% to 40% [Minifie, 1997]. The higher the degree of the starch gelatinization is, the better the elastic structure and also the glossiness of the final product are. On the

other hand, if the degree of gelatinization is very low, the final product will have a bready consistency, and flavourful faults could also occur.
Flour, which is normally used for liquorice production is wheat flour. To get an idea of the suitability of a wheat flour for liquorice production, the falling number has to be determined. In general, those flours should have a falling number which is not less than 200. That means that these flours should have only a low enzyme activity (α-amylase activity) [Hoffmann, et al., 2002].
The α-amylase activity can vary due to weather conditions during the harvest time. Thus, if it is raining more at that time, the α-amylase activity is normally higher. However, if the α-amylase activity is too high, those enzymes will liquefy the starch and produce a sticky goo during the extrusion process [Edwards, 2000].

2.1.4 Bulking Agents

Bulking Agents for example, polyols are usually necessary if sugar is replaced by intense sweeteners, which have high sweetness potency. The reason for this is that sugar produces not only sweetness but also many different functional properties in confectioneries. Typical functional properties of sugar in confectioneries are sweetness, flavour and aroma, volume, texture, shelf-life, colour or moisture [Mitchell, 2006].
In liquids, sugar can be easily replaced by high potential sweeteners; in that case, water is normally used as the bulking agent. In contrast, solid food products like liquorice require special bulking agents. Hence, high potency sweeteners need bulking agents so that the final product will have properties similar to those of sugar [Mitchell, 2006].

2.1.5 Other Ingredients

Other ingredients which are used in the production of liquorice are molasses (treacle), caramel, thickeners, water and salt [Minifie, 1997]. Salt is normally added to improve the final flavour. Caramel as well as molasses are basically used because of the flavours and the sweetness which they provide for the final product, and thickeners (e.g. gelatine) ,on the other hand, are used, in general, to improve the texture of the liquorices.

Furthermore, for some liquorices, also flavours or glazing agents (e.g. beeswax or vegetable oils or carnauba wax) are added to the final product. The main aim of the glazing agents is to prevent the liquorice confectionaries from sticking together. In addition, they are also important for the visual appearance. Finally, also ingredients like potassium sorbate can be used as preservative to increase the shelf life of the liquorice confectionaries. Due to a wide variety of possible ingredients, a large variety of liquorice products can be found on the market [Jackson, 1995; How products are made, 2016].

2.2 Production of liquorice

2.2.1 Extrusion

The production of liquorice has several processing steps, for example, mixing, cooking, shaping, cooling and cutting. The extruder combines the steps mixing, cooking and shaping in one device. Figure 4 shows a schematic representation of a production line which is using such an extruder for the production of liquorice. The production can be also done batchwise in an open steam heated pot, but this is less efficient than the continuous extrusion process. Therefore, the companies are now switching to using extruder for the production of liquorice [Edwards, 2000; Mercier, et al., 1998; Fellows, 2000].

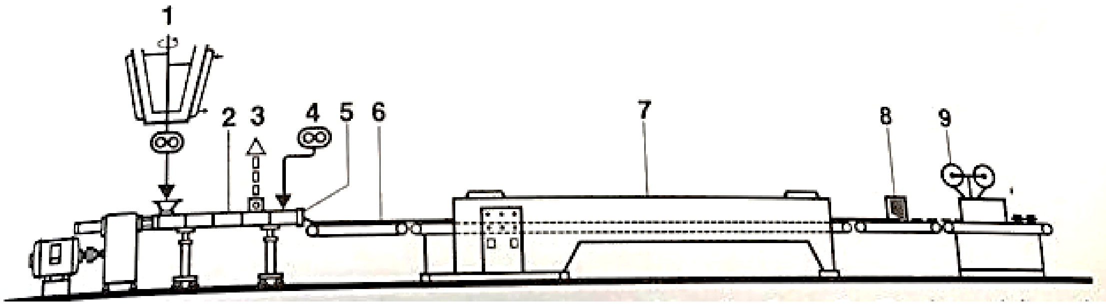

Figure 4. Flow diagram of the liquorice production line [Mercier, et al.,1998]

1=Metering slurry
2=Continua mixing and cooking extruder
3=Venting
4=Metering flavours and colours
5=Die-head
6=Take-off unit
7=Cooling tunnel
8=Cutter
9=Wrapping machine

The continuous production can be achieved by using a scraped surface heat exchanger, which is used to mix and heat up the liquid sugar, wheat flour, liquorice extract, molasses and water [Edwards, 2000]. After heating and mixing in the scraped surface heat exchanger, the ingredients are getting extruded by an extruder. In the extruder, the substances are again getting mixed and cooked but also kneaded under high pressure [Fellows, 2000]. As the product is subjected to atmospheric pressure due to the venting, around 3% of the water flashes off [Edwards, 2000]. If necessary colours and flavours are added to the final product and then the liquorice is cooled down to ambient temperature with the help of a cooling tunnel [Mercier, et al., 1998].

Due to their versatility of application, extruders are used more and more in the food production [Fellows, 2000]. Typically, intermeshing counter-rotating extruders are used for materials with a lower viscosity, for example, liquorice. In these machines those materials can be subjected to low screw speeds that results in a long residence time, which is required for the liquorice production. In addition, the high shear rate also enables the solubilisation of sugar crystals [Frame, 1994]. However, the research of Müller (2012) showed that it is also possible to produce liquorice with a co-rotating twin-screw extruder if liquid sugar is used instead of sugar crystals (Figure 5).

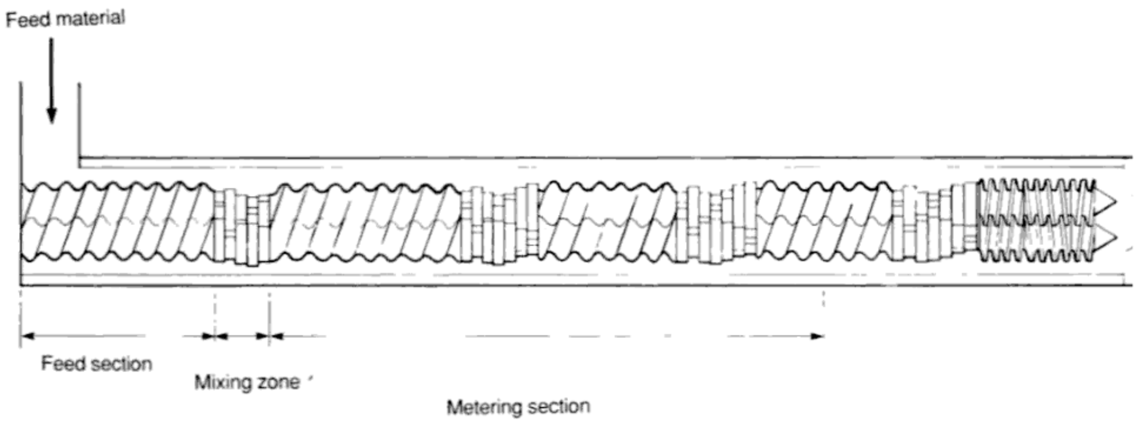

Figure 5. Typical design of a co-rotating twin screw extruder [Frame, 1999].

Figure 5 shows the typical configuration of a co-rotating twin screw extruder with different sections, such as the feed section, the metering section and the mixing zones. The twin screws, which had been used in this research, had the same design as it can be seen in Figure 5.

2.2.2 Structural changes in liquorice doughs during processing

The most important processing step for the texture is the cooking. In this step the fluid mixture is heated up so that starch of the flour is able to gelatinize so that the liquorice gets the desired consistency and texture [Mercier, et al., 1998].

The higher the degree of gelatinization is, the glossier and more elastic the final product is. In contrast, if the degree of gelatinization is relatively low, the final product will have a more bready consistency [Hoffmann, et al., 2002].

It is also important to mention is that during the cooking process, the gelatinization temperature is increasing. The cause for this is the increasing proportion of solubilised saccharides, which leads to an increase of the gelatinization temperature [Hoffman, et al., 2002 and Mercier, et al., 1998].
Another possibility to influence the texture of the final product is to vary the amount of water, which is used for the production. This possibility is common for many different sugar confectioneries [Edwards, 2000].

3 Sweeteners

3.1 Sweeteners and their role in the production of sweets and liquorice

By definition the term sweetener is used for artificial or natural substances with an intense sweet taste. All synthetic sweeteners belong to the group of food additives and have no or very little food energy [Foodlexicon.org, 2016].

Sweeteners are mainly used to replace sucrose and to provide sweetness to all different kind of food products. The market of confectionary, which is using sweeteners instead of sucrose, is growing as the nutritional awareness among the people increases. This trend can be explained due to the adverse health effects of sugar [Nabors, et al, 1986].
The number of people who are suffering from obesity is increasing. Obesity is a serious health problem, which can be traced back to an overconsumption of sugars. This disease goes hand in hand with hypertension, coronary arteriosclerotic heart disease and increased cholesterol in the blood. There are even suggestions that obesity increases the chances for cancer [Nabors, et al, 1986].
Consequently there are many different purposes to use alternative sweeteners, like steviol glycosides or aspartame instead of sucrose. For example the usage of those sweeteners expands the food and beverage choices for nutrition conscious people. Another purpose of sweeteners is to control dental caries because there are substantial evidences that sugar increases the development of dental caries [Nabors, et al, 1986].
However, the choice, which sweetener to take is not only a matter of which one is the healthiest also other properties like technological and economical properties have to be considered to find out if there is a suitability for a particular food [Mitchel, 2006].

Sweeteners have to be used quite often in combination with other sweetener to get a pleasant and comparable taste to sucrose. Normally so called bulk sweeteners, which are mostly polyols (such as Maltitol or Xylitol) have to be mixed with intense sweeteners,

which have a very high sweetness potency. This is necessary because except from maltitol, hydrogenated glucose syrup or Xyltol most of the bulk sweeteners are not sweet enough to replace the sugar if they are used alone. Furthermore a high intake of polyols can often lead to laxative effects [Edwards, 2000].
Important representatives of those sweeteners are aspartame, acesulfame K, saccharine, stevioside or neoesperidine dihydrochalcone (NHDC). Those sweeteners have a high intensity of sweetness and some of them have a sweetness, which is 900 times sweeter than sucrose. Additionally it has to be mentioned that the sweetness intensity of intense sweeteners depends also on the level of use. So with a higher amount of intense sweetener the sweetness potency of the substances is decreasing [Mitchel, 2006].
Some disadvantage of intense sweeteners is that they quite often have bitter or metallic off-flavours. For that they are used quite often with bulk sweeteners (such as maltitol) so that it is possible to hide this off-flavour [Edwards, 2000]. Table 2 shows the relative sweetness for some bulk sweeteners and intense sweeteners according to Mitchel (2006).

Table 2. Relative sweetness of different sweeteners [Mitchel, 2006]

Sweetener	Type of sweetener	Relative sweetness
Sorbitol	Bulk sweetener	0.6
Lactitol	Bulk sweetener	0.4
Maltitol	Bulk sweetener	0.9
Stevioside	Intense sweetener	300
Aspartame	Intense sweetener	180-200
Acesulfame K	Intense sweetener	200

Finally when several sweeteners are used in combination synergy and flavour enhancement can be detected. Especially for aspartame there is a lot of synergy with other bulk or intense sweeteners [Mitchel, 2006]. Synergy means that a mixture of sweeteners provide more sweetness than the same amount of sweeteners if they are used alone [Edwards, 2000].

3.2 Steviol glycosides

3.2.1 Description and technological properties

Based on the opinion of the European Food Safety authorisation (EFSA). Steviol glycosides (E960) are mixtures of steviol glycosides that comprise not less than 95% of steviosides and/or rebaudioside A. Stevioside (Figure 6) is a glycoside which can be found in the plant *stevia rebaudiana* (Bertoni). This plant is mainly found in South America but also cultivated in East and South Asia [Rosenplenter, et al., 2007]. Beside of stevioside, rebaudioside A is another sweetener, which is in the leaves of *stevia rebauiana* (Bertoni). Both sweeteners belong to the group of natural high-potency sweeteners and are the most important sweeteners, which are in this plant [Mitchell, 2006].

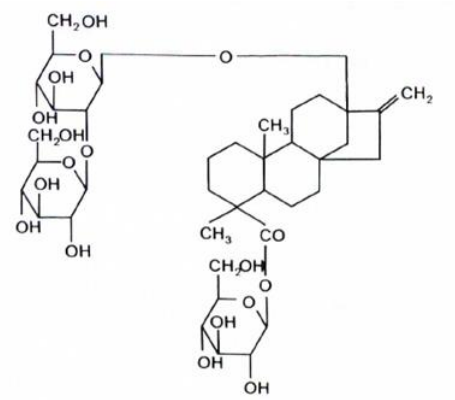

Figure 6. Structure of Stevioside [Rosenplenter et al., 2007]

The table 3 is giving an overview of some steviol glycosides with their relative sweetness that can be found in the stevia plant. According to table 3 it can be seen that the sweetness potency is depending on the polarity of the molecule. A big influence on the polarity in this occasion has the number of hydroxyl groups (-OH). Steviolbioside for example, which has not so many hydroxyl groups has a relatively low polarity and therefore also a low relative sweetness. In contrast Stevioside or Rebaudioside A have a relatively high polarity and so also a higher value of relative sweetness [Mitchel, 2006].

Table 3. Relative sweetness of some steviol glycosides. Sweetness potency measured relative to 0.4% (w/v) sucrose [Mitchel, 2006]

Compound	Relative sweetness
Stevioside	300
Rebaudioside A	250-450
Rebaudioside B	300-350
Dulcoside A	50-120
Steviolbioside	100-125

3.2.2 Regulation

In Japan and South Korea *stevia rebaudiana* (Bertoni) was already approved as a sweetener in 1975. In the EU the approval procedure took much more time, so that the steviol glycosides were finally approved as a food additive (E960) in December 2011. The reason for that was a research in the 1980s, which said that steviol glycosides may cause cancer. However in June 2008, the FAO and the WHO classified steviol glycosides as being harmless [Semler, 2013].

The EFSA determined the ADI-Value (Acceptable Daily Intake) for steviol-equivalents at 0-4mg/kg bodyweight. That leads to an ADI-Value of about 10mg/kg bodyweight for steviol glycosides. This amount is quite low if it is compared to aspartame for example, which has an ADI-Value of 40mg/kg bodyweight [Kienle, 2015].

Nowadays the use of steviol glycosides in food products in the EU is regulated in the Commission Regulation (EU) No 1131/2011. This Commission Regulation determines specific amounts of steviol glycosides, which can be added for different food categories. The maximum level for steviol glycosides of the food category 05.2: *Other confectionery including breath refreshening microsweets*, which is interesting for this liquorice related research is 350mg/kg. It is also said that the steviol glycosides can be only used for confectionary with no added sugar. Therefore, for example in liquorice, it is not allowed to use molasses, because molasses usually has still a very high amount of sugar, which is about 48%, according to the Deutsche Melasse Handelsgeselschaft mbH. Due to this also the liquorice extract has to be without added sugar so that it can be used for the production of liquorice with steviol glycosides.

3.2.3 Commercial sweet/liquorice products containing steviol glycosides

Steviol glycosides can be used for a lot of different products. According to a statistic from 2014, 1.02 million tons of steviol glycosides were used worldwide for food production. Most of the steviol glycosides were used for soft drinks (75.8%) followed by milk products (16.6%), bakery products (6.7%) and other food categories (0.9%) [Enorm Magazin, 2015].

Steviol glycosides are heat stable up to 200°C, which make them perfect suitable for bakery products. Steviol glycosides are also acid stable and not fermentable [Puri et al, 2011]. Table 4 shows some examples of some in Europe produced confectioneries, which are sweetened by steviol glycosides.

Table 4. Confectionaries that contain different sweeteners (Sweeteners are marked as bolded)

Product	Country of manufacture	Ingredients
Sugar free licorice [Halva]	Finland	**Maltitol syrup** Wheat flour Liquorice extract Flavours Thickener (E412) Preservative (E202) Colour (E153) Salt Glazing agents (vegetable oil, E901) **Sweeteners (steviol glycoside)**
Stevi-licorice [Haribo]	Germany	Filler polydextrose Gelatin **Maltitol syrup** Starch Liquorice extract Flavours Cooking salt Glazing agents (beeswax white and yellow, carnauba wax **Natural sweetener: steviol glycosides**
Stevia Gummibärchen [HNK Steviamarkt]	Germany	**Sweetener Maltitol syrup** Gelatin Fruit juice concentrate (apple, pear, peach, grape) Acidifier: malic acid Flavour **Sweetener Steviol glycoside** Glazing agents (vegetable oil) Separating agent: carnauba wax and beeswax
Toms Nellie Dellies Salty Liquorice Stevia [Toms Confectionary Group]	Denmark	Filler polydextrose **Oligofructose** Wheat dextrin Thickener (E414) Gelantin Ammonium chloride Liquorice extract Salt Glazing agents (vegetable oil) Separating agent carnauba wax Flavour **Sweetener steviol glycoside**

3.3 Maltitol syrup

3.3.1 Description and technological properties

Maltitol (Figure 7) belongs to the group of polyalcohols or polyols and it can be manufactured by the hydrogenation of maltose [Nabors, et al., 1986]. It can be also used as a syrup, which is a concentrated solution of maltitol with water. Maltitol, which has the E-number 965, has usually a sweetness potency between 0.6-0.9. As other polyols, maltitol does not take part in Maillard browning reaction, which can be either positive (if browning is not desired) or negative if the browning reaction is important for the final product. The polyol maltitol has due to its molecule structure and its higher amount of hydroxyl groups (-OH) better water binding properties than sucrose. [Mitchel, 2006].

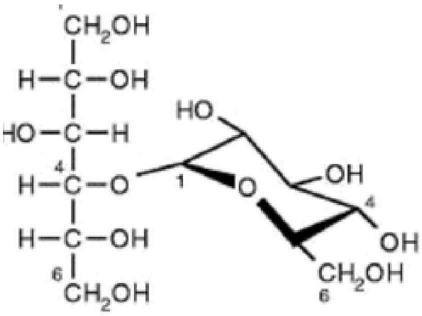

Figure 7. Structure of maltitol

Another aspect, which is worth mentioning is that in contrast to many other polyols like erythritol or xylitol, maltitol has a very low cooling effect, which can be detected if polyols are placed on the tongue. Due to that fact maltitol is often preferred in comparison with the other polyols to replace sucrose [Mitchel, 2006]. Finally as all other polyols maltitol is described as non-cariogenic, because this substance is not able to be fermented by oral bacteria [Mitchel, 2006].

3.3.2 Regulation

In most of the countries of the world, maltitol syrup is permitted for food and pharmaceutical use. Maltitol is a polyol and is therefore considered as a food additive [Mitchel, 2006]. All the food additives are regulated in the directive 89/107/EEC in general. However specific regulations for sweeteners can be found in the directive 94/35/EC. This regulation consists of the permitted amounts of sweeteners, which can be added to different food categories.

According to directive 94/35/EC, maltitol syrup can be used with the principle of *quantum satis* in desserts and similar products and in confectionery. It is also said that maltitol syrup can only be used in “no-added sugar” confectioneries, which makes them good partners for steviol glycosides because they are also only allowed in “no-added sugar” confectioneries. Another thing which is regulated in Article 5 of the directive 94/35/EC is that maltitol has to be clearly marked and there has to be a warning that excessive consumption may induce laxative effects.

4 Material and Methods

4.1 Ingredients

The ingredients used in this study are shown in Table 5. Molasses, which are also normally used as an ingredient in the production of liquorice could not be used for this study, due to the regulation (EC) No 1333/2008.

Table 5. Ingredients

Ingredient	Company	Product
Liquid sugar	Nordic Sugar	Liquid sugar 77
Wheat Flour	Helsingin Mylly Ltd	Wheat Flour
Liquorice extract	Nature MED	Liquorice powder from organic agriculture
Steviol Glycosides	Pure Circle	Alpha (Steviol Glycosides)
Maltitol Syrup	Roquette Nordica Ltd	Lycasin 75/75 Maltitol Syrup
Salt	---	NaCl
Tapped water	---	---

In pre-tests the wheat flour was also compared with full grain spelt wheat flour. Due to the high α-amylase activity of spelt wheat flour, only the wheat flour, which is mentioned in Table 5, was used for production of the liquorice.

The composition of the liquid sugar is according to the company Nordig Sugar: sucrose 23%-31%; glucose 22%-26% and fructose 22%-26% [Noridic Sugar, 2016].
The maltitol syrup which had been used as a bulk sweetener contained 1.4% D-Sorbitol and 73% D-Maltitol (Specification of Roquette Nordica Ltd). This product offers 75% of the sweetness of sucrose [www.Roquette-food.com].

The product of Pure Circle "alpha" has a total steviol glycoside content of more than 95% and the main constituent is rebaudioside A. This product has a sweetness potency of 350. However with an increasing level of use the sweetness potency of the alpha product decrease to a value of 190.

4.2 Characterisation of raw materials

Figure 8 is giving an overview of the tests, which were done to characterize the raw materials. The results of those tests were taken into account, when the final recipes were created.

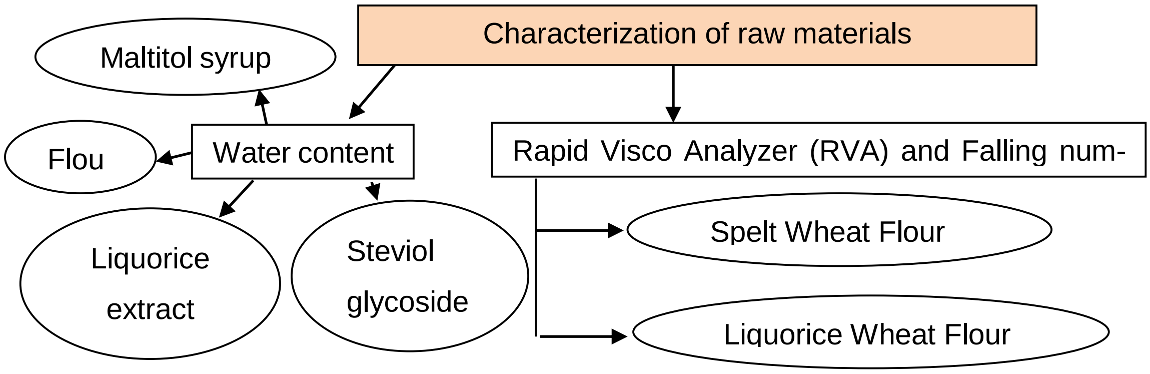

Figure 8. Characterization of raw materials

4.2.1 Determination of the water content

To get the recipes for the final products, it was necessary to determine the water content of the different raw materials (wheat flour, liquorice extract, maltitol syrup, liquid sugar and steviol glycosides). The water content was also needed for the analysis with the Rapid Visco Analyser (RVA^{TM}-device model 4, Newport Scientific Ltd, USA) and for the determination of the falling number (Falling Number 1800, Perten Instrument, Germany).

For the analysation of the water content about 2 to 3g of the wheat flour was dried in an incubator from the company Termaks (130°C) for a period of one hour according to the AACC-Method 44-15A. Afterwards the flour was cooled down to ambient temperature for another hour in a desiccator, which contained phosphorus pentoxide (P_2O_5) as a dehydrating agent. In the end the loss of weight was determined with an analytical balance and thereby also the water content of the flour. The determination was done in triplicate. The water content of the liquorice extract was also measured with the same method like these flours.

The water content of the steviol glycosides and the maltitol syrup was determined with a vacuum incubator (Salvis, 70°C, 20h). For every substance the water content was determined with a three-fold determination. The water content of liquid sugar 77 was taken from the product specification.

4.2.2 Rapid Visco Analyser

The Rapid Visco Analyser (RVA) is used for the testing of starch based products to determine different physical properties, like granule swelling, gelatinisation, pasting and retrogradation [BeMiller et al., 2009 and Newport Scientific, 1998]. In this research this measurement had been done additionally to compare the differences between conventionally wheat flour and full grain spelt wheat.

Before the RVA can be used the right amounts of flour and water had to be calculated. For the calculation of the amount of flour the following formula was needed.

$$S = \frac{86 \cdot 3.5}{100 - M}$$

In this formula *S* is the weight of the flour in grams and *M* is the water content of the flour, which is getting analysed.

The calculation of the amount of water, which has to be added to the flour, is the following.

$$W = 25 + (3.5 - S)$$

In this calculation *W* is the volume of water in ml which has to be added to the flour and *S* is the weight of flour in grams [AACC Method 76-21].

After the right amounts of flour *S* and the right amounts of water *W* had been weighed, the water and the flour was mixed together by putting the stirrer 10 times up and down. The two different flours had been measured with the RVA^{TM}-device model 4 from the company Newport Scientific Ltd. With that measuring device, the generated suspension of flour and water was then subjected to a defined temperature and shear stress program [Newport Scientific, 1998]. For the analyzation of the two flours the program “standard 1” had been used. The temperatures, the speed of the stirrer and the time how long those different actions were lasting can be seen in Table 6.

Table 6. Program Standard 1, which was used with the RVA.

Stage	Temperature/Speed	Program: Standard 1
1	50°C	0 min, 0 sec
2	960rpm	0 min, 0 sec
3	160rpm	0 min, 10 sec
4	50°C	1 min, 0 sec
5	95°C	4 min, 42 sec
6	95°C	7 min, 12 sec
7	50°C	11 min, 0 sec
End of test		13 min, 0 sec

For each flour 3 replicates had been made to get a better accuracy for the results.

With the help of the software, which is used for the RVA, pasting curves had been generated. An example of a pasting curve can be seen in figure 9 below.

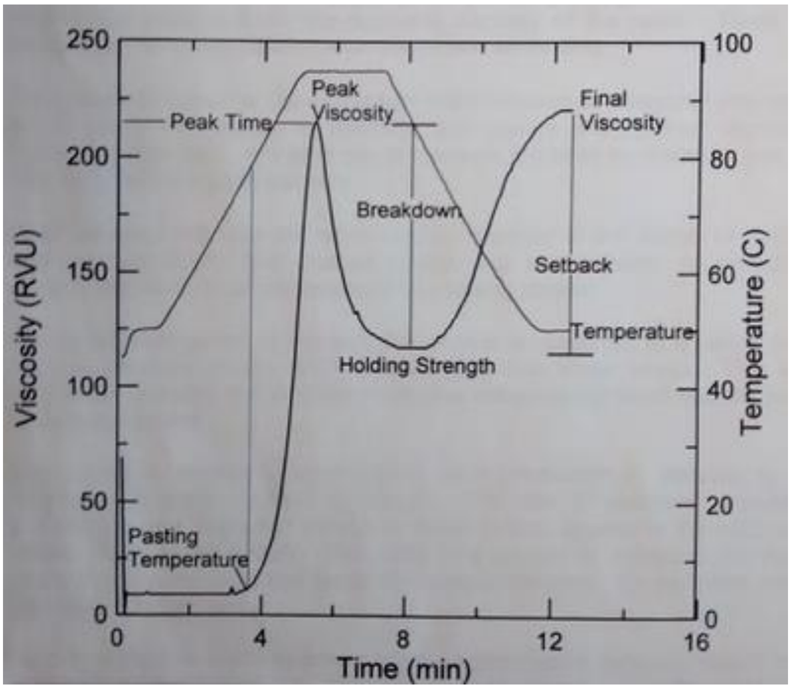

Figure 9. Typical RVA pasting curve showing the common measured parameters [Newport Scientific, 1998]

The pasting curve is giving a lot of different information like the pasting temperature. At that temperature the gelatinization is starting and the starch granules are beginning to swell irreversibly [BeMiller et al., 2009]. Another important fact, which can be seen from the pasting curve, is the peak viscosity. This peak is a measure for the thickening power of a starch, which is for example influenced very much by the present of α-amylase. It also indicates the water-binding capacity of the starch and the peak viscosity is also linked with the final product quality [BeMiller et al., 2009; Newport Scientific, 1998]. Beside those two values (pasting temperature and peak viscosity) also the peak time was selected to characterise the wheat flour. The breakdown, which occurs is due to the alignment of the starch molecules and the setback, weren’t considered for the characterisation [Newport Scientific, 1998].

4.2.3 Falling Number

According to the ICC Standard No. 107/1 of the international association for cereal science and technology the falling number is defined as the time in seconds required to stir and to allow a viscometer stirrer to fall a measured distance through a hot aqueous meal, flour or starch gel undergoing liquefaction due to alpha-amylase activity.

The Falling number is giving information about the α-amylase activity of a flour. The determination of the Falling number is important, because flours, which are used for the liquorice production should have a falling number, which is higher than 200. Otherwise they are not suitable for the production of liquorice, because with a high amount of α-amylase the starch of the flour is not able to build up a proper texture, due to the higher degree of liquefaction [Hoffmann, et al., 2002].

The determination was made with the measurement device “Falling Number 1800” from the company Perten Instrument. The Falling number is based on a flour with a water content of 14%. Therefore the amount of flour, which has to be used depends on the water content. The amount of flour for different water contents can be seen in a table of the ICC Standard No. 107/1.

After the right amount of flour was weighed, the flour was transferred into a dry and clean viscometer tube. Subsequently 25ml of distilled water was added and the viscometer tube, which had been closed with a rubber stopper was shaken vigorously around 40 times in an upright position. After that the rubber stopper was removed and the viscometer stirrer was put inside the viscometer tube. The viscometer tube was put into the water bath and subsequently after 5 seconds the machine started automatically the mixing of the slurry for 55 seconds (2 stirs per second). Then after exact 60 seconds the stirrer was released and the time for a special distance was measured that the stirrer had to go down. The time in seconds which was needed for the stirrer to fall through the aqueous flour together with the time for mixing (60 seconds) is the falling number.

4.3 Recipe

A previous recipe, which had been already used at the University of Helsinki, was modified. Due to the fact that it is not allowed to add additional sugar to confectionery in which steviol glycosides or maltitol is used, the recipe was changed so that there was no more molasses included.

Before using the co-rotating twin-screw extruder, the production was first done in a small scale in a so called kettle test (Figure 10) to see if the proportion of the ingredients especially for the sweeteners like steviol glycosides were suitable for the extrusion. After a few small changes to the estimated recipes, the final recipes (Table 7 and Table 8) had been created, which were then used for the extrusion. The results of the kettle test can be seen in Figure 10

A

B

Figure 10. Results of the kettle test: (A): liquorice with liquid sugar, and (B) liquorice with steviol glycosides and maltitol

The final recipes which were used for the extrusion can be seen in Table 7 and Table 8.

Table 7. Recipe A: liquorice with liquid sugar 77 with different water contents

Ingredients	Water content of liquorice dough (%)		
	21	**23,5**	**26**
Liquid sugar 77 (%)	59.8	57.9	56.1
Wheat flour (%)	34.8	33.7	32.6
Liquorice extract (%)	3.2	3.1	3.0
Salt (%)	0.1	0.1	0.1
Water (%)	2.1	5.2	8,2
Total (%)	100.0	100.0	100.0

Table 8. Recipe B: liquorice with steviol glycosides and maltitol syrup

Ingredients	Water content of liquorice dough (%)		
	21	**23,5**	**26**
Maltitol syrup (%)	61.32	59.38	57.44
Steviol glycosides (%)	0.08	0.08	0.08
Wheat flour (%)	34.79	33.69	32.58
Liquorice extract (%)	3.17	3.07	2.97
Salt (%)	0.11	0.1	0.11
Water (%)	0.53	3.68	6.82
Total (%)	100.00	100.00	100.00

The process diagram of the production trials can be seen in Figure 11.

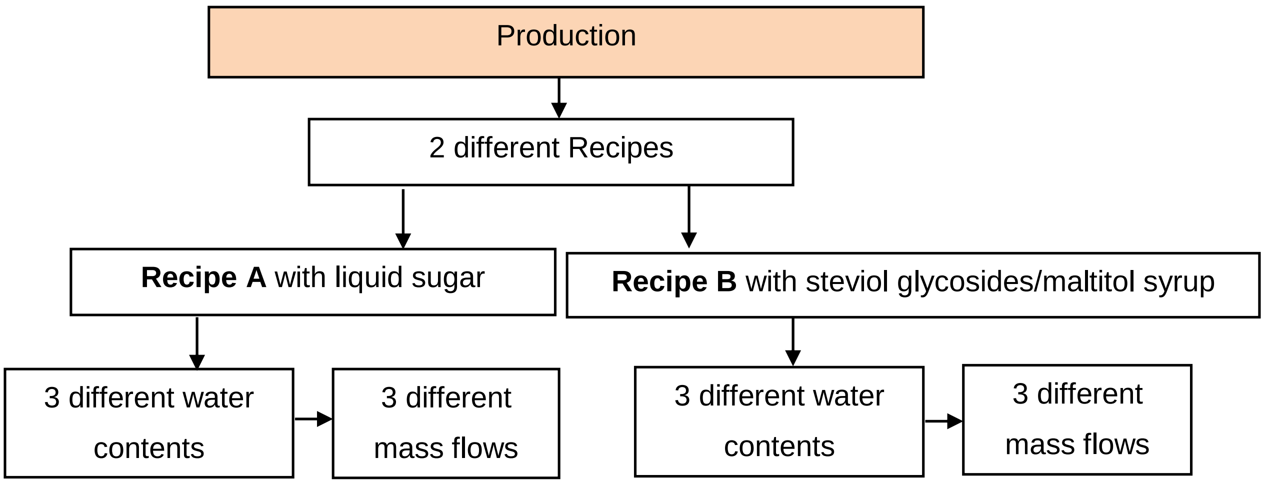

Figure 11. Production Trials

4.4 Extrusion Parameters

Before the production with the extruder, several parameters were determined. To get the right mass flow (70g/min, 100g/min, 130g/min), the corresponding rpm for the peristaltic liquid pump (Watson-Marlow) (liquid part) and also for the flour feeder (Brabender Technologie) (solid part) was necessary. The composition of the solid part was wheat flour and liquorice extract and the composi-tion of the liquid part, which was for the recipe A water, salt and liquid sugar and for the recipe B maltitol syrup, steviol glycoside, salt and water.

The corresponding rpm for a certain mass flow differs, when different substances were used. Due to different compositions the viscosity and the rheological behaviour of the liquid part respectively the solid part are changing, which has an impact on the pumping process [Rao, 2007]. That makes the determination of the rpm for every liquid part and solid part necessary.

To determine the rpm for the desired mass-flow, for several rpms the mass flow was measured. With those values a graph was created in Excel, which described the relation between the mass flow and the rpm for a special liquid or solid composition (Figure 12 and Figure 13)
With the equation, which was given by excel the specific rpm for the special mass flow (70g/min, 100g/min, 130g/min) was calculated. Due limitations of time, the pre-trials for the liquid part were done only with the values of the centre point (23.5% water content).

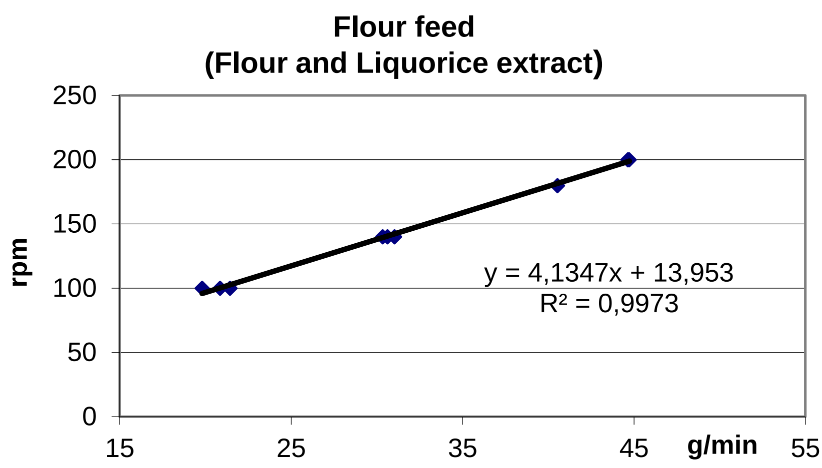

Figure 12. Relation between mass flow and the rpm of the flour feed

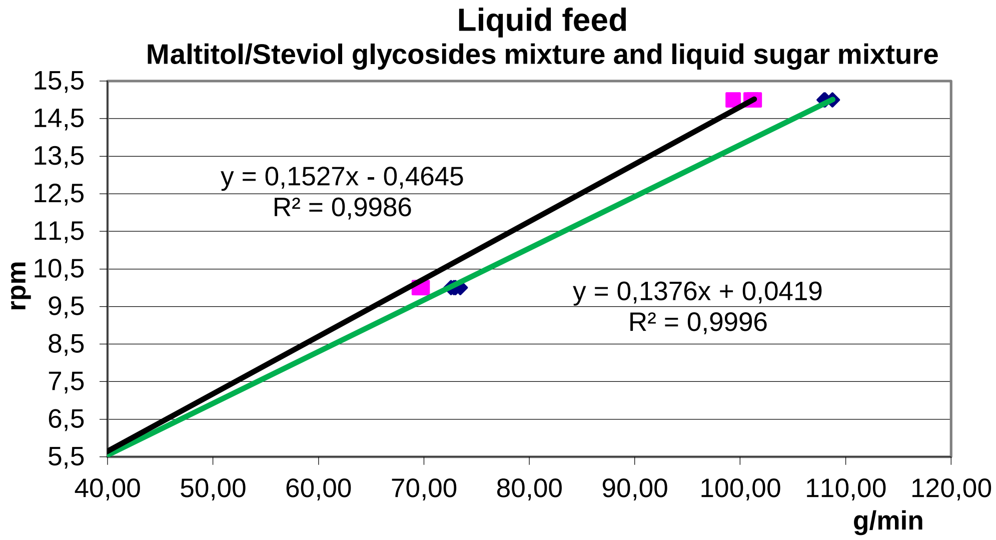

Figure 13. Relation between the mass flow and the rpm of the liquid feet; green line mixture of steviol glycosides and maltitol; black line liquid sugar

4.5 Experimental Design

In total 14 experiments were done, with two different recipes. Recipe A was the recipe in which liquid sugar 77 was used. This recipe was the reference, which was used to compare the difference between liquorice which was produced in the conventionally way with sugar as the sweetening substance and the liquorice in which steviol glycosides was used together with maltitol syrup (recipe B; Table 9).

Table 9: Experimental design of the trials with the co-rotating twin screw extruder (Planning)

Cod-ing	Random-ization	Water content	Mass flow	Recipe
1	2	-1	1	A
2	3	1	-1	A
3	1	-1	-1	A
4	7	0	0	A
5	4	1	1	A
6	6	0	0	A
7	5	0	0	A
8	8	-1	-1	B
9	12	0	0	B
10	9	-1	1	B
11	14	0	0	B
12	10	1	-1	B
13	11	1	1	B
14	13	0	0	B

The two other variables, which were used in this design was the water content and the mass flow. For both variables, 2 levels were used, which is shown in Table 9 as [-1] (lower level) and [1] (higher level). Moreover, for each recipe three centre points were tested [0]. The level of this centre point was lying exactly in middle between the lower level and the higher level. To get more reliable results, the experiments were done in a randomised order. With the values, which had were used in the production the experimental design was looking like this.

Table 10. Experimental design of the trials with the co-rotating twin screw extruder (Production)

	Recipe	Water content [%]	Mass flow to extruder [g/min]	
1	A	21	130	
2	A	26	70	
3	A	21	70	
4	A	23.5	100	Centre point
5	A	26	130	
6	A	23.5	100	Centre point
7	A	23.5	100	Centre point
8	B	21	70	
9	B	23.5	100	Centre point
10	B	21	130	
11	B	23.5	100	Centre point
12	B	26	70	
13	B	26	130	Replicate
14	B	23,5	100	Centre point
16	B	26	130	Replicate
17	B	26	130	Replicate

The trials 16 and 17 were added in the end to get some replicates for the determination of the variance and standard deviation of those experiments. This was necessary because there was some mistake with the centre point, which might could be traced back to weighing mistakes, so it was not possible to get a stable product with a water content of 23.5% and a mass flow 100g/min for the liquorice with steviol glycosides and maltitol syrup.

4.6 Production

The production was done with a co-rotating twin screw extruder (PTW24, Thermo Haake, Polylab System, Germany) as it can be seen in Figure 14. The screws, which had been used for the production had a diameter of 24 mm and a length of 672mm. The die had a diameter of 5mm.

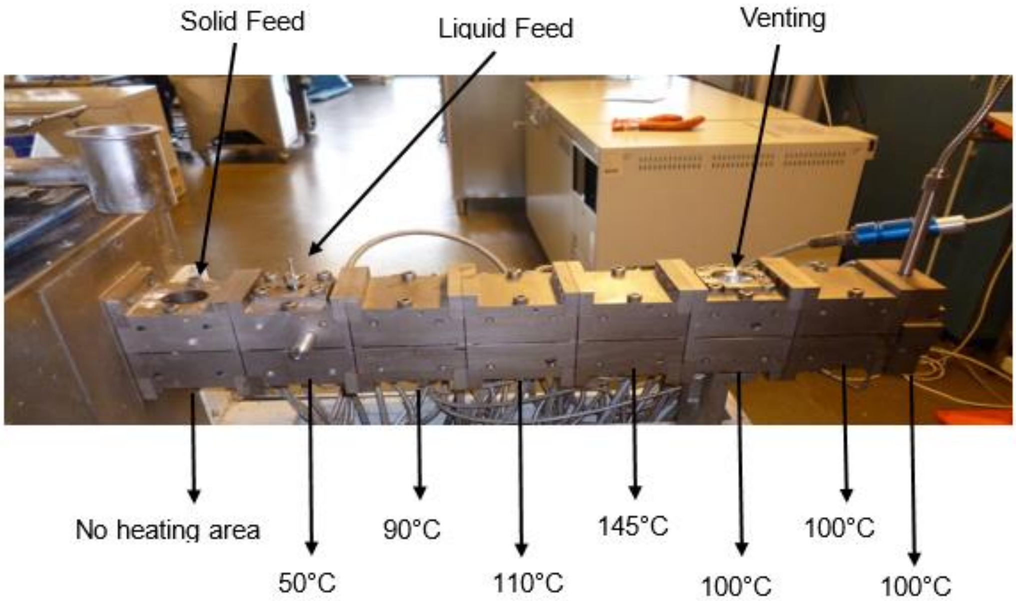

Figure 14. Co-rotating twin screw extruder and the processing temperatures in different sections

The temperature profile used during extrusion, which is shown in Figure 15 can be controlled with a computer program of the company Thermo Haake by “PolyLab System”. The extruder can be divided into seven different sections and the die. In the first section no heating is possible, however for all the other sections and the die heating was possible. With the PolyLab System it was possible to control the temperature for each section individually.

The screw speed was constant (55 rpm) in the trials. The temperatures (Figure 14) was also constant for all trials. The solid part was dosed by a flour feeder (brabender Technology) and the liquid part was added by a peristaltic liquid pump (Watson-Marlow). The batch size for each trial was 7kg. The liquid part was 2kg and of the solid part was 5kg. After the extrusion the liquorice sticks were air dried for about 2 hours and subsequently cut into pieces. All the liquorice samples were packed and the package into plastic bags and the packages were sealed directly afterwards.

4.7 Analysis of the final product

The figure 15 gives an overview of the tests, which were used for the characterisation the produced liquorice, which were produced by the co-rotating twin screw extruder. The results of those measurements for the different liquorices (steiviol glycoside and maltitol or liquid sugar) were afterwards compared with each other.

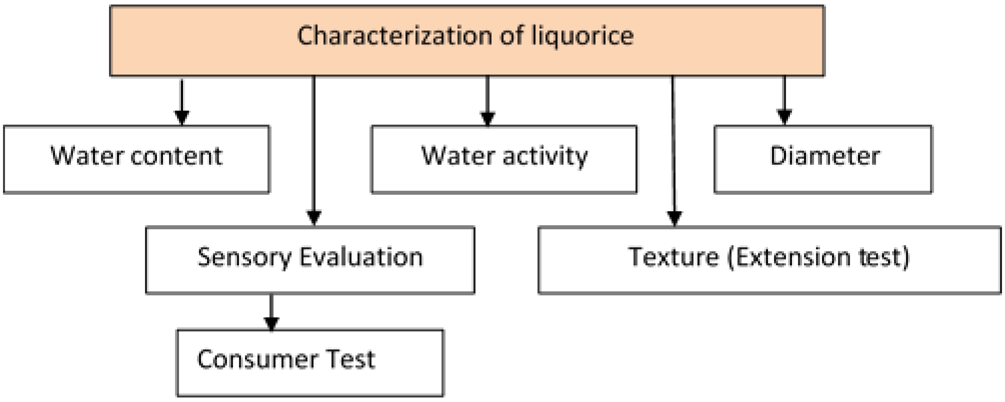

Figure 15. Characterization of liquorice

4.7.1 Water content

The water content of the different liquorice samples were determined in triplicates. In advance the liquorices were cut into small pieces about 5mm to increase the surface for drying and to improve the accuracy of the results (Figure 16). Then for the determination around 3g was weighed on an analytic balance and was subsequently dried in a vacuum incubator (Salvis) for 90 hours at a temperature of 70°C. After that the liquorice samples were also in a desiccator, which contained P_2O_5 (dehydrating agent) for another 72 hours. After that the samples were measured in the first time and then after being again in the desiccator for 96 hours the second time to see if the weight was staying constant.

Figure 16. Liquorice samples which were prepared for drying

4.7.2 Water activity

Water activity is an important value to determine because it is influencing microbial, chemical and physical properties a lot. The properties like stability, flavour, texture or colour are affected a lot by the water activity. If it comes to food preservation the water activity (a_w) is beside the temperature one of the most important values [Barbosa-Cánovas, et al., 2007].

The difference between water activity and water content is that the water content is the total amount of water in the food and the water activity is just a value how much water is bound to food and is therefore not free for chemical or microbiologic reactions [Troller et al., 1987]. Mathematical the water activity can be defined like in this book [Reid, 2007, p.15]:

> Water activity is, at a given temperature, the ratio of the equilibrium partial vapour pressure of water in the system (p_w) to the equilibrium partial vapour pressure $p^0{}_w$ of pure liquid water at the same temperature, which is expressed as:
>
> $$a_w = \left(\frac{p_w}{p^0{}_w}\right)T$$

The measurement of the water activity of the liquorice samples was done with the LabMaster-aw from the company Novasina AG. This instrument is using a special resistive-electrolytic sensor technology to determine the water activity [Novasina AG]. The water activity was measured at a temperature of 25°C.

Because it took quite a lot of time till the partial vapour pressure of water in the system (p_w) was in equilibrium, it was only possible to do 2 measurements for each sample. The measurement for one sample of liquorice took 45 to 60 minutes.

The samples were prepared like those samples for the water content, which means that the liquorices were cut into small pieces to create a higher surface area. Figure 17 shows two filled sample cups for the water activity.

Figure 17. Sample cups for water activity, which are filled with liquorice

4.7.3 Diameter

The diameter of the produced liquorice was measured with a vernier calliper from the company Mahr GmbH (Type: 16ER 150mm / 0.01mm, Germany). To improve the accurateness of the results the diameter was measured 10 times. These measurements were done together with the extension test, because for that method the diameter was needed.

4.7.4 Extension-Test

The texture of the liquorice samples were analysed with the universal testing machine (Instron, Model: 4465, High Wycombe, UK), which can be seen in Figure 18. To be able to make some statement about the texture of the different liquorice samples different attributes (Maximum Load, Extension at the Maximum Load and the Maximum Slope) were measured.

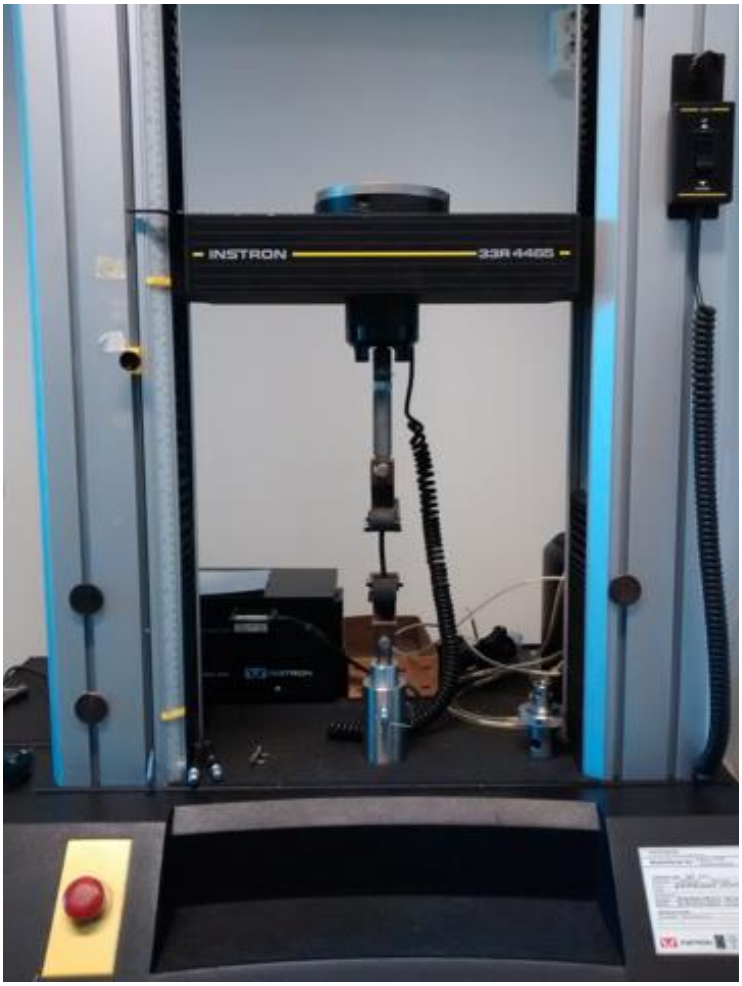

Figure 18. Universal testing machine (Instron, Model: 4465)

The maximum load was the force in Newton, which was required to break the liquorice sample. The program was also giving the maximum extension in mm at this point and the maximum slope of the curve in N/mm as a result. For each liquorice sample 10 different replicates had been determined, to improve the accuracy of the final results. The program used a velocity for the extension of 10mm/min and was stretching them up to a maximum value of 50mm. The start length was 7000mm and the force which was possible to apply was 100N.

Figure 22 shows a typical output of the universal testing machine (Instron, Model: 4465). In that case the output of the liquorice sample 6 is given (liquid sugar; water content 23.5% mass flow 100g/min).

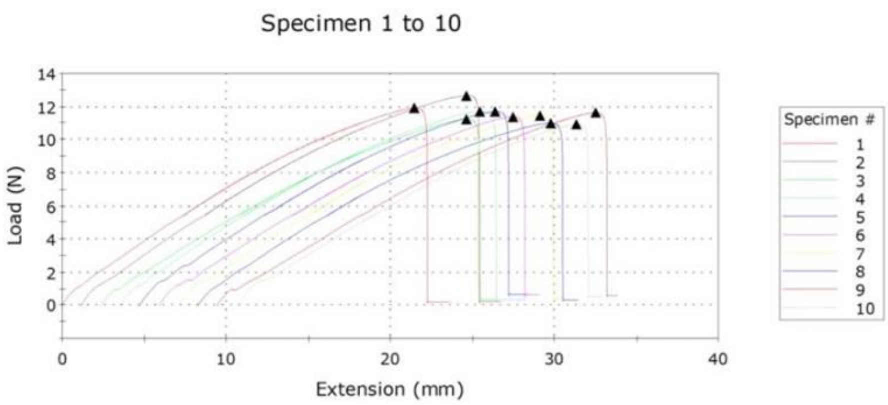

Figure 19. Presentation of the results with Instron (example sample 6)

4.7.5 Consumer test

In the consumer test four different liquorice samples were evaluated against each other. The samples, which had been chosen can be seen in Table11. The centre point of the liquorice with liquid sugar was promising, because of its big diameter and visual appearance and was therefore chosen. Only the liquorice with steviol glycosides and maltitol, which had a water content of 26 % delivered a stable product with similar attributes like those liquorice with liquid sugar. Due to this only those liquorices were taken for the consumer test. Therefore to get comparable results the second liquorice, sweetened by liquid sugar had also a water content of 26%.

Table 11. Liquorice samples, which were used for the consumer test

Sweetener	Water content [%]	Mass flow [g/min]
Liquid sugar	23.5	100
Liquid sugar	26	70
Steviol glycosides and maltitol	26	130
Steviol glycosides and maltitol	26	70

The texture, the sweet-ness, the possible off-flavour and the overall liking of the four different liquorice samples were evaluated. The samples were coded with the tri-digit codes. Also the sample order was randomized for all consumers. The sensory evaluation sheet, which was used in this research can be found in the Appendix 1.

The consumer test was done a week after the production of the liquorice. The test was carried out in the morning at the University of Helsinki and 40 people (n=40), which were asked randomly, if they want to try those liquorice, participate in the consumer test.

4.8 Statistical methods

For the raw material characterization like water content of ingredients, RVA and Falling number only variation and standard deviation of those values were determined.

However for properties like water content, water activity, diameter, maximum load, extension at maximum load and the Young's modulus of the final product a multiple regression analysis was made to see if the parameters water content in the recipe and/or mass flow have an influence on the specific property. If one of the parameters or both have an impact on a certain attribute the regression analysis also shows in which direction the water content or the mass flow changes the characteristic. The multiple regression analysis had been done in with the Microsoft Office program Excel 2013.

Finally the determined values of the consumer test were shown in a bar chart with so called error balks, which have a confidence interval of 95%. That means that the true value is lying in that range of those error balks, with a probability of 95%. The determination of the confidence interval was also done with the Microsoft Office program Excel 2013.
Additionally so called boxplot diagrams had been used, to show the distribution from the evaluation of the properties texture, sweetness and overall liking. The distribution is quite interesting because the opinions of the participants are subjective. Therefore it is good to have an idea how the values are distributed to make an accurate statement, especially if the number of participants is rather small like it is in this case 40. These boxplot diagrams were created with the statistical software "R".

5 Results

5.1 Raw Material Characterization

5.1.1 Water content

Table 12 shows the water contents of the ingredients for the liquorice and the spelt wheat four, which were determined. Unfortunately it was not possible to dry the maltitol syrup completely within the 20 hours at a temperature of 70°C with the vacuum incubator (Salvis). This can be also seen from the high standard deviation 0.2532. Due to this the water content of the specification for the maltitol syrup was used, which was 25%. The specification was also used for liquid sugar 77 from the company Nordic sugar. According to the specification the water content of this liquid sugar was 23%.

Table 12. Water content of ingredients

Substance	Water content [%]	Variance	Standard deviation
Spelt Wheat Flour	9.74	0.00166	0.0407
Liquorice Wheat Flour	13.85	0.00074	0.0271
Liquorice Extract	10.12	0.00064	0.0254
Stevia Extract	5.61	0.00055	0.0235
Maltitol Syrup	19.67	0.0641	0.2532

5.1.2 RVA-Measurement

The Figure 20 shows the results of the Rapid Visco Analyser. The three curves, with the higher peak viscosity are the wheat flour and the other three curves belong to the full grain spelt wheat flour.

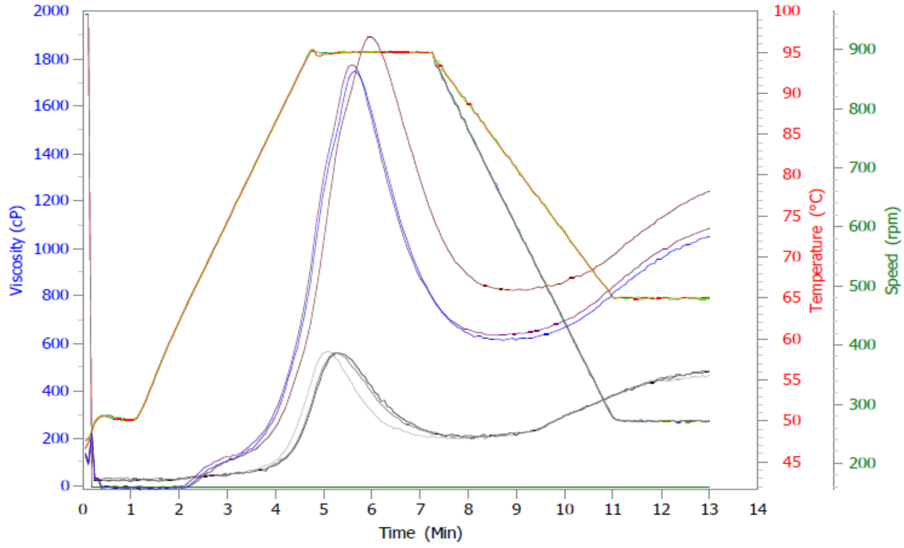

Figure 20. Results of the Rapid Viso Analyzer; smaller peaks: spelt wheat flour; higher peaks: wheat flour

In the Table 13 some important characteristics of the RVA curves are listed in combination with the standard. From that table it can be seen is higher for the spelt wheat flour in comparison to the wheat flour. Due to this the starch of the wheat flour is also starting to gelatinize earlier. However the time, which was needed to reach the viscosity peak was shorter for the spelt wheat flour (5.05 min) in comparison to wheat flour (5.72 min).

Table 13. Peak Viscosity and Pasting Temperature

Flour	Peak Viscosity [cp]	Standard deviation	Pasting Temperature [°C]	Standard deviation
Wheat Flour	1807	75.8046	78.02	8.9959
Spelt Wheat Flour	563	1.7321	89.05	1.6454

5.1.3 Falling Number

Table 14 shows the results of the double determination of the falling numbers for the two different flours. The values of the double determination should not differ more than 5%, otherwise the measurement has to be done again. The liquorice wheat flour had a higher falling number and therefore a lower α-amylase activity than the spelt wheat flour.

Table 14. Falling Numbers of different flours

Flour	1. Measurement	2. Measurement	**Mean Value**	5% Deviation
Liquorice wheat flour	333	336	**335**	16.75 Values are in the range
Spelt wheat flour (full grain)	197	203	**200**	10 Values are in the range

5.2 Characterisation of the final product

5.2.1 Appearance

The optical appearance between those different recipes which had been done with liquid sugar were all looking similar. However in contrast to them the recipes, which had been created with maltitol and steviol glycosides, differed quite much as it can be seen in the Figure 21.

Steviol glycoside/ maltitol (8) WC=21% MF=70g/min	Steviol glycoside/ maltitol (10) WC=21% MF=130g/min	Steviol glycoside/ maltitol (12) WC=26% MF=70g/min	Steviol glycoside/ maltitol (17) WC=21% MF=130g/min	Liquid sugar (7) WC=23.5% MF=100g/min

Figure 21. The visual appearance of diffrent liquorice samples WC=Water content, MF=Mass flow

The liquorice samples (test numbers 8, 10 and 17) produced with steviol glycosides and maltitol, were glossy. In contrast the sample produced with steviol glycosides and maltitol, with a water content of 26% and a mass flow of 70g/min was not glossy at all (test 12). If those 4 samples are compared with the centre point of the recipe with liquid sugar (test 7) it has to be said that under those conditions of sample 12 (water content 26%; mass flow 70g/min) there was barely a difference in the visual appearance to liquid sugar. It has to be also mentioned that those samples, which were glossy (8, 10 and 17) were also very sticky.

5.2.2 Water content

Table 15 shows the water contents of the liquorice after drying, which were produced with liquid sugar. The corresponding standard deviation of the replicates are also shown there.

Table 15. Water content of the produced liquorice Recipe A

Code	Amount of water in Recipe	Mass flow [g/min]	Water content [%]	Standard deviation
3	21%	70	**13.70**	0.1022
1	21%	130	**12.88**	0.1964
2	26%	70	**15.99**	0.1469
5	26%	130	**16.05**	0.2188
4	23.5%	100	**14.18**	0.1733
6	23.5%	100	**14.52**	0.1204
7	23.5%	100	**13.80**	0.1736

Variation of the centre point (4,6,7): 0.1297; standard deviation (4,6,7): 0.3602; Squared average of the measurement standard deviation: 0.0276

After doing a multiple linear regression of the values from Table 15, Microsoft Excel 2013 was given the following output (Table 16).

Table 16. Multiple linear Regression (Water content/Recipe A)

Regression Statistics	
p-Value	0.01038076
Adjusted R Square	0.84717095
Standard Error of the residuals	0.46418515
Observations	7

From those values of Table 15 and 16 it can be seen that for liquid sugar the water content of the final product was increasing with a higher water content in the recipe. However the mass flow didn't show any impacts on the water content.

Table 17. Water content of the produced liquorice (Recipe B)

Code	Amount of water in Recipe	Mass flow [g/min]	Water content [%]	Standard deviation
8	21%	70	**12.22**	0.0420
10	21%	130	**10.15**	0.2196
12	26%	70	**14.02**	0.1455
13	26%	130	**13.47**	0.1318
16	26%	130	**13.44**	0.2776
17	26%	130	**13.60**	0.3970
9	23.5%	100	**11.58**	0.1871
11	23.5%	100	**10.68**	0.0155
14	23.5%	100	**13.35**	0.3363

Variation of the centre point (13,16,17): 0.0072; standard deviation (13,16,17): 0.0850; Squared average of the measurement standard deviation: 0.0524

For the variation, the standard deviation and the linear regression the values of the centre point had been left out, because the variation of those values was too big. Due to that replicates of the sample 13 had been produced (sample 16 and 17).

Table 18. Multiple linear regression (Water content/Recipe B)

Regression Statistics	
p-Value	0,01906891
Adjusted R Square	0.88104046
Standard Error of the residuals	0.49609139
Observations	6

The values for recipe B, which was done with maltitol syrup and steviol glycosides show the same trend that with a higher amount of water in the recipe the water content of the final product was increasing (Table 17).

It can be said that the water contents of the samples, in which maltitol syrup and steviol glycosides were used, are smaller than the samples in which liquid sugar was used as a sweetener.

For the centre point of recipe B the product was not stable, which might explain the big variation between those 3 values. Due to that replicates of the sample 13 were made.

5.2.3 Water activity

Table 19 shows the results of the water activity of the liquorice, which were produced with liquid sugar. The variation of the centre point, the standard deviation and the squared average of the measurement standard deviation are added.

Table 19. Water activity Recipe A

Code	Water content [%]	Mass Flow [g/min]	Water activity
3	21	70	0.66
1	21	130	0.64
2	26	70	0.68
5	26	130	0.71
4	23.5	100	0.67
6	23.5	100	0.66
7	23.5	100	0.67

Variation of the centre point (4,6,7): $5.75 \cdot 10^{-5}$; standard deviation (4,6,7): 0.0076; Squared average of the measurement standard deviation: 0.0002

Table 20. Multiple linear regression (water activity/Recipe A)

Regression Statistics	
p-Value	0.07174745
Adjusted R Square	0.59821429
Standard Error of the residuals	0.01369306
Observations	7

It can be seen that both parameter water content and mass flow had a big influence on the water activity if the liquorice samples were produced with liquid sugar, due to the fact that the values are not varying very much from 0.64 to 0.71 (Table 19). The p-value which is with 0.07 is also quite high, which means that the values for the water activity could vary randomly around the average value (Table 20).

Table 21. Water activity Recipe B

Code	Water content [%]	Mass Flow [g/min]	Water activity
8	21	70	0.64
10	21	130	0.56
12	26	70	0.73
13	26	130	0.72
16	26	130	0.68
17	26	130	0.73
9	23.5	100	0.57
11	23.5	100	0.50
14	23,5	100	0.63

Variation of the centre point (13,16,17): 0.0725; standard deviation (13,16,17): 0.0239; Squared average of the measurement standard deviation: 0.004

Table 22. Multiple linear regression (water activity/Recipe B)

Regression Statistics	
p-Value	0.03651231
Adjusted R Square	0.81656805
Standard Error of the residuals	0.02875181
Observations	6

For the recipe with maltitol syrup and steviol glycosides it can be also concluded, that neither the water content nor the mass flow have a big influence on the water activity of the final product (Table 21). The p-Value is with 0.0365 lower than the p-Value of the recipe with liquid sugar but still it is not possible to make a well-founded statement (Table 22). The values between those two recipes also do not differ much.

5.2.4 Diameter

The properties for diameter couldn’t be measured for the centre point of the liquorice recipe which was produced with steviol glycosides and maltitol. Due to some mistake, which might resulted by weighing mistakes, it was impossible to get a stable product of which the diameter could have been measured. Due to this, the measurement of the texture for the centre point of liquorice with steviol glycoside and maltitol, was also not done. Figure 22 is showing the centre point of the liquorice with steviol glycolsides and maltitol (right) and a stable liquorice extrusion product as it is supposed to be (left).

Figure 22. Liquorice Extrusion products
Left picture: Normal liquorice extrusion product (Sample 1)
Right picture: Centre point of recipe with steviol glycosides and maltitol

Table 23. Diameter of produced liquorice (Recipe A)

Code	WC [%]	MF [g/min]	diameter [mm]	Standard deviation
3	21	70	6.23	0.13693
1	21	130	5.35	0.19722
2	26	70	5.81	0.39153
5	26	130	5.76	0.30714
4	23.5	100	6.28	0.25251
6	23.5	100	6.37	0.07345
7	23.5	100	6.21	0.14894

Variation of the centre point (4,6,7): 0.0064; standard deviation (4,6,7): 0.0802; Squared average of the measurement standard deviation: 0.0567

Table 24. Multiple linear regression (diameter/Recipe A)

Regression Statistics	
p-Value	0.54609383
Adjusted R Square	-0.10872443
Standard Error of the residuals	0.39122746
Observations	7

For the diameter of those liquorices, which were produced with liquid sugar, it can be said that both water content and mass flow did not have a big influence on the diameter. The results show that the difference between the maximum value and the minimum value is just 1.02 mm, which is rather small (Table 23). However small tendencies can be seen that with a higher mass flow for both water contents (21%, 26%) the diameter was decreasing. Unfortunately the p-Value was very high with 0.546, which means that those values can be also just random (Table 24).

Table 25. Diameter of produced liquorice (Recipe B)

Sample	WC [%]	MF [g/min]	diameter [mm]	Standard deviation
8	21	70	3.07	0.35781
10	21	130	3.67	0.23713
12	26	70	6.45	0.08393
13	26	130	5.21	0.10293
16	26	130	5.09	0.21112
17	26	130	5.01	0.14070

Variation of the centre point (13,16,17): 0.0101; standard deviation (13,16,17): 0.1007; Squared average of the measurement standard deviation: 0.0444

Table 26. Multiple linear regression (diameter/Recipe B)

Regression Statistics	
p-Value	0.06345173
Adjusted R Square	0.73485848
Standard Error of the residuals	0.62105287
Observations	6

The same thing cannot be said for the diameter of the liquorices, which were produced with maltitol syrup and steviol glycoside. Because here it can be clearly seen that with an increase of the water content the diameter of those liquorices was also increasing. The mass flow could also have an influence on the diameter but it seems like that the direction of the impact depends on the water content because with a small water content of 21% the diameter is increasing with a higher mass flow. In contrast if the water content is 26% the diameter is decreasing if the mass flow is increasing.
It can be also seen that liquorices, with steviol glycosides and maltitol syrup had smaller diameters in comparison to liquorices, with liquid sugar at a water content of 21%.

5.2.5 Texture (Extension)

Table 27 shows the results of the maximum load for liquorices, which were sweetened by liquid sugar. The standard deviation of the 10 different replicates which were measured can be also seen.

Table 27. Maximum Load (Recipe A)

Code	WC [%]	MF [g/min]	Maximum Load [N]	Standard deviation
3	21	70	14.11	0.4698
1	21	130	3.55	0.3479
2	26	70	10.06	0.5831
5	26	130	6.55	0.6629
4	23.5	100	10.02	0.3117
6	23.5	100	11.57	0.4943
7	23.5	100	11.33	0.6939

Variation of the centre point (4,6,7): 0.7108; standard deviation (4,6,7): 0.8431; Squared average of the measurement standard deviation: 0.2777

Table 28. Multiple linear regression (Maximum Load/Recipe A)

Regression Statistics	
p-Value	0.10430538
Adjusted R Square	0.51362590
Standard Error of the residuals	2.44335432
Observations	7

The mass flow has an impact on the maximum load, if the liquorice had been produced with liquid sugar (Table 27). A smaller mass flow with 70g/min leads to higher results for the maximum load than a higher mass flow with 130g/min. This trend can be seen for liquorice, which had been produced with a water content of 21% as well for liquorice with a water content of 26%. The water content has also an impact on the maximum load. The highest results have been achieved with a water content of 21% and a mass flow of 70g/min. In contrast to that the lowest values have been also achieved with a water content of 21% and a higher mass flow of 130g/min.

From the values in Table 27 it seems that the impact of the mass flow depends on the water content, because with a higher water content (26%) the differences of the maximum load between the higher and the lower mass flow are not that big anymore.

Table 29. Maximum Load (Recipe B)

Code	WC [%]	MF [g/min]	Maximum Load [N]	Standard deviation
8	21	70	0.36	0.1255
10	21	130	1.22	0.3094
12	26	70	11.38	0.6244
13	26	130	3.07	0.4150
16	26	130	1.95	0.2090
17	26	130	2.08	0.2747

Variation of the centre point (13,16,17): 0.3572; standard deviation (13,16,17): 0.6126; Squared average of the measurement standard deviation: 0.1321

Table 30. Multiple linear regression (Maximum Load/Recipe B)

Regression Statistics	
p-Value	0.222829573
Adjusted R Square	0.387418371
Standard Error of the residuals	3.162028885
Observations	6

If the liquorice is produced with maltitol syrup and steviol glycosides, it can be seen that for those liquorice the water content is playing an important role (Table 29). With a low water content (21%) the values which had been determined for the maximum load were very small. By increasing the water content to 26% the values for the maximum load are also increasing. The water content of 26% shows the same trend for the mass flow like it was already seen for the liquorice, which were produced with liquid sugar. So that the maximum load is increasing with a lower mass flow.

A very noticeable aspect is, that the values, for the liquorice samples, which were produced with liquid sugar (Table 27) are significant higher than those measured values of the liquorices, which were sweetened with maltitol syrup and steviol glycosides (Table 29). The only exception on this occasion was the liquorice with maltitol syrup and steviol glycosides with a water of 26% and a mass flow of70g/min (test 12). This sample shows similar values in comparison to the liquorice with liquid sugar.

Table 31. Extension at maximum load (Recipe A)

Code	WC [%]	MF [g/min]	Extension at maximum load [mm]	Standard deviation
3	21	70	29.12	1.7404
1	21	130	11.87	1.1506
2	26	70	22.19	0.9495
5	26	130	13.62	1.6620
4	23.5	100	21.66	0.7220
6	23.5	100	21.94	0.7986
7	23.5	100	22.88	1.1935

Variation of the centre point (4,6,7): 0.4084; standard deviation (4,6,7): 0.6391; Squared average of the measurement standard deviation: 1.5143

Table 32. Multiple linear regression (Extension at maximum Load/Recipe A)

Regression Statistics	
p-Value	0.027773833
Adjusted R Square	0.750017750
Standard Error of the residuals	2.944158866
Observations	7

For the extension at maximum load the measured values (Table 31 and 32) are showing the same trend like those values of the maximum load. Due to this it is very likely that those two parameters maximum load and extension at maximum are related to each other.

Table 33. Extension at maximum load (Recipe B); WC=Water content, MF=Mass flow

Code	WC [%]	MF [g/min]	Extension at maximum load [mm]	Standard deviation
8	21	70	13.88	1.6937
10	21	130	12.40	1.1064
12	26	70	36.87	3.7275
13	26	130	13.50	1.0665
16	26	130	13.12	0.7067
17	26	130	12.84	1.2993

Variation of the centre point (13,16,17): 0.1097; standard deviation (13,16,17): 0.3313; Squared average of the measurement standard deviation: 3.5520

Table 34. Multiple linear regression (Extension at maximum Load/Recipe B)

Regression Statistics	
p-Value	0.177561152
Adjusted R Square	0.473480995
Standard Error of the residuals	7.037051229
Observations	6

The result for extension at maximum load for liquorices, which were produced with steviol glycosides and maltitol syrup show the tendency that with an increasing mass flow the extension at maximum load is decreasing (Table 33). The water content had only an influence in the way that with a higher water content (26%) the impact of the mass flow was much stronger than with the water content of 21%. However despite of the sample, with a water content of 26% and a mass flow of 70g/min, it does not seem that those values are affected much by the water content. It has to be mentioned that the standard deviation of the measurements, were very high which had to be considered when looking at the values.

Table 35. Young's modulus (Recipe A); WC = Water content, MF=Mass flow

Code	WC [%]	MF [g/min]	Maximum Slope (stiffness) [N/mm]	Standard deviation
3	21	70	0.697	0.0840
1	21	130	0.568	0.0458
2	26	70	0.744	0.0992
5	26	130	0.765	0.0630
4	23.5	100	0.664	0.0712
6	23.5	100	0.703	0.0676
7	23.5	100	0.672	0.0509

Variation of the centre point (4,6,7): 0.0005; standard deviation (4,6,7): 0.0219; Squared average of the measurement standard deviation: 0.0050

Table 36. Multiple linear regression (Young's modulus/Recipe A)

Regression Statistics	
p-Value	0.076437422
Adjusted R Square	0.585290224
Standard Error of the residuals	0.041236253
Observations	7

The maximum slope is a value, which describes the stiffness of the liquorice. For the liquorices, which were sweetened by liquid sugar the values of the maximum slope are quite close to each other with the lowest value of 0.568 and the highest value of 0.765 (Table 35). So it is very likely the neither mass flow nor the water content had an impact on the stiffness of those liquorice.

Table 37. Young's modulus (Recipe B); WC = Water content, MF=Mass flow

Code	WC [%]	MF [g/min]	Maximum Slope (stiffness) [N/mm]	Standard deviation
8	21	70	0.036	0.0115
10	21	130	0.143	0.0395
12	26	70	0.500	0.0513
13	26	130	0.391	0.0581
16	26	130	0.216	0.0167
17	26	130	0.258	0.0239

Variation of the centre point (13,16,17): 0.0083; standard deviation (13,16,17): 0.0914; Squared average of the measurement standard deviation: 0.0014

Table 38. Multiple linear regression (Young's modulus/Recipe B)

Regression Statistics	
p-Value	0.194401383
Adjusted R Square	0.440695495
Standard Error of the residuals	0.125375702
Observations	6

The values of the maximum slopes of the liquorices, which were produced with malttol syrup and liquid sugar, show in contrast to the values of the liquorices with liquid sugar bigger differences from 0.036N/mm to 0.5 N/mm. At least for those liquorice the water content seemed to have an influence on their stiffness. So that with a higher water content also the maximum slope is increasing. However the p-value is quite high with a value of 0.194 so that those changes unfortunately can be also random.

Table 39 shows all the considered models for the different response variables (water content, water activity, diameter, maximum load, extension at maximum load, extension at maximum load and Young's modulus) The star at the number shows if the value is significant at a 0.05 level of significance.

Table 39. Formulae to all considered models. x_1= water content; x_2=mass flow; A=Recipe A (Liquid sugar); B=Recipe B (Maltitol syrup and steviol glycosides)

$$\boldsymbol{Water\ content\ (A) = 2.25 + 0.55^{(*)} \cdot x_1 - 0.006 \cdot x_2}$$

$$\boldsymbol{Water\ content\ (B) = 1.61 + 0.54^{(*)} \cdot x_1 - 0.018 \cdot x_2}$$

$$\boldsymbol{Water\ activity\ (A) = 0.45 + 0.009^{(*)} \cdot x_1 + 8{,}33 \cdot 10^{-5} \cdot x_2}$$

$$\boldsymbol{Water\ activity\ (B) = 0.144 + 0.0252^{(*)} \cdot x_1 - 0.00073 \cdot x_2}$$

$$\boldsymbol{Diameter\ (A) = 6.80^{(*)} - 0.001 \cdot x_1 - 0.00775 \cdot x_2}$$

$$\boldsymbol{Diameter\ (B) = -4.97 + 0.4424^{(*)} \cdot x_1 - 0.0095 \cdot x_2}$$

$$\boldsymbol{Maximum\ Load\ (A) = 23.79 - 0.105 \cdot x_1 - 0.1173^{(*)} \cdot x_2}$$

$$\boldsymbol{Maximum\ Load\ (B) = -12.17 + 1.02 \cdot x_1 - 0.084 \cdot x_2}$$

$$\boldsymbol{Extension\ at\ maximum\ load\ (A) = 54.16^{(*)} - 0.518 \cdot x_1 - 0.215^{(*)} \cdot x_2}$$

$$\boldsymbol{Extension\ at\ maximum\ Load\ (B) = -2.68 + 1.93 \cdot x_1 - 0.247 \cdot x_2}$$

$$\boldsymbol{Stiffness\ (A) = 0.20 + 0.024^{(*)} \cdot x_1 - 0.0009 \cdot x_2}$$

$$\boldsymbol{Stiffness\ (B) = -0.92 + 0.055 \cdot x_1 - 0.0014 \cdot x_2}$$

According to these models the water contents of the final product was significantly influenced by the water content in the recipe. The mass flow did not have any significant influence. The water activity was also only significantly influenced by the water content in the recipe. However the impact of the water content was not very big.
The diameter of the liquorice recipes, which were produced with liquid sugar was not influenced by both variables. In contrast to that the liquorice with steviol glycosides and maltitol syrup was influenced a lot by the water content.

The maximum load of the liquorice with liquid sugar was only significantly affected by the mass flow. In contrast to that no variable was affecting significantly the maximum load of the liquorice with steviol glycosides and maltitol. However the water content seemed like that it had a strong influence on those liquorices. The same thing is also valid for the extension at maximum load. Due to this it seemed that those values are very likely influenced by each other. The stiffness of the liquorices, which were produced with liquid sugar was only significantly influenced by the water content. However this influence did not have a big impact. The stiffness of the liquorice with steviol glycoside and maltitol syrup was not influenced significantly.

Finally it has to be said that the p-value (in Excel Significance F) was quite high for every response variable. Only the p-Values for the final water content were in a range of 0.01. Due to this for the other response variables unfortunately it can be only spoken about tendencies but not significant differences, because the results could be also random

5.2.6 Sensory consumer test

Description of the consumer group: In total 40 people participated in the consumer test. More women (72.5%) took part in the sensory evaluation than men (27.5%) and the consumers were in an age between 20 and 61. 2.5% of the test persons ate liquorice almost every day. Another 15% were eating liquorice weekly. The biggest group were the people, who ate liquorice monthly with 50% and 32.5% were eating liquorice less than once a month. With those people the attributes texture, sweetness and overall liking were evaluated.

Table 40 shows the evaluation of the texture. Table 41 shows the evaluation of the sweetness and Table 42 shows the overall liking of the liquorice samples. These Tables are also showing the confidence interval of 95%. Additionally barcharts and boxplot diagrams had been used to exemplify the results.

Table 40. Texture (Sensory Evaluation) WC=Water content; MF=Mass Flow

Sample	Texture intensity	Confidence Interval 95%
Liquid sugar (WC= 23%; MF=100g/min)	**4.55**	0.6873
Liquid sugar (WC= 26%; MF=70g/min)	**4.125**	0.6910
Maltitol/steviol glycoside (WC=26%; MF=130g/min)	**2.125**	0,5121
Maltitol/steviol glycoside (WC=26%; MF=70g/min)	**4.7**	0,6912

Size of test panel 40 people (n=40); 1=soft / 6=hard

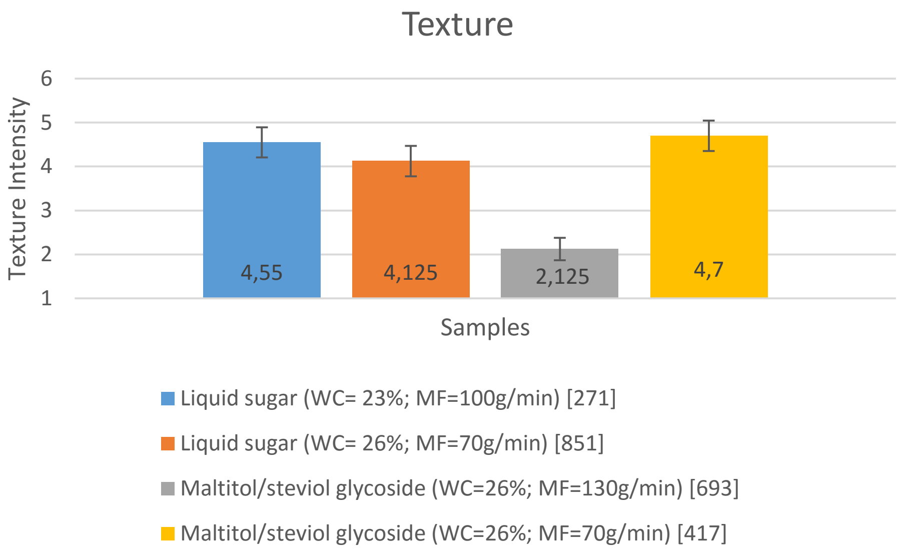

Figure 23. Barchart of the texture evaluation; n=40; 1=soft / 6=hard;confidence interval 95%

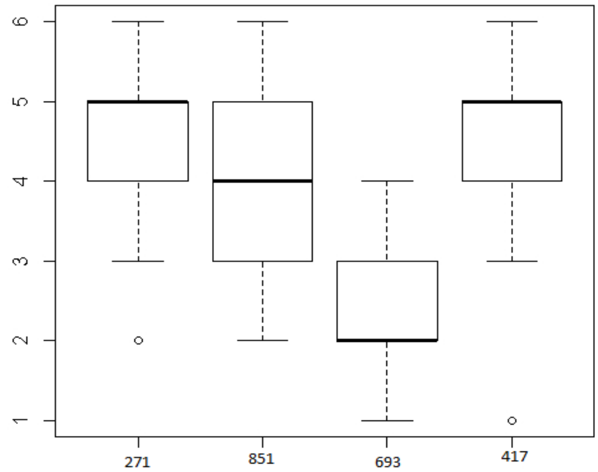

Figure 24. Boxplot diagram, which shows the distribution of the values for texture in quartiles; n=40; 1=soft / 6=hard;
271=Liquid sugar (WC=23%; MF=100g/min);
851=Liquid sugar (WC=26%; MF=70g/min);
693=maltitol/steviol glycoside (WC=26%; MF=130g/min)
417=maltitol/steviol glycoside (WC=26%; MF=70g/min)

The texture was evaluated for all samples quite similar except of one liquorice sample, which was produced with steviol glycoside and maltitol syrup with a water content of 26% and a mass flow of 130g/min. This sample was the only one, which was rated quite low with a value of 2.1 (Table 40/Figure 23 and 24). The other samples instead were rated significantly higher. Those values were in a range from 4.1 till 4.7.

Table 41. Sweetness (Sensory Evaluation); WC=Water content; MF=Mass Flow

Sample	Sweetness	Confidence interval 95%
Liquid sugar (WC= 23%; MF=100g/min)	**3.05**	0,6714
Liquid sugar (WC= 26%; MF=70g/min)	**3.00**	0,7186
Maltitol/steviol glycoside (WC=26%; MF=130g/min)	**3.15**	0,8108
Maltitol/steviol glycoside (WC=26%; MF=70g/min)	**2.725**	0,8552

Size of test panel 40 people (n=40); 1=weak / 6=strong

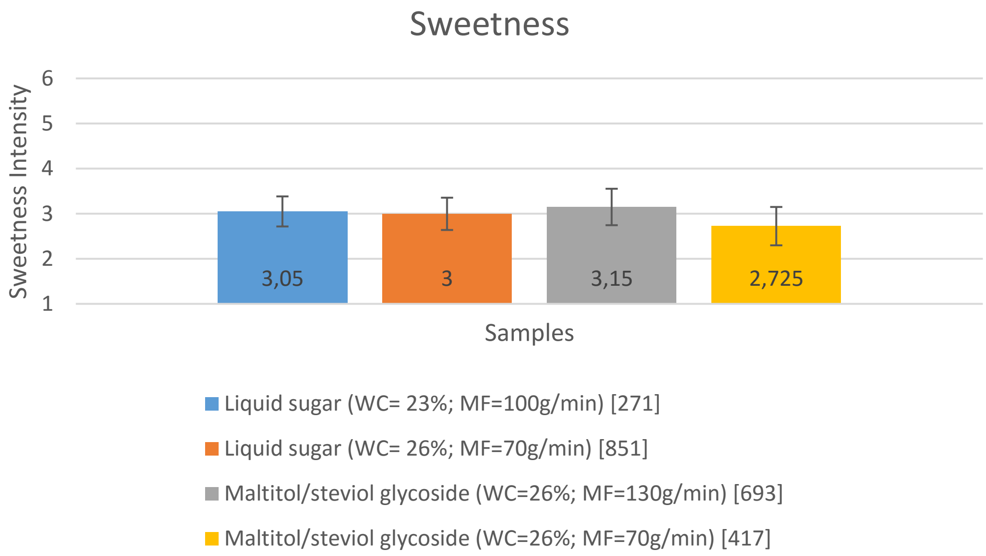

Figure 25. Barchart of the sweetness evaluation; n=40; 1=weak / 6=strong;confidence interval 95%

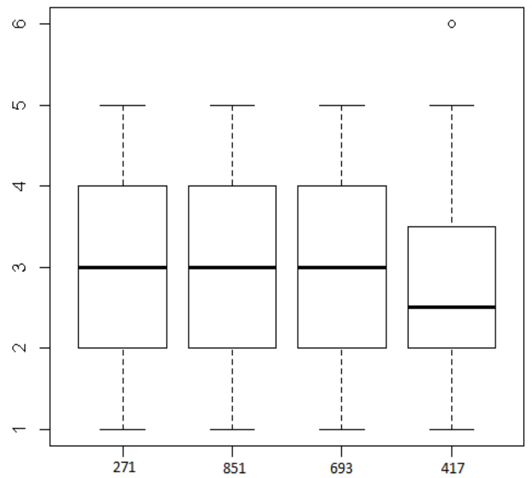

Figure 26. Boxplot diagram, which shows the distribution of the values for texture in quartiles; n=40; 1=weak / 6=strong;
271=Liquid sugar (WC=23%; MF=100g/min);
851=Liquid sugar (WC=26%; MF=70g/min);
693=maltitol/steviol glycoside (WC=26%; MF=130g/min)
417=maltitol/steviol glycoside (WC=26%; MF=70g/min)

The sweetness which was evaluated from the participants was almost the same, so the test persons did not taste the difference in sweetness if the liquorices were sweetened with liquid sugar or with steviol glycosides and maltitol (Table 41). So with the help of the sweetness factor chart it was possible to create a graph and estimate the right proportion of steviol glycosides to get the same sweetness like liquid sugar.
However it has to be mentioned that the statements for the sweetness evaluation of the liquorice samples, which were produced with steviol glycosides and maltitol, had a bigger variation, than those which were produced with liquid sugar.

Table 42. Overall liking (Sensory Evaluation)

Sample	Mean Value of Evaluation	Confidence interval 95%
Liquid sugar (WC= 23%; MF=100g/min)	**3.05**	0.7473
Liquid sugar (WC= 26%; MF=70g/min)	**3.08**	0.8348
Maltitol/steviol glycoside (WC=26%; MF=130g/min)	**3.7**	0.8703
Maltitol/steviol glycoside (WC=26%; MF=70g/min)	**2.7**	0.6912

Size of test panel 40 people (n=40); 1=not much / 6=very much

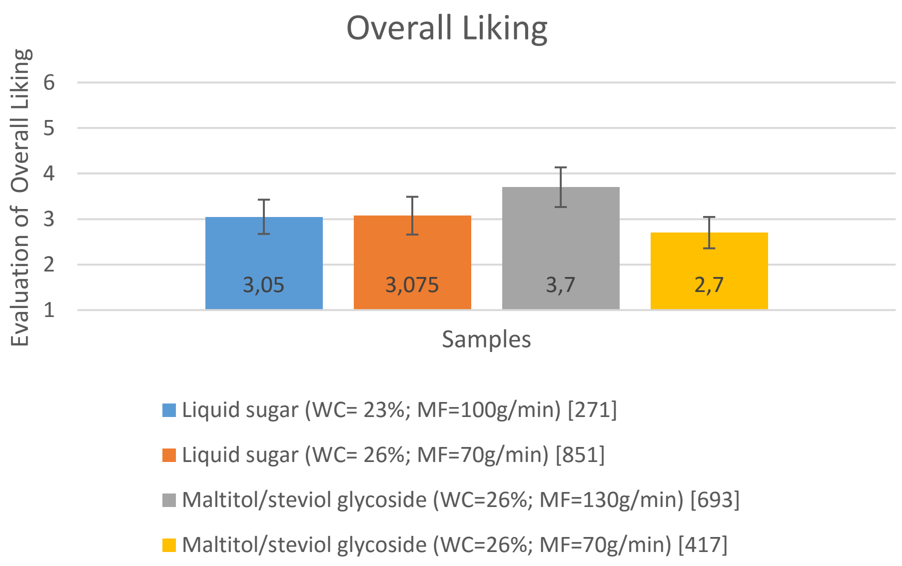

Figure 27. Barchart of the evaluation Overall liking; n=40; 1=not much / 6=very much; confidence interval 95%

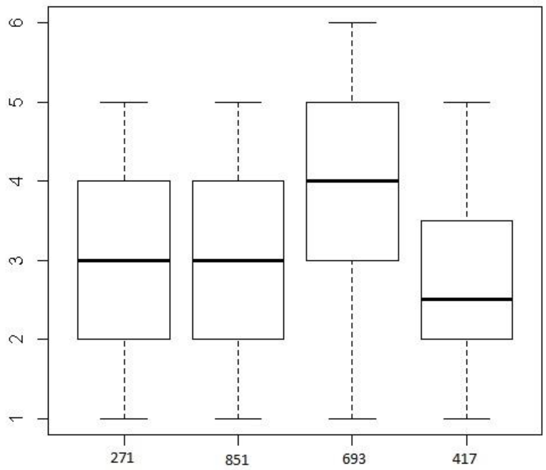

Figure 28. Boxplot diagram, which shows the distribution of the values for overall liking in quartiles;n=40; 1=not much / 6=very much;
271=Liquid sugar (WC=23%; MF=100g/min);
851=Liquid sugar (WC=26%; MF=70g/min);
693=maltitol/steviol glycoside (WC=26%; MF=130g/min)
417=maltitol/steviol glycoside (WC=26%; MF=70g/min)

Between those liquorice, which were sweetened with liquid sugar there was no difference no differences in overall liking at all. Both were rated with a value of 3 on a 6 point scale for overall liking. In contrast to that the samples which were produced with steviol glycosides and maltitol were evaluated quite differently. The liquorice with the water content of 26% and the mass flow of 130g/min was chosen as the best liquorice with a value of 3.7. On the other side the sample with the water content of 26% and 70g/min was only ranked fourth with a value of 2.7 (Table 42/Figure 27 and 28).

Some participants were tasting an off flavour but those people were in the minority with 17.5% for the sample with steviol glycosides and maltitol (WC=26%; MF=130g/min) for the others samples the percentage of participants who determined an off-flavour was even less with 15%. Furthermore it has to be said the attributes, which were mentioned from the participants differed quite much. Attributes, which had been mentioned were honey, peppermint or wood like. So it was not possible to determine any specific off flavour for any of those samples with the test panel of 40 person. Further comments, which were made on the sensory evaluation sheet were that all of the four liquorice samples were not as sweet as conventionally liquorice. Another aspect, which was mentioned quite often was that the flavour which the participants of the consumer test associate with liquorice was not so strong.

6 Discussion

6.1 Characterisation of Flour

The two flours which were compared to get an idea for their suitability for liquorice production were the wheat flour from the Helsingin Mylly Oy and the full grain spelt wheat flour of the company Sunspelt Ltd.

The results of the falling number test were showing that the wheat flour had a significant higher falling number (335) than the full grain spelt wheat flour (200). These results show that the wheat flour has a better suitability for liquorice production because the flours for liquorice production should have a falling number, which is not much lower than 200 according to Hoffmann, et al. (2002). This can be explained that with a low falling number the α-amylase activity is very high. The enzyme α-amylase breaks down the starch even at high temperatures (100°C). Therefore, starch of a flour with a low falling number is not able to build up a proper texture. The reason why spelt wheat flour has such a high alpha amylase activity is that this flour is a full grain flour, which means that the germ and the bran are not separated from the endosperm. However, normally, the most enzymes can be found in the germ and the bran, which is explaining the low falling number [Schünemann, 2006].

The results of the Rapid Visco Analyser support the outcomes of the falling number test. The peak viscosity of the wheat flour (1807) is significantly higher than the peak of the spelt wheat flour (563), which can be also explained by the high amount of alpha amylase in the spelt flour. Another interesting characteristic is that the starch in the wheat flour is starting to paste earlier at a lower temperature (78.02°C) than the starch of the spelt wheat (89.05°C), which might can be explained by the higher amount of shell parts that are in the spelt wheat flour. As the duration of heating is limited in the extruder, a flour which is pasting earlier at a lower temperature should be favoured. An extrusion (co-rotating twin screw extruder) with the spelt wheat flour showed that under similar condition only a sticky formless mass was created

6.2 Physical properties of liquorice

6.2.1 Water content

The water content of the final product was increased with a higher water content for both (liquid sugar and mixture of steviol glycosides and maltitol syrup) liquorice recipes. However, a statistically significant difference of the mass flow could not be detected for either recipes. Only the impact of the water content in those recipes was statistically significant. The higher the water content calculated in the recipe was, the higher the water content of the final product was. Another thing is that the values for the final water content of the liquorices which were produced with steviol glycosides and maltitol were smaller than the recipe in which liquid sugar was used. In this context, it has to be said that those water content values might not be the final ones. After drying the samples for 90 hours in the vacuum incubator and another 72 hours in the desiccator, the first measurement of the samples were done. The second measurement was done after another 96 hours in the desiccator. However, even after that time, the water content was still decreasing about 0.03% in comparison to the first measurement. Due to a lack of time, it was not possible to measure the samples for a third time. In retrospect to the results and to the fact that maltitol syrup is quite a hygroscopic substance, which has higher water binding properties than sucrose, it is very likely that those samples were not totally dry after the second measurement. Hence, at least for the samples with steviol glycosides and maltitol syrup, the time for drying has to be at least several weeks to get a meaningful result.

6.2.2 Water activity

The liquorices produced with liquid sugar and the liquorice produced with steviol glycoside and maltitol did not differ a lot in there water activities. For both recipes, only a statistical significant difference for water content in the recipe, but not for the mass flow, could be detected. However, the impacts of those variables were rather small, and with a p-value of 0.07, it could be also that those changes are just random. This may lead to the conclusion that the water activity depends neither on the water content nor on the mass flow or on the type of sweetener. This is also in line with the outcomes of the research of Müller (2012), which found that the water activity was not influenced by process variables such as water content in the recipe or the mass flow.

However, it has to be mentioned that the liquorice samples were holding the water very tightly and it could not be said, due to a lack of time, if those samples had always been in equilibrium when the water activity was measured. Another aspect was the surface and the amount of the samples. Unfortunately, it was not possible to have always the same amount with the same surface in the measuring chambers for water activity. Thus, these aspects have to be taken into account while looking at those results. Therefore, it is very difficult to make a substantial statement about the water activity.

6.2.3 Diameter

The diameters of liquorice sweetened with liquid sugar (recipe A) did not vary very much. Even the recipe created with a total water content of 21% offered enough water for the starch gelatinization during the extrusion process.

However, it seemed that the mass flow has an impact on the diameter. For both water contents (21% and 26%), the diameter is decreasing, with an increasing mass flow.

The reason for the reduction of the diameter could be the constant screw-speed of the extruder (55 rpm). That means that for a mass flow of 130g/min, more starch of the flour has to gelatinize within the same time in comparison to a mass flow of 70g/min. Thus, if the mass flow is too high, it might be possible that not all of the starch was gelatinized when the final product was coming out of the extruder. Most probably also other factors than only the residence time affect the gelatinization because with a higher mass flow, probably also the mixing effects are more efficient, due to higher shear forces, than if there is less mass in the extruder. However, because of this tendency, it could be that the residence time might have a bigger impact than the increasing shear forces. Furthermore, it has to be mentioned that a co-rotating twin screw extruder was used, because normally intermeshing counter-rotating extruders are used for the liquorice production. In those extruders, the shear forces are higher than in comparison to a co-rotating twin screw extruder and high shear forces are required for liquorice production [Frame, 1994].

A remarkable difference was noticed when the diameters of the liquorices which were produced with liquid sugar were compared with the diameters of the liquorices which were produced with steviol glycosides and maltitol. Liquorice diameters of the recipe with the mixture of the intense sweetener and the bulk sweetener were much smaller with a water content of 21% and the liquorice diameter increased significantly for this recipe if the water content in the recipe was risen to 26%. This leads to the conclusion that the water content of 21% was not sufficient, and there was not enough free water to gelatinize all the starch of the wheat flour [Heldman, et al., 1997]. The reason for that could be the bulk sweetener maltitol because it is more hygroscopic than the liquid sugar, which means that it has higher water binding properties [Mitchel, 2006]. There was propbaly too little water available for the starch to gelatinize completely, due to the higher water binding properties of maltitol. In contrast with a higher water content (26%), the size of the diameter in comparison to the liquid sugar was almost the same. Hence, in that case, enough water was available for the starch gelatinization, which was leading to a bigger diameter.

However, the p-values were quite high for both recipes, which makes it difficult to make a certain statement. The reason for the high p-values was that the liquorice dough which were produced in the extruder was always varying a bit because it was not homogenous. One explanation for this could be that the mixture of the solid part and also that of the liquid part was not always totally homogeneous, which probably had an influence on the extrusion process. In comparison to other researches on the production of liquorice such as those conducted by Kallio (2006) and Müller (2012), the diameters were in a similar range for the recipe which was produced with liquid sugar. This could lead to the assumption that the presence of molasses does not influence the diameter since no molasses was used in the recipes of this research. However, to make a well-founded statement on this aspect, more research is necessary.

6.2.4 Extension Test

Looking at the results of the maximum load and the extension at maximum load, similar tendencies can be seen as for the diameter. Thus, with a higher mass flow the maximum load was decreasing for the recipe with liquid sugar. Focusing on the results of the liquorice sweetened with liquid sugar, there is one value attracting attention, which is the value with a water content of 21% and the mass flow of 70g/min. This measurement value is the highest with an amount of 14.11 Newton. This result leads to the assumption that the proportion of water and starch was probably the best at a water content of 21% for this recipe. Under that condition the starch was able to absorb this amount of water completely. In contrast, for the same mass flow and a water content of 26%, there was probably too much water for the starch to absorb, which decreased the possible maximum load of the final product. This would also explain why the differences in the maximum load caused by the changing mass flow are much higher for a water content of 21% than for a water content of 26% because in that case there is no or not much excessive water for gelatinization.

On the other hand, the values of the maximum load were very low for the recipe with maltitol syrup and steviol glycosides and for the water content of 21%, which can be also explained by the hygroscopic potential of maltitol syrup [Mitchel, 2006]. Therefore, again, more water was needed for the starch gelatinization [Heldmann et. al. 1997]. This can be also seen from the results because with a water content of 26% and a mass flow of 70g/min, the values for the maximum load were quite high. Additionally, at a water content of 26%, the differences between the different mass flows were relatively large for the maximum load. This leads to the assumption that similar maximum load conditions occur for the liquorice sweetened with maltitol and steviol glycoside at a water content of 26% and for the liquorice sweetened with liquid sugar at a water content of 21%.

Another aspect which can be seen by comparing the maximum load and the extension at maximum load is that the samples which were produced with liquid sugar had a higher tensile strength than the liquorice samples which were produced with steviol glycosides and maltitol syrup. That means that the samples, which were produced with the intense sweetener and the bulk sweetener could be stretched with a lower force in comparison to the liquorice which was sweetened with the liquid sugar. This can be also derived from the results of the maximum slope from the curves, which were drawn by the program (stiffness) (Figure 19). This effect might be explained, by the lower density of the liquorice products which had been sweetened with the intense sweetener steviol glycoside and the bulk sweetener maltitol. The lower density can be also traced back to the water binding properties of maltitol, which are higher than those of sucrose.

6.2.5 Appearance

The degree of gelatinization also affected the appearance of the liquorices. The degree of gelatinization was probably lowest in the liquorices prepared with steviol glycoside and maltitol syrup at a water content of 21%, which can be traced back to the hygroscopic character of maltitol. Also the liquorice samples were very sticky and glossy due to a gel-like film adhering to the surface. Furthermore, after the storage in the plastic bags, it was noticed that there was some kind of liquid adhering to the inner surface of those bags and that it seemed to come out from the liquorice. These findings can be explained by the degree of gelatinization because according to Hoffmann et. al. (2002), a higher degree of gelatinization leads to higher water binding properties. Therefore, with a low degree of gelatinization, it is likely that water was set up at the surface of the liquorice after some time, due to that the liquorice became sticky and also glossy.

6.3 Sensory consumer test

The results of the texture analysation show that the value of the liquorice with maltitol and steviol glycoside with a water content of 26% and a mass flow of 130g/min has to be highlighted. In contrast to the other values this value was quite low (2.1). This results were also in line with the measurement values of the texture analyser (Instron. Model: 4465). It had to be said that a lower texture, due to a lower degree of gelatinization, was favoured by the participants of that consumer test. This can be also seen very clearly in the evaluation part of the overall linking.

The sweetness which was determined for the liquorices with liquid sugar and the liquorices which were sweetened by steviol glycosides and maltitol showed similar values. Due to this, it is possible to use the theoretical values for sweetness without large variations also in the practice.

The sweetness of the liquorices, which were produced with steviol glycosides and maltitol syrup showed a bigger variation in the evaluation of the sweetness. The reason for that was that during the production it was not possible to dissolve the steviol glycoside powder completely, which propably led to a product, whose sweetness was varying. For further research, it would be advisable to heat up the solution with water and steviol glycsodes beforehand to improve the process of dissolution and add the other ingredients (salt and maltitol syrup) after all the steviol glycosides are solved. Many participants of the consumer test gave the feedback that all of those liquorice samples, which were tested weren’t so sweet. One explanation for this could be that the molasses which is normally used for liquorice was not used for those recipes because it was not allowed according to the regulation. Molasses has about 48% sugar included, which is therefore offering sweetness as well. However, this loss of sweetness was not considered when the recipes were created. It was also mentioned on the evaluation sheets that the flavours of those liquorices were not so strong this can be also traced back to the loss of molasses because this substance normally also provides some special flavour to the conventionally liquorice products.

The attributes for the off-flavour, if detected, were varying a lot, thus no certain off-flavour could actually be detected. Therefore, steviol glycosides can be used in liquorice production with a proportion of at least 0.08% without any danger of off-flavours. This was not surprising because the off-flavour of steviol glycoside is described as liquorice by Mitchel (2006).

The texture was playing a major role in the rating of the overall liking. This could be seen from many comments made by the participants. They found the low texture of the liquorice sample with steviol glycoside and maltitol syrup with a water content of 26% and a mass flow of 130g/min very pleasant and evaluated this sample as the best liquorice. The maximum load which was exerted on that sample was 3.07 Newton. On the basis of that information it could be said that the liquorice, in general, should have a maximum load around 3 Newton so that the texture is perceived as pleasant. From those results it can be also seen that the texture does not play a tangential role for the overall liking and that it has a similar importance as the sweetness.

Probably the consumer test did not deliver reliable results. This is due to the fact that the informants were not trained to evaluate the attributes texture, sweetness or off-flavour. Therefore, they were rating those values subjectively depending on their opinion, which lead to large distribution around the mean value. This can be seen very well from the boxplot diagrams (Figure 24, 26 and 28). Those diagrams show that for each attribute (texture, sweetness, overall liking) always nearly every number (1 to 6) was used for evaluation. If the informants had been trained, the distribution around the mean value would not be that large and therefore the results would be more reliable. Another aspect was that only 2.5% of the participants were eating liquorice every day. In contrast, most of the informants who participated were eating liquorice not more than once a month or even less (82.5%). It would have been good if more people had eaten liquorice more often because then they would be more used to the taste, which would make them better experts for the evaluation.

7 Conclusion

The aim of this research was to determine if the different sweetener (maltitol syrup and steviol glycosides) in combination with changing extrusion parameters (mass flow and water content in the recipe) have an effect on the final liquorice product when they replace conventional liquid sugar. Therefore, two different liquorices (liquid sugar and mixture of steviol glycoside and maltitol syrup) were produced and compared (water content water activity, diameter, texture and consumer test) with each other.

One achievement of this study was that it was possible to replace the liquid sugar in liquorice with steviol glycosides and maltitol syrup, without any differences in sweetness and with no off-flavour that could be detected. It was also possible to produce liquorices which looked similar and had a similar texture to those liquorice which were produced with liquid sugar. Most properties such as water activity, diameter and texture were not significantly influenced by the extrusion parameters due to a relatively high *p*-value. However, trends could be seen that liquorices which were produced with steviol glycoside and maltitol syrup needed more water to get a similar texture (maximum load) or diameters. Due to the fact that steviol glycoside was only used in a very small amount of 0.08% in those recipes, this substance probably did not have any substantial impact on the extrusion process. However, steviol glycoside was used in combination with maltitol syrup. Depending on the water content of the recipe, the amount of this bulk sweetener ranged from 57.44% to 61.32%. Therefore, it affected the extrusion process quite much. In comparison to liquid sugar, maltitol syrup is a substance which is more hygroscopic and has therefore better water binding properties. This property has a strong impact on the extrusion process because in the presence of maltitol, more water was needed to get similar physical properties (texture, diameter, appearance) than with liquid sugar. Additionally, it has to be mentioned that according to the specification of the product Lycasin 75/75-Maltitol Syrup, also the substance D-Sorbitol was included with an amount of 1.4%. This substance has an even higher hygroscopic character and therefore also higher water binding properties than maltitol [Mitchel, 2006]. Therefore, this substance probably enhances the effects of maltitol.

The determination of the water content proved to be very difficult. Due to the fact that the liquorice confectionaries were holding the water very tightly, it was not even manageable to measure the water content because they had still water after drying them for one and a half week in the vacuum incubator (Salvis) at a temperature of 70°C and in a desiccator with the drying agent phosphorus pentoxide (P_2O_5). Therefore, more time or different

conditions are needed to determine the water content. Also, in the sensory evaluation of the liquorices, better values would have been achieved if the test persons were trained beforehand to evaluate the attributes such as texture, sweetness or off-flavour.
The results for the response variables had a *p*-value, which was quite high so that it was not possible to say if those trends were really existing or if those trends appeared just randomly. The only exception in that case were the results of the final water content. The reason for the high *p*-values could be that the number of replicates were quite low, so therefore outliers had a greater impact. For getting more reliable values, a higher number of replicates for the different analysis would be reasonable.

The results can be utilized if liquorice recipes are created, which contain maltitol syrup and steviol glycosides. With the help of the results the right amount of water can be estimated to get the desired diameter and texture. Furthermore, the results show that liquorice confectioneries with steviol glycoside and maltitol syrup can be produced in a big scale with a co-rotating twin screw extruder when the parameters are set right. Another outcome which can be utilized for the liquorice production in a big scale is that it is important to adjust the residence time in the extruder (screw speed) depending on the mass flow to get the same properties for the final extrusion product.
In further research, it would be advisable to use a water content of 26% for the centre point if liquorice is produced with maltitol syrup and steviol glycosides due to the higher amount of water which is needed. It could also be interesting to see if steviol glycosides are supporting the effect of maltitol syrup. This could be shown by using recipes with different amounts of steviol glycosides in combination with maltitol syrup and analyse those produced products in the end. Unfortunately, due to a lack of time in this study, only one recipe was used with maltitol syrup and steviol glycosides and compared with the liquid sugar.

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Sensory Evaluation Sheet and ethical approval

Testing different natural sweeteners in liquorice

Sensory evaluation sheet Date: ______________

Personal Data:

Age: ________

Male ☐ Female ☐ Other ☐ Smoker ☐ Non-Smoker ☐

How often do you eat liquorice?

☐ Less than once a month

☐ Monthly

☐ Weekly

☐ Almost every day

Evaluation

In front of you, you find four different Samples 271, 856, 693 and 417
Evaluate the following properties by crossing the number

Texture

	soft					hard
417	1	2	3	4	5	6
693	1	2	3	4	5	6
581	1	2	3	4	5	6
271	1	2	3	4	5	6

Sweetness

	weak					strong
417	1	2	3	4	5	6
693	1	2	3	4	5	6
581	1	2	3	4	5	6
271	1	2	3	4	5	6

Off-flavour

417: ◻ No ◻ Yes If yes, describe the off-flavour: ______________________

693: ◻ No ◻ Yes If yes, describe the off-flavour: ______________________

851: ◻ No ◻ Yes If yes, describe the off-flavour: ______________________

271: ◻ No ◻ Yes If yes, describe the off-flavour: ______________________

How do you like the different samples (1=not much/6=very much).

417	1	2	3	4	5	6
693	1	2	3	4	5	6
851	1	2	3	4	5	6
271	1	2	3	4	5	6

Further comments: ____

____

Thanks for participating in this evaluation ☺
Kiitos ☺

Allergens which are included in the product: Wheat flour