

Factors influencing the processing quality of potatoes – development of analytical tools for process monitoring and optimization

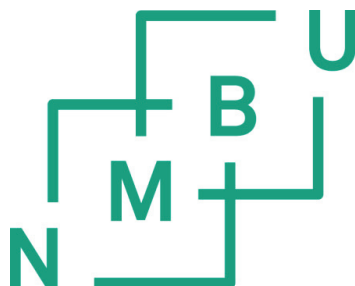
Egenskaper som påvirker potetens prosesskvalitet – utvikling av analyseverktøy for
prosessovervåkning og optimalisering

Philosophiae Doctor (PhD) Thesis

Trygve Helgerud

Department of Chemistry, Biotechnology and Food science
Norwegian University of Life Sciences

Ås 2015



Thesis number 2015:30
ISSN 1894-6402
ISBN 978-82-575-1280-4

Acknowledgements

The work presented in this thesis was carried out at Nofima, The Norwegian Institute of Food, Fisheries and Aquaculture Research, in Ås. The thesis has been written as a part of the project “Optimal Potato” (Project no. NFR-199421/O99) with financial support from The Fund of the Research Levy on Agricultural Products. The project has also been supported by local potato processing industry. I would like to thank Bjørn Olav Drabløs and Mathis Nærby at Buer AS, Anne-Helen Kalhovd at Maarud AS, Stein Rønne and Leif Skjønsby at KiMs Norge AS, Jørn Kongelstad at Hoff SA, and Trygve Kirkerød, Sigvart Grorud and Albrigt Eidstø at Bama Industrier AS. Thank you for supporting the project, and supplying excellent samples for my experiments.

I would like to thank my team of supervisors; Simon Ballance and Nils Kristian Afseth at Nofima, and professor Elling-Olav Rukke at Norwegian University of Life Science (NMBU). The contribution from Svein Knutsen is also greatly appreciated. I would like to thank you all for the supervision my thesis and for all the fruitful discussions. It has been enjoyable to work with you, and I would like to thank you all for always answering my questions.

Bjørg Narum, Karen Wahlstrøm Sanden, Frank Lundby, Lene Ruud Lima and Hanne Zobel are greatly acknowledged for their technical assistance in the lab. I would like to thank Kent Berglund for fabricating odd equipment (like a single tuber underwater weight). I would like to thank previous and present PhD students at Nofima; Anne, Haakon, Jib, Elena, Silje, Nebojsa, Gunna and Sarin, for inspiring discussions, lunch meetings and social activities outside Nofima. In addition, Ulrike, thank you for all the discussions (about bikes and other important subjects).

At last, I would like to thank my family for always supporting me. A big and special thanks to Cecilie and Sigurd. Thank you Cecilie for supporting me through this, and thank you Sigurd, for always smiling when I come home from work.

Ås, January 2015
Trygve Helgerud

Sammendrag

Poteten er et heterogent råmateriale. Produsenter av prosesserte potetprodukter har derfor store prosessutfordringer. Hovedmotivasjonen bak denne avhandlingen er å bistå industrien i å redusere svinn og senke produksjonskostnadene. Målet er derfor å utvikle og forbedre analysemetodikk for bestemmelse av råvarekvalitet, samt å øke kunnskapen rundt produksjonsprosessene. Avhandlingen er bygget opp av fire artikler og to upubliserte forsøk som alle har potetkvalitet som underliggende tema.

Artikkel I og II sikter på å forbedre instrumentelle analysemetoder for prediksjon av tørrstoffinnholdet i hele uskrelte poteter. Artikkel I viser bruken av et nær-infrarød spektroskopi (NIRS) prototype instrument for bestemmelse tørrstoffinnholdet i poteter. NIRS prototype instrumentet sammenlignes med den tradisjonelle undervannsvekten og et kommersielt tilgjengelig NIRS instrument. NIRS prototypen ga noe dårligere prediksjoner enn hva undervannsvekten klarte. Det kommersielt tilgjengelige instrumentet ga dårligere resultater enn både NIRS prototype instrumentet og undervannsvekten. Artikkel II sikter på å finne ut om NIRS prototypen kunne brukes over et samleband (online målinger) Instrumentet ga gode resultater for poteter som beveget seg på et samleband. Det ble konkludert med at NIRS er godt egnet for online målinger av tørrstoffinnholdet i hele uskrelte poteter. Metoden kan dermed være løsningen på noen av utfordringene prosessindustrien møter.

Artikkel III evaluerer tre små håndholdte instrumenter for bestemmelse av glukoseinnhold i poteter. To små blodsuktermålere og et kommersielt instrument beregnet på mat og drikke (RQFlex) ble testet. Alle tre instrumentene er i stand til å gi et estimat for glukoseinnholdet i hel potet. RQFlex instrumentet ga de beste resultater, men er noe mer tidkrevende å bruke. Det ble også funnet at glukoseinnholdet i potetjuice stemmer godt overens med glukoseinnholdet i frysetørket potet. I et upublisert eksperiment ble Fourier transform infrarød spektroskopi (FTIR) av inntørkede juicefilmer testet ut. Resultatene er lovende, men noen utfordringer knyttet til sortsavhengighet og prøveopparbeiding gjenstår.

Artikkel IV identifiserte parametere som påvirker kvaliteten til lompedeig. Retrograderingsgraden og tørrstoffinnholdet til potetene ble funnet som to av de viktigste parametere. Uønskede og tidsavhengige endringer i deigkvaliteten ble funnet å henge sammen med enzymer fra melet. Resultatene fra artikkel IV indikerer at endringer i måten deigen håndteres på kan bote på noen disse problemene.

Abstract

Potatoes are notoriously heterogeneous, and manufacturers of processed potato products are therefore faced with a number of challenges during processing. The motivation behind this thesis is to reduce waste and cut production costs in this industrial segment. The main aim is thus to improve analytical methods for raw material evaluation, hence also improving the knowledge about different processes. This thesis is built around four papers and two unpublished experiments, all of which can be related to raw material quality of potatoes.

Paper I and II aim to improve methods for instrumental prediction of dry matter content in whole unpeeled potato tubers. Paper I shows the use of a Near infrared spectroscopy (NIRS) prototype instrument in comparison to the traditional underwater weight and a commercially available NIRS online instrument, respectively. The NIRS prototype showed slightly lower performance than the underwater weight, but better performance than the commercially available instrument. Paper II focuses on further investigating the possibilities of using the NIRS prototype for online applications. Good prediction performance was obtained for potatoes moving on a conveyor belt. NIRS can be used for online prediction of dry matter content in potatoes, hence potentially solving one of the major challenges in the potato industry.

Paper III evaluates the use of three different hand-held instruments (two portable blood glucose meters (PBGM) and one instrument (RQFlex) made for food and beverage analyses. The RQFlex outperformed the two PBGMs in terms of glucose determination, but were slightly more labour intensive to use. Paper III also evaluates the use of potato juice as a sampling technique. Good correlations were seen for the glucose content in potato juice compared to the glucose content in lyophilized potato. All three instruments were suited for rough estimation of the glucose content in potatoes. An unpublished experiment showed a preliminary investigation of using dry film Fourier transform infrared (FTIR) measurements. The results were promising, but some challenges related to cultivar dependency and sampling technique still have to be overcome.

Paper IV identifies different factors affecting the material properties of “lompe” dough. Degree of retrogradation (i.e. the time between cooking and dough mixing) and dry matter content of the potatoes were found to be crucial to the texture of the dough. Unwanted dough degradation, or softening, shortly after the dough mixing was found to be caused by endogenous flour enzymes. The findings in paper IV indicate that careful dough management may help aid some of the problems the “lompe” producers are experiencing.

List of Papers

The following papers are included in this thesis. They are referred to by their roman numerals.

Paper I:

Helgerud, T., Segtnan, V.H., Wold, J.P., Ballance, S., Knutsen, S.H., Rukke, E.O., & Afseth, N.K. (2012). *Near-infrared spectroscopy for rapid estimation of dry matter content in whole unpeeled potato tubers*. Journal of Food Research, 1(4), 55-65.

Paper II

Helgerud, T., Wold, J.P., Pedersen, M.B., Liland, K.H., Ballance S., Knutsen S.H., Rukke, E.O. & Afseth, N.K. *Towards on-line prediction of dry matter content in whole unpeeled potato tubers using Near-Infrared spectroscopy* (Manuscript, submitted)

Paper III:

Helgerud, T., Knutsen S.H., Afseth, N.K., Stene, K.F., Rukke, E.O., & Ballance S.
Evaluation of hand-held instruments for representative determination of glucose in potatoes (Manuscript, submitted)

Paper IV:

Helgerud, T., Schüller, R.B., Rukke, E.O., Afseth, N.K., Knutsen S.H. & Ballance S.
Factors affecting the material properties of lompe dough (Manuscript, submitted)

Abbreviations

% _{DM}	% dry weight
% _{FW}	% fresh weight
μL	Micro litre
μm	Micro metre
cm	Centimetre
cm ⁻¹	Wavenumber
cv	Cultivar
DM	Dry matter content
DNS	Di-nitrosalicylic acid
e.g.	For instance (from Latin)
EMSC	Extended multiplicative scatter correction
<i>et al.</i>	With others (from latin)
FTIR	Fourier Transform infrared spectroscopy
g	Gram
G*	Complex shear modulus
g/100g	Grams per hundred grams
G'	Loss modulus
G''	Storage modulus
h	Height (of sample)
Hi	High
i.e.	Which means (from Latin)
Im	Imaginary numbers
IR	Infra-Red
IRS	Infrared spectroscopy
kg	kilogram
<i>L.</i>	Latin
lg	Logaritmic
Lo	Low
LVE	Linear vicoelastic
mg	Milligram
mg/mL	Milligram per millilitre
mL	Millilitre

mm	Millimetre
mm/s	Millimetre per second
mmol/L	Milli mole per litre
mNm	Milli Newton meter
NIR	Near infrared
NIRS	Near infrared spectroscopy
Nm	Newton metre
nm	Nanometre
NMR	Nuclear magnetic resonance
\varnothing	Diameter
$^{\circ}\text{C}$	Degree Celsius
PBGM	Portable blood glucose monitor
PCA	Principal component analysis
PLS	Partial least squares regression
R	Reflectance
R^2	Determination coefficient
Rad/s	Radians per second
Re	Real numbers
RMSECV	Root mean square error of cross validation
RMSEE	Root mean square of error estimate
SG	Specific gravity
SNV	Standard normal variate
SV	<i>Sous-vidé</i>
T	Shear stress
$\tan\delta$	Phase angle
W	Water
γ	Strain
γ_L	Strain _{limit}
ω	Angular frequency

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1. Aims of the study

About two thirds of the Norwegian potato crop are turned into processed potato products¹. The heterogeneous nature of potatoes makes it difficult to obtain a high and uniform quality of the final product. For some potato products such as chips and crisps it is well understood how the potato quality affects both processing and product quality. In this case, the challenge is to obtain representative measurements of the potato raw material. For other products such as “lompe” (a Scandinavian product that resembles Mexican tortillas) and *sous-vidé* treated potatoes it is less understood how the different raw material parameters affect production and product quality.

This thesis is defined from the project grant “Optimal Potato” (Project no.: NFR-199421/O99). The project is financed by “The Fund of the Research Levy on Agricultural Products” with 20 % cash contribution from industrial partners. The main goal of this study is to increase product quality and decrease waste and production costs in the potato processing industries. In many cases, an insufficient raw material quality will not be detected until after processing. It is better, both in an environmental and in an economical perspective, to remove low quality raw material before, rather than after, processing.

To achieve this, the effect of potato raw material quality on the production process and product quality needs to be both measured and understood. For fried potato products, dry matter content and the content of reducing sugars are known factors, which influence production process and product quality. However, in order to use this knowledge for potato sorting and process optimization fast and representative measurements of large quantities of potatoes are necessary. The first part of the thesis is therefore aimed at developing improved methods for industrial determination of dry matter content and reducing sugar content in potatoes. The second part of the thesis is aimed at increasing the knowledge of how raw material composition affects the production process and product quality of “lompe” and *sous-vidé* potatoes.

The four different product categories that will be further discussed in this thesis are; “lompe”, *sous-vidé* treated potatoes, crisps and chips. The production of, and challenges associated with, the different product categories will be described in detail in the forthcoming sections. Raw material parameters that are known to affect processing and product quality will be stated and the currently available analytical methods for measuring these parameters will be presented.

2. The potato

In Norway, approximately 330 000 tons² of potatoes (*Solanum tuberosum*, L.) are harvested each year. About 120 000 tons¹ are used for processed potato products, such as crisps, chips, “lompe” and *sous-vidé* treated potatoes. Only 93 000 tons¹ are sold fresh in supermarkets etc. About 55 000 tons are used for starch and alcohol production.

A potato tuber consists mainly of water and starch³. The water content of a mature potato tuber is in general between 70 and 85 % of its fresh weight (%_{FW}). The rest of the weight, called the dry matter content (15-30 %_{FW}), consists of approximately 65-80 percent of its dry weight (%_{DW}) starch and other minor constituents (7-7.5 %_{DW}) such as sugars, cell wall tissue, minerals and proteins. Potato starch is composed of about 80 %_{DW} amylopectin and 20 %_{DW} amylose. Compared to other sources, potato starch has a high degree of phosphorylation with about 0.08 % of the glucose subunits phosphorylated³. A general overview of the main components in a potato is given in Table 1.

Table 1: Main components of a potato. All figures included are taken from available literature, and do not cover all extreme high and low values

	Range:	Unit:
Dry matter content ⁴	16-30	% _{FW}
Starch ³	65-75	% _{DW}
Fibre ⁵	1-2*	% _{FW}
Protein ^{3,6}	0.6-2.1	% _{FW}
Minerals ⁶	1.1	% _{FW}
Lipids ³	<1	% _{FW}
Vitamin C ⁷	0.08-0.15	% _{DW}
Sugars ^{3,6}		
-disaccharides	0.4-6.6	% _{DW}
-monosaccharides	0-3.0	% _{DW}

*Raw tuber with skin

Also internally a potato tuber is heterogeneous and several different zones and parts can be identified within tuber. Some of the zones are differing in their chemical composition. Figure 1 shows a schematic view of the different regions in the potato. It is known that the sugar

content and the dry matter content may differ significantly between the zones. Pritchard and Scanlon⁸ found in 1997 that both dry matter content and sugar content are highest in the outer regions of the tuber. The pith of the tuber is lowest in dry matter content and the parts outside the vascular ring are the richest.

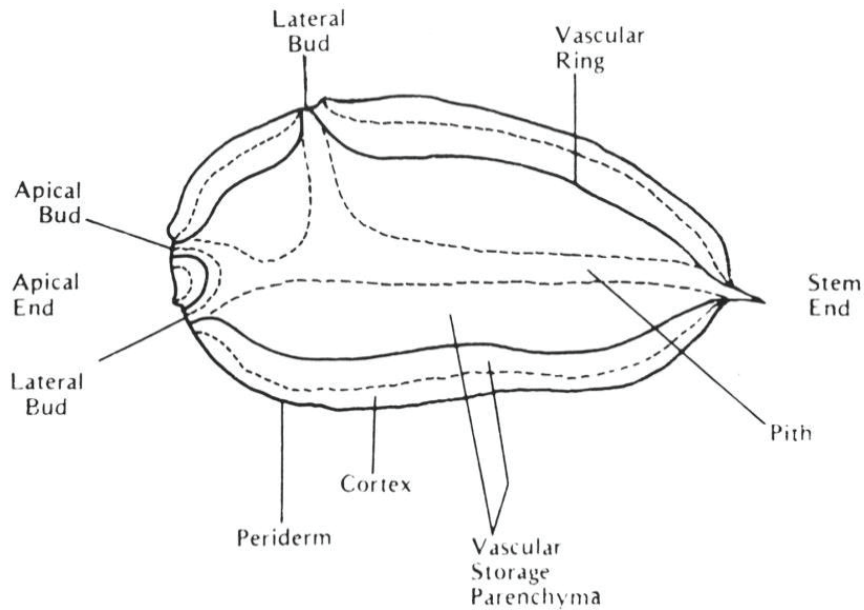


Figure 1: A schematic overview of a potato, showing the names of the different regions inside the potato tuber⁹

3. Relevant potato products

This chapter will give a brief overview of the products involved in this thesis, along with description of the manufacturing. As potatoes can be processed into a large variety of different products, the term “potato processing industry” will in this thesis only be used as a reference to industry making chips, crisps, *sous-vidé* products and “lompe”. Other potato products will not be discussed.

3.1 Deep fried potato products

The largest product category studied in this thesis is the deep fried potato products. Both crisps and chips (UK terminology) are included in this category. A general overview of the process of making crisps and chips is given in Figure 2. Depending on the time of the year, the potatoes may have been stored for as much as 11 to 12 months. After being retrieved from storage, the tubers are cleaned and sorted. Today, sorting is mainly done according to size and visible defects. Very little is done to sort the potatoes based on chemical parameters. The dry matter content and the sugar content (usually only glucose) are checked on site, and batch averages are calculated. Whole lots can be accepted or discarded based on these averages. The

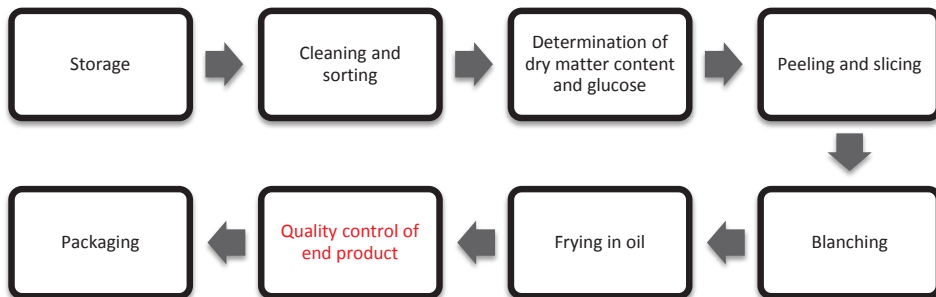


Figure 2: A simplified diagram of the steps involved in the process of making crisps and chips. Minor steps may be included or excluded to form specific products

average dry matter content is sometimes also used to calculate the purchase price of the batch. After sorting, the potatoes are peeled and/or sliced before blanching. The blanching involves of soaking in hot water. Excess water from the blanching is drained off and the slices are fried in oil, before inspection of the final product. Individual chips or crisps can be rejected based

on colour. Whole lots may be rejected if the texture is wrong (e.g. too soft texture). As seen from Figure 2 the rejection of crisps and chips is done at a very late stage of the process. A lot has been done to establish and elucidate the factors affecting the product quality of fried potato products. The quality of fried potato products can mainly be divided into two different categories.

The first is the textural properties. The main contributors to the texture of crisps and chips are dry matter content, frying temperature and frying time (i.e. reducing the water content to the desired level)¹⁰. The frying time will therefore depend upon the dry matter content. The dry matter content of potatoes is known to vary both within and between lots¹¹. This variation will be enhanced if multiple lots are combined during shipping. Since only batch assessment of the raw material is performed, the factories do not know whether or not all the tubers in the lot will give acceptable quality until after further processing. Some may be fried too short and other too long. This implies that resources have been used to produce a product that may have insufficient quality.

The second quality category is the chemical quality of the crisps and chips. This includes taste, colour and unwanted substances formed during the frying. The frying colour of both crisps and chips has been one of the most important attributes of the product since it was put into commercial production. For nearly a century it has been known that sugars and amino acids are of the most important intrinsic factors regulating the colour of fried potato products¹². The reaction pathway leading to the colour formation in heat treated foodstuffs is usually termed the “Maillard reaction”, and was discovered early in the 20th century¹³. The desire to control the frying colour inspired a lot of research concerning the sugar content of potatoes. When researchers started looking into the sugar content of potatoes, it soon became clear that the sugar content may drastically change during the storage season. This phenomenon was enhanced by low storage temperature and was named “cold induced sweetening”¹⁴⁻¹⁶. It is known that storage at 4 °C will cause significantly higher sugar accumulation than storage at 10 °C¹⁷. The mechanism behind the sweetening is believed to be a combination between starch degradation and sucrose cleavage¹⁴. It is therefore necessary to keep a certain temperature when storing potatoes for processing. On the other hand, too high temperature may cause unwanted and too early sprouting of the tubers. The storage temperature may therefore be a compromise between keeping the sugars content low and the potatoes healthy¹⁸. It is, independent of the temperature regime used, important to monitor the sugar content, and its development, closely.

The sugar content of potatoes was a major concern for many years, but only due to the undesirably dark product colour it caused. However, this changed in 2002 when a group of Swedish researchers discovered acrylamide in fried potato products¹⁹. The relationship between the Maillard reaction and acrylamide formation was established shortly after the initial discovery^{20,21}. Acrylamide is well known to be carcinogenic in rats and mice²², but the toxicity and carcinogenic effect on humans is still not fully elucidated²³. However, most authorities are of the opinion that manufacturers should strive to keep acrylamide content to a minimum. Numerous studies have dealt with the formation of acrylamide, and the factors involved^{24,25}. Many methods have been explored in order to reduce the acrylamide content²⁶, but it still seems that the most effective way to control the acrylamide formation is to reduce the reducing sugar content in the potatoes being processed. This can be achieved by either selecting potatoes with a low natural content of sugar or by artificially reducing the sugar content during the process²⁷⁻³⁰. The mentioned blanching process is amongst the counter measures applied to reduce the sugar content, and hence also for reducing the potential of acrylamide formation. Even though some authors indicate that the health impact of acrylamide is lower than anticipated²³. The reduction of acrylamide is still considered one of the major challenges in potato processing industries.

3.2 Dough based potato products (“Lompe” / “Lefse”)

“Lompe” or “lelse” (hereby denoted “lompe”) is a traditional Norwegian, or Scandinavian, product. It probably originated in Scandinavia, in the 19th century³. It is made by kneading cooked, mashed potato and cereal flour into dough. The dough is then rolled into thin sheets (ca. 2 mm, but different thicknesses may be encountered), cut into oval or round shape, before it is baked/cooked by dry convection heat on a griddle (ca. 15 s each side at 225 °C). A brief overview of the process of “lompe” production is given in Figure 3.

“Lompe” is often used as a wrap for a variety of sweet or savoury dishes. The most common of which in Norway is as a wrap for various cooked or grilled sausages/hot dogs. It is industrially produced in large quantities each year. The main challenge encountered during the industrial production of “lompe” is to achieve the correct texture of the dough. As most “lompe” production is mechanized, the dough must have a certain texture to pass through the production line.

Almost all knowledge about “lompe” processing is based on empirical knowledge possessed by the individual bakers. It is common knowledge that the potatoes must be cold

stored for a 12-48 hours (depending on the cultivar) before the potatoes can be used to make “lompe” dough. It also known that the dough must be processed immediately after dough mixing is completed, as it will soften and become nearly impossible to process into “lompe” if left unattended. Another controllable, yet problematic, aspect of “lompe” production is the water content of the dough. The water content of the dough is decided by the water content of the potatoes used. Due to in-batch and batch-to-batch variations, the baker will have to correct the dough texture by adding dry potato flakes (or in some cases water, if the potatoes have a very high dry matter content). Today, these corrections are done based on empirical experience. It would therefore be highly beneficial to base the addition of dehydrated potato flakes on instrumental measurements to ensure consistency in dough texture.

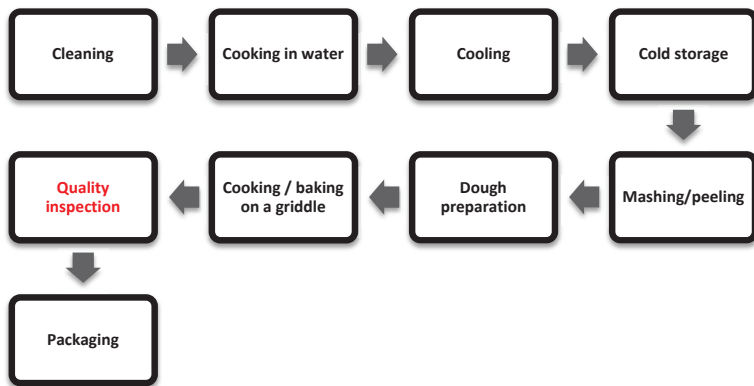


Figure 3: A simplified diagram of the process for production of “lompe”

Although a lot of research has been performed on potatoes, including boiled or mashed potatoes and potato starch, very little research has been performed regarding the ability of the cooked potatoes to mix with cereal flour to form doughs. The only exception is the use of refined potato starch as an additive to products like noodles^{31,32}, but these studies describe the use of raw starch. The challenge in the “lompe” industry still remains; no-one really knows how the dough texture is affected by the different parameters (such as time between heat treating of the potatoes and dough mixing, water content and interaction between the mashed potatoes and the flour).

3.3 Pre-cooked, vacuum packed potato products (*Sous-vidé* products)

Sous-vidé treated potatoes are a convenience product sold for both household and institutional use. They have quite a long shelf life (usually >14 days) and only require a quick reheating

3. Relevant potato products

before being served. The production of *sous-vidé* treated potatoes is shown in Figure 4. After being retrieved from storage, the potatoes are cleaned, sorted according to size and peeled. Since potatoes are prone to enzymatic browning reactions³³, they are dipped in a browning inhibiting solution (usually bi-sulphite or citric acid)³³ after peeling. This also allows some holding time between the different stages of production. The potatoes are packed into vacuum bags of the desired size and submerged in boiling water for a specified time. The cooking time depends on the size of the potatoes and the amount of potatoes in each batch. When the core temperature of the potatoes reaches 92-95 °C, cooling is initiated. The finished product is cold stored to improve shelf life.

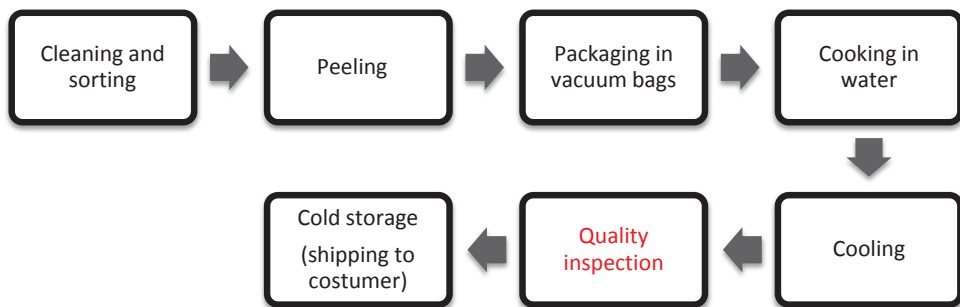


Figure 4: Overview of the process for production of *sous-vidé* treated products

Although *sous-vidé* treated potatoes sound like a desirable and convenient product, it is not easy to produce to a high and even quality. Texture is a key quality trait of cooked potatoes amongst consumers^{34,35}, but as consumers are a diverse group the desired potato texture will greatly change between geographic or demographic groups³⁵. Numerous studies have dealt with different aspects of potato texture (reviewed by Taylor *et al.*³⁵), but few solid conclusions have been made. The relationship mostly agreed upon is the one between cooked potato texture and dry matter content. However, this relationship is not constant and may differ between cultivars³⁶. The general conclusion drawn is that higher dry matter content usually gives mealy texture and low dry matter content gives more waxy, or firm, texture. Starch content is also believed to partly explain the cell disintegration during cooking, which contributes to mealy texture. This relationship is shown to be cultivar-dependant^{36,37}, thus indicating that the disintegration may be affected by other factors as well. Other constituents are also believed to have an influence on the texture. For instance, small granule sizes (<20 µm) are found to contribute to increased soggy texture³⁸. The role of pectin is also studied, and

the results indicate that the enzyme *Pectin methyl esterase* is affecting the texture. Some authors have concluded that higher levels of *Pectin methyl esterase* induce firmer potato tissue after cooking^{39,40}, but more work is needed to fully understand the role of pectin and *Pectin methyl esterase*. Furthermore, some microstructural properties like cell size, and amount of gelatinized starch in the cells are found to be important. However, contradictory conclusions concerning the relationship between these parameters and the texture exist (summarized by Martens & Thybo⁴¹). Some of the challenges seen in everyday production of *sous-vidé* products are uneven texture after cooking and unacceptable hardness in the outer regions of the potato tubers. In addition, some potato batches will leach starchy water from the tubers after or during cooking. This will appear as grey slurry inside the vacuum bags, thus giving an unappealing look of the final product.

Based on present literature, it may be concluded that no universal parameters can yet be used to predict the cooking texture of potatoes. As seen, many of the factors such as dry matter and starch content may give different results for different cultivars. This may in turn indicate that the factors behind potato texture are not fully understood, which is suggested by others⁴⁰, or that the texture is determined by a complex interrelation between all of the above mentioned factors.

3.4 Summary of the challenges

When summarizing sections 3.1-3.3, the need for accurate and representative raw material characterization becomes evident. All of the three processes are mainly relying on quality assessment of the finished product. This indicates that both time and money are spent to produce products with insufficient quality that subsequently must be rejected. However, some key quality parameters could be elucidated from what is known about the different processes. All three industries described rely upon predictions of the dry matter content of the potatoes. Even though dry matter content is not the only factor affecting the product quality, accurate prediction of this parameter may give an important contribution in all the processes discussed above.

The frying industry will in particular benefit greatly from gaining better knowledge about the sugar content of the potatoes being processed. For both dry matter and sugar content, it is beneficial to have sampling methods determining both the batch average values in addition to indicating the variations found within a batch. Producers of *sous-vidé* treated products have a need for better characterization and understanding of their products, along with more rapid methods for raw material assessment. In the case of “lompe” producers there is mainly a need

3. Relevant potato products

to understand and characterize the dough formed from flour and potatoes. Different kinds of mechanical, growth related and pathogen/parasite inflicted defects will also cause losses during processing⁴². These damages and quality defects will not be discussed in this thesis. The same goes for the sugar end disorder⁴³, which is highly uncommon in Norway.

4. Existing and emerging technologies

This section will summarise the technology available for prediction of raw material or product quality. The three main subjects will be identifying the technology to predict or measure dry matter content, sugars and texture. The texture measurements will be focused on the texture of cooked potatoes and the texture of potato based dough.

4.1 Prediction of dry matter content

4.1.1 Traditional methods for prediction of dry matter content

The dry matter content has been the most frequently used chemical quality parameter for potatoes. Since early in the 19th century it has been known that there is a relationship between the density of the potatoes and their dry matter content⁴⁴. The density is expressed as the specific gravity (SG), calculated by the following formula 1:

$$SG = \frac{\text{Weight in air}}{\text{Weight in air} - \text{Weight in water}} \quad (1)$$

where the “weight in water” is the weight of the sample while submerged in water.

In 1880 Maercker *et al.*^{44,45} published a table for converting the specific to dry matter content. According to Lunden⁴, this was not the first publication regarding this relationship, but it was far more accurate and methodical than previous studies. Nissen stated in 1955 that Maercker’s table still was the most commonly used for this conversion⁴⁴.

The use of specific gravity for dry matter determination has been debated for nearly a century, and different studies usually find slightly different models for the conversion. A comparison of the most commonly encountered models is given in Figure 5. The conversion most commonly used in Norway today is the one developed by Lunden⁴ during the 1950s. The linear regression model found by Lunden is shown in equation 2:

$$\% \text{ Dry matter} = 215.73(SG - 0.9825) \quad (2)$$

It is known that the results of these predictions are affected by parameters such as water temperature, or even minute air bubbles trapped in micro cavities at the potato surface^{46,47}. This combined with the large number of similar, but yet different, regression models are a clear indication that the relationship is not constant, and caution is advised when using the underwater weight. It is also known that the relationship between specific gravity and dry

matter content may not be constant within a cultivar. A study performed in 1983⁴⁸ showed a large inter tuber variation in specific gravity for the cv. Bintje.

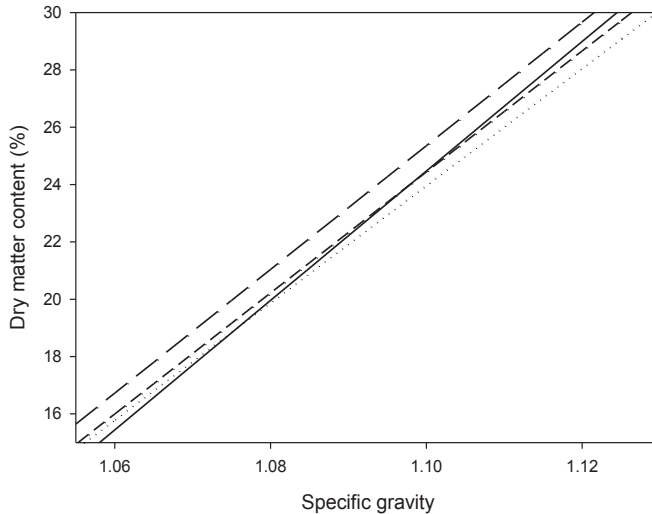


Figure 5: Four of the most common models for conversion of specific gravity to dry matter content are summarized. The models shown⁴ are developed by Lunden (long dashes), Maercker (dots), von Scheele (short dashes) and Kristensen (solid line)

The authors indicated that it was necessary to sample at least in triplicate from each lot of tubers⁴⁸. These findings are supported by other studies^{11,49,50}, although the magnitude of the variations found may vary. As the starch content is correlated to the dry matter content, the specific gravity is commonly used for starch determination too⁴. An EU regulative⁵¹ states that specific gravity (or specifically the underwater weight of 5050 g potatoes) should be used as a reference method in the starch industry for calculating the amount of starch in lots of potatoes. Tables are issued by the EU commission for the conversion of SG to starch content. The use of the underwater weight of potatoes to predict both the dry matter content and the starch content is however disputed⁴⁸. Unfortunately, the specific gravity measured by the underwater weight is not able to detect the variation in dry matter found within a batch of potatoes.

During the first half of the 20th century a new method for graduation of potatoes based on specific gravity was developed (described by Alexander *et al.*⁵² The method uses brine solutions to sort potatoes based on their density (i.e. specific gravity). This method is sparingly described in modern literature, but at least one application has been published. Pflug

et al. published in 1950 the drawings for a machine⁵³, which utilized this method, for grading potatoes into two different categories. An illustration of the machine devised by Pflug *et al.* is shown in Figure 6. It can be seen that the machine has a lot of different mechanical parts and thus is complex to operate and maintain. The brine graduation is very sparingly described in more modern literature, which is probably due to the labour intensive operation and corrosion issues introduced by the brine solution. The method of using specific gravity for dry matter prediction was however not abandoned, and in 1985 Tai *et al.* described what they called the “Grav-O-tater”, which is a computer controlled apparatus for prediction of specific gravity. Computer controlled scales similar to the “Grav-O-tater” are still the most common way to prediction dry matter content in Norway today. The models developed by A.P. Lunden⁴ are usually used to convert specific gravity to dry matter content.

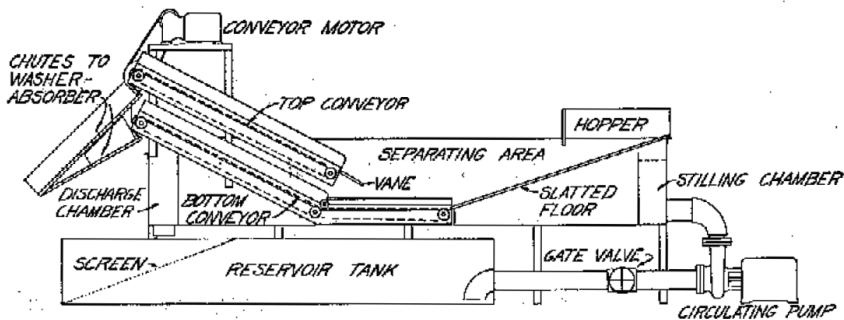


Figure 6: A drawing of the sorting machine devised by Pflug *et al.* in 1950⁵³. The machine uses a brine solution with a pre-determined density to grade the potatoes based on whether they sink or swim. Potatoes were fed from hopper on the top right, and exited via two different chutes on the left

4.1.2 Emerging technology for prediction of dry matter content

The first application of Near infrared spectroscopy (NIRS) to potatoes was seen in the late 1980s, when Dull *et al.*⁵⁴ used NIRS to predict the dry matter content of potatoes. Dull *et al.* used NIRS to predict dry matter content in both intact ($R^2=0.84$) and sliced ($R^2=0.95$) potatoes. This sparked an interest for NIRS as a method for prediction dry matter content.

In general, NIRS is frequently used for rapid, reliable and non-destructive prediction of food quality parameters, such as fat content in meat or meat content in crabs^{55,56}. Sorting of both animal and vegetable based foodstuffs is possible⁵⁷⁻⁶⁰. NIRS measurements are usually used to sort foodstuffs based on the chemical composition, but it has also been used to measure more physical parameters such as the gross meat content of intact crabs⁵⁵.

4.1.2.1 *The working principle of NIRS*

NIRS uses light between infrared and visible light, normally from 760 nm to about 2500 nm and takes advantage of the light's ability to induce molecular vibrations. Vibrations can be induced in molecules, or parts of molecules, that contain molecular bonds with an uneven charge distribution. These bonds are denoted dipole bonds and the dipole strength (i.e. the difference in charge) directly affects the light absorption. The bonds commonly observed as bands in the NIR region are C-H, O-H and N-H groups⁶¹. When light is transmitted into a sample, it will absorb some of the energy of the light. If the right amount of energy is transferred, the molecules in the sample are excited to a higher vibrational energy level. Light at different wavelengths contains different amounts of energy and the excitation of specific molecules in the sample can therefore be seen as absorption bands at specific wavelengths. The absorption bands can be divided into two groups, fundamental vibrations and overtones. The fundamental vibration is caused by the first excitation state, while the overtones are caused by excitation to the second and third excitation state. In addition, combinations of fundamental vibrations can be observed. Most fundamental vibrational states occur in the energy regions of the infrared light (IR). Thus, mostly overtones and combination bands are seen when using NIRS. The bands seen in the NIR region are often broad, compared to bands in the IR region⁶¹. Since the absorption, and thus the vibrations are specific to each chemical bond, it is possible to identify and quantify compounds based on the light absorption of the sample⁶¹. A spectrometer usually records the intensity of the light exiting the sample, and converts it into absorbance via Lambert-Beers law, which is given in equation 3:

$$\text{Absorbance} = \log \frac{I_0}{I} \quad (3)$$

Here I is the transmitted light and I_0 is the incident light.

Three different sampling configurations are commonly used to record NIR spectra. An overview of the three methods can be seen in Figure 7. The first method is transmission measurements, where light is sent through the sample (i.e. the light source and the detector is on opposite sides of the sample). The second is interreflectance measurements, which optically or physically blocks direct surface reflection and hence, measures light that has travelled to a certain depth in the sample. The third sampling method is the reflectance measurements, which measures the surface reflection of the sample. All three configurations can be used with

or without contact between the light source/detector and the sample. Absorbance spectra can then be used for multivariate regression, with or without pre-processing.

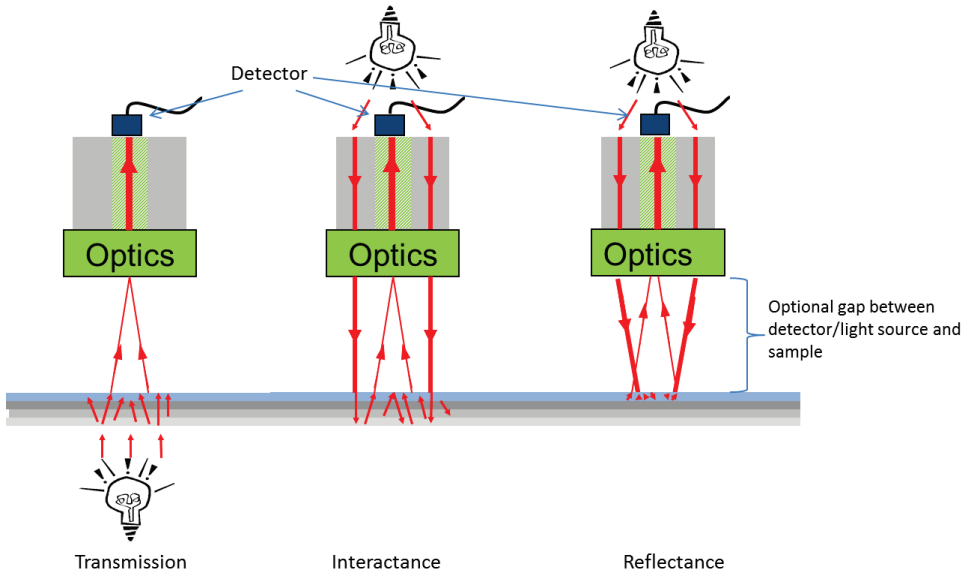


Figure 7: The three most common sampling principles for near infrared spectroscopy. The detector used for NIRS is a spectrometer. From left to right: Transmission, interactance and reflection measurements

4.1.2.2 Pre-processing and multivariate regression

Pre-processing is a term used to denote mathematical techniques for removal of artefacts and unwanted physical effects in the spectra⁶². One of the most basic forms of pre-processing is the normalization of the spectra. Normalization removes multiplicative effects by dividing each variable in the spectrum with a constant. Selection of this constant may depend upon the application, but the average of all samples is commonly used⁶². NIR spectra are commonly pre-processed by a technique called Standard Normal Variate (SNV) transformation. SNV transformation was designed to separate physical and chemical variation in interactance spectra⁶³. The SNV transformation is performed on each spectrum individually, by first calculating the average value of all the spectrum variables (i.e. each wavelength measured) and subtract this from the original spectrum values. SNV transformation will remove most spectral features caused by physical phenomena, such as sample thickness. Furthermore, the

standard deviation of the spectrum is calculated and the normalized spectrum is divided by the standard deviation. Figure 8a shows spectra before pre-processing has been performed. Figure 8b shows the same spectra after SNV transformation has been applied. A range of other pre-

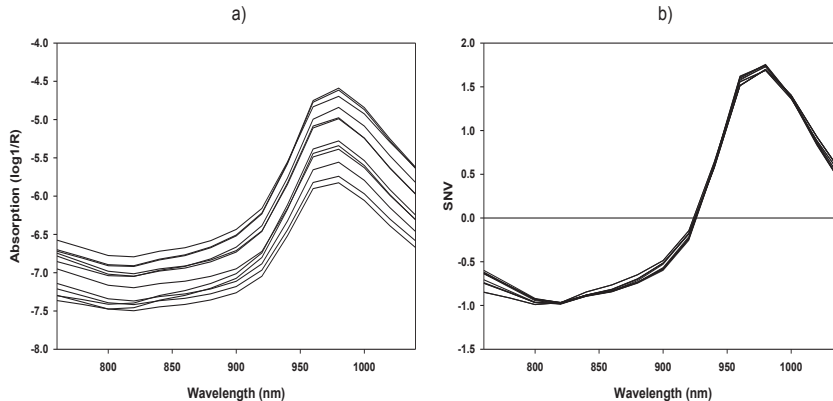


Figure 8: Spectra before (a) and after (b) SNV transformation. The spectra shown are from potatoes

processing techniques do exist⁶⁴, but will not be discussed in the context of NIRS.

After sufficient pre-processing has been performed, the spectra can be used for multivariate regression. Ultimately, the regression model will be used to calibrate the instrument, and allow prediction of attributes of the sample. Many different algorithms exist for multivariate regression⁶⁵. One of the most commonly used is called Partial Least Squares (PLS) regression⁶⁶. The PLS regression is decomposing the spectra data matrix (\mathbf{X} matrix) into scores and loadings, much similar to a Principal Component Analysis⁶⁵. However, in PLS regression the decomposition of the \mathbf{X} matrix is guided by the response variables matrix (\mathbf{Y} matrix), hence maximizing the covariance between the two matrices. The PLS method is decomposing the co-variation between \mathbf{X} and \mathbf{Y} matrices into a series of factors, where the first factor is representing the main variation. Based on this, a regression model can be created from the most relevant variation in both \mathbf{X} matrix and the \mathbf{Y} matrix⁶⁵.

After creation of the PLS regression model, its performance must be checked, or validated. One way to perform the validation is to use an independent set of samples. This gives a thorough validation, but does require a large number of samples and hence, is often expensive⁶⁵. An alternative is to use a method denoted “Cross validation”. This method creates a model based on all samples except one (sometimes several samples can be kept out), followed by a prediction of the sample that was kept out. Then a new model is created, but

this time keeping the next sample out of the calibration. When all samples have been kept out once, the performance of the model can be calculated. The final regression model created is the average model of those calculated. The performance of the PLS model is usually given as the determination coefficient (R^2) and the Root Mean Square Error of Cross Validation. The latter is calculated by equation 4:

$$\text{RMSECV} = \sqrt{\frac{\sum_{i=1}^I (\hat{y}_i - y_i)^2}{I}} \quad (4)$$

where i denotes the samples from 1 to I . y_i and \hat{y}_i denotes the reference value and the predicted value, respectively.

4.1.2.3 NIRS for prediction of dry matter content in potatoes

After the initial report by Dull *et al.*⁵⁴, the use of NIRS for prediction of dry matter content is demonstrated in a number of different reports. A variety of different sample preparations is described. A review written by López *et al.*⁶⁷ in 2013 summarized the literature on NIRS regarding potatoes. It showed some interesting trends about the prediction performances of NIRS for prediction of dry matter content in potatoes. The use of high resolution laboratory instruments is frequently shown and it is clear that the highest determination coefficients ($R^2=0.98-0.99$) are achieved when using pulped potato samples, as shown in the studies by Haase *et al.*⁶⁸⁻⁷⁰. NIRS for prediction of dry matter content in various homogenized samples, with determination coefficients ranging from 0.58 and upward, were found. The bulk of the studies report determination coefficients from 0.81 to 0.97. Studies concerning whole potatoes do in general show lower prediction performance ($R^2 < 0.90$) than those dealing with homogenized samples⁶⁷. Subedi and Walsh⁷¹ obtained in 2004 similar results ($R^2=0.85$) as Dull *et al.*⁵⁴, for prediction of dry matter content in whole potato. However, they only subjected a small potato cube, from directly beneath the measurement location, for reference analysis. Subedi and Walsh⁷¹ also showed that potato slices could be measured while travelling on a conveyor belt. They obtained a determination coefficient of 0.95 for the slices.

Attempts have been made to make equipment suited for use in parallel with a raw material stream (at-line). In 2010 Brunt *et al.*⁷² performed a two part study, investigating the possibility of developing a NIRS based method for at-line determination of dry matter content in homogenized samples. They obtained a determination coefficient of 0.94. The aim for the

second part of their study was to develop the final measuring apparatus they employed the under-water weight approach⁷³. They did, however, use NIRS for prediction of starch and coagulating protein. Most studies do correlate NIRS measurements with the dry matter content of the potatoes directly, but exceptions are found. Chen *et al.*⁷⁴ predicted the specific gravity of potatoes ($R^2=0.90$) by using NIRS⁷⁵.

The feasibility and working principle of NIRS for prediction of dry matter content in potatoes are well documented. Still, the application of NIRS in factories and sorting facilities is not well established. This is probably due to the lack of high speed and high through-put instrumentation needed for such applications.

4.2 Measurements of sugars

4.2.1 Traditional methods for determination of sugar content in potatoes

The amount of reducing sugars in potatoes has become increasingly more interesting as the wish to control the colour of the chips and crisps became stronger. Due to the low amount of sugars found in potatoes, sensitive and precise methods are required for determination. The use of such methods in an industrial environment also calls for methods that are easy to use, fast and cheap. This chapter will discuss some of the most important methods used for quantification of sugars in potatoes today.

Traditionally, a wide variety of chemical approaches have been used to quantify sugars in various food and plant samples⁷⁶. During the first half of the 20th century various wet chemistry methods were the only options. Methods such as the di-nitro salicylic method (DNS) are reported to give good results for measuring reducing sugars^{77,78}. Other test methods such as the glucose oxidase based enzymatic test kits, for instance those supplied by Megazyme (Megazyme Ltd, Ireland) opened for easy and reliable sugar quantification. Yellow Spring Instruments (YSI) released a semi-rapid instrument for glucose determination in the mid-1970s⁷⁹. The YSI instrument (current model: “YSI 2950 Select, Biochemical analyser”) is commonly used as a reference method for determination of reducing sugars in plant and animal based fluids. Unfortunately, the YSI instrument requires a certain amount of training to use, and it is not portable. In addition to all the mentioned methods, a range of different chromatographic techniques is available, like ion exchange chromatography⁸⁰. The advantage with these methods is that they can determine glucose, fructose and sucrose. However, many of these methods are both time consuming and labour intensive and they are not suited for rapid industrial use. Some methods do also require use of various chemicals and

instrumentation not commonly found at a potato processing factory. Methods suited for daily use in an industrial environment should ideally give the results within a few minutes, are easy to operate and require minimal sample preparation.

In the early 1990s a new type of glucose measuring technology emerged⁸¹. Glucose test strips originally emerged in the field of medicine. The very first tests consisted of small tablets that were dissolved in the patients urine, or as an enzyme coated test strip⁸¹. The colour of the urine solution or urine treated test strip was matched against a colour scale supplied along with the kit. In the late 1980s, Louwes and Neele^{82,83} published two studies showing that the Reflocheck glucose strips (Boehringer Mannheim, Mannheim, Germany) could be used to predict the colour of potato crisps. They found the test strips to correlate well with the frying colour, but slightly overestimating the glucose content.

In the 1970s and 1980s a series of new blood glucose meters, termed portable blood glucose monitors (PBGM) were developed⁸¹. These meters were designed to measure the glucose in blood, rather than in urine. They used glucose oxidase / peroxidase coated test strips, which was the same as the early test strips. The new instruments used colorimetric determination of the colour of the test strips, some with the option for manually readings (i.e. comparing the test strip to a colour scale)⁸⁴. The first report utilizing a PBGM was published early in the 1990s⁸⁵. The PBGM was easy to use, with application of potato cell sap / potato juice to a small sample stick/strip. Excess potato juice was after a given time (commonly 1 minute) wiped off and the test strip was inserted into the monitor itself. A glucose reading was shown in the display of the instrument. A study by Coleman *et al.* showed the use of a PBGM, without any modification of the system, and the results were encouraging ($R^2=0.82$)⁸⁵. Good correlation between glucose measured with a PBGM and frying colour of the potato crisps were reported⁸⁶, along with a modified procedure to measure fructose⁸⁷. In 1996 a report of a modified system was published, accommodating a wider range of glucose levels (5-50 mmol/L, with $R^2=0.76$)⁸⁸. However, the evolution of PBGMs has been rapid and the technology demonstrated by Coleman *et al.*⁸⁵⁻⁸⁷ is now obsolete⁸¹. During the 1990's the technology of the PBGMs evolved into a newer enzyme technology⁸⁴ and a more user friendly interface⁸¹. There were no longer a need to first apply the sample, wait, and then wipe off the excess. This sample handling was seen as a major drawback with the older PBGMs, as it allowed the user to heavily distort the reading⁸¹. The new PBGMs the test strips are inserted into the monitor and the sample is then applied by capillary suction into the test strip, thus eliminating most of the human factor^{84,89}. The monitors themselves now used electrochemical

detection, instead of the colorimetric detection found in the older ones. The sample volume has decreased from 50 to 1 μL ^{84,89}.

Test strips are now available with different enzyme combinations. Glucose dehydrogenase combined with flavin adenosine dinucleotide or glucose dehydrogenase combined with pyrrolo quinolone quinone are two of the most common enzyme combinations in moderns PBGMs⁸⁴. Glucose oxidase /peroxidase combinations can also be found. A schematic overview of how the enzymatic test strips work is shown in Figure 9⁸⁴. The glucose present in the sample solution is oxidized into Glucono lactone by the main enzyme, which is then reducing a mediator. The mediator is oxidized at the detection electrode, also present in the test strip. The potential is read by the PBGM, and converted into a glucose reading.

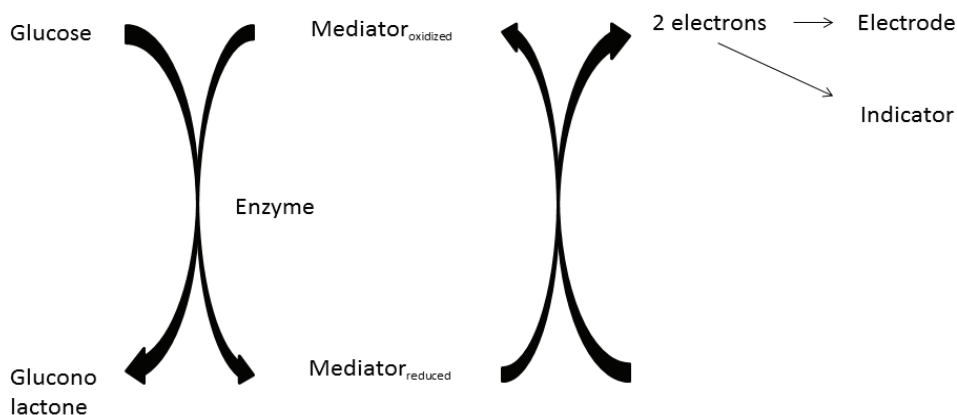


Figure 9: Scheme of enzyme and mediator action for enzyme strips in portable blood glucose meters⁸⁴

Although frequently used in potato processing industry, little research has been performed on the quality of the more modern PBGMs for determination of glucose in potato juice. Available literature about the new PBGMs is restricted to one conference proceeding⁹⁰, which reported a determination coefficient ranging from 0.78 to 0.92 for the correlation between two PBGMs and a reference method. However, the newer monitors are demonstrated for fermentation broths, giving determination coefficients in the range of 0.83 to 0.99 between glucose content measured with two PBGMs and a YSI analyzer⁹¹.

In addition to the mentioned PBGMs, one alternative is available from Merck Millipore (Merk KGaA, Darmstadt, Germany). This instrument is called RQFlex and is equipped with Reflectoquant test strips. The technology is similar to the older PBGMs. Strips supplied with the instrument are soaked in sample solution, wiped off and inserted. The enzyme technology

used is glucose oxidase peroxidase combination, with colorimetric detection. A wide variety of different Refectoquant test strips are available, and the RQFlex is the only rapid instrument capable of determining other sugars than glucose. The drawback is that different test strips must be used for each sugar, and the procedures for both the total reducing sugar strips and the sucrose strips are considerably more time consuming than the glucose test. The RQFlex is very sparingly described in available literature, and further discussion of this instrument can be found in section 5.2.

4.2.2 Spectroscopic techniques for prediction of sugars content in potatoes

Along with the evolution of spectroscopic techniques, the development of methods for sugar prediction followed. Attempts^{70,92,93} have been made to use NIRS for prediction of total reducing sugar content in potatoes, but good results have not been obtained. Mehrubeoglu *et al.*⁹² investigated NIRS for prediction of sugars in two potato cultivars (Russet and Chipping). Low prediction performance ($R^2=0.62$ and 0.52) was obtained for the two cultivars individually. If combining the two cultivars into the same regression model, the prediction became useless ($R^2=0.27$). Others have reached similar conclusions for use of NIRS for sugar prediction in potatoes⁷⁰. The use of IR and Fourier transform infrared spectroscopy (FTIR) has been widely demonstrated for various food components^{94,95}. Sugar analyses of juice from sweet fruits such as apples are also demonstrated⁹⁶. Another well-established use of FTIR is for raw material characterization in the dairy industry. Commercial instruments such as the MilkoScan (FOSS, Denmark)⁹⁷, and LactoScope (Delta Instruments, The Netherlands)⁹⁸ utilize FTIR to predict various components and properties of milk and other dairy products. The specification supplied by the vendors of these instruments state that they are accurate at a “0.8-1 %” (assuming weight %) level for lactose (milk sugar). Unfortunately, the amount of glucose in potatoes used for processing is usually well below 2 % (equivalent to 20 mg/mL)⁹⁹. This renders these commercially available instruments useless for glucose prediction in potato tubers.

The sugar concentration in potato juice does not seem to be easily determined by traditional spectroscopic methods. One solution is to remove the water in the sample, for instance by drying the sample. The FTIR dry film approach was first introduced in the early 1990's¹⁰⁰, and has since been applied to applications like qualitative blood analysis¹⁰¹, characterization of microorganisms¹⁰² and prediction of fatty acid composition in milk samples¹⁰³. This approach involves that the sample is pipetted onto a silicon plate, dried into a

thin film, and measured in transmission mode by a FTIR instrument. The drying, and hence also the pre-concentration of the samples, allows detection of otherwise undetectable amounts of analyte. Even though many applications of FTIR dry film analysis can be found, no literature regarding the use of FTIR dry film on potato juice has been found.

4.3 Rheological and textural measurements

4.3.1 Measurements of dough quality

At present, very few instrumental measurements are performed on “lompe” dough at the processing facility. The industrial “testing” of “lompe” dough is based on hand feel. A variety of scientific reports can be found on bread dough, and various other dough types. It is common to measure parameters such as water absorption and the falling number (i.e. Hågerbergs falling number, which is a measure of the general enzyme activity in the flour) of the flour to predict dough quality. Similarly, the volume of the finished bread is commonly found in literature concerning flour doughs, as a quality measure of the dough. None of these flour specific quality traits will be considered in this thesis. Therefore the main focus will be on test methods suitable to characterize dough for “lompe” production, as limited knowledge about “lompe” dough is available. The main method for “lompe” dough characterization is expected to be rheology, to assess the structural behaviour of the dough. The following sections will give a brief introduction to the rheology relevant for experiments performed in this thesis.

4.3.1.1 Classical rheological measurements

The term “rheology” originates from the Greek word “rhein” which means “to flow”. Rheology is seen as the science about flow and deformation, and is most commonly used to determine properties such as viscosity and elasticity of substances. Very simplified, one can say that rheological measurements are carried out by placing the sample between two plates. The plates are then carefully put into movement, and the response of the sample is measured. Response variables connected to the motion are then mathematically converted into rheological parameters such as viscosity. Rheological behaviour can be separated into two extremes, ideally viscous samples, and ideally elastic samples¹⁰⁴. An ideally viscous sample can be illustrated by very low-viscous mineral oil. It moves freely, and will not spring back once moved. An ideally elastic sample can be illustrated by a piece of spring steel. It can be slightly bent, but will spring back into its original shape once the force is removed. There is

an unlimited number of samples that are both viscous and elastic. Such samples are denoted as viscoelastic and are commonly divided into viscoelastic liquids and solids, based on whether they are most viscous or most elastic. A graphical illustration of the differences, along with some examples, is given in Figure 10. A paste is typically a viscoelastic liquid, while a piece of rubber is usually a viscoelastic solid. Most doughs are viscoelastic solids.

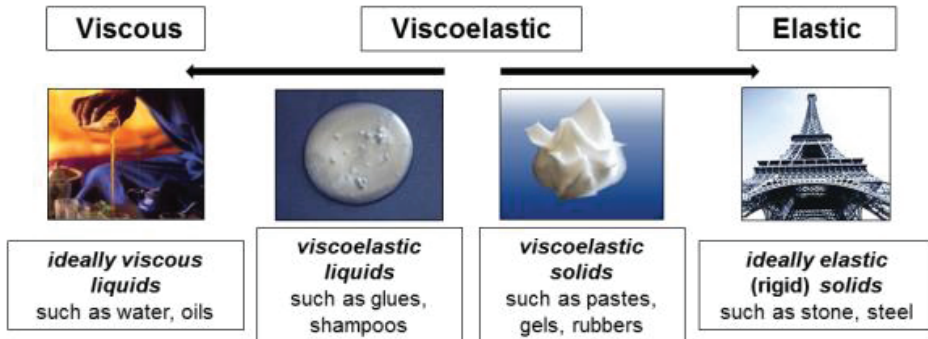


Figure 10: Rheological behaviour can be divided into elastic, viscous and everything in between, which is denoted viscoelastic¹⁰⁴

To be able to measure liquids, solids, or any sample in between, a number of different sample holders have been developed. The sample holders are usually denoted “measuring geometry”. During the measurement, the sample is confined within the measuring geometry. The two most common sample geometries are known as “plate-plate” and “bob-cup”, which are shown in Figure 11. The plate-plate measuring geometry can be divided into parallel plate (Figure 11b) and cone plate (Figure 11c). All three geometries can be found in different sizes, and are usually made in accordance with ISO or DIN standards¹⁰⁴. The cone plate is not suitable for dough studies, since it has strict requirements to the maximum particle size in the sample. Both bob-cup and parallel plates can be used for measurement of doughs. Modern rheometers can operate in two fundamentally different modes. The measuring geometry can either rotate or oscillate (i.e. turn a controlled number of degrees back and forth). Rotational measurements are mainly used for liquid samples, and are of limited interest to dough analysis. Oscillatory measurements are better suited for solid samples. Such experiments can be performed without destruction of the sample structure. A few basic parameters are set by the user before the start of the measurement.

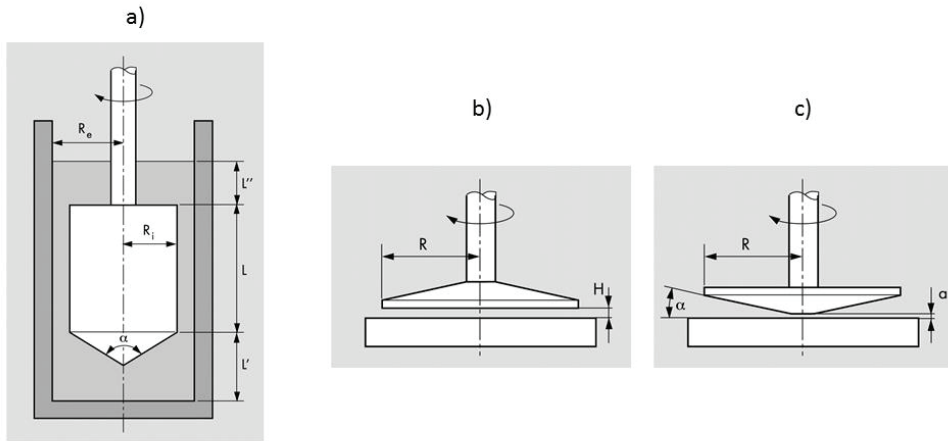


Figure 11: The three most common measuring geometries used in rheological analysis¹⁰⁴. a) bob-cup, b) parallel plate, c) cone plate

Oscillatory measurements is commonly performed in two different ways, either by setting the shear stress (T), or by setting the strain (γ)¹⁰⁴. Controlled strain is the measurement setting that will be further discussed. The strain is defined by equation 5¹⁰⁴:

$$\gamma = \frac{s}{h} \quad (5)$$

where s denotes the distance (i.e. the amplitude of oscillation) and h is the sample height. The strain is dimensionless, but a strain equal to 1 means that measuring geometry has moved a distance equal to the sample height. Concerning the plate-plate geometry, this distance is measured at the outer edge of the plate. Furthermore, the frequency of the oscillation will impact the results of the measurement. This frequency is denoted angular frequency (ω), and is commonly set to 10 radians per second (rad/s)¹⁰⁴. As both the angular frequency and the strain are predefined by the user, one must take steps to find the correct values. The definition of “correct” will depend on the wanted outcome of the measurement. In the case of dough assessment, one often wants to look at dough properties at rest, i.e. the measurement should not distort the internal structure of the sample.

Before further discussing the internal structure of a sample, a few key terms should be explained. The complex shear modulus (G^*) is one for the most used response variables from a rheometer and is given by Hook's law, equation 6

$$G^* = \frac{T(t)}{\gamma(t)} \quad (6)$$

where $T(t)$ and $\gamma(t)$ are given as time variables. $T(t)$ has the unit Pascal (Pa). G^* is given the unit Pa, since $\gamma(t)$ is dimensionless¹⁰⁴. The internal structure of a sample is commonly denoted by the storage and loss moduli (G' and G'' , respectively). G' will contain information about how the sample is able to store energy from the deformation (i.e. the elastic properties of the sample). The G'' is containing the information about how structure is lost during the applied deformation, i.e. the viscous part of the sample. If $G' > G''$ the sample is considered a solid, and vice versa, if $G' < G''$ the sample is considered a liquid¹⁰⁴. The G^* value, and hence also G' and G'' will depend on the measuring condition applied. G' and G'' are derived from G^* by vector decomposition, as illustrated in Figure 12. The relationship between G' and G'' is expressed as the tangent of the angle ($\tan\delta$) between the two vectors G' and G'' . $\tan\delta$ is used to describe the interrelation between G' and G'' . For instance, a decrease in $\tan\delta$ will signify that G' is increasing in proportion to G'' ¹⁰⁴.

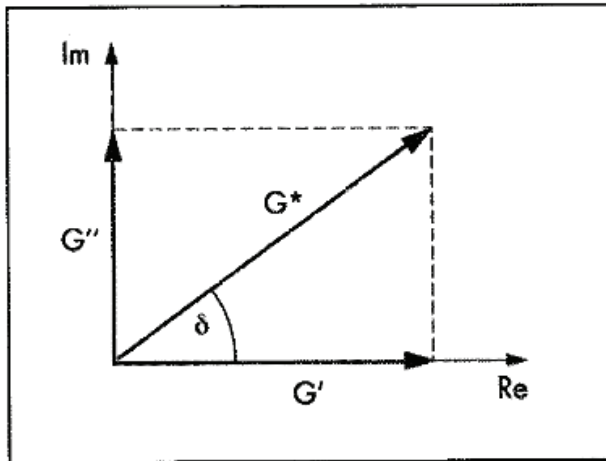


Figure 12: The vector decomposition¹⁰⁴ of G^* into G' and G''

For samples like dough, it may be of interest to perform measurements that do not affect / destroy the internal structure of the sample. The range of strain values that can be applied

without destruction of the samples internal structure is denoted the linear viscoelastic range (LVE-range). Within this range, a sample is per definition at rest¹⁰⁴. In order to determine the LVE-range, an amplitude sweep is performed. An example

of an amplitude sweep is shown in Figure 13. The strain (i.e. the amplitude of the oscillation) is set to a very low value (e.g. 0.01) at the beginning of the experiment, but does ramp up to high values (e.g. 10 or 100) within a user defined time span. The LVE-range is defined when the storage (G') modulus is starting to decrease. The strain at this point is often denoted strain_{limit}, (γ_L). The point where the curves for G' and G'' are intersecting is called the flow point and marks at what strain the sample is turning into a flowing liquid. After establishing the LVE-range, a wide number of measurements can be performed. For instance time dependent measurements can be run, measuring changes in the sample as function of time.

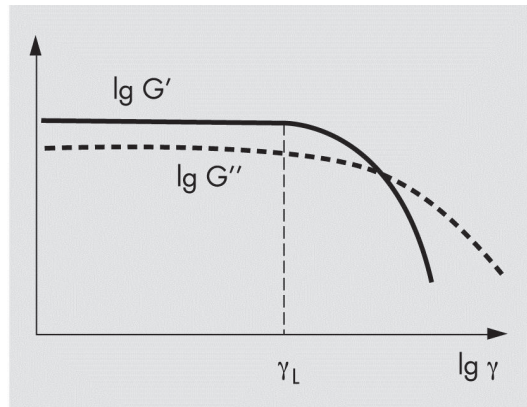


Figure 13: An amplitude sweep¹⁰⁴ is used to determine the LVE-range of a sample, based on the behaviour of G' and G'' . The scale of the y-axis will depend on the sample; Pascal (Pa) is usually the unit.

Both axes are logarithmic

4.3.2 Texture of boiled potatoes

In this thesis, textural measurements of potato products will be restricted to *sous-vidé* treated products. The texture of cooked potatoes are quite extensively studied in the literature, and both sensory and mechanical methods have been applied¹⁰⁵. Methods such as grid extrusion¹⁰⁶ have been demonstrated, along puncture and cut force tests¹⁰⁵. It is claimed that it is not possible to fully predict the sensorial properties of potatoes by mechanical or instrumental measurements¹⁰⁵. Attempts using NIRS and nuclear magnetic resonance (NMR) spectroscopy have been made¹⁰⁷, but neither NIRS nor NMR alone is able to give good predictions of the texture. From section 3.3 it is clear that dry matter content can contribute to mealiness / waxiness of cooked potatoes. This may explain why NIRS and NMR can give a partial prediction of the potato texture. A combination of uniaxial compression, and NIRS or NMR is able to predict 45 to 70% of the sensorial quality¹⁰⁷. The maybe most studied instrumental

characterization method for cooked potatoes is the uniaxial compression method. Although unable to predict the sensory quality of the potatoes, the uniaxial compression is capable of predicting some parameters with good accuracy. Uniaxial compression tests are performed by careful and controlled compression of the sample, while measuring the compression resistance. A compression to 25 % of the original height is commonly used in combination with 10 mm high samples. The diameter, or cross section, of the sample can vary, but 10 mm is frequently used¹⁰⁸. Hardness, fracturability, springiness and firmness are all parameters well within what the uniaxial compression can predict^{41,109}. Figure 14 shows a typical curve from a uniaxial compression test. Hardness and fracturability can be measured as the peak force as indicated in the figure. Other parameters such as chewiness and cohesiveness can be calculated from the areas (A_1 and A_2) below the curve^{109,110}.

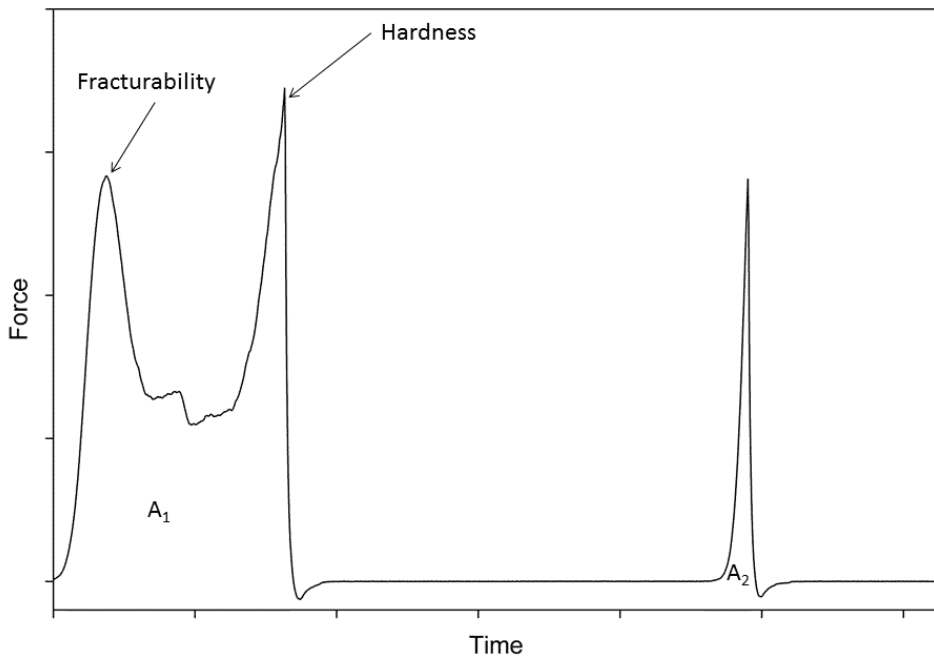


Figure 14: The curve of an uniaxial compression test

Hence, uniaxial compression will give information about the texture, but not the taste and mouth feel desired by the consumer. Uniaxial compression must therefore be used in tight conjunction with the producers to find the quality they want for their product. For industrially producers of *sous-vidé* potatoes, it may be advantageous to know the dry matter content of each individual tuber. This may allow sorting into different groups, requiring different

4. Existing and emerging technologies

processing parameters (e.g. cooking time). This is mainly applicable if one wants to obtain even quality and not necessary accurately predict the sensorial properties of the product. Exact sensorial prediction will require sensorial testing of the product ¹⁰⁵.

5. Results and Discussion

5.1 Paper I and II: NIRS for online prediction of dry matter content

Today, the potato processing plants only measure dry matter content as an average of each batch. Hence, the variation of the dry matter content is not assessed on an individual tuber level, and subsequent sorting is not possible. The development of NIRS for prediction of dry matter content in potatoes was summarized in chapter 4.1.2. A few studies have shown prediction of dry matter content in whole potatoes, but very few industrial applications of NIRS for dry matter prediction have been found. Many sorting solutions for potatoes do exist, like sorting of outer defects or green tubers. Although some of these solutions may have the potential for sorting by dry matter content, none of these seems to be commercially available yet. One example is the Insort Sherlock sorter (Insort GmbH, Kirchberg an der Raab, Austria), but presently the Insort webpage¹¹¹ still reports the sorting by dry matter content as an application “under development”. This lack of good methods for online prediction of dry matter content, and the desire to develop such, was the main motivation behind paper I and II.

A previously developed prototype NIRS instrument was available for testing⁶⁰. This instrument was designed with low resolution and high sampling speed. A schematic overview of the light path of the instrument is given in Figure 15. The instrument is equipped with two 100 Watt light bulbs (at the moment, Pk22s supplied by General Electric, Fairfield, CT, USA) and a light collection tube (marked B in Figure 15)

physically blocking all surface reflection. Folkestad *et al.*⁶⁰ describe the instrument as having two 50 Watt bulbs, but this has later been changed to two 100 Watt bulbs. The spectrometer installed in the prototype reads 30 equally spaced wavelengths (channels), ranging from 449-

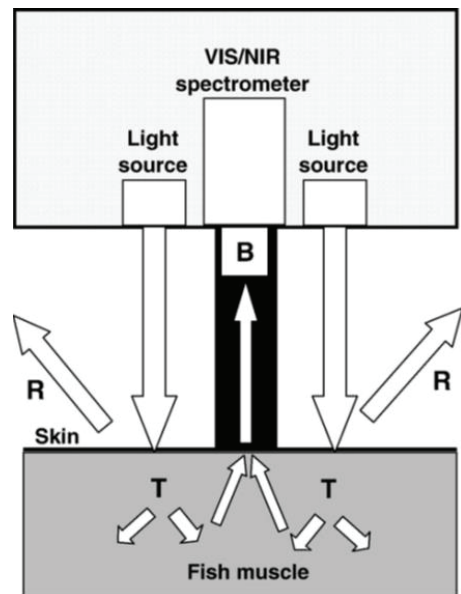


Figure 15: Schematic overview of the light path of the NIRS prototype instrument developed by Nofima⁶⁰. Potato tissue was probed, instead of fish muscle. (R) – Reflected light, (B) – Optics, collecting the light, (T) – Light transmitted through sample

1050 nm. This range was divided into two sub-ranges, 449-744 nm representing the visible light and 760-1050 nm representing the NIR light. In both paper I and II, the visible light was excluded from the measurements, as both potato skin and potato flesh shows large colour variation. The skin and flesh colour is cultivar specific, and no indications of a relationship between flesh/skin colour and the dry matter content has been found.

5.1.1 Contribution from paper I

Paper I deals with the basic application of the NIRS prototype instrument for assessment of dry matter content, compared to the traditional method (the underwater weight) and a commercially available NIRS instrument supplied by QVISION (now part of TOMRA Sorting Solutions, Asker, Norway). The underwater weight was constructed in-house to accommodate single tuber measurements. The commercially available instrument was termed the “2D instrument” in paper I, as it supplied a two dimensional image. In this image, each pixel consisted of a spectrum ranging from 760-105 nm with 15 equally spaced channels. The commercial instrument was designed to monitor a conveyor belt up to 60 cm wide, and to handle conveyor belt speeds up to one meter per second. Recordings consisted of the same wavelengths as the NIRS prototype instrument. The main questions to be answered by paper I were; Was the NIRS prototype (termed the “1D instrument” in paper I) capable to accurately predicting the dry matter content of whole unpeeled potato tubers? How deep into the potato tuber could the light penetrate? Furthermore, could the NIRS prototype outperform the traditional potato underwater weight measurements?

The first part of paper I demonstrates that underwater weight of single potato tubers correlated well with the dry matter content ($R^2=0.97$ and $RMSECV=0.68\ %_{DM}$). This was to be expected. However, there is an offset when comparing underwater weight with the reference measurements. Nearly all samples were predicted to be an offset of about 1 percentage. If other regression models were used, a better fit could be obtained. Figure 16 shows the results of using the regression models⁴ obtained by Lunden, Maercker and Kristensen, respectively, are shown. As can be seen, the model obtained by Kristensen appears to fit closest to the target line. This illustrates that the conversion from specific gravity is an estimate, and should be treated as such. It also illustrates the importance of having all parties in a supply chain using the same model.

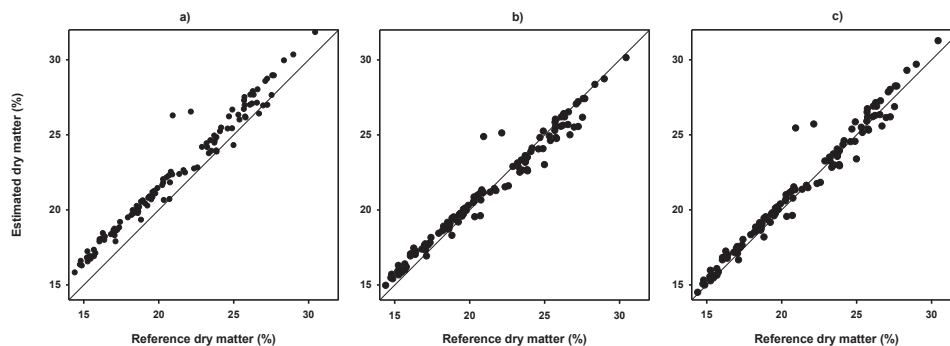


Figure 16: Dry matter content of samples from paper I, predicted by three different regression models⁴; a) A.P. Lunden, b) Maercker and c) R.K. Kristensen. The solid line is a target line, illustrating a perfect relationship between predicted and measured values

The NIRS prototype instrument turned out to work well for prediction of dry matter content in whole, unpeeled potato tubers. A PLS-regression model with 5 factors gave a determination coefficient of 0.95 and an estimation error (RMSECV) of 0.91 %_{DM}. The regression coefficients fit well with known overtones¹¹² of water and starch absorption bands (further discussion in paper I), and no artefacts were found in the spectra. The commercially available instrument, described in paper I, showed poor performance ($R^2=0.83$, RMSECV=1.68 %_{DM}, 9 PLS-factors) compared with the NIRS prototype instrument. The instrument was used to measure potato tubers while they were travelling along a conveyor belt.

As a part of paper I, the light penetration depth was estimated for both of the two instruments used. The term light penetration depth will be used, although in reality, it is the depth from which light is reflected. A photo of a potato being measured by the NIRS prototype instrument is shown in Figure 17a, and the light penetration is illustrated in Figure 17b. It was found that the spectra were changing when the potato sample was thinner than 20 mm, indicating that the light penetrated 20 mm into the potato tuber. Figure 18 shows a schematic drawing of the internal distribution of two potato tubers, and it can be seen that the dry matter content decreases toward the centre of the potato tuber⁶. Similar gradients in dry matter content have also been found by others^{8,113}. This further implies that the light penetration depth, and hence the depth of the measurement will affect the predictions.

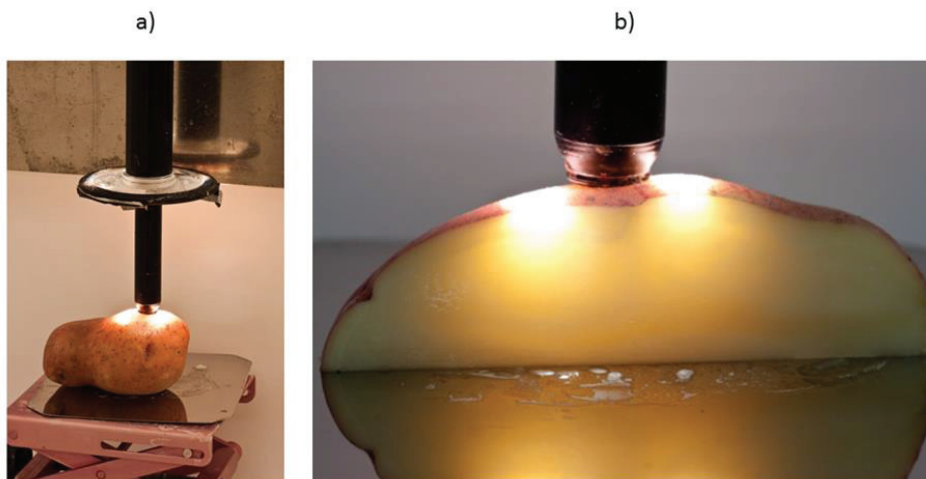


Figure 17: The NIRS prototype instrument used in paper I. a) A view of the light focusing lens and the light collection tube. b) illumination of a quartered potato tuber, with the light collection tube in the middle

Measurements of the light penetration depth revealed a penetration depth of only 10 mm for the commercially available instrument. The shallow light penetration is most probably caused by the reduced output power of the source of illumination in the commercially available instrument. The online instrument used one 55 Watt bulb (supplied by the manufacturer), while the NIRS prototype instrument uses two 100 Watt bulbs. It is likely that shallow penetration is one of the main reasons for low prediction performance. After this experiment, it was suspected that the motion of the potato tubers was also contributing to the low performance. As the ability to

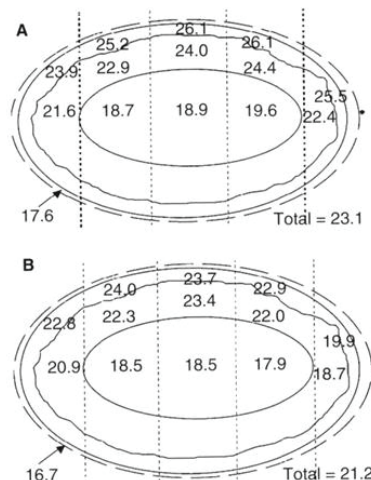


Figure 18: The internal dry matter distribution throughout a potato, reported by Storey⁶. a) is an example of cv. Saturna and b) is an example of cv. Russet Burbank⁶

measure potatoes while they travel along a conveyor belt is essential to develop online applications, further investigations of such measurements became therefore the subject of paper II.

5.1.2 Contribution from paper II

The main aim of paper II was to investigate if the NIRS prototype could be used in a more industrially applicable mode. This meant investigating if the instrument could be used without contact between the light collection tube and the sample, and if samples on a moving conveyor belt could be measured. A part of this was to see if the single line of spectra recorded from a potato moving past the detector, contained sufficient spectral information to build a prediction model. The first part of paper II compared measurements with and without a gap between the sample and the light collection tube. Both contact and contactless configuration gave determination coefficients of 0.89. This is lower than what was obtained in paper I ($R^2=0.95$). As no obvious reasons for the lower prediction performance were found, one can only speculate if this was due to factors such as larger sample set, differences in cultivars or different sample surface morphology. The second and most important question in paper II was whether the NIRS prototype could be used for online measurements. The NIRS prototype was installed above a conveyor moving at 15 cm/s, which gave approximately 25-30 spectra per tuber. The spectra from the potato tuber were then extracted from the raw recording with an in-house built MATLAB (MATLAB, V.7.10, The MathWorks, Natick, MA) routine. This allowed full customization of the extraction in terms of the number of spectra extracted, and the criterion, or threshold for the extraction. The thresholding routine removed the spectra recorded of the conveyor belt and of the ends of the potato. An illustration of the recording before and after the thresholding routine is given in Figure 19a and b. All spectra from each tuber were averaged before they were used for PLS-regression. The approximate positioning of the spectral recordings is shown in Figure 20. Removed spectra are illustrated in a light grey colour in Figure 20, and those used for PLS-regression in red and black. The total number of recordings for each tuber was dependent on the size of the tuber, and the number given in Figure 20 may therefore not be correct for all samples. The threshold gave an average of 15-20 spectra for each potato tuber. The smallest tubers yielded less than 10 spectra after the threshold was applied. Higher prediction performance ($R^2=0.92$) was achieved, when using the spectra from the moving samples for PLS-regression. As the results

in paper II indicate, the number of spectra recorded is of relevance to the prediction performance.

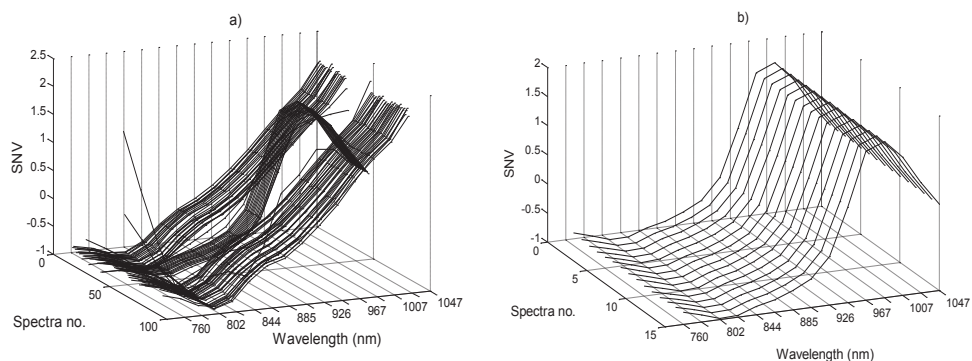


Figure 19: a) Absorption spectra from one online recording, with spectra recorded from a potato showing a clear peak at 967 nm. b) Remaining spectra after a thresholding routine has been applied.

Note that all spectra are SNV transformed to make the differences easily visible

To further investigate the impact of the number of spectra, a selection of the three spectra from the centre of each tuber were extracted and used for prediction. These spectra were from the same area of the tuber as the stationary measurements were taken, and are indicated by the red arrows in Figure 20. When PLS-regression was performed with the three centre spectra as a representation of the whole tuber, the prediction performance was lower ($R^2=0.89$).

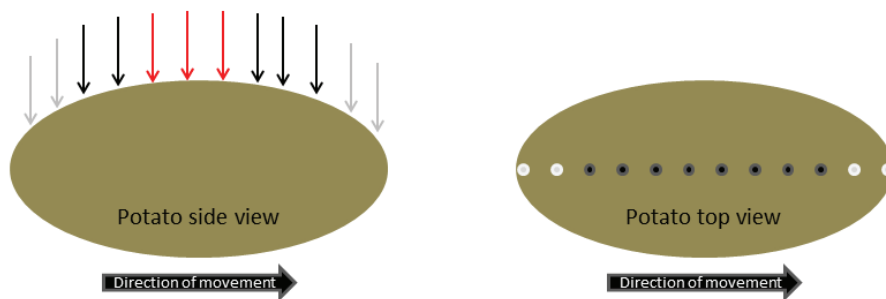


Figure 20: During the online measurements, spectra were recorded along the axis of the tuber. The arrows or dots indicate where spectra were recorded. The total number of spectra was dependant on tuber size

The loss in prediction performance was not drastic, but significant ($p < 0.05$). When only three spectra are used, the chances of interference increases which may also contribute to the lower prediction performance. The prediction performance obtained with the three centre spectra is not significantly different from that obtained with the stationary measurements. This may indicate that the larger sampling area provided by a moving sample may give a more representative sampling technique. When comparing Figure 20 and Figure 18 it becomes clear that each spectrum should yield different dry matter values, as this is not the same over the potato tuber. In paper II it is described how the prediction model developed for whole tubers was used to predict the individual spectra from each tuber. The results are described in detail in paper II, but in summary; they indicate that the dry matter gradient within the tuber is detected by the NIRS prototype instrument.

The ability to detect internal differences in the potato tubers shows that the method is robust, and probably suited for industrial use. As mentioned previously, the commercially available instrument shown in paper I was not able to produce spectra that correlated well with the dry matter content. The NIRS prototype, on the other hand, was capable of doing this. Hence, a few key differences between the instruments should be noted. The commercially available instrument records spectra at a higher rate than the NIRS prototype instrument. The most notable difference is maybe the light source used in the two instruments. Without further testing, it is not possible to conclude whether or not the commercial online instrument would perform better with a more powerful light source, but it may be a feasible explanation for the lack in performance. The goal of both studies was to take one more step toward online application of NIRS for dry matter prediction and the methods demonstrated can be easily adapted to fit an industrial conveyor belt.

5.2 Paper III: Evaluation of hand-held methods for representative determination of glucose in potatoes

Even though very little research exists on the performance and reliability of portable blood glucose monitors (PBGM) for determination of glucose in potato juice, quite a few companies do use them. In Norway it is common practice to use a PBGM for quality monitoring. Producers of chips and crisps rely on these small hand-held instruments, along with test frying, for their daily monitoring of glucose. The determination of glucose, however, is also important to control and minimize the formation of acrylamide. In addition to the PBGMs, at least one other alternative is commercially available; the RQFlex/Reflectoquant system (Merk

KGaA, Darmstadt, Germany). This instrument is described in section 4.2.1 and was evaluated as a possible rapid instrument for glucose determination in potatoes. To the best of my knowledge, no scientific publications exist regarding the use of the RQFlex for sugar analysis in potatoes. Paper III aims to elucidate whether the PBGMs and the RQFlex instrument really do give reliable results. This work can be divided into two parts. First, to investigate if the instruments themselves perform well, and secondly, to investigate if the sampling technique currently in use (i.e. the use of potato juice) gives a reliable picture for the glucose content in potatoes.

5.2.1 Hand held instrumentation for glucose determination

The two PBGMs used in paper III was one LifeScan OneTouch Easy Ultra (LifeScan Inc. USA), and one Bayer Contour (Bayer GmbH, Germany). Both gave determination coefficients of 0.92 when compared to the reference analysis. This is a quite good, but the error estimations for the Bayer monitor (RMSEE=1.12 mg/mL) and the LifeScan monitor (RMSEE=0.54 mg/mL) reveals that the determinations are not as good as they should be. Especially the Bayer monitor performed poorly at values above 2 mg/mL, as can be seen from Figure 21. The RQFlex outperformed both of the two PBGMs, as better prediction performance ($R^2=0.96$, RMSEE=0.24 mg/mL) was achieved. In additions, some problems with over and underestimation by the two PBGMs were experienced. The RQFlex, which is purpose built to measure glucose in potato juice, gave a more linear relationship and no over or underestimation.

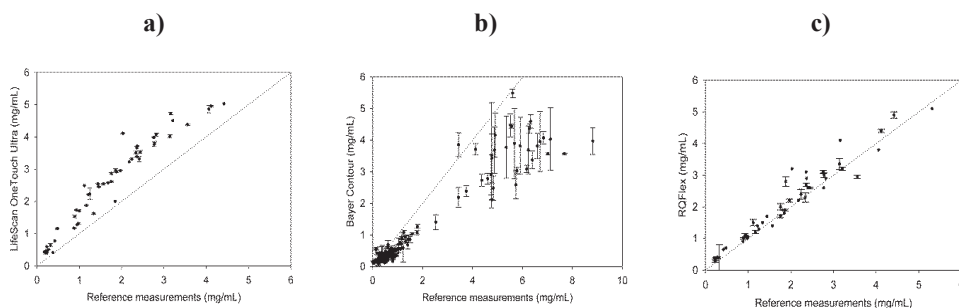


Figure 21: A summary of the performance of the handheld instruments from paper III.. A) the LifeScan monitor, b) Bayer Monitor, c) RQFlex instrument

5.2.2 The use of potato juice for glucose determination

Sampling of potato juice represents a fast and convenient sampling method, as it requires no extraction of solid potato matter. However, if the amount of glucose in the juice is not representing the total glucose content in the potato tuber, the use of juice for sampling is questionable.

The results from paper III indicate that a good relationship between glucose content in juice and in lyophilized potato powder may be hard to achieve. A determination coefficient of 0.91 was found for glucose content in juice compared to the glucose content in lyophilized potato powder. The potato tubers, used in paper III, for this comparison were halved along the longitudinal (i.e. stem to bud) axis. According to the findings of Pritchard and Scanlon⁸, this should yield two halves with comparable sugar content. An illustration of the glucose distribution in two different cultivars can be seen Figure 22.

The findings of Pritchard and Scanlon⁸, do however point at a another major source of errors. In Figure 22 it can be seen that the glucose content is differing both from stem to bud and from pith to cortex. It is therefore a risk of extracting juice with biased glucose content. In paper III it was conclude that this combined with general lack of ability to evenly extract the juice, were the main reasons for the low determination coefficient achieved for the relationship between potato juice and lyophilized material.

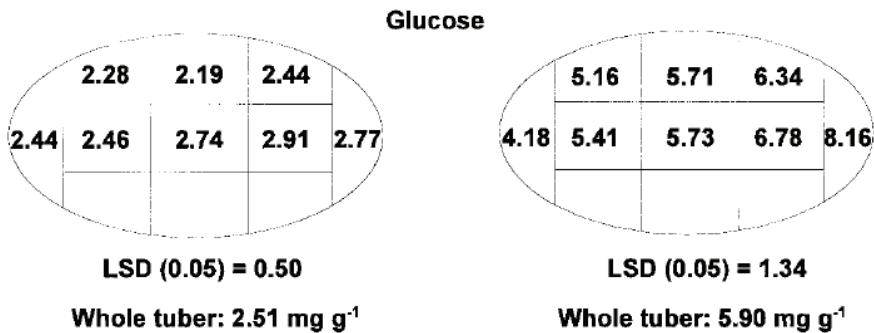


Figure 22: Internal sugar distribution in potatoes of cv. Russet Burbank (right) and cv. Shepody (left), as found by Pritchard and Scanlon⁸. LSD denotes the least significant difference. ($n=20$, and values are given based on fresh weight)

5.2.3 General comments on the use of rapid methods for glucose determination

One of the aims of paper III is to investigate how the two PBGMs and the RQFlex perform, regarding determination of the glucose content in potatoes. The performances are given in section 5.2.1. Further correlation between the glucose content in potato juice and lyophilized powder is given in section 5.2.2. If one is combining these findings, it becomes clear that the use of PBGMs on juice samples made with a household juice extractor is not very accurate. The RQFlex does exhibit better performance on the actual juice analysis, but it will still suffer from lack of representative sampling associated with the juice extraction. None of the above discussed instruments, or extraction methods are suited for highly accurate determination, but they are able to give indications of high or low glucose content.

It is well documented that both glucose and fructose are contributing to the Maillard reaction, and hence also the acrylamide formation¹¹⁴. Since the rapid methods described above are only measuring glucose, the correlation between glucose and fructose becomes of importance. A determination coefficient of 0.93 was found between glucose and fructose, measured in the lyophilized potato powder. This correlation is probably good enough to use the glucose content to give a rough estimate for the fructose content. It is also corresponding well to studies performed by others^{99,114}. This means that the small and hand-held instruments may provide a measure for the total reducing sugar content in a sample. This is desirable for the industry, as both glucose and fructose are contributing to the colour and acrylamide formation¹¹⁴.

5.3 Paper IV: Factors affecting the material properties of “lompe” dough

“Lompe” production has a long tradition on Norway, and similar products can be found in other Scandinavian countries. Even though “lompe” is manufactured on an industrial scale, the quality of the dough is solely based on the baker’s experience. The main challenges in “lompe” production are described in section 3.2, but can be summarized as; what is affecting the texture of the “lompe” dough? The need for proper understanding of the dough for making “lompe” formed the main motivation for paper IV. Since very little research has been performed on “lompe”, this study can be seen as a first survey of how potato properties are affecting the dough. As stated in section 3.2 the “lompe” dough will be ruined after a short amount of time (<30 min) if left unattended after mixing. When first developed, the dough must be processed into “lompe” quickly. This is the quite opposite of regular bread dough. If

yeast is used as a raising agent, the dough is commonly allowed to stand for hours after dough development. It has been found that regular flour dough will reach a steady state after an initial relaxation¹¹⁵. To find the reason for this time dependent degradation was one of the main aims of paper IV. The other main aim was to elucidate why potatoes must be cold stored for 12-48 hours before they are ready for dough formation. In addition, the understanding of the effect of water content was important.

The dough preparation in paper IV was performed with a DoughLab-instrument (Newport Scientific Inc., USA), which is a Farinograph type instrument. However, it was not used as a Farinograph and the results shown in this thesis cannot be compared to traditional Farinograph measurements. The instrument was used due to its computer controlled mixing speed and time, in addition to its capability to record the torque (given in Newton metre (Nm)) needed to mix the dough. The investigation in paper IV showed that the G' modulus of the dough started to decrease immediately after dough mixing and probably also during dough mixing. This is in accordance with empirical knowledge amongst "lompe" bakers. At first this was thought to be caused by bacterial growth, as wild yeasts may be present in flour. It was soon discovered that antibiotics did not affect the reduction in G', but the addition of silver nitrate had a great effect. Silver nitrate is known as a general enzyme inhibitor, and has been demonstrated for inhibition of flour enzymes¹¹⁶. It was not firmly established whether the amylase found in the flour was the sole cause, or if other flour enzymes also contribute. It was clear that flour enzymes were causing the dough structure to degrade. The main difference between regular flour dough and "lompe" dough is the gelatinized starch. The gelatinized starch in the potatoes are no longer tightly packed into granules, but transformed into a gel with more loosely packed amylose and amylopectin chains. A cross section of a starch granule compared to free amylose / amylopectin chains is shown in Figure 23. Amylases can attack the starch granules and cause damage, but the starch granules can be seen as a filler material within the gluten network¹¹⁵. The network would therefore not be degraded in the same way if attacked by enzymes. This indicates that the gelatinized starch possesses a completely different role in the dough. It was therefore suspected that the gelatinized starch may be an active part in the network formation. This may also explain the large impact of enzymatic degradation. No scientific reports have been found about inclusion of gelatinized starch in gluten networks. However, some models describing mixed and filled gels¹¹⁷ may be suitable to explain the structure in "lompe" dough. It is worth keeping in mind that "lompe" dough contains gelatinized starch, raw starch granules from the flour and the gluten proteins from the flour. In addition a small amount of potato proteins is

present. Although, enzymatic degradation was pointed out as the main reason for the dough degradation, more research should be performed to fully identify the role of gelatinized starch in the dough structure. Meanwhile, the producers should be able to use this information to aid their production difficulties. This can be achieved either by selecting flours with a low amylase activity, or by keeping the doughs as cold as possible to minimize the enzyme activity in general.

The other main parameter affecting the dough consistency was found to be the time between heat treatment of the potatoes and the dough formation. In paper IV it was shown that retrogradation of the gelatinized potato starch is the most probable factor causing the need for this holding time. The theories describing starch retrogradation are not conclusive^{117,119}. The retrogradation rates for different potato cultivars are not well studied, but further studies of how different potato cultivars retrograde and if this rate can be influenced may help the “lompe” producers. As production must be planned days ahead, it is difficult for the producers to meet the day to day variations. Hence, better knowledge about what rate the different cultivars retrograde will be most helpful to improve the production, and something that should be looked into.

The dry matter content of the potatoes is largely determining the water content of the dough, and was found, in paper IV to have a direct impact on dough consistency. This is as expected, as water content is known to affect dough consistency in other types of dough¹¹⁵. Better methods for determination of water or dry matter content may therefore greatly aid the “lompe” producers in their strive to make uniform dough batches. It is worth noting that the study performed in paper IV did not aim to find the optimal “lompe” dough, but rather to use one recipe to produce model dough for elucidation of the factors affecting the dough

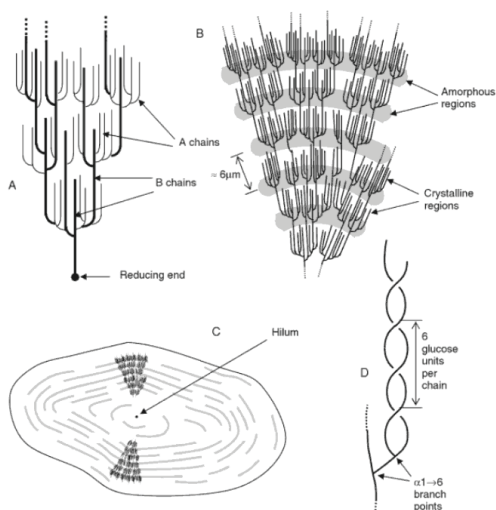


Figure 23: A schematic overview¹¹⁸ of starch and starch granules A and B shows a representation of amylopectin and D shows a representation of loose amylopectin. C shows a cross section of a starch granule. D shows the suspected helices formed by amylose chains

properties. Dough made from the recipe used in paper IV was not processed into a final product, and no sensorial evaluation was performed.

5.4 Unpublished experiments

5.4.1 *Dry Film Fourier Transform Infrared analysis of sugars in potato juice*

The rapid techniques presented in paper III all have one drawback in common. The two PBGMs cannot, by design, measure fructose and sucrose. The RQFlex instrument can measure both fructose and sucrose, but this requires multiple test strips. It is more time consuming than the glucose measurements as both the fructose and the sucrose measuring strips are more time consuming to use than the glucose strips. Therefore, rapid methods for determination of all three sugars in potato juice are needed. Paper III generated a set of juice samples with corresponding data of their sugar content. The samples were analysed with a FTIR dry film approach to investigate if this was a feasible measure for sugar determination in potato juice.

Materials and methods

A total of 249 samples, all of which from paper III, were analysed according to the following procedure. Reference data from paper III were used in the calibration process. All samples were stored in a freezer and thawed on the day of the spectroscopic analysis. Preserved potato juice samples (15 μ L) were transferred directly, in triplicate, to well plates (silicon, 96 wells) and dried in a vacuum desiccator with silica gel at room temperature. FTIR measurements were performed using a high-throughput instrument (eXTension, HTS-XT with Tensor 27 Spectrometer, Bruker Optik GmbH, Germany). The instrument was equipped with a deuterated L-alanine doped triglycine sulfate (DLaTGS) detector. Spectra were recorded in transmission mode in the spectral region from 4000 to 400 cm^{-1} with a resolution of 4 cm^{-1} and an aperture of 5.0 mm. Background spectra (40 scans) of the silicon substrate were collected before each sample measurement to account for variation in water vapor and carbon dioxide. Further processing of the spectra was performed using The Unscrambler X software (V.10.2, CAMO PROCESS AS, Oslo Norway). The average spectra for each sample were calculated before the second derivate of the spectra was found using the Savitzky-Golay algorithm¹²⁰, using a polynomial degree two and a window of 13 points in total. Furthermore, the spectra were subjected to extended multiplicative scatter correction (EMSC)¹²¹. Spectral regions from 486-1884 and 2685-3085 cm^{-1} were used for data analysis. Regression models were developed using PLS-regression⁶², with the sugar concentrations determined by the reference method (section 2.4) used as reference values. The optimal number of PLS factors

was determined by full cross validation. RMSECV was determined in the software. Reference analysis was performed according to materials and methods in paper III.

Results

All samples from paper III were used in the same IR prediction. A PCA analysis (plot not shown) of all replicate spectra recorded showed good grouping of the replicates, indicating good reproducibility of the dry films. Details about the regression models obtained are shown in

Table 2, along with determination coefficients and error estimates (RMSECV). The prediction performance was acceptable, but for the higher sugar concentrations the uncertainty seemed to increase drastically. Due to the natural variation in sugar levels, only samples from cv. Peik are found in the region above 4 mg/mL. If cv. Peik is removed, the performance of the regressions models is improving, but the number of PLS-factors is increasing. The prediction of glucose, excluding cv. Peik is shown in Figure 24b.

Table 2: Performances of the different FTIR dry film PLS regression models obtained

Prediction:	R ²	RMSECV (mg/mL)	No. of PLS- factors	No. of samples	Range (mg/mL)
Glucose	0.92	0.64	3	238	0.02 - 12.95
Fructose	0.93	0.33	4	238	0.01 - 7.34
Sucrose	0.86	0.38	5	238	0.20 - 7.33
Glucose exc. Cv. Peik	0.95	0.31	6	200	0.02 - 9.72
Fructose exc. Cv. Peik	0.94	0.16	8	200	0.01 - 4.87
Sucrose exc. Cv. Peik	0.88	0.37	6	200	0.20 - 7.33

From the individual regression coefficients (not shown) it is clear that peaks characteristic for each of the sugars coincide with the different absorption bands found by Dupuy *et al.*¹⁰⁰. For instance, the regression coefficient for fructose is dominating at 815 cm⁻¹, which is a characteristic absorption band for fructose. The same goes for sucrose bands at 920

and 970 cm^{-1} (C-OH stretch at the anomeric position) and a band at 1030 cm^{-1} (OH-deformation / C-O stretching) for glucose¹²².

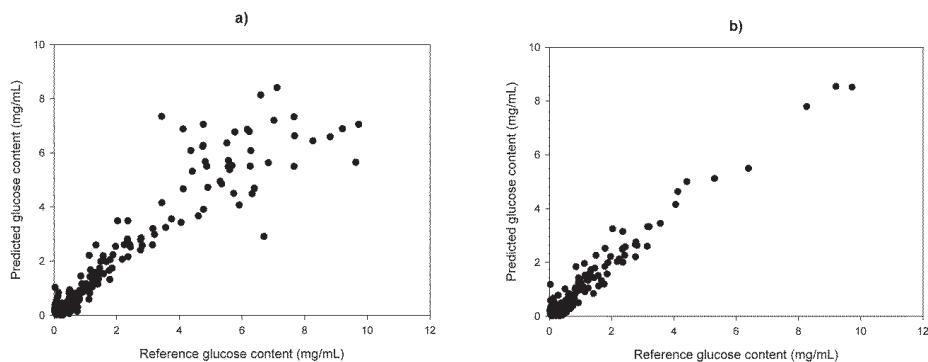


Figure 24: The FTIR predictions obtained, a) including cv. Peik, and b) excluding cv. Peik

Even though the regression coefficients are indicating that the models are based on signals from the actual sugars, some work still remains before the FTIR dry film is ready for commercial use. Some aspects that will have to be addressed are: Firstly, what seems to be a cultivar dependant challenge must be addressed, as a sample independent calibration is desired. Hence, the calibrations must be extended to a larger number of cultivars to elucidate if a single calibration can be used for multiple cultivars, or if cultivar specific models must be obtained. Secondly, alternatives for sampling and sample handling must be elucidated. In the current study, the samples were diluted in methanol, followed by a heat treatment to prevent enzymes from degrading the sucrose. It may be beneficial to seek sample handling protocols that can deal with fresh potato juice, without any heat treatment.

5.4.2 NIRS for sorting of *sous-vidé* treated potatoes

Although some methods for determination of potato texture exist¹⁰⁷, their use is not common amongst producers of *sous-vidé* treated potatoes. A small experiment was undertaken to investigate if the methods developed in paper I and II could help predicting the cooking texture of *sous-vidé* treated potatoes. The aim of this study was therefore to investigate if dry matter content predicted by NIRS can be used to sort a batch of potatoes into two different texture categories. Both *sous-vidé* treated potatoes and regular boiled potatoes were prepared, in case the *sous-vidé* processing would cause changes in the texture. This study aimed to give

indications whether or not better sorting, based on dry matter content, may facilitate the basis for obtaining a more evenly textured product.

Materials and methods

Potatoes from cv. Fakse, cv. Folva and cv. Mandelpotet were obtained from local processing industry. The potatoes were cleaned in tap water and allowed to reach room temperature. The dry matter content of the three different batches used was determined by NIRS, by the method described in paper I and II. The average dry matter content in each batch was determined and potatoes with a dry matter content close to the median value ($\pm 0.5\%$ DM) were discarded. Potatoes with dry matter content above were classified into a batch with high dry matter content, and potatoes with lower dry matter content classified into a batch with low dry matter content. After classification, all tubers were peeled and each category was divided into two lots. One used for regular cooking in boiling water, the other vacuum packed (PA/PE 20/70 my, EmbaNor AS, Norway) and heat treated in boiling water. Each sample consisted of 3 tubers, 3 samples from each category were prepared. This equals a total of 9 tubers per category. After cooking, the samples were cooled to 4 °C and stored for 48 hours. A long probe connected to a temperature logger (Ebro, Ingolstadt, Germany) was inserted into the middle of one potato for each sample. This was only done on samples that were vacuum packed (to protect the temperature logger). The loggers were used to ensure that the same core temperature of 92-95 °C was reached for all samples.

Texture was measured by uniaxial compression tests. Disks were cut across the long axis of the tubers, and small cylinders ($h=10$ mm, $\phi=10$ mm, $n=4$) were cut from the slices. The cylinders were cut midway between the centre and the outer edge of the tuber. Texture was analysed with a TA-Xt-plus Texture Analyser (Stable Micro Systems, Surrey UK) with a cylindrical probe ($\phi=20$ mm) and a 5 kg load cell. A probe speed of 0.25 mm/s was used, and the samples were compressed to 25 % of the original height.

Total starch and amylose:amylopectin ratio were determined according to the method described in paper IV. The replicate and sample variance was determined by standard deviation and Student's T-test (MiniTab 16, MiniTab Inc, USA). Replicates were averaged for each sample (i.e. an average was found for the individual tubers in a sample) before statistical analysis was performed.

Results

The two parameters “hardness” and “fracturability” were chosen to represent the texture of the potatoes. A summary of the hardness of each sample as shown in Figure 25. A Tukey’s pairwise comparison revealed no significant difference ($p < 0.05$) between the samples within each cultivar. Details about sample grouping are given in Table 3.

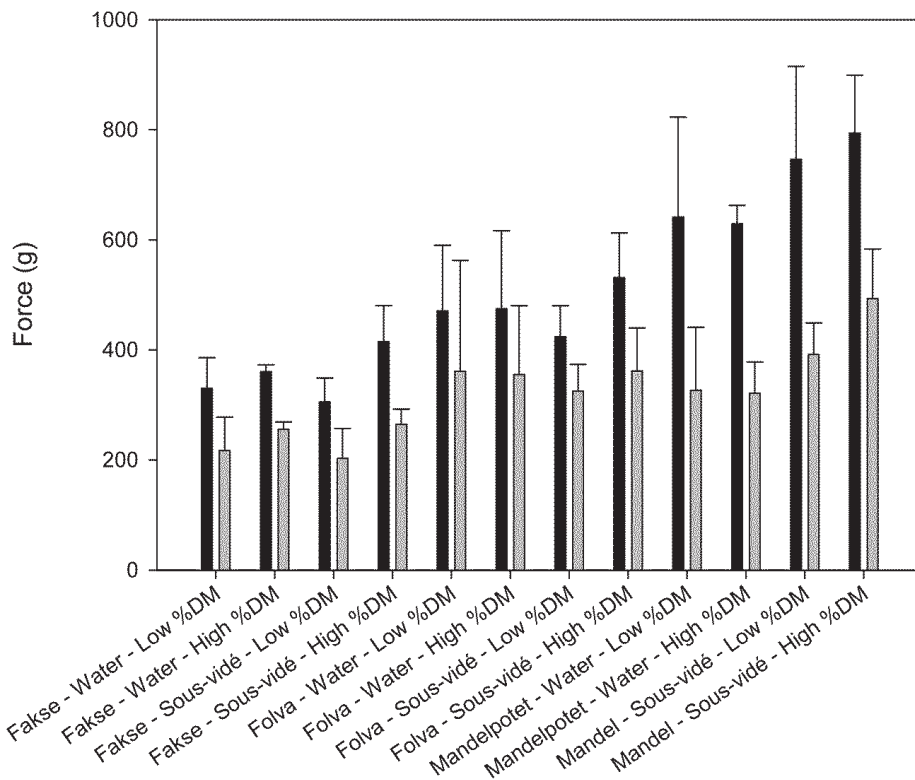


Figure 25: The hardness (black bars) and fracturability (grey bars) for the samples used in this study.

The standard deviation is given as error bars

No significant differences were found between samples of the same cultivar, but some significant differences were seen between cultivars. Difference between cultivars was to be expected, as differences in both starch composition and dry matter content can be seen. Total starch and amylose amylopectin ratios of the different cultivars are given in Table 4. As shown, there is no difference in starch content or amylose amylopectin ratio between cv. Folva and cv. Fakse, while it is a significant difference between these two cultivars and cv. Mandelpotet. It is also a large difference in average dry matter content (no significance

testing performed). The difference in chemical composition seems to be the reason for the difference between the cultivars. Quite large standard deviations for hardness and fracturability were found between samples. This is illustrated by the error bars in Figure 25. A lot of research has been performed to investigate texture measurements, and the relation between measuring parameters and the results has been studied^{108,123}. It has been found that parameters such as the speed of the measuring probe may affect the results. Possibilities for optimization of measurements were not investigated in the current study. From the results presented it can be concluded that prediction of dry matter content, by NIRS, was not capable of separate potatoes from a specific cultivar into different texture categories. The NIRS instrument was the same as described in paper I and II, which means it was a low resolution instrument, capable of predicting the texture of the potatoes. The spectra were used for dry matter prediction, and no further multivariate analysis was applied to predict cooking attributes. Although the capabilities of predicting dry matter content, it is unlikely that the low resolution will yield enough information to predict cooking quality.

Table 3: The different sample treatments, and the sample grouping based on the measured hardness and fracturability

Cv.	%DM	Treatment	Grouping*						
			Hardness			Fracturability			
Mandelpotet	Hi	SV	A					A	
Mandelpotet	Lo	SV	A	B				A	B
Mandelpotet	Hi	W	A	B	C			A	B
Mandelpotet	Lo	W	A	B	C			A	B
Folva	Hi	SV	A	B	C	D	E	A	B
Folva	Lo	SV			C	D	E	A	B
Folva	Hi	W		B	C	D	E	A	B
Folva	Lo	W		B	C	D	E	A	B
Fakse	Hi	SV			C	D	E	A	B
Fakse	Lo	SV					E		B
Fakse	Hi	W			C	D	E	A	B
Fakse	Lo	W				D	E	A	B

*Based on a Tukey method, 95% confidence

Although there did not seem to be a significant correlation between dry matter content and texture, this may be due to the small dry matter range used. The findings presented in paper II indicate that a potato batches can have large range of dry matter content, and that ± 1 or 2 percent may be more suitable. Depending on the application, one may want to choose a broader range of dry matter contents in order to elucidate the texture differences. In the current study, only tubers ± 0.5 % from the median dry matter content was removed. The use of NIRS for prediction of cooking quality is a subject worth further investigation.

Table 4: An overview of the samples used for texture analysis of *sous-vidé* -treated potatoes. Standard deviation is given in parentheses, where applicable

Cultivar	Average dry matter* (%FW)	Total starch** (%DM)	Amylose : Amylopectin-ratio**
Mandel	25.5	76 (2.9) ^A	20:80 ^A
Folve	20.0	70 (1.5) ^B	23:77 ^B
Fakse	17.6	69 (4.6) ^B	23:77 ^B

*Dry matter values predicted by NIRS, **Samples that do not share a letter are significantly different

6. Concluding remarks and future prospects

All the work presented in thesis share a common feature: the intra- and inter-heterogeneity of potatoes, and the associated processing challenges. Paper I and II deal with NIRS for improved assessment of the dry matter content in potatoes. Paper III evaluates the use of small hand-held instruments for glucose determination in potatoes. Paper IV aims to improve the understanding of how the potato raw material affects the production of “lompe”.

The results of paper I and II show that a NIRS prototype instrument is well suited for predicting the dry matter content in both stationary potatoes and potatoes moving on a conveyor belt. The use of NIRS for prediction of dry matter content was thus shown to be feasible for adaption to online use. The results from paper II indicate that the NIRS prototype instrument can even detect some of the internal gradients of dry matter content found in potato tubers. These findings are encouraging, and it is well worth developing this instrument further. Currently, the NIRS prototype is configured for measuring a single line of potatoes. This approach could be used to measure a stream of potatoes and thus calculate the average dry matter content for each time unit (e.g. every second). This can provide a good indication of both the average dry matter content in the batch along with the high and low values. A subsequent development stage is the ability to measure and predict the dry matter content for each and every potato tuber passing on the conveyor belt (i.e. on multiple lines). However, this is simply a matter of instrument development. The technology in the NIRS prototype has proven capable for achieving this, and it should be possible to adapt this technology to wider sample streams. The ultimate goal would be to allow for precise sorting of large batches of potatoes into sub-batches with a narrow range of dry matter content. This was the intention of Pflug *et al.*⁵³ which made sorting equipment based on graduating the tubers according to density in brine solutions. The equipment devised by Pflug *et al.* had its obvious drawbacks, such as the intricate mechanical system and corrosive brine solutions. NIRS has none of these drawbacks. Further research should therefore focus on the development of this instrumentation to fully adapt it to online sorting of potatoes in an industrial environment. The best option in order of making processed potato products with an overall improved and even quality is to use potatoes of well defined and even dry matter content. In this way, processing conditions like cooking time can be more finely tuned, resulting in considerable reductions in energy consumption, time and waste quantities. This can be achieved by sorting the raw potato batches into sub-batches.

Small hand-held instruments allow for fast and easy sampling of the glucose content in potatoes and are frequently used by manufacturers of fried potato products. Although in frequent use, very few scientific publications concerning their capabilities can be found. Paper III shows that the two PBGMs and the RQFlex instrument were capable of providing an estimate of the glucose content in potato juice. The RQFlex showed better performance than the two PBGMs, but were slightly more time consuming to use. A good correlation between the glucose content in potato juice and lyophilized potato powder was found. This makes potato juice a viable option for quick and easy assessment of the glucose content in potatoes. Although capable to measure the glucose content, the hand-held instruments cannot easily determine fructose and sucrose. In one of the unpublished experiments presented, the use of dry film FTIR was investigated. The results are encouraging, but indicate that some challenges are still to be overcome. Dry film FTIR can provide a tool for semi-rapid prediction of all three sugars in potato juice. It was, however, found that at least one cultivar gave poor results, but the reason for this was not elucidated. The further focus on sugar analyses should therefore be twofold. There are different PBGMs with different enzyme technologies available on the market. It is possible that other brands or models may perform differently than those shown in this thesis. Due to the obvious drawback of only determining glucose, it should also be put effort into investigating and improving the dry film FTIR approach. There is a need to investigate if cultivar specific calibrations are needed, or if a universal one can be developed. Furthermore, it would be beneficial to simplify the sample handling. Better and more reliable sugar analyses will greatly improve the product quality and reduce acrylamide contents in fried potato products. A more even product quality and lower acrylamide content will in turn cause fewer products to be rejected in the quality control, and hence reduce waste. This will be even more effective if combined with dry-matter-content-based sorting.

Paper IV investigates the factors affecting the material properties of “lompe” dough. It was concluded that retrogradation, activity of endogenous flour enzymes and dry matter content of the potato tubers were three of the main parameters affecting the dough texture. Although answering some questions about the factors affecting the dough texture, paper IV also opens a few new intriguing questions. The interactions between gelatinized starch, gluten and starch granules are poorly understood. This is a subject that would benefit from further investigation. If the dough structure can be thoroughly elucidated, along with knowledge about the enzyme effect, this could help the “lompe” producers make more stable doughs. The use of flour with low enzyme activity may also be of great interest, and can potentially aid

some of the dough stability issues. This will in turn give a more reliable process and less waste. The holding time, caused by the retrogradation of the starch, is also causing some challenges. This creates a need for cold storing large amounts of cooked potatoes, and it makes it hard to meet rapid fluctuations in the market demand. There are two obvious ways to reduce the holding times. Cultivars with a naturally high rate of retrogradation can be used, or one can seek to artificially increase this rate. The challenges caused by the variation in dry matter content in the potatoes, and subsequently varying water content in the dough, can be aided by the findings of paper I and II. As the dry matter content of the potatoes can be measured after cooking and mashing, it is believed that an adaption of the instrumentation presented in paper I and II is straight forward. Accurate prediction of the water content allows for less human-based dough adjustments, and hence also less room for human error along the process. Further studies of “lompe” dough could also look into the use of different stabilizers and other conditions to improve dough quality, although this may interfere with the wish to maintain a clean label on the product.

The three first papers presented in this thesis focus on analytical tools for better assessment of raw material quality. The quality parameters provided by these tools are not only suited for process control. They can also be used for better understanding of how the raw material quality affects the processing. Regarding prediction of dry matter content, this will be especially true for the industries producing *sous-vidé* treated potatoes. Closely monitoring the raw material stream going into the process may give vital clues about how the process is affected by parameters such as dry matter content. Manufacturers of chips and crisps do not only need to understand their processes, they also need to understand how the raw material is developing during the storage season. Improved methods for sugar determination, combined with better predictions for dry matter content will help. The ability to foresee the changes in sugar levels during storage will allow producers to process a batch before the sugar levels rises to much, and to determine what batches that may be stored for the longer period of time. All in all, this will help reducing waste, and subsequently production costs.

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Papers I-IV

Paper I

Near-infrared Spectroscopy for Rapid Estimation of Dry Matter Content in Whole Unpeeled Potato Tubers

Trygve Helgerud^{1,2}, Vegard H. Segtnan¹, Jens P. Wold¹, Simon Ballance¹, Svein H. Knutsen¹, Elling O. Rukke² & Nils K. Afseth¹

¹ Nofima AS, Postboks 210, Ås, Norway

² Department of Chemistry, Biotechnology and Food Science, Norwegian University of Life Sciences, 1432 Ås, Norway

Correspondence: Trygve Helgerud, Nofima AS, Postboks 210, Ås N-1431, Norway. Tel: 47-6497-0100. E-mail: trygve.helgerud@nofima.no

Received: August 23, 2012 Accepted: September 7, 2012 Online Published: October 25, 2012

doi:10.5539/jfr.v1n4p55

URL: <http://dx.doi.org/10.5539/jfr.v1n4p55>

Abstract

The dry matter is one of the main quality parameters of raw and processed potatoes. In the present study, the potential of utilizing high throughput commercially available NIR interactance systems for dry matter determination in whole unpeeled potato tubers is investigated. The performance of a 2D NIR interactance instrument was compared with that of a 1D NIR interactance instrument and a traditional underwater weight apparatus. A total of 114 tubers were assessed individually with both of the NIR instruments (760-1040 nm), the underwater weight and an external reference method (freeze drying). The 1D interactance instrument obtained better prediction results than what the 2D instrument could achieve ($R^2=0.95$, RMSECV=0.91, and $R^2=0.83$, RMSECV=1.65, respectively). The underwater weight obtained the highest explained variance ($R^2=0.97$), but the estimation was biased by approximately 1.5% (by weight). The poorer prediction performance of the 2D NIR interactance system can be partly explained by the lower penetration depths of the light compared to the 1D NIR interactance systems.

Keywords: near-infrared spectroscopy, potato, dry matter determination, on-line measurements

1. Introduction

On a global basis the potato (*Solanum tuberosum* L.) is grown with yields in millions of metric tons per year. The trend in potato consumption shows that the amounts used for processing has been steadily increasing over recent years (Loy, Riekert, & Steinhagen, 2011). A potato, however, is highly heterogeneous. During factory processing, unacceptably high variations in raw potato quality means that different processing parameters have to be periodically adjusted and optimised in order to obtain consistent end product quality and reduce waste quantities. The features most commonly assessed prior to industrial processing of potatoes include internal and external defects along with dry matter content and glucose levels. Dry matter is one of the variables required to calculate and estimate frying or cooking time, and hence the texture, color and acrylamide content of the end-product. Wrongly determined dry matter content may thus lead to an unacceptable quality of whole batches of processed potato products.

Traditionally, the preferred method for dry matter evaluation has been the underwater weight of the tubers. The relationship between specific gravity and dry matter has been investigated numerous times, and slightly different approximations of this relationship have been obtained for tubers of different growth years or geographic origins (Haase, 2003; Lunden, 1956; Nissen, 1955; Schippers, 1976). The accuracy of the approach is, however, disputed (Lunden, 1956; Nissen, 1967; Wilson & Lindsay, 1969), which is indicated by the large number of different approximations. It is also clear that this method is not suited for on-line measurements, and the technique is as such unable to assess the dry matter variation within a batch of potatoes. The need for better batch characterization along with the development of better technological solutions has led to a significant interest in methodology such as Near-Infrared (NIR) spectroscopy. In NIR spectroscopy, near-infrared light is absorbed at specific frequencies due to vibrations of molecules within the sample. The molecular vibrations are specific for the functional groups of each molecule and can therefore be used to quantify components such as water or starch. NIR spectroscopy is non-invasive and may provide high sampling rates, making the technique

ideal for on-line assessment of food components.

The first reports concerning NIR spectroscopy for dry matter analysis of potatoes were published in the late 1980s (Dull, Birth, & Leffler, 1989). Dull et al. reported that NIR light in the region of 800-1100 nm in transmittance mode (light source and detector on opposite sides of the sample), could be used to determine dry matter in whole unpeeled potato tubers (Dull et al., 1989). As a proof of principle the authors reported correlations (R) of 0.9178 for whole unpeeled tubers, and 0.975 for thin-sliced tubers (Dull et al., 1989). Since then a number of feasibility studies have been published, most measuring mashed or sliced tubers, excludes the possibility of non-invasive measurements. Explained variances (R^2) between 0.58 and 0.99 for dry matter determination have been achieved with reflectance measurements, where mashed tubers as expected provided the highest explained variances (Brunt & Drost, 2010; Haase, 2011; Scanlon, Pritchard, & Adam, 1999). Studies have also concluded that even though feasible estimations of dry matter and starch could be obtained, the minor constituents such as glucose, crude protein and recoverable protein are difficult to predict (Fernandez-Ahumada et al., 2006; Haase, 2011; Scanlon et al., 1999). More recently, automated routines for dry matter determination have also been explored. Brunt and Dorst (Brunt & Drost, 2010; Brunt, Smits, & Holthuis, 2010) made a prototype off-line set up for determination of dry matter and starch. For this automated system the underwater weight approach was used for the dry matter, and NIR reflectance of mashed potato tubers were used for starch determination (Brunt et al., 2010). On-line procedures for estimation of dry matter and fat content in processed potato products have been described previously (Pedreschi, Segtnan, & Knutsen, 2010).

It is well known that the dry matter content is unevenly distributed throughout a potato tuber. Generally, the dry matter content is highest in the outer layer (parenchyma) and lowest in the middle layer of the tuber (pith). In addition, the gradient of dry matter content is not necessarily linear through the tuber or constant from one tuber to the next (Storey, 2007). Thus, in order to develop automated NIR instrumentation for analysis of whole unpeeled tubers, the sampling approach has to provide adequate sampling volumes. With a transmittance set-up this could be achieved, but the set-up would require instrumentation both above and below a conveyor belt and would thus be challenging to fit on-line. The reflectance approach presents obvious limitations due to reduced sampling volumes. The interactance approach, on the other hand, could potentially provide adequate sampling volumes, and the instrumentation could be easily situated above the conveyor belt. Subedi and Walsh recently published the first NIR interactance investigation on intact tubers (Subedi & Walsh, 2009). A system capable of measuring samples on a conveyor belt was also investigated, but only for sliced tubers. The authors concluded that short wave NIR (750-950 nm) in interactance mode was well suited for rapid assessment of potatoes. Using a high resolution laboratory instrument they achieved an explained variance (R^2) of 0.87 for whole unpeeled tubers. When they measured sliced tubers, the explained variance (R^2) increased to 0.92. For moving sliced tubers the explained variance (R^2) decreased down to 0.82.

Whereas Subedi and Walsh investigated NIR interactance approach for dry matter content assessment using a high-resolution laboratory instrument, there are no known studies where commercially available and industrially applicable instrumentation has been evaluated for the same purpose. Thus, in the present study, the potential of utilizing high throughput commercially available NIR interactance systems for dry matter content determination in whole unpeeled potato tubers is investigated. The instruments used in this study have previously been applied at-line and on-line for measuring main components in food systems like meat trimmings and fish fillets (ElMasry & Wold, 2008; J. P. Wold, O'Farrell, Hoy, & Tschudi, 2011). The instruments provide lower spectral resolution than reported in previous publications, hence enabling faster spectrum acquisition, higher signal to noise ratio (SNR) and higher sample volumes per time unit. The objectives of the study are three-fold: (1) To utilize 1D NIR interactance for stationary analysis of whole unpeeled potato tubers; (2) to utilize a commercially available 2D NIR interactance system to provide on-line estimation of whole unpeeled tubers; and (3) to compare the performance of the NIR instrumentation to the performance of the underwater weight. All measurements were conducted on single tubers in order to eliminate in-batch variation (Cole, 1975).

2. Materials and Methods

Potato tubers were acquired from a potato packaging facility (varieties Asterix, Folva and Celine) and a chips manufacturer (varieties Bruse and Saturna). For the main part of the experiment 20 tubers from each of the varieties with red skin (varieties Asterix, Bruse and Celine) and 30 tubers from each of the varieties with yellow skin (varieties Folva, Saturna) were selected. After cleaning in tap water the tubers were left overnight to dry and reach an even temperature (room temperature at 21°C). The tubers were selected so that they had a size distribution similar to that found in potato processing industry.

2.1 1D NIR Interactance Measurements

1D NIR interactance measurements were performed using a prototype VIS/NIR interactance instrument allowing a spectral resolution of 30 equally spaced channels in the VIS/NIR region (449-1040 nm) (Folkestad et al., 2008). The spectral acquisition time was set at two seconds. Two equal sized squares were illuminated by two 50 Watt halogen lamps (OSRAM, Augsburg, Germany), and backscattered light was collected through a collection tube. Measurements were done with contact between the sample and the collection tube, which effectively blocked all direct reflections from the sample surface. The 15 channels in the visible range (446-760 nm) were not used. Ten tubers (five with red skin and five with yellow skin) were used to estimate the penetration depth into the tuber of light from the 1D NIR interactance instrument. This was performed by recording a spectrum with a tuber placed first on a non-reflective (black plastic plate) and then on a reflective background (glossy metal plate). The tuber was then thinly sliced and measurement repeated. The distance where one could see a difference between the two surfaces was recorded as the light penetration depth. A series of 60 tubers were measured at three different places: stem, bud, and along the middle of the longitudinal axis. Data obtained were used to determine the best measuring position for NIR measurements on each tuber.

Each single tuber was measured three times on each side. Each tuber was rotated approximately 60° between each measurement. Measurements were taken at the center of the longest axis. This position was shown to have a dry matter content closest to the average of the whole tuber (Pritchard & Scanlon, 1997).

2.2 2D NIR Interactance Measurements

Single tubers were scanned with the commercially available QV500 (QVision AS, Norway). The QV500 is equipped with 15 equally spaced channels in the NIR region (760-1040 nm), and is able to scan a conveyor belt moving with a speed of up to one ms⁻¹. The resolution perpendicular to the moving belt was 60 pixels distributed on a 50 cm wide area. Light was transmitted via a rotating mirror onto the potato. Backscattered light from the interior of the potato was collected in the spectrophotometer. The optical lenses were configured to block direct reflections, hence only light that had traversed the tuber was collected. As for the interactance instrument the light penetration depth was determined. A series of ten tubers (five with red and five with yellow skin) were measured with a non-reflective and a reflective surface underneath, with successively thinner samples. For the determination of dry matter content in intact tubers, each tuber was scanned once at each side. The distance from the detector to the conveyor belt was 31 cm.

2.3 Specific Gravity Measurements

After spectral acquisition tubers were suspended from a load cell (Teda Huntleigh, Model no. 1004, accuracy of 0.017%) and the weight was measured in air and under water. The specific gravity (SG) was calculated based on equation 1.

$$SG = \frac{\text{Weight in air}}{\text{Weight in air} - \text{Weight in water}} \quad (1)$$

Utilizing equation 2 an estimate of the dry matter content was calculated (Lunden, 1956).

$$DM = 215.73(SG - 0.9825) \quad (2)$$

After measurement tubers were dried with tissue paper and placed in small plastic beakers.

2.3.1 Reference Analysis

Prior to freeze drying (Christ Gamma 1-16) tubers were carefully sliced into small pieces with a sharp knife and frozen to -40°C. After freeze drying, remaining potato material was ground to a fine powder in a household blender (Moulinette, Moulinex, Italy). The remaining moisture in samples was determined in a moisture analyzer (Sartorius L420P/YTC01L Thermo Control). Total amount of dry matter content was calculated based on the weight before and after freeze drying.

2.4 Data Analysis

Data from 1D NIR interactance measurements were imported into Unscrambler X, V 10.1 statistical analysis software (CAMO PROCESS AS, Oslo, Norway). The average of six replicate measurements was calculated. Averaged spectra were standard normal variate (SNV) transformed (Barnes, Dhanoa, & Lister, 1989), and a regression model was developed by partial least squares regression (PLSR) (Martens & Næs, 1989), with dry matter percentage obtained by freeze drying as reference values (Wold, Martens, & Wold, 1983). The optimal number of PLSR factors was determined by full cross validation. The reference value, and the predicted value, of every sample were used to calculate the prediction error of the cross validated calibration model, expressed as

the root mean square error of cross validation (RMSECV). The RMSECV value is defined by the following equation:

$$RMSECV = \sqrt{\frac{\sum_{i=1}^I (y_i - \hat{y}_i)^2}{I}} \quad (3)$$

Where i denotes the samples from 1 to I . y_i and \hat{y}_i denotes the reference value and the predicted value, respectively.

For the data acquired by the 2D NIR interactance system, a segregation routine had to be applied in order to separate the potato tuber from the conveyor belt. This was performed by comparing the ratio between the absorption of the wavelengths of 800 and 980 nm. The threshold was set so that all pixels with a ratio above 0.04 were selected as potato tuber, and the center of the tuber was automatically calculated. An area of 1/10th of the total width was selected on each side of the center point, and 1/10th of the height above and below the center. The spectrum of each pixel within a selected area was extracted and averaged. This was performed by an in-house built MatLab routine (MatLab, V. 7.10, The Mathworks Inc., Natick, MA). All spectra were exported to Unscrambler software and the spectra from both sides of the tuber were averaged. The spectra were used in PLS regression towards the reference analysis without any pre-treatment. Regression was validated by full cross validation.

3. Results and Discussion

The potatoes used in this study were selected to give as large variation in both skin color and dry matter content as possible. The color of the potato flesh was yellow for all tubers, with slight variation in the shades of color. An overview of the dataset used for the experiment is provided in Table 1. The dataset ranges from 14.4% to 30.5% in dry matter content, which covers most of the natural variance found amongst potato tubers (Burton, 1989). The samples also provided a good distribution of dry matter content amongst the tubers within each variety. A total of six samples had to be removed from the data set due to abnormalities such as rot and internal cavities.

Table 1. An overview of the potato dataset used in this study

Variety	Skin Color	Min % DM	Average % DM	Max % DM	SD DM	n
Asterix	Red	18.0	20.1	21.8	1.0	19
Bruse	Red	21.4	26.4	30.5	1.9	19
Celine	Red	14.8	18.1	20.3	1.6	19
Folva	Yellow	14.4	16.6	19.7	1.4	29
Saturna	Yellow	22.2	24.4	27.0	1.4	28
Total	-	14.4	21.0	30.5	4.1	114

3.1 Specific Gravity

The specific gravity of a potato is easily transformed into an estimate of the dry matter content. Due to its feasibility and low cost this has been the method of choice for dry matter content determinations in industry since it was introduced. The underwater weight used in the current study was configured for evaluation of single potato tubers. The values obtained from specific gravity measurements are shown in Figure 1a. Estimated values had a very good explained variance with reference values ($R^2=0.98$). The RMSECV was estimated to be 0.65, but all measurements made with the underwater weight were approximately 1.5 weight-% higher than the reference value. This is clearly shown in Figure 1a and indicates that a consistent deviation is present in all measurements, or that the model used has a small offset.

As previously mentioned a number of different approximations exist for converting specific gravity to dry matter percentage. Five of the industrially utilized regression models are shown in Figure 1b, the uppermost one being that used in this study. This is the regression model used by the Norwegian potato industry. Apparently, a careful selection of regression models may give an accurate estimation without any significant bias.

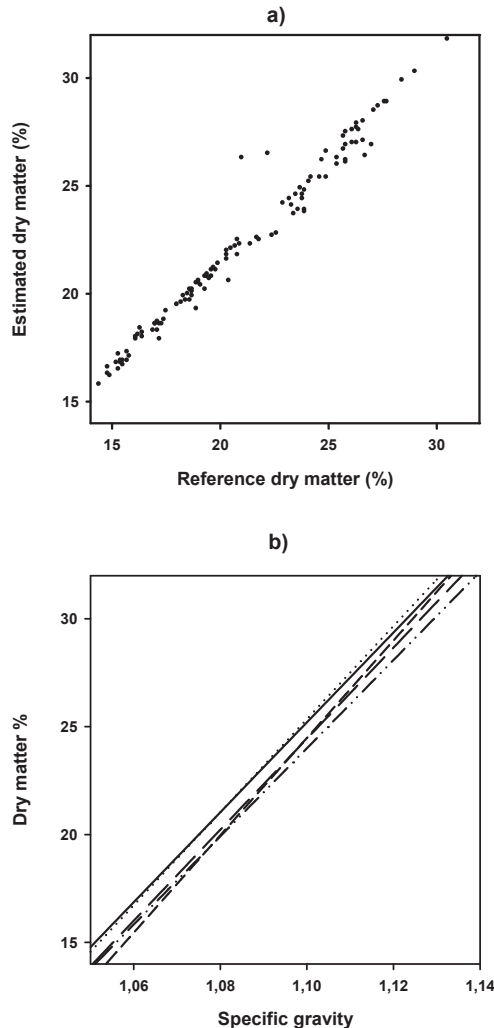


Figure 1. a) The dry matter values obtained from specific gravity measurements and the reference values obtained by freeze drying. b) Some of the most common regressions (Lunden, 1956) for converting underwater weight into dry matter content

3.2 1D NIR Interactance Measurements

The surface topography and especially heterogeneity of the potato tuber will clearly affect the NIR acquisition. To better understand how each sample affects the measurements, light penetration depths in 10 tubers were measured by 1D NIR interactance (data not shown). A penetration depth of up to 20 mm into unpeeled tubers could be achieved. An experiment measuring 60 tubers at three different places (stem, bud and middle) showed that measurements taken at the middle of the longitudinal axis of each tuber gave the highest explained variance (R^2) and the lowest RMSECV for estimation of dry matter content (data not shown). This is in accordance with the findings of Pritchard and Scanlon (Pritchard & Scanlon, 1997).

Figure 2a and 2b shows the absorption and the SNV corrected spectra, respectively, obtained from the 1D NIR

interactance measurements. As one can see there is a large absorption band at 960 to 980 nm, which is typical for the second OH-water overtone (960-970 nm). Spectra were related to reference values using PLS regression. The performance of the developed model is provided in Figure 3a. PLSR regression provided a high explained variance value ($R^2=0.95$) and an RMSECV value of 0.91, with a model based on five PLSR factors. Regression coefficients (Figure 3b) show that the main positive contribution to the model is found in the area where the second overtone for the carbohydrate OH-stretch absorption band (901 nm) is known to appear (Williams & Norris, 1990). The second OH-water absorption overtone is known to appear at 960 nm, which seems to give a positive contribution to the dry matter content determination. The absorption in the region 960-980 nm may thus partly be ascribed to starch. The carbohydrate OH-stretch is known to give a strong absorption band at 979 nm, which may explain the positive contribution from the wavelengths in the region 960-980 nm (Williams & Norris, 1990).

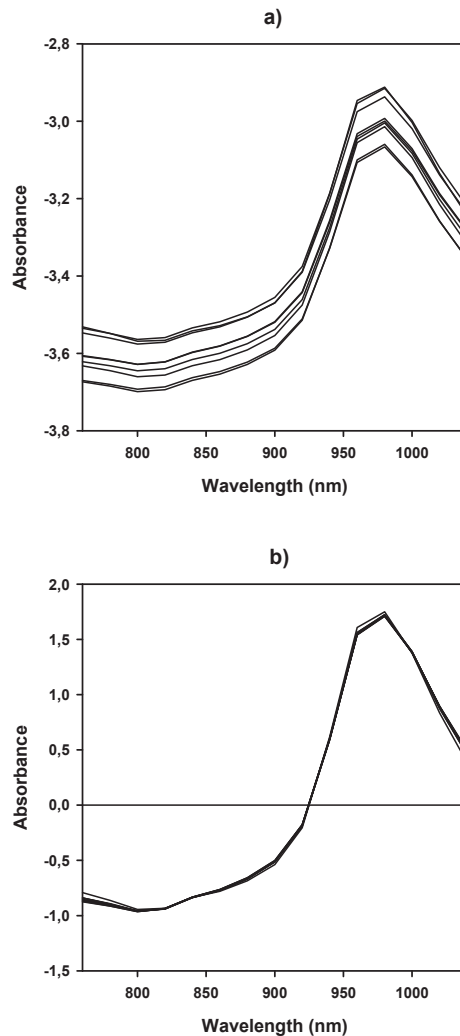


Figure 2. Absorbance a) and SNV-transformed spectra b) of whole unpeeled potato tubers acquired by 1D NIR interactance

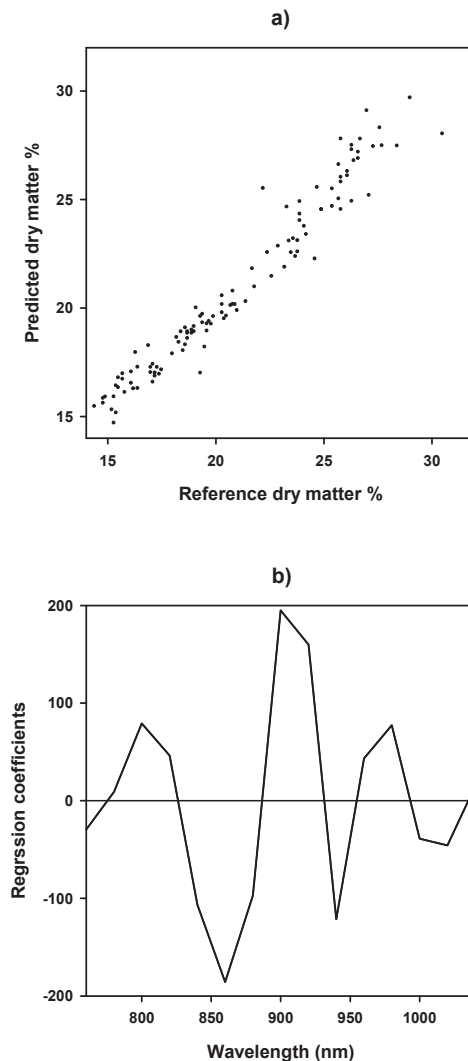


Figure 3. a) The relationship of predicted dry matter obtained by 1D NIR interactance against reference values obtained from freeze-drying. b) Regression coefficients as a function of wavelength obtained by PLS regression used to predict the dry matter in whole unpeeled potato tubers

3.3 2D NIR Interactance Measurements

In contrast to a 1D NIR interactance instrument, the 2D NIR interactance system creates an array of spectra in a short period of time, enabling it to monitor and assess an incoming stream of tubers in a factory. The 2D NIR interactance system obtained spectra similar to those shown for the 1D NIR interactance system. The 2D NIR interactance system could only measure 10 mm into unpeeled tubers. In contrast to 1D interactance measurements, 2D NIR interactance spectra were not SNV corrected prior to PLSR regression. This may indicate that the 2D NIR interactance system is less vulnerable to physical variation in e.g. sample surface. Figure 4a shows the regression result, providing an explained variance (R^2) of 0.83 and a RMSECV value of 1.65. The 2D NIR interactance system thus provided a significantly lower R-square value and a higher prediction

error than what was obtained by 1D NIR interactance measurement. From Figure 4b one can see that the regression vector is based mainly in the same absorption bands as the model obtained with the 1D instrument.

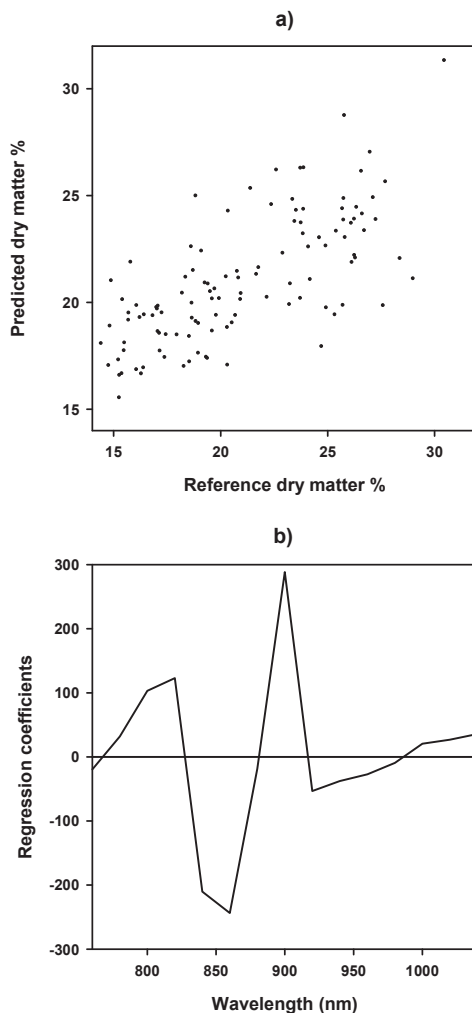


Figure 4. a) A scatter plot of the predicted dry matter values predicted by the 2D interactance instrument plotted against the reference values. b) The regression coefficients from the regression models used to predict the dry matter in whole unpeeled potato tubers

To be able to predict the dry matter content accurately, the 2D NIR interactance system has to distinguish between the sample and the conveyor belt. For the current study it was desirable to assess each tuber individually, but in an industrial application an average of the whole width of the conveyor can be determined, if necessary. The tubers used in this experiment varied in size. A single segregation routine was therefore not sufficient, and a double criterion was developed. After the first extraction the pixels were counted, and if the number of pixels exceeded a certain value the image was re-sampled with a new and higher threshold. This had a dual purpose: 1) to assure that the area used to extract spectra only contained potato matter and not parts of the conveyor belt; and 2) to ensure that an area with the same relative size, compared to the whole tuber, was selected for the sampling.

3.4 General Discussion

A summary of predictive performances of the three methods is shown in Table 2. Underwater weight is the method with largest deviation, but this is not truly comparable since an already existing regression model was used. The explained variance is still very good, and a careful selection of regression model might even further reduce the error.

Table 2. An overview of the R-square and RMSECV values obtained in the experiments

	R ²	RMSECV	Pre-treatment	number of PLSR factors	Average estimated value for all samples
1D Interactance measurements	0.95	0.91	SNV	5	21.0
2D Interactance measurements	0.83	1.68	none	9	21.0
Specific gravity	0.97	0.65	none	-	22.3
Reference value	-	-	-	-	21.0

One of the most important differences between the techniques investigated in this study is the ability to assess the variation of dry matter content in batches of potato tubers. The major drawback with the underwater weight is that it can only be used with small sample volumes. In the Norwegian potato industry the common sample size is 2 x 5 kg of tubers per 10 metric tons. The 1D NIR interactance system is not suited for assessment of samples on a conventional conveyor belt, but can be incorporated on a single line conveyor belt. The 1D instrument can be configured to record multiple spectra per second, and could hence monitor single tubers going down for instance a chute prior to sorting. The instrument used in the present study is a prototype, but a commercially available version exists (QPoint, QVision AS, Norway). The 2D NIR interactance system suffers from higher prediction error than the two other approaches, but the system can easily assess each tuber in the whole batch and calculate a batch mean. Then the user can decide whether the output should include both maximum, minimum, and average values, or just the latter. The higher prediction error is a typical tradeoff when one desires higher speed and larger throughput volumes. There will always be a trade off in each individual application whether the prediction accuracy, SNR or sampling speed is most important.

The dry matter content in potato tubers does not only vary within a batch, and there is also a considerable variation within each tuber (Storey, 2007). Light will travel a shorter distance relative to size, into a large tuber, than into a small one. Hence, it may be beneficial to adjust the regression model according to the actual size of the tuber, keeping in mind that the dry matter content is unevenly distributed within the tuber. For 2D NIR interactance measurements, if desirable one could include the same size threshold used for the segregation routine and say that tubers exceeding a specified size should be estimated by a different regression model. This model should thus to some degree compensate for the fact that the measurement is taken from the outermost layer of the largest tubers. In addition, light from the 2D NIR interactance instrument penetrated to a shallower depth into each tuber than the light from the interactance system, hence sampling a smaller part of the dry matter content gradient. This could explain some of the prediction error, especially when considering the large intra-tuber variation in dry matter content. Other factors that may have contributed to a high estimation error of the 2D NIR interactance system might include shorter measurement times and reduced SNR compared to the 1D system.

Pre-treatment of the data is an important part of data modeling, and SNV-transformation is one of the most frequently utilized techniques. In contrast to data obtained with a point measurement system, the 2D NIR interactance data did not undergo SNV-transformation before regression. Performing regression on SNV-transformed data yielded a model (data not shown) with slightly poorer performance than when using absorbance data. The reason for this was not investigated.

One issue not discussed so far is the sampling speed of the 2D NIR interactance system. The QV500 is rated to

handle a conveyor belt speed up to one ms^{-1} , with a sampling width of 50 cm. This would accommodate the possibility of measuring large quantities in a short time frame. One drawback with both the 1D and the 2D NIR interactance systems is the low spectral resolution. The interactance system has the ability to measure 30 channels, 15 of these originating from the visible light region. The 2D NIR interactance system features the same 15 channels in the near infrared region. The visible part of the spectra was discarded since tubers of different color were used. The regression coefficients for the 1D and 2D NIR interactance systems (shown in Figure 3b and 4b, respectively) show that the second overtone of the OH stretch from carbohydrates dominates the regression coefficients. Since the regression model estimates dry matter content, this indicates that it can be difficult to estimate other tuber attributes, like starch content and composition, without a significant increase in spectral resolution of the measurement system.

4. Conclusion

A robust NIR-based calibration model is usually continuously developed to take into account all expected physical and chemical variation. For industrial purposes it may thus be beneficial to develop calibrations based on the varieties used on site, and leave other varieties out of the equation. The calibrations presented here should therefore not be seen as universal, and factors such as variety, surface topography, growth conditions and diseases may affect the prediction performances. The results, however, are still expected to provide realistic insight into the predictive performances of the commercial instrumentation investigated. This study shows the feasibility of using high speed industrially available 1D and 2D NIR interactance instrumentation for dry matter content assessment in single potato tubers. The performance of the 1D NIR interactance instrument exceeded the performance of a 2D NIR interactance instrument ($R^2=0.95$, RMSECV=0.91 and $R^2=0.83$, RMSECV=1.65, respectively). Better predictions could possibly be achieved by optimization of the light-configuration in order to obtain a largest possible penetration depth. Even though the 1D NIR instrument is primarily built for stationary use, it can easily be mounted and used on a single line conveyor belt or narrow chute.

Acknowledgements

Martin Høy, Bjørg Narum and Karen W. Sanden are acknowledged for excellent technical assistance. Financial support from the Agricultural Food Research Foundation (Oslo, Norway) and the industrial partners (KiMs Norge AS, Maarud AS, Buer AS, Hoff SA and Bama AS) in the project nr. 199421/O99 is greatly acknowledged.

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Paper II

Towards on-line prediction of dry matter content in whole unpeeled potatoes using Near-Infrared spectroscopy

Trygve Helgerud^{1,2}, Jens P. Wold¹, Morten B. Pedersen^{1,2}, Kristian H. Liland ^{1,2}, Simon Ballance¹, Svein H. Knutsen¹, Elling O. Rukke² & Nils K. Afseth¹

¹) Nofima AS - Norwegian Institute of Food, Fisheries and Aquaculture Research, PB 210, N-1431 Ås, Norway

²) Norwegian University of Life Sciences, Department of Chemistry Biotechnology & Food Sciences, N-1432 Ås, Norway

Corresponding author: Trygve Helgerud, Nofima AS, PB 210, N-1431 Ås, Norway.
Tel: 47-6497-0100. E-mail: trygve.helgerud@nofima.no

Abstract

Prediction of dry matter content in whole unpeeled potatoes is a desired capability in the processing industry. Accurate prediction of dry matter content may greatly reduce waste quantities and improve utilization of the raw material through sorting, hence also reducing the processing cost. The following study demonstrates the use of a low resolution, high speed NIR interactance instrument for prediction of dry matter content in whole unpeeled potatoes. Three different measuring configurations were investigated: 1) off-line measurements with contact between the potato and the light collection tube; 2) off-line measurements without contact between the potato and the light collection tube; and 3) on-line measurements of the potatoes. The offline contact measurements gave a prediction performance of $R^2= 0.89$ and $RMSECV=1.19$. and similar prediction performance were obtained from the off-line non-contact measurements ($R^2=0.89$, $RMSECV=1.23$). Significantly better ($p=0.038$) prediction performance ($R^2=0.92$, $RMSECV=1.06$) were obtained with the on-line measuring configuration. Thus, showing the possibilities of using the instrument for on-line measurements. In addition it was shown that the dry matter distribution across the individual tuber could be predicted by the model obtained.

1. Introduction

The potato processing industries, and especially the frying and starch industries, are heavily dependent on reliable measurements of dry matter content. This is an important quality parameter frequently used to determine both the cost of the raw material and, most importantly, the subsequent choice of processing parameters. Today, most processing plants adjust their processes based on dry matter content predicted by simple gravimetric methods, which are based on the density of the potatoes [1, 2]. These predictions are based on batch samples (typically 5 kg samples), which implies that any process adjustments are based on average dry matter content of a small subset, and not of a whole batch of potatoes. The variation within a crop is then not taken into account. This variation is found to be significant, and it depends on factors such as growing conditions and choice of cultivar [3, 4]. The ability to accurately predict the dry matter content, and the resulting possibilities for continuous sorting, would allow for a more uniform stream of raw material. This is expected to lead to easier processing and a significant decrease in waste quantities.

Near-Infrared (NIR) spectroscopy is a frequently used tool for rapid and reliable prediction of quality parameters in a wide variety of foods [5, 6]. This technology can then give input for devices for sorting of both animal and vegetable based foodstuffs [6-8]. NIR measurements with subsequent sorting is usually based on the chemical composition of the foodstuff, but it has also been used for more physical parameters such as the gross meat content of intact crabs [9].

NIR analyses have been extensively studied for potatoes in recent years. These studies have, however, often been restricted to homogenized samples like potato pulp, sliced potatoes and cooked potato mash [10]. The prediction of dry matter content in such samples is highly accurate, as the water is sensitively probed [10]. Haase and co-workers [11, 12] obtained correlations (R^2) around 0.98 for prediction of dry matter content of potato pulp. Also the starch content, which is closely correlated with dry matter content in potatoes, can be predicted with high accuracy in homogenized samples using NIR. Minor constituents like sugars are generally reported to yield lower prediction performance [10], although some authors have achieved decent predictions ($R^2=0.82$) of total reducing sugar content in homogenized samples [13].

Despite the promising results for using NIR for prediction of dry matter in homogenized samples, the use of NIR on whole unpeeled potatoes is only

infrequently encountered in the literature. Transmission studies performed by Dull and coworkers [14] paved the way for investigation of whole unpeeled potatoes, and moderate prediction performance ($R^2=0.84$) was obtained for dry matter of intact potatoes. Subedi and Walsh [15] obtained similar predictions ($R^2=0.85$) of dry matter in their NIR analysis of whole potatoes, but in reality only a small cube of potato tissue directly below the measured area was used for reference analysis. Another approach that has shown decent performance is the use of NIR spectroscopy for prediction of the specific gravity (i.e the density) of potatoes [16]. The drawback with this approach is the conversion of specific gravity to dry matter content. The equations used for this conversion are often old and many different models exist, indicating that the relation between specific gravity and dry matter content might depend on several different factors [17].

In all above-mentioned studies, high resolution laboratory instruments were utilized, lacking the speed necessary for on-line industrial applications. The present authors recently showed that a rapid NIR interactance system of low spectral resolution (15 NIR channels) can be used for predicting dry matter ($R^2=0.95$) in whole unpeeled potatoes off-line [4]. The light penetration depth in intact potatoes was found to be approximately 20 mm. On the other hand, when employing a commercial on-line NIR interactance imaging system, lower prediction performance ($R^2=0.83$) was obtained [4]. The lack in performance for measurements of whole unpeeled potatoes, with the latter instrument, can most likely be ascribed to shallow light penetration (<10 mm) into the potato combined with the heterogeneous distribution of dry matter content within the potato tuber [18-20]. The dry matter content may differ significantly from the outer to the inner part of the potato tubers [21]. Similar results were reported for on-line studies of sliced samples ($R^2=0.85$) [15]. Despite recent efforts, there is still no commercially available system for on-line prediction of dry matter in whole unpeeled potatoes.

In the present study, a rapid prototype high speed, low resolution NIR interactance system (described in [4, 5]) is demonstrated. The aim of the current study was to investigate and evaluate further use of this instrument for online prediction of dry matter content in whole unpeeled potatoes, using three different sampling configurations: 1) off-line measurements with contact between the potato and the light collection tube; 2) off-line measurements without contact between the potato and the light collection tube; and 3) on-line measurements of the potatoes.

The impact of the intra tuber dry matter gradient on the prediction performance was also investigated, as this was suspected to affect the prediction of dry matter content in samples moving past the detector.

2. Materials and methods:

2.1 Dataset and sample preparation

A total of 240 potatoes from 7 different potato cultivars were obtained from either commercial suppliers or local potato processing industry. An overview of the cultivars and number of samples are provided in Table 1. All samples were stored at 4 °C prior to analysis. Samples were cleaned by hand in tap water and equilibrated (16 hours) to ambient temperature (18 °C), also allowing the outer skin to dry prior to spectral acquisition.

2.2 Spectral acquisition

A prototype NIR interactance instrument was used for spectral acquisition [5]. Whole potato tubers were illuminated with two 50 Watt halogen bulbs (Osram, Augsburg Germany) and backscattered light was collected through a collection tube. The instrument was equipped with a detector recording 30 equally spaced channels from 449-1040 nm [5], thus recording both visible and near infrared light. In this study the 15 channels from 760-1040 nm were used, thus excluding the visible part of the recorded spectrum. The NIR measurements were performed in three different ways: (1) the potato was placed in direct contact with the light collection tube of the NIR instrument, henceforth denoted “off-line contact measurements”. This collection geometry has been previously studied [4]; (2) the potato was placed approximately 10 mm below the light collection tube of the NIR instrument, henceforth denoted “off-line non-contact measurements”. This configuration was used to mimic the optical path length encountered in typical on-line situations; and (3) on-line collection of spectra of potatoes moving along a conveyor belt below the collection tube, henceforth denoted “on-line measurements”.

For both off-line contact and non-contact measurements a collection time of 2 seconds was set. Measurements were performed in triplicate, rotating the sample 60° in the horizontal plane, between each measurement. Spectra were acquired from the center of the longitudinal axis, which is by others found to be the location with dry matter content closest to the average of the potatoes [21]. On-line measurements were performed by sending the potatoes along a conveyor belt moving at 15 cm/s with the detector mounted directly above the conveyor belt. Each potato was exposed to the light for about 1/3 of a second. The sampling rate for the spectrophotometer was 80 spectra per second, and each measurement lasted for 5

seconds. Each moving potato was measured in triplicate, but was not rotated between measurements. They were placed on a small metal ring to stabilize them and prevent them from rolling around on the conveyor belt.

2.3 Reference analysis

Each individual potato tuber was carefully grated into strips (cross section of 2x3 mm) with a bench top grater (Hallde RG-100, AB Hällde Maskiner, Sweden). Three sub-samples (20±1 g) of each potato tuber were immediately placed in aluminum pans and dried for 48 hours at 105 °C in a forced fan oven. Dry matter percentages were calculated based on the weight before and after drying. The standard error of the reference method (S_{ref}) was calculated by the following equation (1):

$$S_{ref} = \sqrt{\frac{\sum_{i=1}^I S_i^2}{I \cdot N}} \quad (1)$$

Where S_i denotes the standard deviation for each sample, I denotes the number of replicates and N denotes the number of samples.

2.4 Data processing

Spectra from off-line contact and off-line non-contact measurements were pre-processed using an in-house MATLAB routine (MATLAB, V.7.10, The MathWorks, Natick, MA). Averaging of spectra from each sample was performed, and all spectra were log-transformed into apparent absorbance values. Spectra from the on-line measurements were processed by another in-house built MATLAB routine, separating potato spectra from conveyor belt spectra. NIR spectra from the conveyor belt differed from spectra obtained from potatoes. Separation of the two types of spectra could be based on intensity differences in the raw spectra, observed at four different wavelengths (i.e. the intensity of 800 nm had to be greater than the intensity of 760 nm and the intensity difference between 880 nm and 820 nm had to be greater than 350). Finally, an average of the three replicate measurements from each potato was calculated.

In addition to the averaged spectra representing a whole potato tuber, the MATLAB routine provided the individual spectra from each potato tuber. Each individual spectrum represented a movement of 2-3 mm along the axis of the potato.

These were used for calculation of the gradient in dry matter content along the potato.

Further processing was performed using the Unscrambler X software (V.10.2, CAMO PROCESS AS, Oslo Norway). The standard normal variate (SNV) transformation [22] were applied to spectra for both types of measurements. Subsequently, regression models were developed using Partial Least Squares (PLS) regression [23], with the dry matter values determined by oven drying used as reference values. The optimal number of PLS factors was determined by full cross validation. Root Mean Square Error of Cross Validation (RMSECV) was estimated by the following equation (2):

$$\text{RMSECV} = \sqrt{\frac{\sum_{i=1}^l (\hat{y}_i - y_i)^2}{l}} \quad (2)$$

where i denotes the samples from 1 to l , y_i and \hat{y}_i denotes the reference value and the predicted value, respectively. Principal Component Analysis (PCA) was performed in the Unscrambler X software to elucidate the variation within the dataset.

Analysis of variance of the cross validation (CVANOVA) [24] were performed, including a two-way ANOVA and pairwise comparison (Tukey's), using MiniTab (MiniTab v16, MiniTab Inc. USA), allowing significance testing of model differences.

3. Results and Discussion:

The dataset comprised 240 potatoes of 7 different cultivars. An overview of the samples is provided in Table 1. The dry matter range spanned from 14.7 to 31.5 %, thus covering most of the natural variation found in potatoes [19]. The standard error of the reference method was found to be 0.18 %.

3.1 Off-line contact and non-contact measurements

Spectra recorded using the off-line contact configuration gave a PLS regression model with R^2 value of 0.89 and an RMSECV value of 1.19 (5 PLS factors). SNV-transformed spectra of the potatoes along with regression coefficients of the 5 factor PLS model are shown in Figure 1a. When examining the regression coefficients, it became evident that the main contribution to the regression model comes from a region around 890-930 nm. A smaller contribution can be found around 960-980 nm. The region around 960 nm is known to contain the second overtone from the O-H bond in water molecules [25]. This may seem contradictory, but the C-H bond in starch may, according to Williams and Norris [25], have a strong absorption band at 979 nm. This may explain the positive contribution from this area. 878 and 901 nm are also pointed out as two starch absorption bands by Williams and Norris [25]. The regression coefficients are very similar to those found by others [15], except that they are shifted about 30 nm to the right in relation to the spectra. Subedi and Walsh [15] used the second derivative of absorption spectra, rather than SNV-transformed absorption spectra, which may explain the shift in regression coefficients. The regression coefficients are also almost identical to those found in a previous study by the current authors [4]. The prediction performance, however, is slightly lower than what was achieved in the previous study, which may be ascribed to a larger dataset and different selection of cultivars in the current study.

The spectra obtained from the off-line non-contact configuration yielded a prediction performance comparable to the off-line contact method, with $R^2=0.89$ and $RMSECV=1.23$ (5 PLS factors). The difference in performance was not significant ($p=0.482$). SNV-transformed spectra and regression coefficients can be seen in Figure 1b. It should be noted that the regression coefficients are found to be very similar to those found for the off-line contact measurements, which indicates that the non-contact configuration does not significantly alter the output of the NIR

measurements. The ability to perform non-contact measurements is essential in order to develop a procedure for on-line measurements.

3.2 On-line measurements

On-line measurements require that the sampling procedure can distinguish between the samples and the conveyor belt. One way to do this is to utilize the spectral difference between the background (e.g. the conveyor belt) and the sample. In Figure 2a a section of the recording, before the threshold was applied, can be seen. It contains both the potato spectra and the conveyor belt spectra (both SNV-transformed). The potato is represented by the spectra with a clear peak around 970 nm. NIR spectra after removal of the conveyor belt-spectra can be seen in Figure 2b. All spectra extracted from one potato tuber were averaged and used for PLS regression. A regression model based on the on-line measurements gave good prediction performance ($R^2=0.92$, RMSECV=1.06, 6 PLS factors). The model obtained from the on-line measurements was significantly better than the model from the off-line non-contact measurements ($p=0.038$), but not significantly better than the model from the off-line contact measurements ($p=0.362$). Example spectra from the on-line measurements, along with the regression coefficients of the corresponding PLS model, are shown in Figure 3. The spectra and the regression coefficients do not differ much from the ones acquired from the off-line results. All regression results are summarized in Table 2.

To evaluate if the extraction criterion was correct and if the number of spectra extracted from each potato tuber could be reduced, PLS models were made extracting fewer spectra from each sample. First, a model was made using only the three spectra from the middle of each sample. This model had a significantly ($p=0.036$) lower prediction performance ($R^2=0.89$ and RMSECV=1.25) than the one using all spectra from each potato tuber. Secondly, two spectra from each end of the potato tuber were removed to see if removal of potential interference from the conveyor belt and reduced light scattering effects caused by the curvature of the potato tuber would improve the prediction performance. This also gave a model with slightly lower, but not significantly lower ($p=0.926$), prediction performance ($R^2=0.91$ and RMSECV=1.11) compared to the model using all spectra. This may also indicate that the threshold used to distinguish between the sample and conveyor belt worked as intended. This shows that a further reduction in the number of extracted spectra

by removing spectra from each end of the sample will remove information rather than noise.

The average size of the potatoes used in this study was 50 mm across the shortest axis. At the conveyor belt speed used, this should theoretically result in an average of 20 spectra per potato using the current equipment. After extraction of the sample spectra from the conveyor belt, an average of 15-17 spectra were left, hence further indicating that the threshold was effectively removing enough to avoid interference from the conveyor belt and the outermost ends of the potato.

Given the above-mentioned measurement conditions, each individual NIR spectrum will represent 2-3 mm of movement along the potato axis. This could allow for prediction of internal dry matter differences along the main axis of the potato. Thus, the individual NIR spectra from each potato were extracted, and dry matter content of each spectrum was predicted using the PLS model established with the averaged spectra from the on-line measurements, as described above.

It should be noted that the PLS regression used for this was made based on spectra and dry matter content values averaged over whole potatoes. The dry matter along the potato axis may not fully correspond to the average value in the potato [21], hence values predicted for individual spectra along the potato may not fully correspond to the actual value found in the potato. The interrelation between the different spectra should, however, be correctly predicted. The variation in dry matter content across the potatoes fell mainly into three different categories. Examples of internal dry matter distributions along the main axis of potatoes are provided in Figure 4. The first category consisted of potatoes with nearly constant dry matter content across the potato axis. A prediction example of this is shown in Figure 4a. Note that three replicates of the same potato are shown side by side. The second category consisted of the potatoes where the dry matter content increased from one end of the measurement to the other. Whether this is seen as decrease or increase depends on the direction of measurement, as can be seen in Figure 4b. The trend shown in Figure 4b is believed to be the gradient found along the longitudinal axis (perpendicular to equatorial axis). The third category contained potatoes where the dry matter decreased towards the center, but then increased again when approaching the other end of the potato, shown in Figure 4c. This is believed to be the dry matter gradient from the stem end towards the bud end of the potato (longitudinal axis). The two different dry matter gradients shown in Figure 4a and b

were representative for most of the potatoes analyzed, and the results are in good compliance with the findings of Peiris and co-workers [26]. This relationship is also established by other authors [18, 21]. For some potatoes, however, peculiar and deviating results were obtained, which also was clear from the specific prediction errors obtained from the software. This might indicate that the shape of the potato was irregular and hence not allowing accurate prediction, or that the water gradient within the tuber may not be constant for all potatoes. Overall, the internal gradients and the corresponding interpretations serve as a strong indication that the NIR approach is feasible for internal dry matter content predictions.

3.3 General discussion and further development

The off-line contact measuring configuration, described in section 3.1 (summary given in Table 2), should theoretically give the best prediction result, due to the elimination of all surface reflection and stray light of the measurements. The off-line non-contact and the on-line measurements do allow some surface reflection and stray light to be collected. The regression model shows, not surprisingly, a slightly, but not significantly lower prediction performance for the off-line non-contact compared to the off-line contact measurements. However, the on-line measurements provided a significant increase in prediction performance compared to the off-line non-contact configuration, but the PLS model needed one additional PLS factor, as described in section 3.2. Since the dry matter content varies along the length of the tuber, the on-line measurements might therefore give a better spectral representation of the dry matter content in the tuber and a correspondingly better prediction. Another possible explanation is that the recording and averaging of multiple spectra across a moving potato may eliminate most of the contribution from local skin defects, such as scabs and scurfs common on potatoes [27]. In this study three replicates of each potato tuber were averaged before the calibration model were obtained. However, based on a PCA analysis of the replicates (data not shown) and the information in Figure 4, no large replicate variance is seen. Hence, one replicate may be enough if the instrument is mounted above a conveyor belt.

One of the most important aspects of on-line prediction of the dry matter content is speed. According to Brunt & Dorst [28], the starch industry can receive up to 250 000 kg , or 10 truckloads, of potatoes per hour during peak season. The speed of the raw material flow, of course, depends on the number of conveyor belts used to

handle the incoming raw material. It is unlikely that this amount can be handled by one single conveyor belt and, which may require multiple instruments to ensure good coverage. Brunt and co-workers made a prototype machine for prediction of starch and dry matter content in potatoes [29]. They briefly investigated the use of NIR for dry matter content prediction, but in the end traditional gravimetric prediction of the dry matter content was used. The authors stated that 10 samples per hour would suffice for their needs, each sample containing 3-5 kg. To sample one sample (5 kg) for each 10 000 kg is also common when using the gravimetric method for determining dry matter content. This equals 25 samples per hour (total of 125 kg of tubers), if calculating with the before mentioned amounts of potato. The conveyor belt in the current study was moving at 15 cm/s during the on-line measurements. Based on the number of spectra obtained from each potato tuber (approx. 15 spectra per tuber) it seems obvious that the speed can be increased. The prediction performance was largely maintained, provided that spectra used for dry matter content are samples from the entire length of the tuber. If increasing the speed to 25 or 30 cm/s the system would be able to measure nearly 300 individual tubers (with size of approximately 50 mm) per minute. This should be well within 10 samples per hour, and maybe also as much as 25 samples, allowing for different sampling regimes. Hence, the speed of the system demonstrated in the current study should have the capabilities to be incorporated in an on-line application. It is, however, expected that the current approach could be used at significantly elevated conveyor belt speed.

Robustness and variety independence are two key features in order to develop an industrial method for determination of dry matter content. The method used cannot be cultivar dependent or be affected by the skin color of the potato. A PCA analysis of average spectra from all on-line measurements is thus provided in Figure 5. As can be seen, there seems to be a slight grouping along PC2. However, dry matter content and cultivar is slightly confounded, as shown in Table 1. Labelling the samples in the PCA plot with the corresponding values for dry matter content, reveals that the dry matter content is explaining the variation seen along PC2. This, rather than cultivar dependent differences, seems to be a more likely explanation for the slight grouping seen in Figure 5. The PCA also indicates that the measurements are unaffected by the skin color. The two red colored cultivars (Asterix and Bruse) do not differ from the cultivars with yellow skin (Fakse, Folva, Mandelpotet, Saturna and Sava) along any of the two first PCs. This could also be related to the fact that the skin of the potatoes

is only about 0.25 mm thick [19], and the interactance instrument sends light up to 20 mm into an unpeeled potato [4]. Hence, the skin should give an insignificant contribution to the spectra in this configuration. Noticeable differences due to flesh color was not seen, and is also unlikely, since all cultivars used had yellow or light yellow flesh color.

4. Conclusion

The current study shows that the use of a high speed NIR interactance instrument with a low resolution spectrophotometer measuring 15 wavelengths is a feasible approach for on-line prediction of dry matter content in potatoes. The extraction of spectra from the background could be fully automated, and the dry matter content could be rapidly and accurately predicted on-line for individual potatoes. The model obtained from on-line measurements was significantly better than the model obtained from the off-line non-contact configuration and as good as the ones obtained from measurements in the off-line contact configuration. This was ascribed to a more representative sampling when measuring in the on-line configuration. Even dry matter gradients along the longitudinal axis of individual potatoes are reflected in the NIR spectra. The current measurement configuration only allows for measurement of one row of single potatoes, but it is expected that further instrumental development could increase expected sample throughput. The development of a user friendly instrument with the necessary hardware adapted to suit industrial environments is the natural next step in this development.

Acknowledgement:

Financial support from the Norwegian Research Levy on Agricultural Products in projects 199421/O99 and 225347/F40 is greatly acknowledged.

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FIGURES:

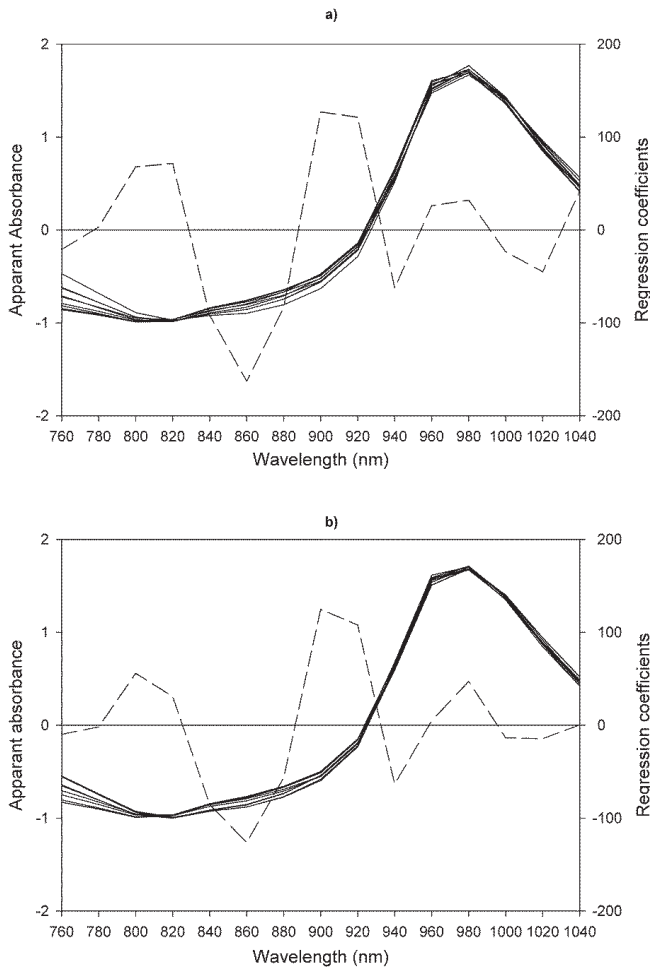


Figure 1: Example spectra (solid lines) and regression coefficients (dotted lines) for the models obtained from the off-line contact **(a)** and off-line non-contact **(b)** measurements. Spectra shown are SNV-transformed apparent absorption spectra. The models obtained from contact and non-contact measurements are both made with 5 PLS factors.

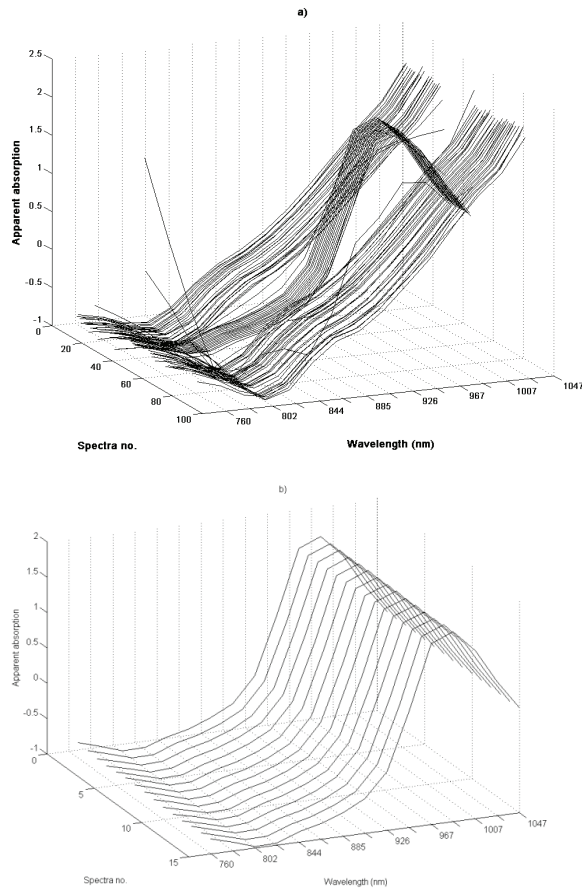


Figure 2: a) A section of the recording from an on-line measurement of a potato. Spectra with a peak at 950-990 nm represent the potato, the rest of the spectra represent the conveyor belt. All spectra seen are SNV-transformed. The sample shown is from the cultivar Asterix. b) Spectra remaining after removal of all spectra representing the conveyor belt.

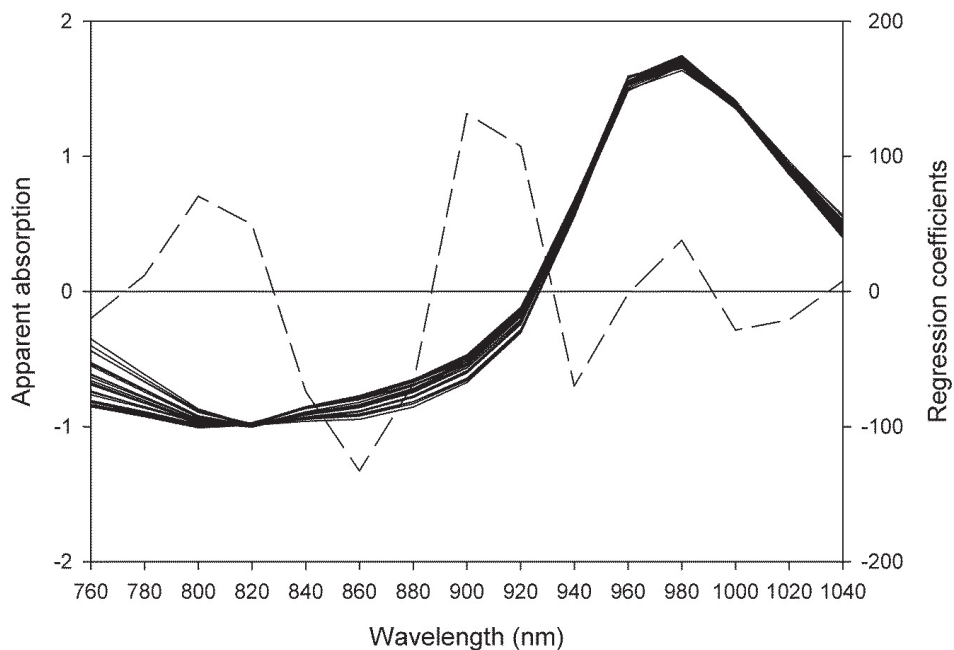


Figure 3: Spectra (solid line) from potatoes moving past the detector. Each spectrum shown contains the averaged spectra from one sample, and is SNV-transformed apparent absorption spectra. Regression coefficients (dotted line) are shown for the prediction model (6 PLS factors) obtained for prediction of dry matter content in potatoes moving along conveyor belt.

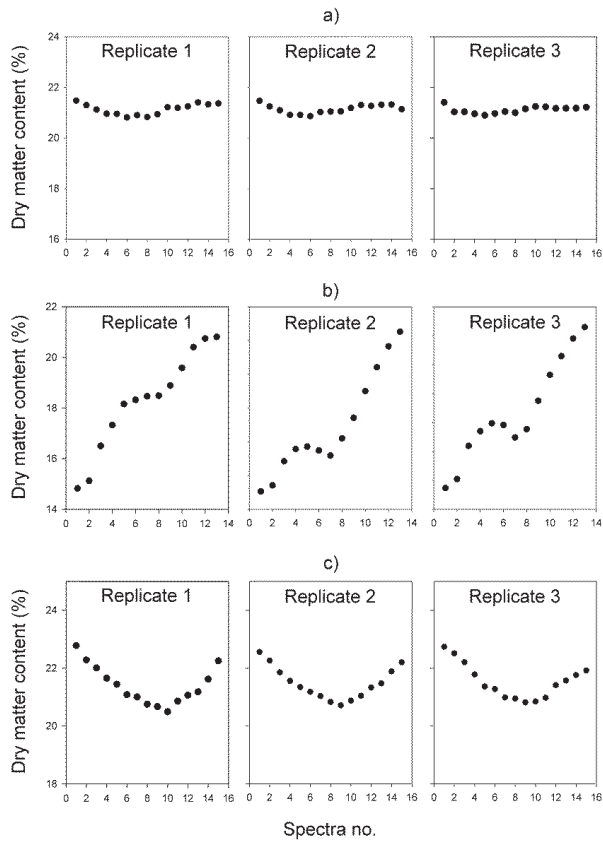


Figure 4: Three different patterns emerged when dry matter content was predicted from the individual spectra recorded from a sample. Note that three replicates are shown side by side. **a)** a sample with rather homogenous dry matter content. **b)** increasing (or decreasing, if potatoes was turned the other way before measurement) dry matter along the potato length. **c)** potatoes with lower dry matter content at the center, and higher dry matter content in the periphery.

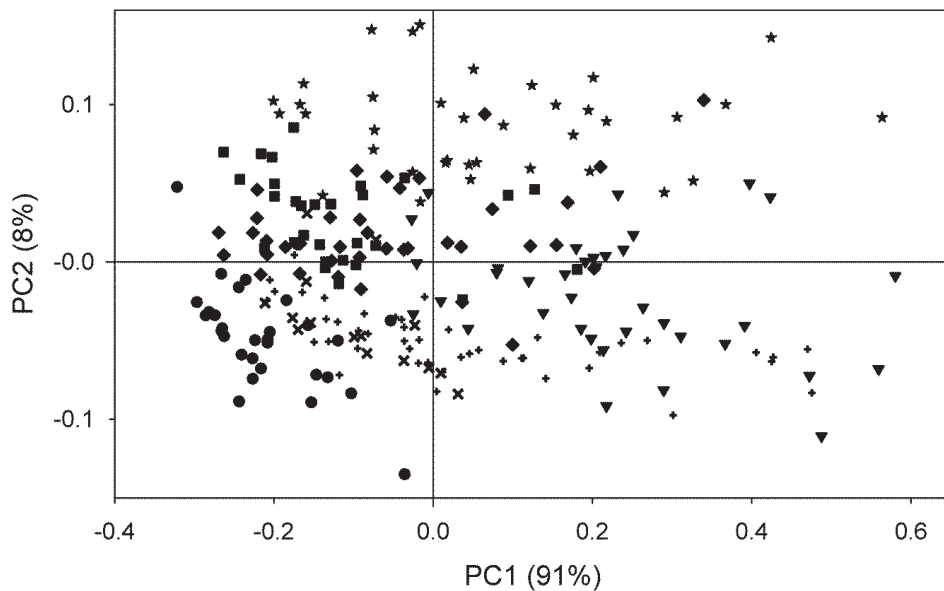


Figure 5: a) A PCA analysis of the averaged spectra from the on-line measurements showed no clear grouping of the cultivars, Asterix (●), Bruse (★), Fakse (▼), Folva (x), Mandel (■), Saturna (◆) and Sava (+). PC1 explains 92% and PC2 explains 8% of the variance

TABLES:

Table 1: An overview of the samples used (DM = dry matter content)

Cultivar:	Min. DM	Average DM	Max. DM	St.Dev DM	No. of samples	Skin color	Flesh color
Asterix	14.7	19.1	23.5	2.0	30	Red	Light yellow
Bruse	25.4	28.1	31.5	1.3	40	Red	Light yellow
Fakse	17.7	20.3	22.6	1.3	40	Yellow	Light yellow
Folva	17.1	20.3	23.8	1.4	30	Yellow	Light yellow
Mandelpotet	21.9	25.9	29.3	2.1	30	Yellow	Yellow
Saturna	21.8	25.4	28.3	1.4	40	Yellow	Light yellow
Sava	18.2	20.4	22.4	1.1	30	Yellow	Yellow
All samples	14.7	23.0	31.5	3.7	240	-	-

Table 2: An overview of the performance of the different regression models obtained.

Measuring configuration:	R²	RMSECV (%)	No. of PLS-factors
Off-line contact ^{a,b}	0.89	1.19	5
Off-line non-contact ^b	0.89	1.23	5
On-line ^b	0.92	1.06	6
On-line – 2 end-spectra removed ^b	0.91	1.11	6
On-line – 3 middle spectra ^a	0.89	1.25	6

^{a,b}) Measuring configurations not shearing a common letter show significantly different prediction performance.

Paper III

Evaluation of hand-held instruments for representative determination of glucose in potatoes

Trygve Helgerud^{a,b,*}, Svein H. Knutsen^a, Nils K. Afseth^a, Kim F. Stene^{a,b}, Elling O. Rukke^b and Simon Ballance^a

^a) Nofima AS - Norwegian Institute of Food, Fisheries and Aquaculture Research, PB 210, N-1431 Ås, Norway

^b) Norwegian University of Life Sciences, Department of Chemistry Biotechnology & Food Sciences, N-1432 Ås, Norway

*Corresponding author: Trygve Helgerud, Nofima AS, PB 210, N-1431 Ås, Norway.
Tel: 47-6497-0100. E-mail: trygve.helgerud@nofima.no

Abstract

In this study, the use of two modern portable blood glucose monitors (PBGMs) and a fruit juice specific system (RQFlex) from Merck were evaluated for determination of glucose contents in potato juice, as a representation of the whole potato. The two PBGMs (LifeScan OneTouch Ultra Easy and Bayer Contour) both provided squared correlation coefficients (R^2) of 0.92, when compared to reference analysis based on Anion Exchange Chromatography (HPAED-PAD). The LifeScan monitor gave an error estimation of 0.54 mg mL^{-1} , but overestimated the glucose content by 20 %. The Bayer monitor gave an error estimation of 1.12 mg mL^{-1} and underestimated the glucose content by 40 %. The RQFlex system outperformed ($R^2=0.96$, error estimation of 0.24 mg mL^{-1}) both the PBGMs, but this system is slightly more labor intensive to use than the PBGMs. A squared correlation coefficient of 0.91 was found when comparing the glucose content in potato juice and lyophilized potato powder, indicating that sampling of potato juice may be introducing uncertainties to the measurements.

1. Introduction

One of the major challenges in production of fried potato products, such as chips and French fries (US terminology), is the unwanted formation of acrylamide, which is formed through the Maillard reaction. Reducing sugars and asparagine are known as the precursors, determining the extent of acrylamide formation (Mottram et al. 2002; Stadler et al. 2002). Potatoes are heterogeneous both in sugar content and sugar distribution (Pritchard and Scanlon 1997) and can metabolize sugars throughout the storage season (Sowokinos 2001). This calls for rapid, cheap and simple tools for quantification of reducing sugars, both during storage and in subsequent processing steps. Hence, to be industrially applicable, the methods for sugar determination must be rapid (i.e. the result must be available within a few minutes), cheap and easy to use. Time consuming sample preparation should thus be avoided.

Traditionally, enzymatic kits, wet chemistry laboratory tests or chromatographic approaches have been employed in order to determine the reducing sugar content. All of these methods are accurate and reliable, but fairly time consuming (often >1 hour), labor intensive and do require trained personnel. No rapid methods for determination of fructose and sucrose are available, but for glucose some methods have emerged. In the 1990's the first report utilizing a Portable Blood Glucose Monitor (PBGM) to rapidly determine the glucose content of potatoes was published (Coleman et al. 1993). They showed a squared correlation coefficient (R^2) of 0.82 (no error estimation provided) between glucose content measured by an AccuCheck III monitor (Boehringer Mannheim, Canada Ltd, Dorval Quebec) and chromatographic analysis. Coleman et al. also demonstrated the use of PBGM for prediction of frying quality of chips (Coleman and Tai 1999). Another attempt made in 1996 by Misener et al. (1996), which demonstrated the use of a modified PBGM (ExacTech, MediScience Inc, Cambridge, MA) for glucose determination, showed a squared correlation coefficient of 0.76 (no error estimation provided).

The PBGM demonstrated by Coleman et al. during the 1990s, were based on glucose oxidase-peroxidase impregnated test strips. The strips were soaked in the sample solution, wiped clean after exactly 15 seconds, and then inserted into the monitor itself, with colorimetric detection to read the strips. Newer enzyme technologies (Hones et al. 2008) and more user friendly interfaces (Clarke and Foster 2012) have emerged in recent years, whereas the PBGM used by Coleman et al. are obsolete and no longer for sale (Clarke and Foster 2012). The newer type of test

strips are inserted into the monitor before sample application and thus provides fully automated analysis (Heinemann 2010). Thus, this removes the human interference from the analyses. Both the exact timing and the wiping, performed by the operator, were seen as drawbacks with older PBGMs (Clarke and Foster 2012). The enzymes used in the newer PBGMs are either a glucose oxidase-peroxidase combination or a glucose dehydrogenase combined with a mediator like flavin adenine dinucleotide. The latter is reported to be less prone to interference from, amongst others, ascorbic acid which may be abundant in potatoes (Burton 1989; Heinemann 2010). The new generation PBGMs also uses electrochemical detection, in contrary to the older monitors using colorimetric detection (Clarke and Foster 2012; Hones et al. 2008). The new PBGM technology is only briefly described for use on potato juice (Olsen et al. 2011), but they show promising results (R^2 of 0.83 to 0.99, no error estimation provided) for fermentation broths (Choy et al. 2007).

Another commercially available and portable instrument is the Merck Millipore (a subdivision of Merck KGaA, Darmstadt, Germany) RQFlex/Reflectoquant system. This system is made for analysis of, amongst other, different sugars in plant juice. It uses glucose oxidase peroxidase coated test strips and is based on colorimetric detection. Additional enzymatic test strips are supplied by Merck Millipore for measuring sucrose and total reducing sugars, but these require longer analysis time (>10 min per samples). Even though this instrument is commercially available, the authors are not aware of any scientific publications regarding the use of the RQFlex with Reflectoquant test strips for analysis of glucose in potatoes.

Rapid and reliable methods for glucose determination are essential for industrial applications, and many companies use a PBGM for rapid and convenient sugar determination. Thus, the main aim of this study is twofold; 1) to evaluate the suitability of two modern PBGMs and the RQFlex/Reflectoquant system for determination of glucose in potato juice and; 2) to investigate how the measurements of potato cell sap / juice correlate to the actual sugar content in the lyophilized potato tissue. Based on these data, the also interrelation between different sugars in the potato materials will be investigated.

2. Methods and Materials

2.1 Samples and general sample preparation

A total of 250 potato tubers, of 6 different cultivars, were collected in two batches from producers of chips and French fries. All samples were stored locally at 8 °C, in order to minimize the impact of cold induced sweetening. The tubers in batch 1 were used to test the RQFlex system and one of the PBGMs (OneTouch Ultra Easy (LifeScan Inc. USA)). The tubers in batch 2 were used to test the reliability of juice sampling, the interrelation between the different sugars, and to test the second PBGM (Bayer Contour (Bayer GmbH, Germany)). An overview of the cultivars, number of tubers, and batch sizes is given in Table 1. The tubers were cleaned in lukewarm water and dried with a paper towel on the morning of the day they were analyzed. The tubers in batch 1 were analyzed according to pathway a) shown in the flow chart provided in Figure 1, and tubers in batch 2 were processed according to both pathway a) and b). Samples from batch 2 were carefully sliced into halves along the stem-bud axis before processed into pathway a) and b), respectively.

2.2 Analysis with the PBGMs and the RQFlex

Potato juice was extracted with a household juice maker (HR1865/00, Phillips, The Netherlands) that was cleaned with water between each extraction. The potato juice samples from batch 1 were stored at -40 °C before analysis, while the juice samples from batch 2 were analyzed immediately. The analysis was performed by inserting the strips into the PBGM and then dipping a spatula into the potato juice, lifting a droplet onto the tip of the spatula. This was done to mimic a blood droplet on a fingertip. The end of the test strip was brought into contact with the drop, and the required amount of sample was automatically absorbed into the test strip. Analyses with the PBGMs were performed in duplicate.

Samples from batch 1 were also analyzed using a Merck Millipore RQFlex Plus (Cat.no.116955) instrument with the corresponding Reflectoquant glucose strips (Cat.no. 116720) supplied from Merck Millipore (Merk KGaA, Darmstadt, Germany). Potato juice samples were diluted in distilled water (1:50) in volumetric flasks. The test strips was submerged in the diluted sample solution, and subsequently the instrument was started. After soaking for 15 seconds, excess sample solution was wiped off on a paper tissue and the strip inserted into the RQFlex instrument. The reading (in mg L⁻¹) was shown in the instrument display after 60 seconds. Samples were analyzed in duplicate.

After the analysis with PBGM, samples from batch 2 were diluted with an equal amount of methanol (*puriss p.a.*, Sigma Aldrich), whereas samples from batch 1 were used as is. Sub samples (1 mL each) were centrifuged (15 600 g, 5 min, ambient temperature) in a table top centrifuge (IEC Centra M2, International Equipment Company, USA) to remove any solid residues in the potato juice. The supernatant was transferred to a 1.5 mL screw top vial and placed in boiling water for 15 minutes. This was performed to inactivate the enzyme *acid invertase*, which hydrolyzes sucrose into fructose and glucose (Sowokinos 2001). The sample vials were stored in a freezer prior to further analysis.

2.3 Reference analysis

For samples of batch 2, the second halves of the potato tubers (pathway b, Fig 1) were carefully sliced into smaller pieces and frozen in liquid nitrogen before lyophilization. The lyophilized samples were finely ground in a small kitchen blender (Moulinette, Part of GroupeSEB, France). These dried and powdered samples were analyzed with a modified version of the method of Elmore et al (2007). Each sample was weighed (200 mg \pm 10 mg) into a screw top tube (15 mL). Aqueous methanol (1:1 mixture) was added (15 mL), along with internal standard (100 μ L trehalose (10 mg mL⁻¹)). The samples were extracted for 15 minutes at a shaker table (250 rpm), followed by a resting period of 15 minutes. A sub sample of the supernatant (1.5 mL) was transferred to a small sample tube and centrifuged (13500 rpm, 15 min). Aliquots of the supernatant were diluted 1:10 with aqueous methanol (50 %) and filtered (0.22 μ m syringe filter) into vials. Samples were prepared in duplicate. The sugar content in the lyophilized samples is given in mg g⁻¹ of the dry weight (based on weight before and after lyophilization).

Aliquots of the juice samples (100 μ L) from both batch 1 and 2, prepared as described in section 2.2, were diluted with aqueous methanol (50 %, 1375 μ L) and internal standard (25 μ L, trehalose (10 mg mL⁻¹)). The samples were centrifuged (13500 rpm, 15 min) before dilution (1:10) with aqueous methanol (1:1 mixture) directly in 3 mL syringe, and filtered (0.22 μ m syringe filter) into vials.

All samples were analyzed by High-Performance Anion Exchange Chromatography with Pulsed Amperometric Detection (HPAED-PAD). Samples from batch 2 were analyzed with a Dionex ICS 5000⁺ chromatographic system (Thermo Scientific Inc., USA). It consisted of an AS-AP auto sampler, an ICS 5000⁺SP pump

and an ICS 5000⁺DC combined column oven and detector compartment. The detector unit installed was an ICS 5000⁺ED pulsed amperometric detector, with an Au working electrode and an Ag/AgCl reference electrode. Waveform A from the Dionex technical note 21 (Dionex 1998) was used. The system was equipped with a CarboPac PA-1 anion exchange column and a CarboPac PA-1 guard column. The column was kept at 25°C at all times. An isocratic elution program was set up, with sodium hydroxide (100 mM) for 15 minutes, followed by washing for five minutes with sodium hydroxide (100 mM) in sodium acetate (500 mM). The column was reconditioned for five minutes with sodium hydroxide (100 mM) before the next injection. Injection volume of 20 µL and a flow rate of 1 mL min⁻¹ were used for all samples. Samples from batch 1 were analyzed by the same protocol and the column system, except that a preceding instrument of the same brand and type described by Elmore et al. (2007), was used.

2.4 Statistical analysis

Linear regression, yielding the squared correlation coefficient (R^2), were performed in Sigma Plot (ver.11.0, Systat Software Inc, USA). The difference quotient from the linear equation was used to calculate the percentage over- or underestimation of the instruments. The term target line is used to illustrate perfect correlation (i.e. $R^2=1$). Root mean squared error of estimation (RMSEE) was calculated based on equation (1) and used as an error estimate in the study:

$$RMSEE = \sqrt{\frac{\sum_{i=1}^I (X - X_{ref})_i^2}{I-1}} \quad (1)$$

Here I is the number of samples, X is the measured value, X_{ref} is the reference value. The scaling of the glucose concentration, according to dry matter content, was performed using the following equation (2):

$$\text{Sugar content per dry weight} = \frac{A * B}{C} \quad (2)$$

Here A is the concentration (mg mL) of glucose in the potato juice, B is the amount of juice given as mL, C is the amount of dry matter in the potato tuber, calculated from the weight loss after freeze drying, given in gram. The sugar content per dry weight is given in mg g⁻¹. Amount of juice and dry matter was based on the weight before and

after lyophilization taking the assumption that the density of the potato juice is 1 mg mL⁻¹.

3. Results and Discussion

3.1 The sampling of potato juice

In practice all rapid methods for determination of glucose in potato tubers are utilizing the potato juice. This allows for faster sampling and requires no time consuming extraction of solid potato matter. Hence, these analyses depend upon a stable and reliable relationship between the glucose content measured in potato juice, and actual glucose content in the potato. In this study, the reference analysis showed that the glucose content in the lyophilized powder of batch 2 ranged from 0.1 to 28.9 mg g⁻¹. This is within the range of what has previously been found in processing potatoes (Amrein et al. 2003; Knutsen et al. 2009). The glucose levels measured in the juice ranged from 0.02 to 12.95 mg mL⁻¹. A squared correlation (R^2) of 0.91 between the glucose content found in potato juice and the glucose content found in lyophilized potato powder as obtained, as seen in Figure 2. Even though good correlation between the glucose content in the juice and the lyophilized material was obtained, it can be seen from Figure 2 that there are some deviations. One possible explanation may be the difference in dry matter content in the potatoes used in this study. It is well known that there are large inter tuber variations regarding dry matter content of potato tubers (Cole 1975).

When scaling the glucose content of the juice samples according to dry matter content, as shown in Figure 3, the correlation remains mainly the same ($R^2=0.91$) as found for comparison of juice and lyophilized material (Fig 2). This result indicates that scaling according to dry matter content does not compensate for the deviation between the glucose content found in juice samples compared to that found in the lyophilized samples. Hence, there must be other factors causing the discrepancies.

As the juice and pulp are separated by the household juicer, and not extracted into a solvent, a part of the glucose will be left in the pulp, which has been described for other pulped fruits and vegetables (Gunaseelan 2007). It is also known that different parts of the tuber yield different sugar concentrations in the juice (Pritchard and Scanlon 1997). This combined may lead to inconsistent glucose content in the juice extracted and may indicate that the main contribution to the deviation between the juice samples and the lyophilized samples comes from incomplete and inhomogeneous juice extraction. This should be kept in mind when using potato juice for prediction of glucose content in potato tubers.

3.2 Performance of the two PBGMs and the RQFlex for determination of glucose in potato juice

49 samples of batch 1 were within the detection range of the first PBGM (LifeScan OneTouch Ultra) and glucose values ranged from 0.2 to 5.0 mg mL⁻¹. Figure 4 shows a scatterplot between the glucose values from the LifeScan monitor and the reference analysis. A squared correlation coefficient of 0.92 and an estimation error of 0.54 mg mL⁻¹ were obtained for the first PBGM. Batch 2 were analyzed with the second PBGM (Bayer Contour), and consisted of 152 samples after removal of samples outside the detection range. The glucose content ranged from 0.08 to 5.5 mg mL⁻¹, measured with the Bayer-monitor. A scatterplot of the performance is shown in Figure 5. The Bayer monitor obtained a squared correlation coefficient of 0.92 and an estimated error of 1.12 mg mL⁻¹. Although the squared correlation coefficient is identical to that found for the LifeScan Monitor, it can be seen from the error bars in Figure 4 and 5 that there are differences. The LifeScan monitor also seems by visual inspection to give less variation between the parallels than what the Bayer monitor does. This is confirmed by the estimated errors, as the Bayer monitor gave a estimated error value of 1.12, compared to 0.52 for the LifeScan-monitor. Judging by the error bars, the Bayer monitor seems to yield unreliable results for glucose contents above about 4 mg mL⁻¹. Samples with glucose content above 2 mg mL⁻¹ were all from potatoes of cv. Peik. It was not elucidated if this was induced by the instrumentation, the sampling or specific constituents of this particular potato cultivar.

Neither the Bayer monitor, nor the LifeScan monitor seem to give a fully linear relationship throughout the detection range, as can be seen in Figure 4 and 5. The Bayer monitor seems to underestimate almost all samples, whereas the LifeScan monitor seems to slightly overestimate (Fig 4 and 5). The difference quotient from the linear regression indicates that values obtained by the Bayer monitor are, in average, approximately 40 % lower than the values obtained by the reference analysis. The LifeScan monitor seems to overestimate by about 20 %. Similar trends were found in a study of how the same two PBGMs were performing on fermentation broths (Choy et al. 2007), with a LifeScan monitor that overestimated and a Bayer monitor that underestimated. With respect to the differences between the two monitors, it should be kept in mind that two different sample series were used and that individual differences in these two may influence the results. However, taking into account the

results of Choy et. al. (2007) who found similar differences in performance when comparing two identical PBGMs for measurements in fermentation broths, this indicates that the difference in performance may most likely be ascribed to the monitors themselves.

Another important aspect in the determination of glucose in potato juice is the risk of interference from other constituents of the potato juice. The LifeScan monitor is based on glucose oxidase technology (Heinemann 2010), and is therefore seen vulnerable to the oxygen levels in the sample (Öberg and Östenson 2005). The Bayer monitor on the other hand, is based on glucose dehydrogenase technology and is therefore seen as less vulnerable to, but not completely unaffected by, the oxygen levels (Öberg and Östenson 2005). If oxygen levels in the samples are causing the lower prediction performance of the Bayer monitor, this may be explained by the sample preparation. The ripper blade inside the juice machine is introducing a lot of air into the juice samples during the juice extraction. Samples from batch 2 was used directly after juice extraction, but samples from batch 1 underwent a freeze-thaw cycle due to storage, which may have altered the oxygen levels in the juice, hence affecting the enzymes in the test sticks. However, a freeze thaw cycle is not possible to perform in an industrial environment, and the PBGM of choice should be able to predict the glucose content of fresh potato juice.

It should be noted that the two PBGM used are both giving the blood glucose as serum/plasma glucose, i.e. the values have been converted from whole blood glucose to serum glucose. Glucose content in the serum is 10 to 15 % higher than the content in whole blood (Holtkamp et al. 1975). This implies a corresponding instrument-induced overestimation of the glucose content in the potato juice. The conversion equations are usually proprietary and not available to the customer. Therefore, it is important that everyone using PBGMs for glucose analysis in potatoes should know whether their PBGM is showing values for whole blood or serum equivalents.

The RQFlex system (Fig 6), which is designed to measure the glucose content in samples like fruit juice, showed an overall good performance ($R^2=0.98$, $RMSEE=0.24$ mg mL⁻¹). The samples had a range from 0.3 to 11.1 mg mL⁻¹ glucose. Visual inspection of Figure 6 indicates that the RQFlex gives predictions close to the target line, with no over or underestimation. Good consistency between the replicates was

also seen. This indicates that the RQFlex is well suited for determination of the glucose content in the potato juice.

3.3 Advantages and drawbacks

A summary of the advantages and drawbacks of the different instruments evaluated in the study are given in Table 2. The RQFlex system, which is designed to measure glucose in plant juice, gave a higher squared correlation coefficient and lower estimation error compared to what was obtained with the two PBGMs. The Bayer monitor gave higher estimation error than the LifeScan monitor. The RQFlex also determined the glucose content without the over or underestimation that were seen with the two PBGMs (Fig 4, 5, and 6). When looking at the convenience of operation, the two PBGMs are less time consuming than the RQFlex. In selecting one of these 3 instruments then one has to weigh-up the trade-offs between ease of use and accuracy. It is often sufficient in an industrial setting to just broadly classify potatoes into groups of low, medium and high reducing sugar. In this case ease of use may be more appropriate for selection choice than accuracy.

Since at present only glucose can be measured rapidly by hand-held instruments one must use this value as a proxy to estimate total reducing sugars (fructose + glucose) Based on the sugar content in the lyophilized samples from batch 2 determined by reference method. Scatterplots of the correlation between the different sugars can be found in Figure 7. An R^2 of 0.93 was found for the correlation between the amount of glucose and fructose. It was found that the amount of glucose was 1.6 times higher than the amount of fructose. A very weak relationship between glucose or fructose and sucrose ($R^2= 0.43$ and 0.38 , respectively) was found. This corresponds well to findings in other studies (Amrein et al. 2003; Knutsen et al. 2009) The results suggest that glucose can be used to estimate the total reducing sugar content. Sucrose, on the other hand exhibit low correlation with the reducing sugars. Regarding process control in processing industries, the sucrose level is of less importance, since it is not contributing to the Maillard reaction (Amrein et al. 2003).

The use of potato juice as a sampling method may introduce a higher level of uncertainty. As can be seen from Figure 2 it seems that the relationship between the glucose content in the extracted potato juice does not correspond fully with the glucose content in the actual potato tuber. Presently the authors would conclude that the use of potato juice for sugar determination should be subjected to individual

evaluation by the industry using it. Further testing should be carried out to firmly establish whether the juice can be used for reliable quantification of sugars in potato tubers, and if this relationship is truly cultivar independent.

4. Conclusion

The LifeScan and Bayer monitors both gave squared correlation coefficients of 0.92, when compared to HPAEC analysis. The error estimations were 0.54 and 1.12 mg mL⁻¹ for the LifeScan and Bayer monitor, respectively. The RQFlex showed a higher squared correlation coefficient ($R^2=0.96$) and a lower error estimation (0.24 mg mL⁻¹) than the two PBGMs. Although all three instruments are capable of determining the glucose content in potato juice, the RQFlex is more accurate than the two PBGMs, but also slightly more labor intensive. The current study also suggests that analysis of juice itself may induce uncertainties to the measurement. A squared correlation coefficient of 0.91 was found for the relationship between glucose in juice samples and lyophilized samples. Further work may be focused on identifying and quantifying the limitation of the potato juice as part of a suited and representative sampling method to be combined with any rapid or simplified analytical system. However, the current study shows that PBGMs are able to rapidly and easily determine the glucose in potato juice, with accuracy sufficient for industrial use.

Acknowledgement: The authors greatly appreciate the technical assistance from Hanne Zobel. Financial support from the Norwegian Research Levy on Agricultural Products in projects NFR-199421/O99, FFL-225352 and FFL-225347/F40 is greatly acknowledged.

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TABLES:

Table 1: The number of samples collected from each batch, and the cultivars used.

Cultivars:	No. of samples	
	batch	batch
	1	2
Saturna	25	80
Lady Claire	-	40
Bruse	-	40
Peik	-	40
Asterix	25	-

Table 2: Advantages and drawbacks with the two PBGMs and the RQFlex system for glucose determination

Instrument:	Advantage:	Drawback:
Bayer Contour	+Fast and easy to use +Cheap	-Underestimates glucose values -High RMSEE value -May be affected by other potato constituents or oxygen
LifeScan OneTouch	+Fast and easy to use +Cheap	-Moderate RMSEE values -May be affected by other potato constituents or oxygen
RQFlex	+Cheap +Accurate (low RMSEE-value) +Larger measuring range (due to the dilution step)	- Require more sample preparation than the PBGMS - More time consuming

FIGURES:

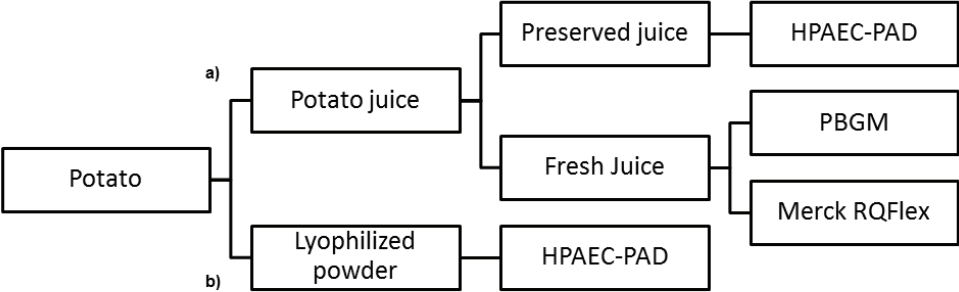


Fig 1 Schematic overview of the sampling methods used in the current study. Not all analyses were applied to all samples

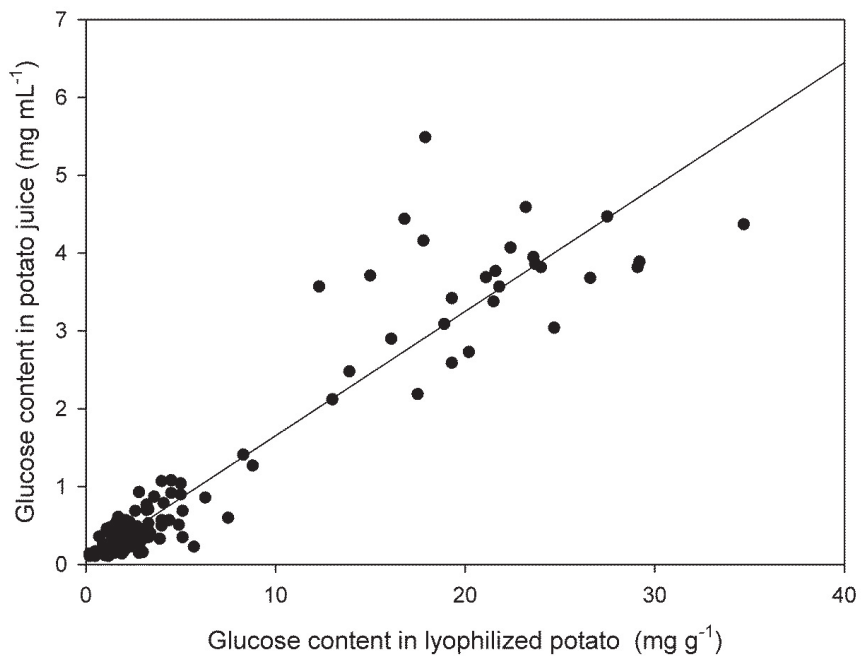


Fig 2 The relationship ($R^2=0.91$) between the glucose content in potato juice (mg mL^{-1}) and lyophilized potato (mg g^{-1}), both determined by HPEAC-PAD. The linear regression line is shown (solid line)

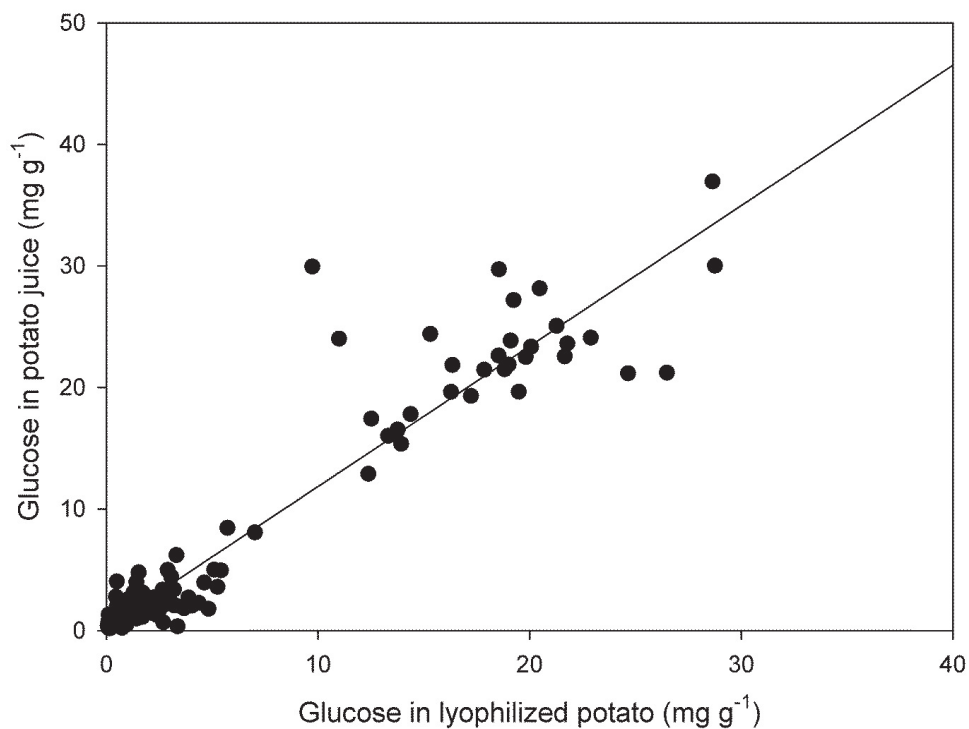


Fig 3 The relationship ($R^2=0.91$) between the glucose content in the potato juice (mg g^{-1}) and lyophilized potato (mg g^{-1}), both determined by HPEAC-PAD. Glucose values in juice samples have been corrected for dry matter content. The linear regression line is shown (solid line)

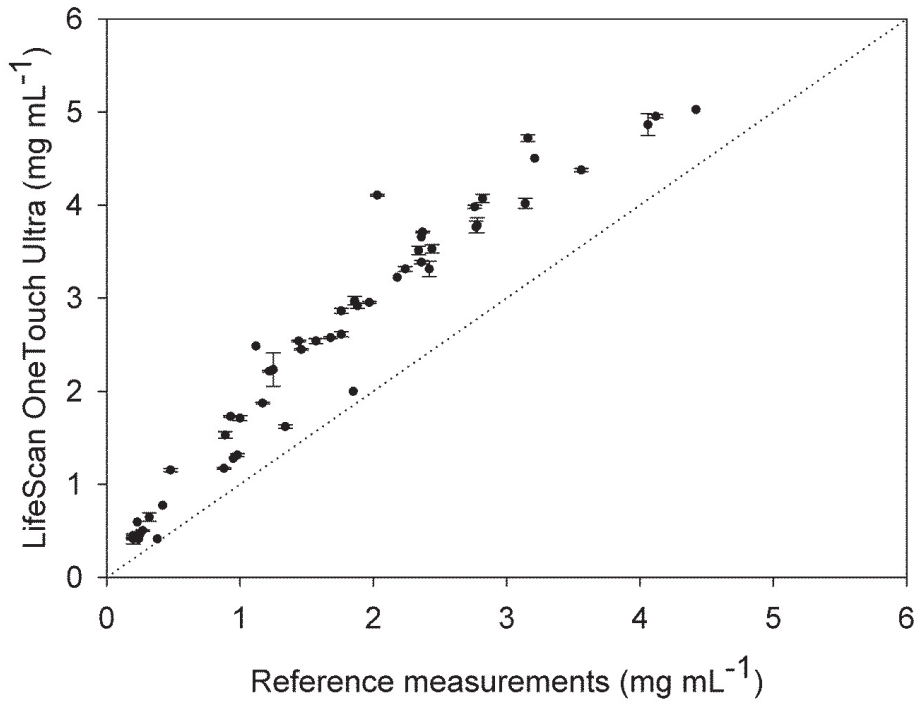


Fig 4 The performance ($R^2=0.92$, $RMSEE = 0.54 \text{ mg mL}^{-1}$) of the LifeScan OneTouch predicting glucose content in potato juice (mg mL^{-1}). The middle dots plotted are average values of the replicates, and the error bars represent individual replicate values. The dotted line is the target line

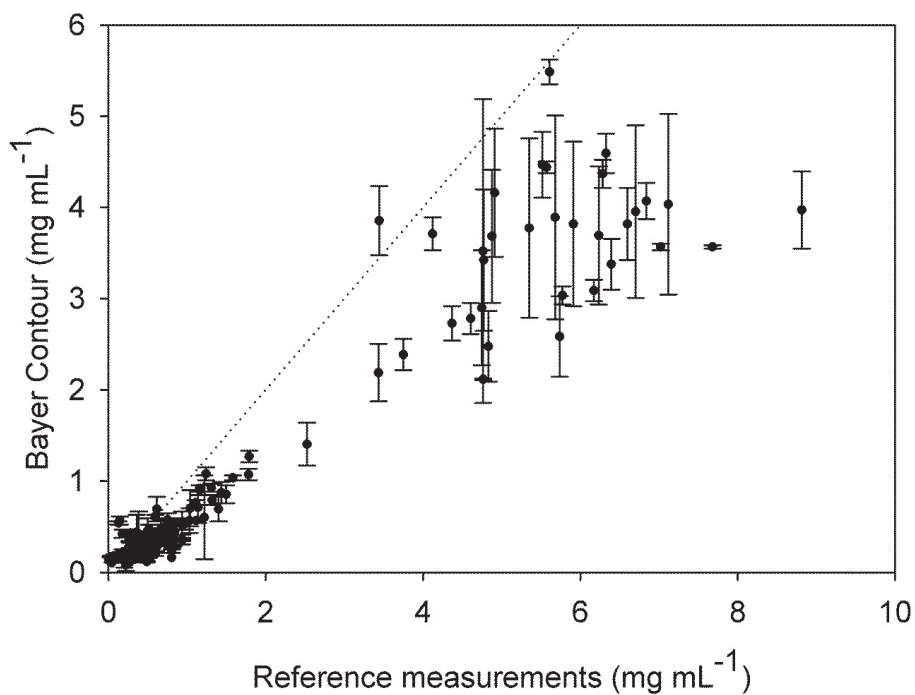


Fig 5 The performance ($R^2=0.92$, $RMSEE = 1.12 \text{ mg mL}^{-1}$) of the Bayer Contour predicting glucose in potato juice (mg mL^{-1}). The middle dots plotted are average values of the replicates, and the error bars represent individual replicate values. The dotted line is the target line

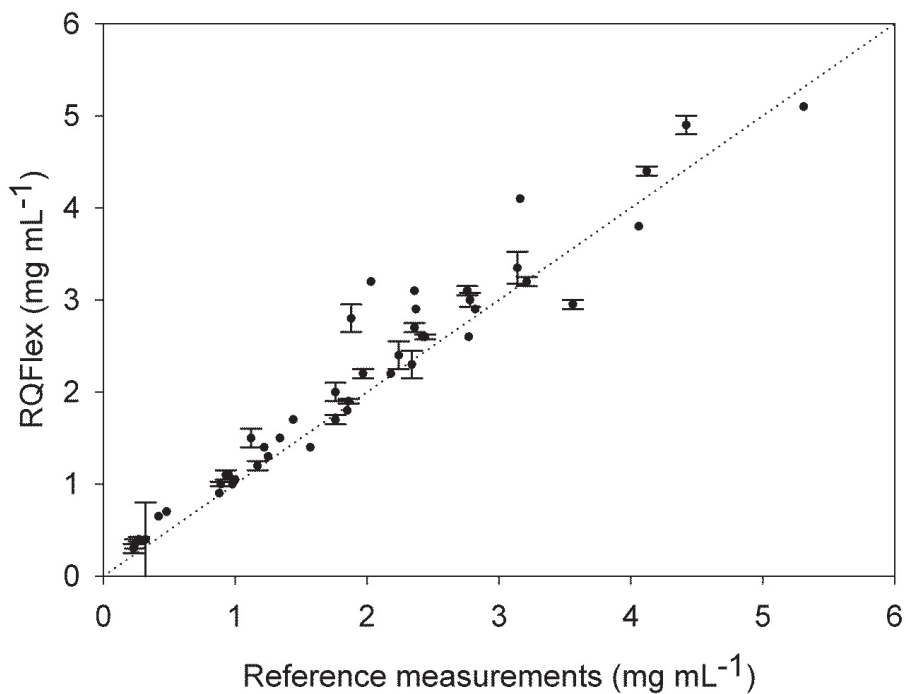


Fig 6 The performance ($R^2=0.96$ RMSEE = 0.24 mg mL^{-1}) of the RQFlex system predicting glucose in potato juice (mg mL^{-1}). The middle dots plotted are average values of the replicates, and the error bars represent individual replicate values. The dotted line is the target line

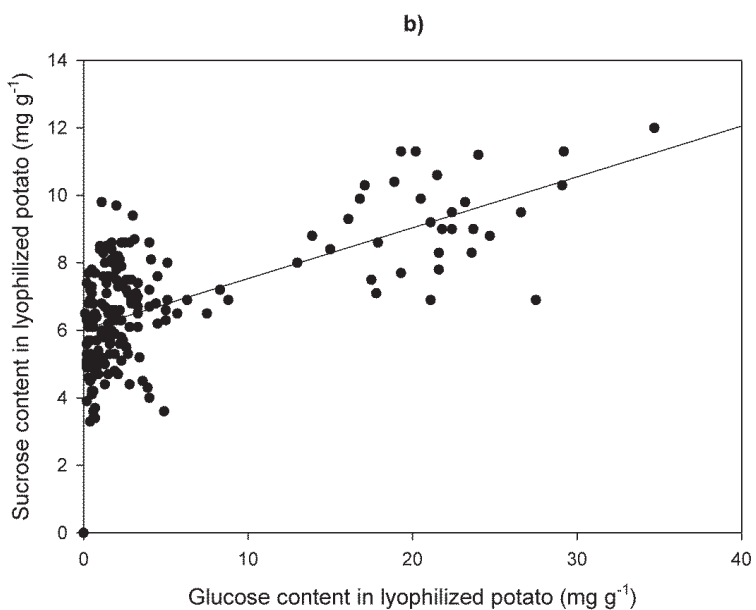
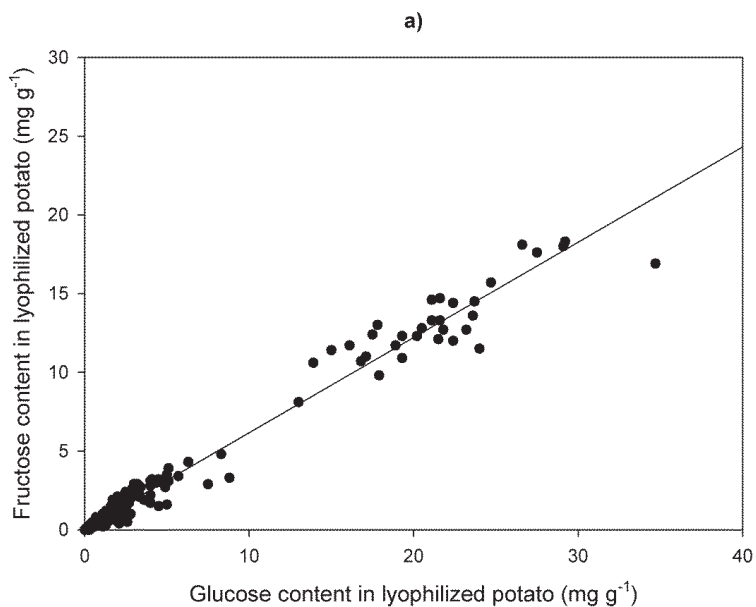


Fig 7 The interrelation between the major sugars found in lyophilized potato powder. **a)** Fructose and glucose ($R^2=0.93$) and **b)** sucrose and glucose ($R^2= 0.42$). The linear regression line (solid line) is shown in both a and b

Paper IV

Factors affecting the material properties of lompe dough

Trygve Helgerud,^{a,b} Reidar B. Schüller,^b Elling O. Rukke,^b Nils K. Afseth^a Svein H. Knutsen^a and Simon Ballance^a

^{a)} Nofima AS - Norwegian Institute of Food, Fisheries and Aquaculture Research, Pb 210, N-1431 Ås

^{b)} Norwegian University of Life Sciences, Department of Chemistry Biotechnology & Food Sciences, N-1432 Ås

Corresponding author: Trygve Helgerud, Nofima AS, Pb 210, N-1431 ÅS, Norway.
Tel. +47 64970100, E-mail: trygve.helgerud@nofima.no,

Abstract

Lompe is a potato based soft flat bread originating in Scandinavia. It is made from dough comprising cooked and cold-stored mashed potatoes subsequently mixed with cereal flour. Poor understanding of how the raw material affects the dough limits the process optimization and increases production cost. This study investigates how the properties of the potato raw material affect the rheological properties of the dough. Three factors were identified as the main contributors to dough quality and dough stability. 1) Water content of the potato raw material is the main contributor to the overall gel strength of the dough, since no extra water is added. Lower water content, gave higher gel strength. 2) Retrogradation of the potato raw material was found to have great impact on the mixing properties of the dough. Unless the potatoes were sufficiently retrograded the dough would collapse and become sticky before mixing cycle was finished. Different retrogradation rates for the two cultivars studied, were found (24 hours for of cold storage needed for an old Scandinavian cultivar and 48 hours for cv. Saturna). 3) Endogenous enzymes in the flour was found to cause dough break down after mixing, hence being instable over time (<1 hour after mixing).

Keywords: Lompe, potato, Saturna, Dough properties, retrogradation, α -amylase of flour.

1. Introduction

Cooked, cold-stored and mashed potato can be kneaded together, usually with wheat flour, to form dough. Moisture present in the potatoes is usually sufficient to form dough, but water and/or potato flakes can be added if necessary. From similar dough, potato based unleavened bakery products are made, such as Italian potato based pasta (Gnocchi) (L. Alessandrini, Balestra, Romani, Rocculi, & Dalla Rosa, 2010). Another product is a type of unleavened potato-cereal flour soft flat bread known in Norway as lompe or lefse (hereby referred to as lompe). It probably originated in Scandinavia, in the 19th century (Burton, 1989). It is made by rolling potato-cereal flour dough into thin sheets (ca. 2 mm), cutting it into a flat oval or round shape, before it is baked by dry convection heat on a griddle (ca. 15s each side at 225°C).

Lompe is often used as a wrap in a variety of sweet or savory dishes. The most common of which in Norway is as a wrap for various cooked sausages/hot dogs. Even though lompe can be found in a few countries outside Scandinavia such as USA, to the best of our knowledge neither scientific research on lompe nor on the material properties of dough made from a combination of cereal flour and cold mashed potato has been published. Lack of knowledge makes commercial bakeries producing lompe unable to fully optimize their production, which leads to higher waste volumes and increased production costs. One major challenge encountered during processing is related to deterioration in dough stability and subsequent change in consistency at rest, which increase production difficulties.

Although cereal flour quality is well understood and closely monitored during industrial baking, potato quality in lompe baking, and its role in dough formation is much less understood. The material properties of boiled potatoes have been extensively studied (Anette Kistrup Thybo & Martens, 1999; van Marle et al., 1997), and studies have been conducted on dough made from rice starch mixed with potato starch in concentrations up to 50% (Sandhu, Kaur, & Mukesh, 2010). This study concluded that higher noodle quality could be achieved using potato starch, judging by parameters such as cooking loss and slipperiness of the product. Addition of potato starch to flour-water mixtures has also been studied to some extent (Sarker et al., 2008), showing that raw potato starch may help stabilize wheat flour dough. Material properties of mashed potatoes, with or without additives such as olive oil, have also been studied extensively (Alvarez & Canet, 1999; Moledina, Haydar,

Ooraikul, & Hadziyev, 1981; Peksa, Apeland, Gronnerod, & Magnus, 2002). Many of the above mentioned potato based products also contain egg, milk or other types of emulsifiers, none of which is found in lompe.

One other unique feature found in lompe production is the cold-storage interval between cooking the potatoes and the dough production. It is common knowledge amongst bakers that the potatoes need cold-storage for a certain period before they can be used to make workable/useable lompe dough. The need for cold-storage of the cooked potatoes before baking implies that the potato starch must be at least partly, retrograded before they can be used to make lompe dough. Retrogradation is therefore believed to be a key factor affecting the lompe dough formation, hence introducing potato starch retrogradation as a new aspect not found in conventional cereal dough preparation. Numerous investigations have been conducted upon the process of retrogradation, yet it is not fully understood (Chaudhary & Adhikari, 2010; Ottenhof & Farhat, 2004).

It is also known that the tuber quality will vary between cultivars, seasons and during storage (Pinhero, Coffin, & Rickey, 2009). This enhances and complicates the role played by the potato component as a functional ingredient. Knowledge about the influence of potato properties on dough formation is even more important, since it is easier to determine the potato properties and adjust the processing parameters, rather than to modify the potato raw materials. The goal of this study is to identify and investigate factors suspected to affect the properties of potato based dough for industrial lompe production. This includes identifying the reasons behind the limited life-time (<1h) for viable use in production of resting dough at room temperature. It is also known by bakers that different potato cultivars often used in commercial lompe production behave differently during dough formation and subsequent processing. The main dough related parameters investigated were: 1) the effect of cooked and cold-stored mashed potato water content; 2) the effect of the degree of potato starch retrogradation and; 3) the effect of endogenous enzymes introduced when flour is mixed with mashed potato. In addition microbiological activity and general potato starch properties (amount and amylose/amylopectin ratio) were also taken into consideration.

2. Materials and Methods

2.1 *Potato raw material*

Ready to use, freshly made, peel-free, potato mash from two different cultivars (cv.) Saturna and an undisclosed Scandinavian cultivar (hereby denoted Scandinavian cultivar) were acquired from a commercial lompe bakery. Prior to mashing the potato tubers had been cooked in boiling water, followed by cooling and cold-storage (4°C), for 48 hours for cv. Saturna and 24 hours for the Scandinavian cultivar, unless otherwise specified.

2.2 *Dough preparation and in-situ dough mixing analysis*

The dough consisted on fresh weight basis (g/100g) of 79.6 g mashed potato, 20 g wheat-rye flour mixture (8:1 ratio) and 0.4 g sodium chloride. Flour of standard quality (Falling number was approximately 250 for the wheat flour and 130 for the rye flour) were used. The flour-potato ratio was kept constant throughout the experiments. Mixing was performed with a DoughLab instrument (Newport Scientific Inc., USA) using DoughLab software (ver.1.3.0.185). A 300 gram capacity mixing bowl was used, except for experiments involving silver nitrate which were scaled down due to the toxicity of silver nitrate and performed with a 50 g capacity bowl. The potato mash was premixed for 2 minutes at 64 revolutions per minute (rpm). The dry ingredients were added and the blend mixed at 64 rpm for 12 minutes before final mixing at 120 rpm for 40 seconds. This formed the dough. Data for the last 40 seconds are not shown in the figures. The torque needed to rotate the mixing blades was recorded in Newton meter (Nm) as a function of mixing time and is hereby referred to as mixing torque. The mixing bowl of the DoughLab was maintained at 15°C. When specified, the following were added along with the flour; antibiotics (*Penecillin G* and *Kamamycin*, Sigma-Aldrich, USA) at 0.01 mg/100 g_{dough} to inhibit bacterial growth; and/or silver nitrate (AgNO₃, Sigma-Aldrich (USA)) at 3.5 mg/g_{dough} (Meredith, 1970) as a general enzyme inhibitor.

2.3 *Rheological analysis of dough at rest*

After dough preparation, a sample was quickly (5 min) transferred to the measuring system of the rheometer (Physica MCR301, Anton-Paar GmbH, Austria). The rheometer was controlled by Rheoplus software (Ver.3.40, Anton-Paar GmbH, Austria). Two standardized geometries were utilized during the experiments: 1) 25

mm parallel plate geometry (Anton-Paar, PP25-TG, with P-PTD200/62/TG lower plate); or 2) bob-cup (Anton-Paar CC27 with C-PTD200 temperature control for the cup). When using the parallel plate geometry a guard ring consisting of a layer rapeseed oil was added on top to limit evaporation effects along the perimeter of the plate. When using parallel plate measuring geometry the gap was set to 1 mm. Parallel plate gave easier sample application, but noisier measurements than with the bob-cup. The use of Bob-cup was more time consuming and the dough was subjected to higher stress when the bob was lowered into the cup containing the sample. Nevertheless, when using two standardized (i.e. in accordance with the ISO and DIN standards) geometries the absolute G' and G'' values are comparable after the sample has come to rest (i.e. after the first few minutes) (Mezger, 2011). All experiments were performed at 15°C, unless otherwise stated. Conversion of physically measured parameters to loss (G'') and storage modulus (G') were performed automatically in the software. Amplitude sweeps were performed by varying the amplitude (strain, γ) from 0.01 to 100% with fixed angular frequency (ω , 10 rad/s). Direct strain oscillation (DSO) was used. It was assumed that there were no non-measurement related changes in the dough during the short time span of the amplitude sweep. The plateau of the Linear Viscoelastic Range (LVE-Range) was calculated in the Rheoplus software and the criterion for strain limit (γ_L) was set to the point of 1% decrease in G' -value. This strain limit is commonly used for gel like samples (Mezger, 2011). Time dependent experiments were therefore performed with strain ($\gamma=0.1\%$) within the LVE-range at fixed frequency (10 rad/s). Each sample was measured for up to 12 hours (unless otherwise specified). No resting time was applied after insertion into the measuring system. Hence, differences in the first 15 minutes of each measurement may be seen. These variations occur when the samples are equilibrating to the newly applied measuring geometry. G' for time zero ($G'(t_0)$) was therefore found by linear backwards extrapolation of the G' -values between 10 and 60 minutes.

2.4 Other physical and chemical analysis

2.4.1 Water content

Water content (g/100g weight of total mass) of the potato mash was determined in triplicates after oven-drying at 105°C for 48 hours.

2.4.2 Total Starch and amylose/amylopectin ratio in cv. Saturna and the Scandinavian cultivar

Raw potato tubers (20 kg) were cleaned in tap water and peeled in an abrasive peeler (2 min) and sliced into cubes (1 cm³). A sub-sample (1.4 kg) was immediately frozen in liquid nitrogen before it was lyophilized (Yglesias & Jackson, 2005). Dry samples were ground on a centrifugal mill (0.5 m² screen), (Retch, Germany). Enzymatic test kits, (Meagzyme Ltd., Ireland), were used for total starch (Kit no. K-TSTA 07/11. Mono- and disaccharides were removed by ethanol washing, followed by starch degradation with thermo stable α -amylase and amyloglucosidase. Samples for amylose/amylopectin ratio determination (Kit No. K-AMYL 07/11) were first washed with ethanol to remove mono- and disaccharides. The samples were then dissolved in dimethyl sulfoxide and placed in boiling water, with subsequent precipitation of amylopectin by addition of *Concanavalin A* (supplied in the kit).

2.4.3 Enthalpy of starch retrogradation in cooked cold-stored potato

Samples were prepared by boiling potato tubers in vacuum bags and placed at cold-storage (4°C). After a given number of hours, specified in the results section 3.4, the samples were coarsely ground by a meat grinder (Kenwood Major, Kenwood, England) and frozen in liquid nitrogen and lyophilized (Yglesias & Jackson, 2005). A sample of potato powder (30 mg) was placed in a closed aluminum pan along with water (90 μ L) and left overnight at room temperature to swell before differential scanning calorimetry (DSC), (DSC823^e, Mettler-Toledo, Switzerland). The DSC temperature program was as follows: start temperature 10°C, a temperature gradient of 5°C/min for 22 minutes with end temperature of 120°C. A silicone filled pan was used as reference. Calibration was performed with an indium reference sample (6.72 mg). This method measures the melting endotherms of the amylose and amylopectin crystals formed during retrogradation. Integration of the heat endotherms was performed in the region from 45-75°C.

2.4.4 Statistical analysis

The replicate variance between samples subjected to chemical analysis, were determined by a student's T-test (MiniTab 16, MiniTab Inc, USA). Linear regression was performed in SigmaPlot (ver. 11.0, Systat Software LTD, USA)

3. Results and Discussion

3.1 Formation/Preparation of lompe dough

The overall shapes of the mixing torque curves for lompe dough formation are similar to those of traditional bread dough (Mani, Eliasson, Lindahl, & Tragardh, 1992; Migliori & Correr, 2013). The first 2 minutes of dough formation for cv. Saturna, and first 5 minutes for the Scandinavian cultivar, consists of a phase where dry flour is incorporated with the moist potato mash (Figure 1). In this phase the mixing torque shows large variations due to the initial formation of separate potato-flour clumps. At 2.5-3 min for cv. Saturna (water content: 78.0 g/100g_{potato}), and 5-6 min for the Scandinavian cultivar (water content: 78.7 g/100g_{potato}), mechanically stable dough is formed as indicated by a stable plateau in the mixing curves (Figure 1), which continues until mixing was completed at 12 minutes. If mixing were continued for more than 15-20 minutes the gluten network started to break down and the dough became stickier. This was observed as a sharp drop in mixing torque curves (data not shown). This breakdown is similarly observed in overmixed bread dough and has been ascribed to either physical or chemical rupture of linkages within the gel/dough structure (Meredith & Bushuk, 1962; Okada, Negishi, & Nagao, 1987).

While in bread baking one can produce dough to specified, and desired water content, for potato based doughs such precise control of the water content is not possible. Differences in water content between two lompe doughs explain a major part of the variation in mixing torques at plateau. A general decrease (linear fit $R^2=0.46$) in mixing torque at plateau is shown in Figure 2 for lompe dough, as a function of increasing water content. Similar trends are also observed for bread doughs, but with less variation (Farahnaky & Hill, 2007). For lompe dough made from potato mash with water content of 75-78 g/100g_{potato} a mixing torque at plateau of more than of 3 Nm was required. Lompe dough made with potatoes with a higher water content (>78 g/100g_{potato}) gave mainly mixing torques below 2.5 Nm. Variation in lompe dough water content are presently unavoidable when using mashed potato. During production, dough consistency is controlled during preparation by hand feel of an experienced baker who can make adjustments by either adding water or potato flakes.

Another observation made was that the Scandinavian cultivar gave more noisy mixing torque curves (Figure 1) than when mixing dough with cv. Saturna. Upon visual inspection of the cooked cold-stored and mashed potato from the

Scandinavian cultivar gave a visually drier and more crumbly texture than cv. Saturna. Perceived texture difference may be attributed to cell wall structure and how the potato tissue reacts upon cooking, peeling and mashing (A. K. Thybo, Martens, & Lyshede, 1998) The perceived crumbliness of the potato mash may, hence, explain some of the noise seen in the mixing torque curve in Figure 3a and 3c. This was however, not investigated further.

3.2 Material properties of lompe dough

Amplitude sweeps of lompe doughs revealed, as expected, that it behaves as a viscoelastic solid at rest (<1% strain), with G' greater than G'' (Figure 4). Amplitude sweeps also showed that typical lompe dough, independent of the cultivar used, and water content of the potato mashed used, had a linear viscoelastic range (LVE) that spanned to a strain value from 0.01% to approximately 1% (Figure 4). The yield point ($G'=G''$) of the dough was calculated to between 20 and 30% strain. Very little difference in G' and G'' values between cv. Saturna and the Scandinavian cultivar was found (Figure 4). Accordingly the water contents of the potato mash used to make both these doughs are similar, 78.0 and 78.7 g/100g_{potato} for cv. Saturna and the Scandinavian cultivar, respectively. Like with differences in mixing torque plateau values, it can be seen from Figure 2 that small differences in water content of the mash, and hence the lompe dough itself, have a major impact on the relative gel strength of the dough at rest. Lompe dough made from potatoes with the highest water content (78 g/100g) were generally softer ($G'(t_0)=22-24$ kPa) than those made from potatoes with slightly lower water content (75-76 g/100g_{potato}; $G'(t_0)=34-36$ kPa) shown in Figure 2. Again, bread doughs, like most polymer gels, follow the same trend (Navickis, Anderson, Bagley, & Jasberg, 1982).

3.3 Gel stability of resting dough (15min-12hrs)

In lompe dough without any additives, G' decreased as a function of resting time (Figure 3b). This decrease occurred at a constant rate of 250-370 Pa/min (Figure 3b (1)-(3)), independent of the potato cultivar used in the dough. Decrease in G' for up to two and a half hours can be seen in Figure 5a, but the constant rate of decrease in G' continued for at least 12 hours (Data not shown). In Figure 5b the $\tan\delta$ values corresponding to the G' values shown in Figure 5a are shown. A small increase in $\tan\delta$ indicates that the internal structure of the dough, is slowly weakened or broken

down when it is at rest. An increase can be seen for curve (1-2) and (4) in Figure 5b. Curve (3) in Figure 5b does not increase, but stabilizes at $\tan\delta=0.22$. An explanation for this was not found. With time, the dough is also perceived to become more soft and sticky. It is known that the textural properties of regular wheat flour dough may change during the first hour of rest and thereafter is considered stable (Letang, Piau, & Verdier, 1999). As can be seen from Figure 5a the decrease in G' was linear. This suggests that the decrease is caused by a phenomenon not found in regular wheat flour dough.

The gel-strength of freshly prepared dough as a function of temperature (4-40°C, Figure 3b) was nearly linearly related ($R^2=0.96$). An increase in temperature causes a decrease in gel-strength, visually observed as increase in softness. This is typical for flour based dough and many gels between 4-40°C (Farahnaky & Hill, 2007). Lompe dough measured at 4°C (Figure 3b) showed little change in G' for the first 50 minutes, and only a minimal decline (17 Pa/min) for longer measurements (up to 2.5 h, data not shown). The rate of decrease in G' showed a near linear relationship with temperature up to 40°C ($R^2=0.99$). At first the loss in G' was suspected to be caused by microbial growth/fermentation, as gas production and an off-odor was observed after 6-24 hours storage of the dough at room temperature. The addition of the antibiotics to the dough (Figure 3d (3) and (4)) did not influence its rheological behavior compared to the control (Figure 3b (1) and (2)). Corresponding mixing torque curves can be seen in Figure 3c curves (3) and (4), and seems to be unaffected by the addition of antibiotics. The other possible culprit for softening of resting lompe dough was the initiation of endogenous enzymatic activity from flour when exposed to moisture in the potato mash.

Lompe dough with added silver nitrate showed that the gel-strength of resting dough was higher than for control samples (no silver nitrate added, Figure 3d (1) and (2)), and over time the G' showed an increasing trend (approx. 1200 Pa/min). Also for samples measured for an extended period of time an increase in G' was seen (Figure 5a (5) and (6)). $\tan\delta$ values for samples containing silver nitrate showed a decreasing trend (Figure 5b (5) and (6)) which further supports that the gel strength increases as a function of time. The increase in gel strength (G') of resting dough may be caused by syneresis (water loss from polymer network), which is a slow process found in many gel based foodstuffs and is often seen as exudation of water, caused by a time dependent de-swelling of a gel (Draget et al., 2001). When a dough

sample was placed in a closed plastic container, an increase in surface moisture was observed, within the timespan of the rheological measurement. $\tan\delta$ for the doughs with silver nitrate (Figure 5b (5) and (6)) have a lower overall value, which may indicate that syneresis is taking place (Mezger, 2011). To some extent syneresis may also occur for dough without silver nitrate, but it is undetectable in the measurements due to the actual slow breakdown of the dough polymer network. Samples containing silver nitrate also have a higher $G'(t_0)$, 61.0 kPa for dough made with the Scandinavian cultivar and 40.6 kPa for samples made with cv. Saturna (Figure 3d and Figure 2). This is believed to be due to inhibition of enzyme activity during dough mixing. Mixing torque curves for doughs containing silver nitrate are shown in Figure 3c curves (3) and (4), but it should be noted that these samples were mixed in a small 50 grams bowl and hence, they required much lower overall mixing torques. Although, the mixing torque values cannot be directly compared to the measurements from the 300 grams bowl, the shape of the curve remains similar. Samples with silver nitrate, measured for prolonged times (exceeding 12 hours, data not shown) had stable, or even increasing, G' and G'' values. Hence, the activity of endogenous flour enzymes, such as α -amylase, can be seen as a feasible cause of the loss in gel strength observed during and after dough mixing. This also agrees with the observed temperature dependent stability. Enzymes can also be found in potatoes, but the activity is known to significantly decrease, or disappear, during cooking or heat treatment (Laura Alessandrini, Romani, Rocculi, Sjöholm, & Dalla Rosa, 2011).

3.4 Retrogradation of potato starch

Figure 6a shows mixing torque curves for dough made with cv. Saturna. As can be seen from Figure 6a lompe dough made from potatoes stored cold for 6 hours post-cook reaches mixing torques in the initial phase similar to dough made from potatoes subjected to 48 hours of cold-storage (Figure 1), but after about 2 minutes the torque decreases drastically. The final mixing torque is about one third of that required to mix dough made from potatoes cold-stored for 48 hours. Potatoes from cv. Saturna, cold-stored for 24 hours, do show a higher mixing torque peak at 2 minutes, but then declined and plateaued at the same values seen for the dough made with potatoes stored for 48 hours. However, perceived stickiness is much greater and the dough is

hence not suited for commercial baking. First when reaching 48 hours of cold-storage, mixing curves with stable plateau between 2 and 12 minutes are acquired. If the Scandinavian cultivar was cold-stored for 3 hours the dough (Figure 6b) yielded a mixing curve with a sharp decline after about 3 minutes. After a cold-storage period of 9 hours the Scandinavian cultivar was able to form dough that remained stable for 5-6 minutes before the decline in mixing torque started, but after 24 hours dough with a stable mixing torque curve could be produced. The same trend became evident when looking at the gel strength in dough (Figure 7a). Extrapolated $G'(t_0)$ for dough made with cv. Saturna was found to be 8.1 kPa, increasing to 17.6 kPa after 24 hours and reaching 26.4 kPa for dough made with potatoes cold-stored for 48 hours. Correspondingly, after 3 hours of cold-storage the Scandinavian cultivar gave dough with $G'(t_0)$ of 5.1 kPa. $G'(t_0)$ increased to 12.6 kPa for dough made with potatoes stored for 12 hours and 26.4 kPa after a storage interval of 24 hours. There seems to be a linear increase in $G'(t_0)$ as a function of the cold-storage interval (see linear fit in Figure 7a). Based on the data shown in Figure 6 and 7 it seems like the increase in $G'(t_0)$ may be cultivar dependent.

The most obvious time dependent starch property is retrogradation of potato starch. From the time dependent measurements it seems that the rate of retrogradation varies from the two cultivars (Figure 6 and Figure 7a). The rate of retrogradation will effectively influence the degree of retrogradation and hence the processing quality of the dough. A DSC investigation of the two potato cultivars did yield some indications about why the potato tubers require cold-storage after cooking. The retrogradation enthalpies (ΔH) obtained from the DSC are shown in Figure 7b, and indicate the energy needed to melt the amylopectin crystals (Abd Karim, Norziah, & Seow, 2000). Both the Scandinavian cultivar and cv. Saturna show retrogradation enthalpies in the region of 0-1 J/g_{lyophilized} within the first 4 hours of cold-storage. After 6 hours the enthalpies for the two cultivars seem to separate. Enthalpies for the Scandinavian cultivar seem to increase faster, and reach approximately 3 J/g_{lyophilized} after 24 hours. Samples from cv. Saturna needed 48 hours to reach the same level, and continues to increase up to 3.6 J/g_{lyophilized} after 72 hours. Samples of the Scandinavian cultivar do not show further increase in retrogradation enthalpies after 24 hours. The retrogradation enthalpies support the data shown in Figure 5, telling that cv. Saturna needs a cold-storage interval of 48

hours, and the Scandinavian cultivar an interval of less than 24 hours, before it is suited for baking.

Investigation of batch-samples of the two cultivars used in this study revealed that the starch content is quite similar (Table 1). The two cultivars contain from 61 to 68 g/100g_{dry weight} starch, but showing no significant difference ($p=0.24$). The same conclusion can be elucidated from the values for the amylose content, which ranges from 21 to 23 g/100g_{dry weight} of the total starch ($p=0.16$). This means that total starch content based on fresh weight will be greater. A difference in starch would, however, not explain the time dependent difference between the two potato cultivars. Since the amylose/amylopectin ratio of the two cultivars is similar, the DSC results indicate the amylopectin in the Scandinavian cultivar retrogrades faster than the amylopectin found in cv. Saturna. It is also known that amylose retrogrades much faster than amylopectin (Abd Karim, Teo, Norziah, & Seow, 2000). The starch content of the two potato varieties used in this study was investigated, but the difference in amylose-amylopectin ratio is not large enough to fully explain this phenomenon. It was not elucidated why the Scandinavian cultivar seems to retrograde faster. This requires further investigation. However, results obtained from DSC measurements correspond well with the mixing torque curves and G' values as a function of cold-storage time. Therefore, the rate in retrogradation may be a feasible explanation of the difference storage intervals required to form stable dough.

In regular bread dough, the starch granules act a gel filler in-between the gluten structure (Abang Zaidel, Chin, & Yusof, 2010; Peressini, Peighambardoust, Hamer, Sensidoni, & van der Goot, 2008). It is well known that alteration of gluten content will affect dough properties. However, in the current study the wheat flour amount, and hence also the gluten amount, were kept constant. It may also seem that the retrograded starch is playing a different role than the starch granules do in traditional bread baking. Models describing so called "mixed and filled gels" (Brownsey & Morris, 1988) may be a more appropriate description for lompe dough seen in the current study.

4. Conclusion

Water content and cold-storage interval of the potato tubers seem to be the two most important potato related factors affecting dough formation. Although these two factors do determine the dough properties, an exact definition of acceptable dough will depend on the type of processing equipment used, and the amount of mechanical and manual dough handling used during the lompe production. However, based on the results of the current study, some general features of dough suited for mechanical processing may be indicated; Mixing torque should reach a plateau after 2-4 minutes, and remain stable until mixing is finished at 12 minutes. The mixing torque for the mixing period between 4 and 12 minutes should be, at least, 2 Nm. This value will depend on the type of equipment used, but it may seem beneficial to seek $G'(t_0)$ values in excess of 20-25 kPa. The $G'(t_0)$ value is depending on the water content of the mashed potato used, and lower water content leads to higher $G'(t_0)$ values. It seems that water content above 79 g/100g_{potato} in the potato mash should be avoided, and potato tubers do require a certain interval of cold-storage after cooking. The required cold storage interval may be cultivar dependent, as the Scandinavian cultivar requires an interval of between 12 and 24 hours, and cv. Saturna needs approximately 48 hours. Improved short term dough stability was achieved by adding an enzyme inhibitor, leading to the belief that endogenous flour enzymes are degrading the dough polymers structure during and after dough mixing.

Acknowledgement:

The authors would like to thank Lars Axelsson and Ida Rud for their advice on the selection of antibiotics. We would also thank Norwegian Research Levy on Agricultural Products for financial support in projects "Optimal Potato" (NFR-199421/O99), FFL-225352 and FFL-225347/F40.

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TABLES:

Table 1: Chemical properties analysis of cv. Saturna and the Scandinavian cultivar, showing total starch, amylose/amylopectin ratio. Two batches from each cultivar were used, and the mean values are shown. (N=4 ± S.D.).

	Total starch (g/100g_{fresh weight})	Amylose (g/100g_{starch})
Scandinavian cultivar	68.2 ± 6.60	21.1 ± 1.5
Saturna	60.8 ± 5.90	22.8 ± 2.1

-

FIGURES

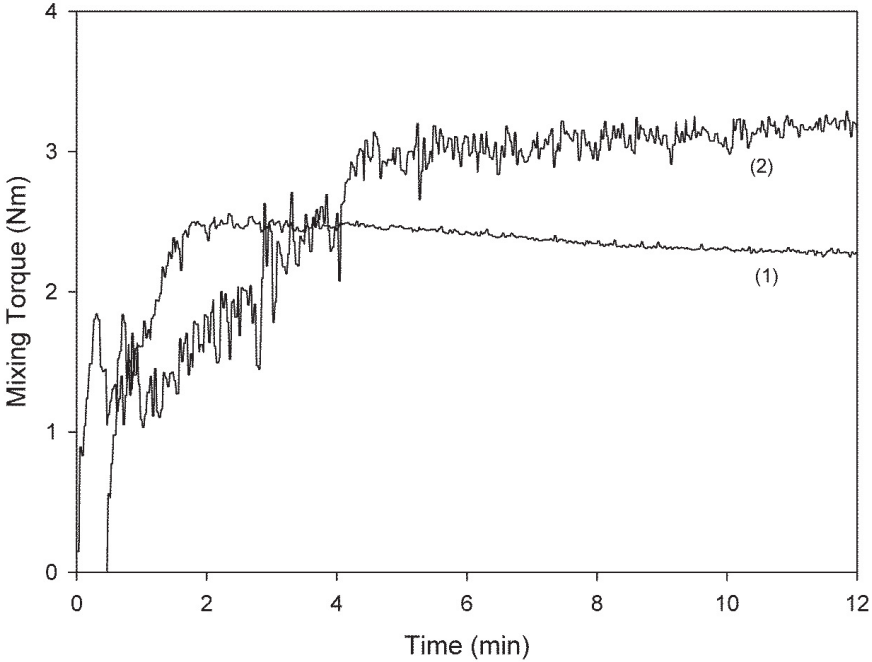


Figure 1: Mixing torque curves for dough formation with cv. Saturna (water content of 78.7 g/100g_{mashed potato}) (1) and the Scandinavian cultivar (water content 78.0 g/100g_{mashed potato}) (2).

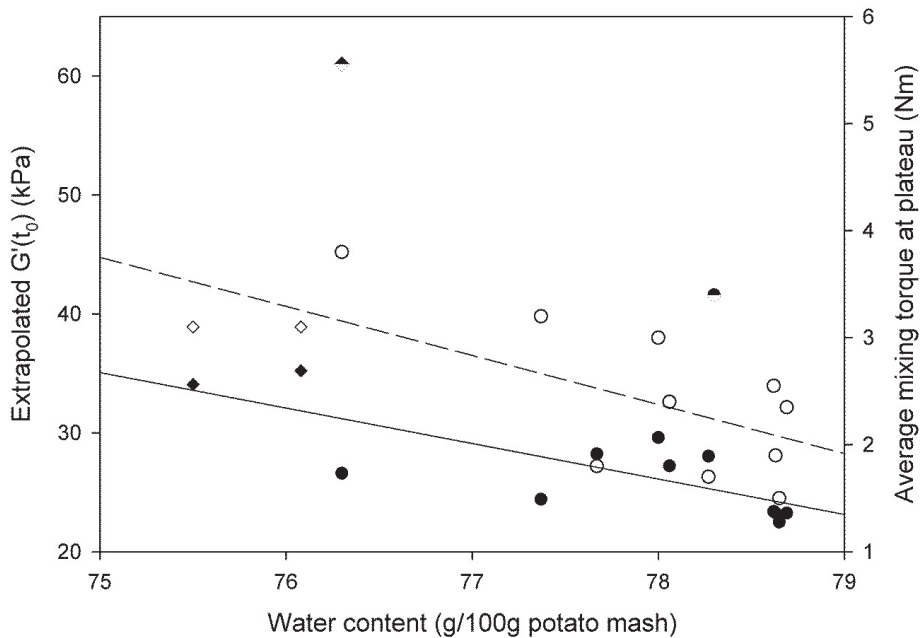


Figure 2: $G'(t_0)$ (closed circles for cv. Saturna and closed diamonds for the Scandinavian cultivar) and average mixing torque (Nm) values (open circles for cv. Saturna and open diamonds for the Scandinavian cultivar) for doughs made from potato mash of different water content. Linear regression for G' values are shown as a solid line and linear regression for mixing torque values are shown as dashed line. The two half-filled symbols (circles for cv. Saturna and diamonds for the Scandinavian cultivar) are representing $G'(t_0)$ value for dough with added silver nitrate (not included in the two regression models).

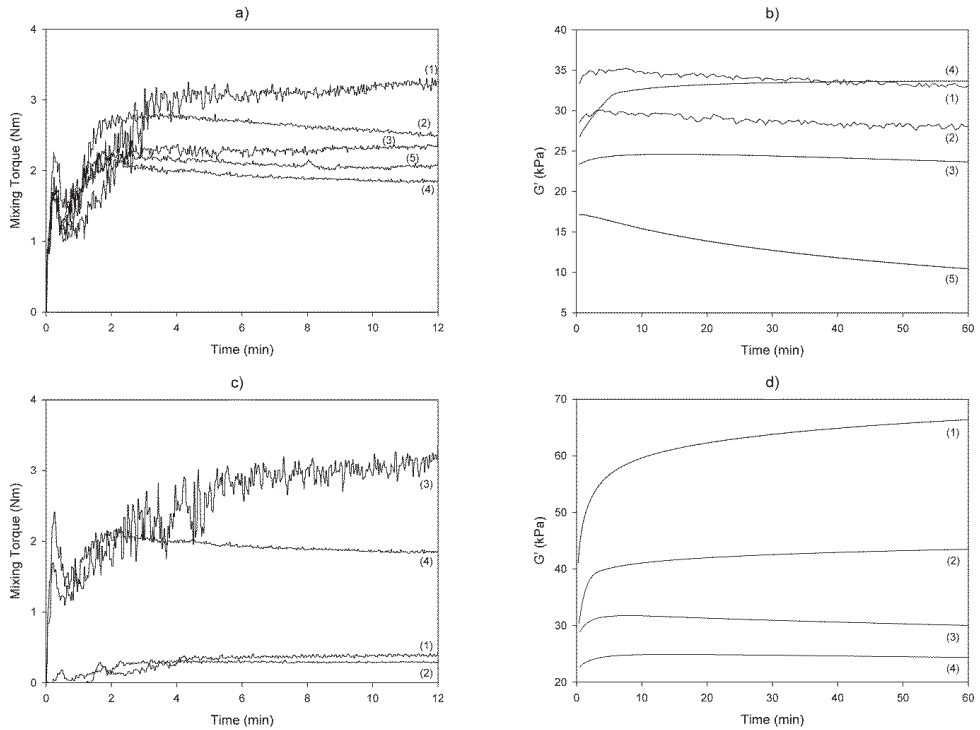


Figure 3: a) Mixing torque curves for lompe doughs prepared from cv. Saturna , with different water contents of 76 g/100g_{potato} (1), 78 g/100g_{potato} (2) and 79 g/100g_{potato} (3-5) b) Gel strength (G') in the samples in a). Sample (1-3) measured at 15 °C, sample (4) at 4°C and sample (5) at 40°C. c) Mixing curves for four doughs made with the Scandinavian cultivar (1-2) and cv. Saturna (3-4). Sample (1) and (2) are with added silver nitrate. Noted that dough (1) and (2) was mixed in a 50 g bowl at the DoughLab instrument, hence the low overall mixing torques d) The gel strength of the doughs given in c). Note: samples b) (1-2) was measured with parallel plate, all other samples were measured with bob-cup geometry.

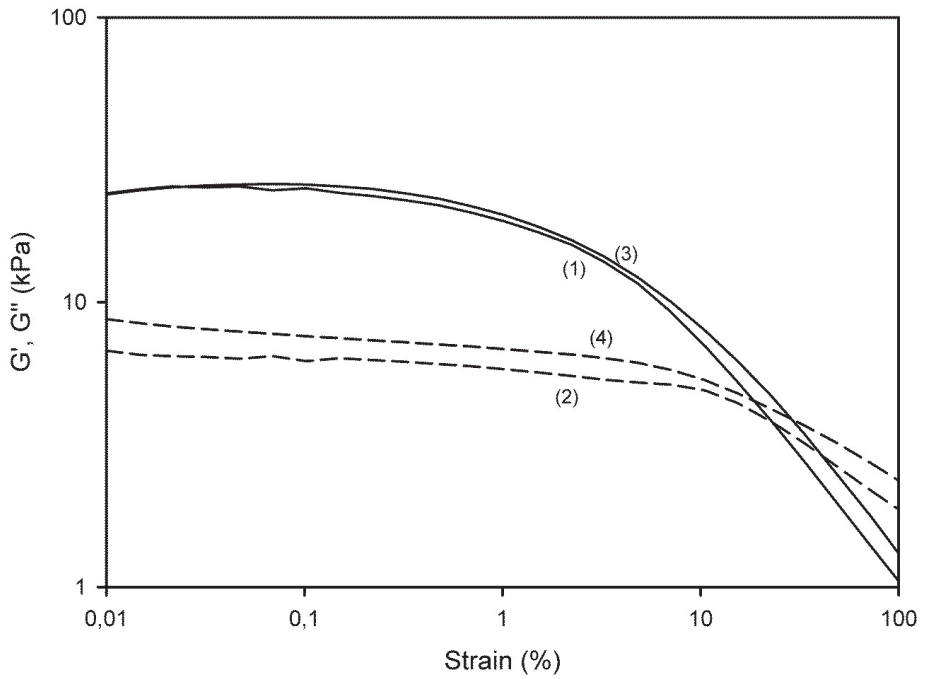


Figure 4: Amplitude sweep for dough made with cv. Saturna (G' (1) G'' (2)) and the Scandinavian cultivar (G' (3) and G'' (4)).

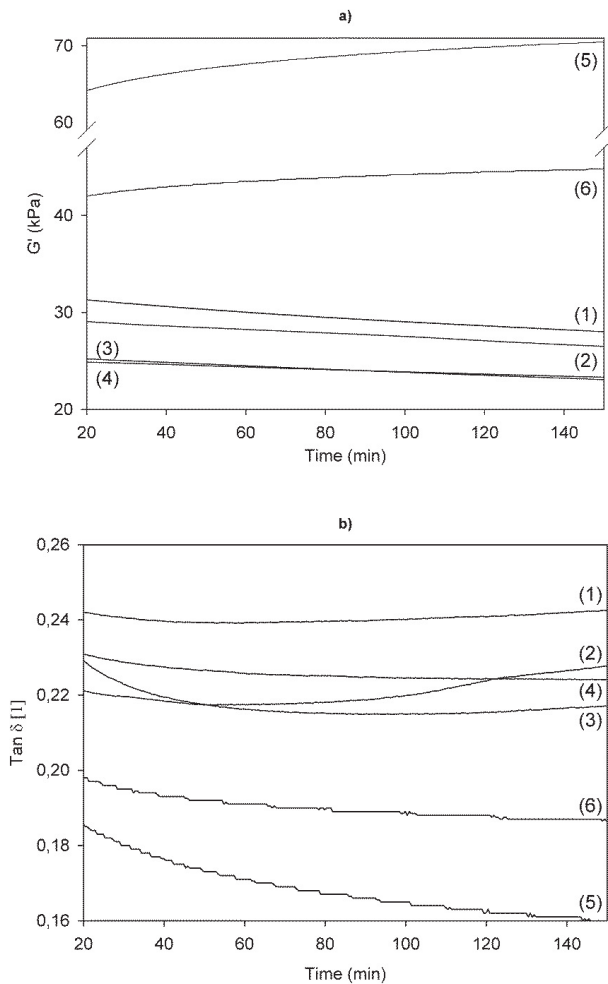


Figure 5: a) Doughs, made from the Scandinavian cultivar (1-3) and cv. Saturna (4-6). Sample number (5) and (6) contain silver nitrate. **b)** The $\tan \delta$ values for samples with corresponding numbers (1-6) in a). All measurements performed with bob-cup measuring geometry.

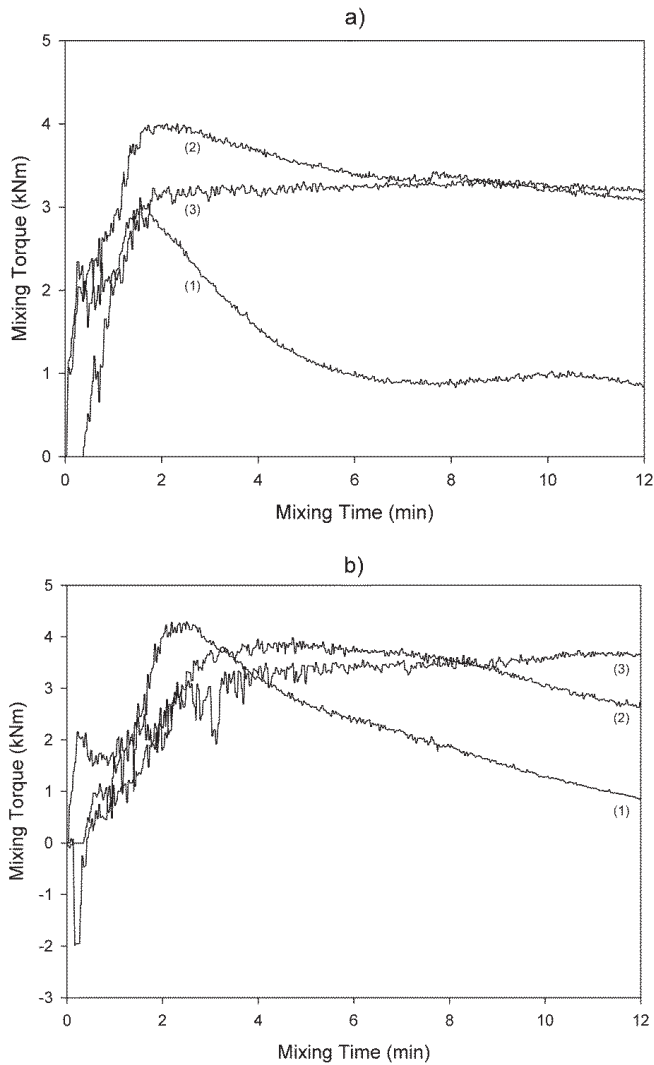


Figure 6: a) Mixing torque curves for doughs based on cv. Saturna. The potato raw material was cooked and cold-stored for 6 h (1), 24 h (2) and 48 h (3) before dough mixing. **b)** Mixing torque curves for doughs based on the Scandinavian cultivar. The potato raw material was cooked and cold-stored for 3 h (1), 9 h (2) and 24 h (3) before dough mixing.

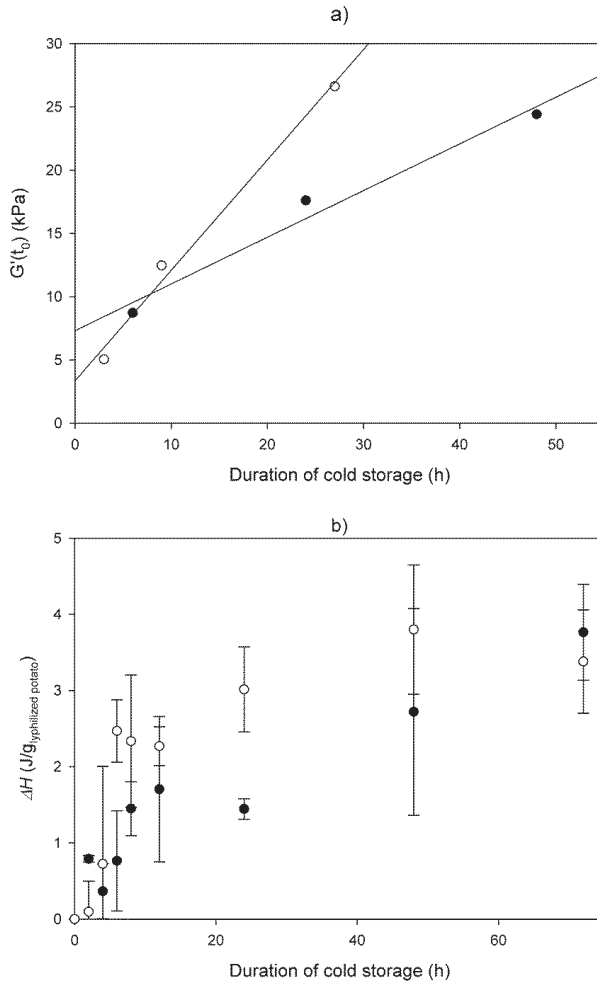


Figure 7: a) Extrapolated $G'(t_0)$ values for doughs made from potatoes from cv. Saturna (filled circles) and the Scandinavian cultivar (open circles) subjected to different durations of cold storage. **b)** Retrogradation enthalpy (ΔH , J/g_{lyophilized}) for the melting of retrograded amylose crystals in cooked and cold-stored potato samples of cv. Saturna (filled circles) and the Scandinavian cultivar (open circles).