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Investigation of drying behavior and color development of beef slices for development of non-invasive monitoring approaches

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„Mit dem Wissen wächst der Zweifel“

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List of publications

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Chapter 3: von Gersdorff, G.J.E, Porley, V., Retz, S.K., Hensel, O., Crichton, S.O.J., Sturm, B. (2017). Drying kinetics and quality parameters of dried beef (biltong) subjected to different pre-treatments, Drying Technology 36, 21-32. https://doi.org/10.1080/07373937.2017.1295979

Chapter 4: von Gersdorff, G.J.E.; Kirchner, S.K., Hensel, O.; Sturm, B. (2021). Impact of drying temperature and salt pre-treatments on drying behavior and instrumental color and investigations on spectral product monitoring during drying of beef slices. Meat Science.

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von Gersdorff, G.J.E., Crichton, S., Retz, S., Hensel, O., Sturm, B. (2016). Drying of fresh organic beef with different pre-treatments, Proceedings of the 20th International Drying Symposium (IDS 2016), Gifu, Japan, 07.08.-10.08. 2016, B-6-5

von Gersdorff, G.J.E., Crichton, S., Retz, S., Hensel, O., Sturm, B. (2017). Organic Beef Drying, 2nd Nordic Baltic Drying Conference, Hamburg, Germany, 07.-09. June 2017

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von Gersdorff, G.J.E., Kirchner, S., Hensel, O., Sturm, B. (2020). Development of sustainable drying strategies for beef drying. Organic World Congress 2020, 21.09.-27.09.2020, Rennes, France (accepted abstract)

Kurzfassung

Die Trocknung von Fleisch hat weltweit eine lange Tradition und industriell getrocknetes Fleisch wird zunehmend als fettarmes, eiweißreiches Lebensmittelprodukt geschätzt. Die Konvektionstrocknung ist in der industriellen Konservierung von Lebensmitteln weit verbreitet und muss hohe Prozesseffizienz und hohe Produktqualitäten bieten, welche widersprüchlich zueinander sein können. Ziel dieser Arbeit war es, den kumulativen Einfluss von Reifung, Kühllagerung, Marinieren und Trocknungstemperatur auf das Trocknungsverhalten und die Farbveränderung von Rindfleisch hinsichtlich der Entwicklung optimierter Trocknungsstrategien zu untersuchen. Weiterhin wurden Vorhersagealgorithmen, die Hyperspektral- und Labordaten miteinander verbinden, entwickelt, um zukünftig herkömmliche, invasive Messungen durch nicht-invasive Messsysteme zu ersetzen und über kontinuierliche Echtzeit-Messungen ein tieferes Verständnis für die Veränderungen im Produkt zu erlangen. Die Ergebnisse zeigten einen deutlichen Einfluss der Fleischreifung und des Einfrierens/Auftauens auf das Trocknungsverhalten, ebenso des Marinierens und der Lufttemperatur. Der Einfluss der verschiedenen Faktoren wirkte sich leicht auf die Farbveränderung im Laufe der Trocknung aus, führte aber zu keinen sichtbaren Unterschieden der Proben nach der Trocknung. Vorhersagemodelle von hoher Genauigkeit zur spektralen Messung von Feuchte und Farbe konnten erfolgreich entwickelt werden. Durch die Selektion von Wellenlängen wurden die Modelle jeweils vereinfacht. Die Robustheit konnte unter Einbeziehung der verwendeten Einflussfaktoren über den Versuchsverlauf deutlich gesteigert werden und abschließend sogar ein Gesamtmodell zur gleichzeitigen Vorhersage von Feuchte und Farbparametern und auf nur 10 Wellenlängen vereinfacht werden. Die Ergebnisse zeigen, dass produktspezifische Trocknungsstrategien zu effizienteren Prozessen führen können und die spektrale Produktüberwachung eine vielversprechende Methode ist, gerade hinsichtlich der zukünftigen Entwicklung von produktgesteuerten, „intelligenten Trocknungssystemen“.

Abstract

Meat drying has a long tradition worldwide and industrially dried meat is increasingly valued as a low-fat, high-protein food product. Convection drying is widely used in industrial food preservation and has to provide high process efficiency and high product qualities, which can be contradictory to each other. The aim of this work was to investigate the cumulative influence of ripening, cold storage, marinating and drying temperature on the drying behavior and color change of beef with respect to the development of optimized drying strategies. Furthermore, prediction algorithms combining hyperspectral and laboratory data were developed to replace conventional invasive measurements with non-invasive measurement systems in the future in order to gain a deeper understanding of the changes in the product via continuous real-time measurements. The results showed a clear influence of meat maturation and freezing/thawing on drying behavior, as well as marinating and air temperature. The influence of the various factors slightly affected the color change during drying, but did not result in any visible differences between the samples after drying. Prediction models of high accuracy for spectral measurement of moisture and color were successfully developed. By selecting wavelengths, the models could be simplified in each case. The robustness could be significantly increased by including the influencing factors used over the course of the experiment, and finally even an overall model for the simultaneous prediction of moisture and color parameters and simplified to only 10 wavelengths. The results show that product-specific drying strategies can lead to more efficient processes and that spectral product monitoring is a promising method, especially with regard to the future development of product-controlled "intelligent drying systems".

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List of Abbreviations

a*	redness
ANOVA	Analysis of Variance
B	Blind
b*	Yellowness
BA	Bland-Altman
°C	Celsius (degree)
DGE	Deutsche Gesellschaft für Ernährung
DR	Deming regression
ΔE	Color change
HSI	Hyperspectral imaging
Kg	Kilogram
L*	Lightness
LOA	Limits of agreement
MC	Moisture content
MR	Moisture ratio
MSI	Multispectral imaging
nm	Nanometer
PBR	Passing-Bablok regression
PLS	Partial least squares
R^2	Regression coefficient
RGB	Red Green Blue
RMSE	Root mean square error
S	Salt
S+V	Salt and vinegar
VNIR	Visual and near infrared

1 General Introduction

1.1 Background

Drying is one of the oldest preservation methods for food and helps to decrease post-harvest losses. Dehydration reduces the moisture content (MC) inside the product and lowers the amount of water available for microorganisms and enzymes and, thus, deterioration processes. Dried foods further have a lower bulk weight and volume than fresh produce, providing an opportunity to reduce transportation costs.

Food products can be classified in different groups in terms of drying, which are low hydrated (e.g. grains, oil seeds), highly hydrated (e.g. fruits and vegetables, meat, milk, herbs), intermediate products (e.g. pasta, sausages, sugar) and by-products for feed (e.g. whey, pulps, mash) (Bonazzi and Dumoulin, 2011). Industrial food drying is known to require high amounts of energy with to date often low energy efficiencies of on average 35 - 45 %, but it can also be as low as 10 % (Mujumdar, 2007). That results in unnecessarily long drying times and, therefore, high energy demands and affects the product quality negatively (Bonazzi and Dumoulin, 2011). However, the 21st century is characterized by a growing demand of processed foods and increased sustainability and, therefore, forcing the industry to meet the environmental and social concerns regarding energy demands, food losses and quality (Galanakis, 2018) and, thus, to provide more sustainability to this sector.

In recent years, dried meat products have become increasingly popular as a low-fat snack food. It is further a highly nutritional protein source, which is popular with sports enthusiasts, e.g. hikers and bikers, due to its low packaging size and weight. Therefore, there is a growing market with turnovers measured in billions of US dollars for meat snack products in Europe and the United States, with increasing forecasts (Grand View Research, 2017; Nielsen Research, 2017). Further, dried meat could present an additional source of income especially for small and medium scale farmers. However, concerning the high environmental impact of meat, the high amount of land used for livestock nutrition (FAO, 2006) and the often low efficiencies of drying processes mentioned above, efficient meat drying strategies are of a great importance in light of environmental and economic aspects and, therefore, high product and process efficiencies. Further, optimized drying strategies offer a high potential regarding minimal processing principles and the avoidance of critical food additives required for organic products at the level of organic associations, whose requirements go far beyond the EU regulation for organic products (BÖLN, 2003).

To optimize drying processes, it is imperative to understand the occurring changes inside the product. Commonly used conventional laboratory measurements provide a sound basis for data acquisition during the drying process to monitor the product. Simultaneously, these measurements require an interruption of the process and certain analysis can be destructive and time consuming, making a close monitoring by invasive measurements difficult to achieve. In contrast, non-invasive measurements offer a real-time alternative that enables continuous measurements. With a view to dynamic and individualized, so-called *smart drying* processes, non-invasive product monitoring can further provide in-process feedback control parameters. In view of the limitations in data acquisition and processing with respect to low computing times required for embedded systems, the techniques must be as simple as possible. Spectral applications present one method in terms of non-invasive data acquisition and hyperspectral imaging (HSI) provides a useful tool to identify the most important information (wavelengths) for the desired product parameters to develop simple (multispectral) systems (Kamruzzaman et al., 2016). Method comparisons can be utilized to evaluate the accuracy between laboratory and ‘new’ method measurements and further provide the parameters to calibrate the spectral measurement system.

The MC is a main parameter of a product to monitor the drying progress and behavior concerning the development of efficient drying strategies. Additionally, product color is of increased importance as it influences the appearance of meat products and is known to significantly influence the buying decision of consumers (Font-i-Furnols and Guerrero, 2014). Meat characteristics are influenced by several factors related to the handling post-slaughter or treatments prior to processing, which, besides the drying conditions, can be assumed to influence the characteristics during dehydration as well.

Against the background of the development of optimized drying strategies, the present study intends to investigate the impact of post-slaughter handling, seasoning, drying air temperature and cumulative effects on the drying behavior and color development of beef slices during convective drying. In the context of non-invasive real-time measurements and related future development of smart drying applications, the study further intends to investigate the applicability of spectral measurements of MC and color of beef slices during dehydration, providing innovative approaches in terms of increased model robustness and simplification of prediction models.

1.2 Research questions and objectives of the study

The optimization of drying processes requires the monitoring of the product to understand the changes occurring inside during processing. For this, laboratory measurements provide a sound basis, but are limited with regard to the provision of real-time information. HSI offers the potential to identify the relevant spectral information to be used for simplified non-invasive real-time monitoring applications, such as those, e.g. required for product related control of smart processing devices.

Although the impact of post-slaughter handling and seasoning on meat quality is well investigated, less is known about their impact on the drying behavior, the color development and the spectral response of beef slices during convective drying. Therefore, the aims of the study were to increase the scientific knowledge 1) on the drying behavior and the development of the color of beef slices during drying and 2) on spectral monitoring principles for MC and the international standardized CIELAB color pattern of beef during drying regarding a future development of non-invasive and real-time product monitoring systems.

Several research questions were addressed: (i) How do the post-slaughter and pre-drying handling of beef (maturation, freezing/thawing, seasoning), related cumulative effects and the drying temperature impact the drying behavior and the color development of beef slices during convective drying? (ii) Do hyperspectral data and laboratory measurements allow developing robust prediction models for non-invasive MC and color monitoring of beef slices throughout the drying process? Moreover, if yes, (iii) to which extend can the models be simplified to be utilized in real-time monitoring systems in beef drying processes?

Therefore, the specific objectives of the study include:

1. To investigate the drying behavior and color development of beef slices subjected to different pre-treatments, maturation stages and cold storage conditions (Chapter 3)
2. To evaluate the impact of drying temperature and salt pre-treatments on the drying behavior and color development and to investigate spectral monitoring of beef slices during drying (Chapter 4)
3. To conduct a method comparison between real-time spectral and laboratory based measurements of MC and color parameters during drying of beef slices (Chapter 5)

To achieve the objectives, the drying behavior and CIELAB color pattern of beef slices was monitored invasively by laboratory measurements. With regard to non-invasive monitoring systems, additionally, HSI was applied to collect as much information as possible to develop prediction models. The most important information in terms of MC and CIELAB L*, a* and b* values was extracted by wavelengths selection to proof the development of simplified models applicable for embedded real-time monitoring systems for beef drying processes.

1.3 Approaches and investigated factors

This section provides a tabular overview about the materials and methods used and the factors investigated in this thesis (Table 1-1). Detailed information on the experiments is given in the individual chapters (Chapters 3-5) related to the work conducted.

Table 1-1: Materials, methods and investigated factors

Materials (i) / Methods (ii) / Investigated factors (iii)	**Chapter**
ANOVA (iii)	4
a_w measurement (ii)	4
Beef – l. dorsi, m. semimembranosus (i)	3, 4, 5
CARS-PLS (ii)	3
CIELAB measurements (iii)	3, 4, 5
CIELAB prediction (ii)	3, 4, 5
Cold storage (cooling freezing) (ii)	3, 4, 5
Colorimetry (ii)	3, 4, 5
Drying (ii)	3, 4, 5
Drying behavior (iii)	3, 4
Hyperspectral imaging (ii)	3, 4, 5
MCUVE-PLS (ii)	3
Method comparison (ii)	5
MC measurement (ii)	3, 4, 5
MC prediction (ii)	3, 4, 5
Moisture distribution visualization (ii)	3
RMSE (ii)	3, 4
PLSR (ii)	3, 4, 5
PRESS (ii)	5
Pre-treatments (i)	3, 4, 5
Wavelengths selection (ii)	3, 4, 5

1.4 Structure of the thesis

The present Chapter 1 gives an overview and introduction into the research topic of the study and further focusses on the research objectives, approaches and investigated factors and the structure of the thesis. Chapter 2 comprises the state of the art related to dried meat products, post-slaughter and pre-processing handling of meat and introduces the reader to food drying principles, optimization and online product monitoring techniques. Chapter 3 deals with the investigations based on laboratory measurements on drying behavior and color development of beef slices dried at 70 °C with regard to different seasonings and different beef states (fresh, fresh-frozen thawed, matured and matured-frozen thawed). Further, individual prediction models related to each raw material and seasoning regarding spectral MC and CIELAB color pattern measurements were developed and evaluated. The impact of different drying temperatures (50, 60, and 70 °C) and salt solutions on drying behavior and color development of beef slices was investigated in Chapter 4. Additionally, prediction models for MC and CIELAB L*, a* and b* values with an increased robustness against the influencing factors ‘drying temperature’ and ‘pre-treatment’ were developed. In Chapter 5, data acquired for Chapter 3 and 4 was utilized to develop robust prediction models irrespective of the beef origin or pre-drying handling for MC or CIELAB color pattern, providing innovation for this research topic. Moreover, an entire model for simultaneous prediction of the investigated parameters was developed. With regard to the application of spectral measurements into real-time monitoring systems, the models were simplified by wavelengths selection and evaluated by method comparisons. Chapter 6 provides a general discussion of the key investigations of Chapters 3 - 5 and offers a critical review of the thesis and future research options. Chapter 7 is the summary of the thesis in English and German language.

1.5 References

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2 State of the Art

2.1 Dried meat products

Drying of meat is popular across the world. Dried meat products provide a shelf stable protein source and, therefore, can secure a balanced diet where no refrigeration is available. In Europe, sausages and ham are common dried meat products, but in recent years, meat snacks like jerky or biltong that are popular in the US and South Africa, respectively, have gained an increasing interest (Grand View Research, 2017) and reflects the trend for high protein nutrition (Arenas-Jal et al., 2019) or related specific diets like, e.g. *paleo.* In Germany, beef jerky is the most popular, however, there is no legal definition, but it is considered that the treatments applied guarantee an absolutely safe product (LGL, 2018). The labeling of other dried meat products is diverse, like *charqui* in Brasil, *khaddid* in Maroko, *sharmoot* in Sahelian Africa or *Yukpo* in Korea (Purnomo, 2012), what all can be translated with “dried meat”. However, the processing, which also depends on the local conditions and availabilities, varies greatly (Feiner, 2006). Whole muscle products are usually dried at ambient conditions or in aging chambers and are sliced after dehydration, while sliced and ready to eat meat snacks are dried in hot air driers.

The raw material used for common dried meat products like *jerky* or *biltong* ranges from housed livestock to game from the local environment (Jones et al., 2017) and can be assumed to be dependent on the local eating habits and availabilities of the raw material. Comparisons showed higher unsaturated fatty acid concentrations for pork jerky in comparison to beef jerky, which might influence the sensorial properties, while after equivalent drying times lower moisture contents and water activity were investigated for pork jerky (Yang et al., 2009). Muscles from different livestock showed different contents of water, fat and protein, which influenced the composition of lamb, mutton and beef jerky, while it did not influence the sensory properties evaluated by panelists (Sutton et al., 1993), but could affect the properties during storage. Carr et al. (1996) observed a high desirability regarding emu jerky in comparison to beef or turkey jerky, which was related to the highest shear force, while the flavor of beef jerky was ranked the highest. Besides the production of an microbial stable product, quality determination in dried meat focuses less on vitamin contents like for fruits and vegetables, but on changes of lipids and proteins, which could lead to off-odors and undesired color changes due to heat induced rancidity (Souza et al., 2013).

Against the background of the crucial impact of meat products on human health (Richi et al., 2015) and with a consumption of 59.5 kg/capita and year in Germany (Statista, 2020) twice as high as recommended (DGE, 2015), it is worth mentioning that the contribution of meat drying to global food security and reduction of post-harvest losses is considered rather small compared to dehydration of fruits and vegetables. However, meat drying offers an opportunity in terms of value addition of underutilized cuts, e.g. due to local preferences like for pork tenderloin or leg, which, for example, is much less consumed in Korea compared to other cuts (Yang et al., 2009). Underutilized cuts further offer lower raw product prices and, thus, lower production costs compared to high-quality cuts. In some cases, this might require a careful consideration and evaluation of final product properties and consumer acceptance, as challenges appeared concerning those issues for jerky produced from cattle heart or tongue (Miller et al., 1988).

2.2 Post-slaughter and pre-processing handling of meat

2.2.1 Muscle structure and meat development

The skeletal muscles of mammals consist of water (75 %), protein (22 %) fat (1 - 2 %) as well as of vitamins and minerals (1 %) (Honikel, 2002). So-called myofibrils are bundled by the sarcolemma (cell membrane) to build muscle fibers, which again are bundled to fascicles. The sarcolemma contains the sarcoplasm. Muscle proteins consist of stromal (10 - 20 %), sarcoplasmic (30 %) and myofibrillar proteins (50 - 60 %) (Boland et al., 2018). The stromal proteins (elastin and collagen) provide the connective tissue to stabilize the muscle fiber bundles, fascicles and the entire muscle and tendons and connect the muscle to the bone. The sarcoplasmic proteins are responsible for metabolic functions inside the muscle, like myoglobin for the oxygen transport and various enzymes, of which some are directly involved in meat development processes post-slaughter (Boland et al., 2018). Myofibrillar proteins (myofilaments) consists of contractile, cytoskeletal and regulatory proteins and build so-called sarcomeres. With making up 70 - 80 % of the total protein content, the contractile proteins actin (thin filaments) and myosin (thick filaments) are the main proteins of the sarcomere (Frontera and Ochala, 2015). Microscopic investigations identified characteristic bands and lines present in the sarcomere. These are protein dense so-called A-bands and less protein dense I-bands (Goll et al., 1984), further recognizable H-zones and according M-lines in the middle of each sarcomere as well as sarcomere bounding Z-disks, marking the edges of each sarcomere. The tenderization of meat is highly related to degradation of I-bands and Z-disks (Nowak, 2011).

Immediately after slaughter, the pre-rigor phase begins, in which oxygen is still available in the myoglobin and hemoglobin to resynthesize ATP (adenosine triphosphate) from ADP (adenosine diphosphate) and phosphocreatine enzymatically. After phosphocreatine is exhausted, ATP is synthesized anaerobically by the glycolysis of glycogen into lactic acid, which leads to a decrease of meat pH. With the exhaust of glycogen, the concentration of ATP and, thus, the elasticity of the muscle decreases. That leads to the rigor mortis phase and a rigidity of the muscle due to an irreversible linkage of actin and myosin filaments (Hamm et al., 1980), referred to as actomyosin (Huff-Lonergan et al., 2010). In the rigor mortis phase, the meat has a low water binding ability, because the pH is close to the isoelectric point (IEP, pH = 5.5) of muscle proteins and is firm and tough (Honikel, 1986). According to Lana and Zolla (2016), the conversion from muscle to meat begins with the rigor mortis phase. The relevant enzymes in the post-rigor phase pushing the meat conversion processes are calpains, cathepsins, caspases and the 20S proteasome (Kemp et al., 2010). The pre-rigor and rigor mortis phase induce several mechanisms like the release of calcium from sarcoplasmic proteins into the sarcoplasm (Vignon, Beaulaton and Ouali, 1989) or the decrease of pH, which induces the activation of calpain or the release of cathepsins from lysosoms (Lana and Zolla, 2016). Lana and Zolla (2016) pointed out that to date, the process of meat development is not definitely determined. This is also due to contradictory results regarding in-vivo investigations and might further be influenced by other processes like apoptosis and autophagy of muscle cells. However, proteolytic post-rigor processes as well as denaturation (induced by low pH) and oxidation processes lead to the release of rigor mortis in the muscle and especially softens the myofibrillar structure (Ouali et al., 2006), which affects several quality parameters of meat such as tenderness, taste, and color. The slight increase of the pH during the post rigor phase further increases the water holding capacity (Hamm, 1972). The maturation time recommended to take place at -1 - +7 °C is dependent on the meat species and is referred to be 36 hours for poultry, at least 60 hours for pork and 2 - 6 weeks for beef (Honikel and Schwägele, 2006).

2.2.2 Freezing and thawing of meat

Freezing treatments primarily affect the water molecules in meat, which according to Hamm (1963) are located in the myofibrils (70 %), in the sarcoplasmic space (20 %) and in the extracellular space (free capillary water). A very small part (<10 %) of the water is truly bound to the proteins due to the bipolar structure of the water molecules and is resistant to freezing (Fennema, 1985). The highest percentage of water is immobilized by electrostatic, osmotic and capillary forces (Puolanne and Halonen, 2010) and is highly affected by freezing treatments (Huff-Lonergan and Lonergan, 2005).

Freezing treatments are known to induce moisture losses after thawing due to protein denaturation of mainly myosin (Wagner and Añon, 1985), which decreases the water holding capacity (Farouk et al., 2004; Leygonie et al., 2012). This phenomenon is increased by low freezing rates according to the formation of larger ice crystals at higher freezing temperatures and, therefore, a bigger damage of meat compounds during thawing process (Mortensen et al., 2006). The drip mainly contains sarcoplasmic proteins due to their high water solubility (Farouk et al., 2004). Further, increased storage duration under frozen conditions were investigated to increase the drip after thawing, which could be explained by the formation of bigger ice crystals by recrystallization (Ngapo et al., 1999), which can lead to almost equal thaw drips of slow (-20 °C) and rapid frozen (-80 °C) meat at extended storing times (Farouk et al., 2004). Thus, rapid freezing is particularly important for short-term storage. Moreover, high thawing rates have been investigated to decrease the drip after thawing (Gonzalez-Sanguinetti et al., 1985), which is explained by less recrystallization to bigger ice crystals, and was supported by Ngapo et al. (1999) with higher drip for meat thawed in a refrigerator compared to thawing by microwaves or at room temperature. Most research studies focus on the reduction of drip caused by freezing and thawing treatments. However, it has been determined that parameters such as texture, flavor, appearance, microbial and nutritional value are impacted as well (Leygonie et al., 2012). This is due to the breakdown of muscle structure caused by the formation if ice crystals and the resulting denaturation of muscle proteins, but also due to oxidation of lipids and proteins during freezing and thawing. This is initiated by pro-oxidative factors like the release of enzymes from the mitochondria and lysosomes due to disruption of muscle cells by ice crystals (Leygonie et al., 2012) and increases at increased storage times (Vieira et al., 2009). The myoglobin is partly denatured and, thus, is more susceptible to autooxidize to metmyoglobin, which results in less redness compared to unfrozen meat (Leygonie et al., 2012).

2.2.3 Seasoning

Seasoning of meat with salt and spices is a common pre-treatment to increase the flavor of meat and meat products and is known to increase microbial safety and shelf life (Björkroth, 2005; Weiss et al., 2010). Seasoning can further prevent oxidation of lipids and proteins (Weiss et al., 2010), but can also lead to increased oxidation processes (Ke et al., 2009; Maqsood et al., 2015). Additionally, functional properties, e.g. water binding capacity, tenderness or color of meat and processed meat can be influenced by seasonings (Aktas et al., 2003; Andrés-Bello et al., 2013; Burke and Monahan, 2003; Goli et al., 2014; Yusop et al., 2010). In this context, the pH and the salt concentration play a major role, since both parameters influence the functionality of meat proteins.

Sodium chloride (NaCl) induces a solubilization and a swelling of myofibrillar proteins and increases the water binding in meat. This is due to a stronger binding of Cl^-- ions than of Na^+-ions to the proteins leading to an increase of negative charges inside the myofibrils, which further leads to a repulsion of molecules and, thus, to swelling (Hamm, 1972). The negative charges further shift the IEP to a lower pH, which supports the swelling and water binding capacity at pH values above the isoelectric point (Hamm, 1986).

The decrease of pH in meat by acid seasonings below the IEP increases the water holding capacity due to swelling of meat proteins induced by net positive charges and osmotic pressure (Gault, 1985). Acid treatments further lead to solubilization of myofibrillar proteins (Ke et al., 2009) and might lead to denaturation of myofibrillar and sarcoplasmic proteins and, thus, affecting the water binding capacity and the color of meat (Aktaş and Kaya, 2001).

However, simultaneous addition of salt and acid has been shown to increase the drip or cooking loss (Goli et al., 2014; Medyński et al., 2000), but was shown to be highly dependent on the salt concentration (Goli et al., 2011). This can be explained by a salt induced decrease in electrostatic repulsive forces at pH values below the IEP (Hamm, 1961).

2.3 Principles and advances of food drying

The principle of drying is the removal of water from a product. Apart from freeze drying that utilizes the principles of sublimation, evaporation is widely used for moisture reduction of food. Heat increases the evaporation rate and is supplied by convection, conduction or irradiation. There is a wide variation regarding industrial food drying techniques (rotary, spray, drum, deep and fluidized bed, tray, belt drying etc.) to meet the specific requirements of the final product with convection drying being the most used (Mujumdar and Law, 2010). For this, hot air is used as the drying medium. Food belongs to the hygroscopic materials in that moisture is partially sorptively bound. As a result, compared to non-hygroscopic materials, they cannot be dried to moisture content 0, but only to equilibrium moisture content (Mersmann et al., 2005). During the dehydration process, water and water steam diffuses to the surface of the product, which is forced by different temperature and pressure gradients occurring inside the product (Mujumdar, 2007). The drying process at a constant drying temperature can be separated in three phases (Mersmann et al., 2005), which are characterized by a complex interaction of heat and mass transfer affecting the final product quality (Mühlbauer and Müller, 2020). According to Mersmann et al. (2005), in the first phase of drying, the surface moisture is evaporated that avoids the heating of the product and results in a constant drying rate that ends with an inflexion point. In the second phase, the drying rate decreases due to the maximum hygroscopic moisture at the product surface that expands into the product. This ends evaporation so that the product heats up and vapor diffuses through the expanding dry layer to the surface until the maximum hygroscopic moisture is present throughout the product. This is characterized by a less significant inflexion point that initiates the third drying phase, in which water vapor still diffuses through the product, but the drying rate is constant until it decreases to 0, indicating the equilibrium moisture content has been reached. In addition to the temperature, the relative humidity, the velocity, as well as the thermal conductivity, the specific heat capacity and the density of the drying air influence the drying process (Mühlbauer and Müller, 2020).

Food processors face several challenges, as they need to produce nutritional, safe and shelf stable products, economical, fulfill consumer’s perception in terms of quality and sustainability (Chen, 2008). Concerning the need for higher CO_2 savings and potential future governmental regulations to meet the climate agreement (UNFCCC, 2015) and to reduce the global food waste significantly by 2030 (UN, 2015), the development of drying processes with a highest level of product and process efficiency are essential.

Dehydration leads to a reduction of MC, but other product parameters are impacted as well, which leads to changes in flavor, appearance and texture and can decrease the content of valuable compounds. In this context, drying needs to be regarded as a quite complex and dynamic phenomenon (Aghbashlo et al., 2014). In contrast, the product analysis is usually carried out only after drying (Martynenko, 2017) so that the most drying processes can be regarded as a *black box* with regard to changes occurring inside the product, which poses the risk for both product and process inefficiencies. Optimization of drying processes that considers only energetic issues were investigated to influence the product quality negatively and vice-versa (Mujumdar, 2007). Therefore, in recent years, the investigation of the drying process as a holistic approach has gained more and more interest (Tsotsas and Mujumdar, 2014). In this context, e.g. the idea of smart drying strategies offers a big potential to consider both product and process efficiency.

2.3.1 Smart drying

Smart drying implicates the knowledge of what occurs inside a product and the drying environment during drying by continuous monitoring to adjust the drying parameters consequently according to the monitored data to achieve a predefined high product quality and acceptable energy efficiencies (Su et al., 2015), resulting in individualized of drying processes. However, fully developed smart control systems are very complex and require a multidisciplinary effort by involving control theory, expert systems, automation, computer vision (CV), sensor fusion, operations research and artificial intelligence (AI) (Martynenko, 2018b).

To date, less smart or intelligent drying applications for biomaterials are available, which might be due to challenges in the interpretation of the AI language for the practical needs of drying (Martynenko, 2018a). Traditional control systems are widely used but are unable to achieve complex control objectives (Kondakci and Zhou, 2016). However, these objectives can be achieved by intelligent real-time control systems due to the ability of self-learning (Sun et al., 2019). Further, today´s knowledge about changes inside the product during drying is often insufficient, which influences the development of drying control strategies and, thus, intelligent applications negatively (Martynenko, 2018a).

A simple approach regarding smart drying is the termination of drying processes based on moisture content monitoring instead of time, to achieve efficient drying times (Moscetti et al., 2018). Smart drying approaches become more complex as soon as continuous product measurements should be integrated into the control of drying parameters (air temperature, velocity and humidity) throughout the process. In this context, Sturm (2018a) showed a high potential for increased efficiency and product quality by product temperature controlled drying of apples achieved by the dynamic control of air temperature at different air velocity and humidity. Recent smart drying applications focus on control of the drying environment by product monitoring systems, like for example CV or spectroscopy techniques (Su et al., 2015), whereas so called *product quality driven adaptive control systems* (Sturm, 2018b; Sturm et al., 2018) require parameters measurable non-invasively and in real-time to enable immediate control.

2.3.2 Product monitoring approaches

One of the first techniques to meet the requirements of real-time and non-invasive product monitoring during drying regarding shape, size, color, and texture changes and, therefore, to replace traditional (laboratory) techniques that were designed for quality control after drying (Martynenko, 2017) was CV technology (Aghbashlo et al., 2014). CV aims to imitate the human eye, which naturally leads to limitations with regard to the color space used (red, green, blue) as it is represented only by visible wavelengths (Gómez-Sanchis et al., 2013; Xu and Sun, 2017). Therefore, traditional CV is useful when quality attributes are linked to a product’s extrinsic characteristic like the physical parameters of drying (Sun et al., 2019), but is limited regarding internal characteristics and composition (ElMasry et al., 2012). Conversely, spectroscopic measurements offer increased spectral information and, thus, enable the measurements of intrinsic parameters, however, due to the point detection, show disadvantages regarding high spatial resolution (Gómez-Sanchis et al., 2013).

HSI combines CV and spectroscopy and provides spatial and spectral information simultaneously (Yang et al., 2017) by acquiring so called *3-D hypercubes* with two spatial and one spectral dimensions, which enables a more complete description of distribution of ingredients and is also named as *chemical imaging* (Gowen et al., 2008). Due to the large amount of data and the corresponding computational challenges, HSI is difficult to implement in online applications (ElMasry et al., 2012). However, HSI offers considerable possibilities regarding the development of simple multispectral imaging (MSI) systems by using it to determine specific wavelengths for certain parameters (Burger and Gowen, 2011).

HSI and MSI have already been shown to provide a high potential regarding food quality and safety analyses or to monitor food during processing (Amigo et al., 2013; Liu et al., 2017; Ma et al., 2019; Wu and Sun, 2013). In terms of meat, spectral imaging has been successfully investigated in terms of non-invasive measurements of technological and chemical parameters, detection of contamination, defects and authenticity (Cheng et al., 2017; Feng et al., 2018; Kamruzzaman et al., 2015; Prieto et al., 2018) and product monitoring during various meat processing techniques, respectively (Liu et al., 2017). In terms of meat processing, HSI was successfully used for the prediction of moisture content and distribution during convective drying (Wu et al., 2013) or moisture content and color prediction during microwave heating (Liu et al., 2018). The models could be simplified in view of MSI applications by selecting specific wavelengths. However, the robustness of the models against variations in raw material quality or processing conditions is often mentioned for future research (Feng et al., 2018; Su et al., 2020) but rarely implemented.

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3 Drying behavior and quality parameters of dried beef (biltong) subjected to different pre-treatments and maturation stages*

3.1 Abstract

The drying behavior of fresh, matured and frozen beef, marinated with 0.5 % salt, 1 % salt, salt and vinegar and blind samples, dried at 70 °C, was investigated. Weight and color (CIELAB) were measured and images of the samples were created with a hyperspectral imaging camera. Results show that the marinade and the type of beef influences the drying behavior of beef, but not the final color. Results from the hyperspectral imaging show, that it is possible to build good fitting prediction models resulting in high R^2 (min. 0.81, max. 0.98) and low RMSE (min. 0.08, max. 2.35) for moisture content, *a** and *b** values.

Key words: beef drying, pre-treatment, maturation stage, hyperspectral imaging, VNIR

3.2 Introduction

Drying is one of the oldest methods of food preservation with records on meat drying dating back to 20,000 BC (Hayashi, 1989). While in developing countries, dried meat products provide a valuable protein source, in industrial countries it is more common as a healthy, high protein snack food. For example, there has been a growing demand for meat snacks among young adults in the United States, with an increase by 18 % between 2010 and 2015 (McLynn, 2015). Meat snacks are mainly produced from beef, but the demand for jerky from turkey is also growing (McLynn, 2015). Currently, the most popular types of dried meat snacks are *biltong* (South Africa), *charqui* (South America) and *jerky* (North America). However, compared to the United States and South Africa, the meat snack market is considered underdeveloped in Europe and salami style, rather than jerky style products, dominate the market (Wells, 2015). The manufacturing of jerky products is diverse in terms of drying methods (sun/air, solar/thermic etc.), the raw materials used (meat from different species, different seasonings, whole muscle, stripes or ground) and pre-treatments (e.g. cooking before drying, seasoning etc.). Lean meat was observed to dry quicker and to show lower product temperature than minced meat dried under the same conditions (Karabacak et al., 2014).The raw meat is usually

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treated in various ways before drying in order to produce a microbiologically safe and tasty meat snack. Cooking the meat before the drying process has been shown to reduce pathogens like *E. coli* O157:H7 (Albright et al., 2003; Harrison et al., 2001), *L. monocytogenes* and *Salmonella* (Harrison et al., 2001), and additionally leads to increased drying rates (Hii et al., 2014). Spices can also be utilized to reduce microbial growth. The osmotic pressure and lower water activity induced by ions affects microbial survival and growth negatively and, thus, increase shelf life of processed meat products. Regarding prevention of microbial survival and growth, a low pH value of the marinade is essential (Calicioglu et al., 2003a, 2003b; Yoon et al., 2005, 2009), as low pH environments increase the sensitivity of pathogens to second lethal stresses (SLS) like drying and low water activity (Shadbolt et al., 2001). However, salt can also act as a pro-oxidant regarding lipids and fresh meat pigments when used in low, flavor enhancing concentrations (maximum 3 % of the final product) and the oxidation potential depends on impurity of the salt by the presence of metallic components (Overholt et al., 2016). Shelf life mainly depends on oxidation of compounds in meat, such as proteins and lipids, as it leads to changes in taste (e.g. rancidity) and appearance. For this reason, meat products are often seasoned with antioxidants before drying. A very common antioxidant is sodium nitrite which additionally inhibits pathogens by interfering with several metabolic processes and for this reason is very hard to replace (Weiss et al., 2010). However, particularly in organic production, non-artificial alternative preservatives that pose no risk to health are required. Furthermore, the drying step itself can lead to increased protein degradation as it was investigated in air dried fish compared to microwave or freeze drying (Hu et al., 2013), which can lead to a poor rehydration behavior (Mounir, 2015). Therefore, understanding the effects of ageing and freezing/thawing on beef prior to drying is crucial for the optimization of the drying process in terms of efficiency and quality. The structural integrity of the meat affects the water holding capacity and hence the drying behavior. One of the most important factors affecting the drying of meat is the ability of the myofibrils to shrink and squeeze water out to extrafibrillar regions where it can be lost as drip. In order for this to occur, the myofibrils and the cross links between them must be intact for the shrinkage to be translated down the fiber. Hence, fresh beef will dry faster due to it not being subjected to the damage caused by ageing or freezing. Maturing leads to structural breakdown and blockage of drip channels, whereas freezing leads to wider drip channels but increases water binding during drying (Retz et al., 2017).

A survey amongst very small and small jerk producers found that drying temperature ranged from 51.6 °C to 93.3 °C. However, a number of producers did not know the temperature and dried until the product was finished, which was based on experience rather than measurement

(Lonnecker et al., 2010). This shows that there are no clear processing guidelines within the industry.

Currently, process monitoring and control, if even present, are predominantly invasive. A non-invasive method tested successfully in some food processes is hyperspectral imaging, which allows the prediction of quality parameters, based on models that were designed from measured (in the lab) and hyperspectral data, respectively. The method is based on wavelength selection and, therefore, is very specific regarding products and quality parameters. Hyperspectral imaging was successfully applied regarding several quality parameters in foods (Wu and Sun, 2013a). As the method is relatively expensive, a further step could be to transfer the results to a CCD camera (with sensitivity in the VNIR), in combination with selective lighting (Chi et al., 2010), or with filters (Tominaga, 1999).

In this study, the effect of seasoning fresh and matured beef, as well as the influence of freezing and thawing on drying behavior and quality changes represented by color, were investigated. Using hyperspectral imaging, investigations were made into evaluating the feasibility of quality metric prediction using wavelengths in the VNIR waveband. Investigations were also carried out to evaluate different spectral pre–processing techniques. The vast changes in surface texture of beef during drying was the reason for the utilization of a number of different pre-processing methods. Furthermore, quality metric predictions, chromaticity and moisture content were evaluated and reduced dimensionality methods were also tested.

3.3 Materials and Methods

For the experiment, organically produced topside of three two-year-old crossbred (Simmental x Limousine) bulls from the same farm, slaughtered in the same abattoir, was used. Thus, the tests were conducted in triplicate. Of each bull, 1.5 kg fresh (1 week after slaughter, stored at 4 °C ± 1 °C), 1.5 kg matured (3 weeks after slaughter, stored under the same conditions like the fresh) and 1.5 kg frozen (-15 °C, 7 days) of each variation of beef were used. The fresh/matured frozen beef was thawed for 24 h at 4 °C in a fridge before it was prepared for drying. The beef was cut with the fiber into slices of 5 mm thickness using an electrical slicer (Graef, Allesschneider Vivo V 20, Arnsberg, Germany) and cut with a knife into 50 x 50 mm pieces. The samples were seasoned and stored for 22 hours at 4 °C. For each drying session, three different seasonings were prepared (eight slices/pre-treatment). The first was 0.5 % salt, the second 1 % salt and the third 5 % apple vinegar plus 0.5 % salt (S+V) (m/m based on raw beef weight). In addition, there were also reference samples, kept untreated (blind). Due to the small volume, the samples were not tumbled for an equal seasoning, but sprinkled by hand. The slices were dried at 70 °C for six hours in a tray dryer (HT mini, Innotech Ingenieursgesellschaft mbH, Germany). Before seasoning, directly before drying and hourly during the drying process, the samples were weighed, *L**, *a** and *b** values were determined at three points per sample to gain an average color values and hyperspectral images were taken. After the drying process the samples were dried at 105 °C in an hot air oven (SLE 500, Memmert GmbH, Germany) for 24 hours according to AOAC (1990) to determine the final moisture content. Moisture content (MC, wet base) was determined from the weighing data and rest moisture determination. The moisture ratio was calculated with the equation below:

$$\mathrm{MR} = \frac{\mathrm{M} - \mathrm{M_e}}{\mathrm{M_i} - \mathrm{M_e}} \tag{1}$$

with M as moisture content at any time, M_i as the initial moisture content and M_e as the equilibrium moisture content. According to Rayaguru and Routray (2012) (Rayaguru and Routray, 2012) it was simplified to:

$$\mathrm{MR} = \frac{\mathrm{M}}{\mathrm{M_i}} \tag{2}$$

The drying rate DR was calculated with:

$$\mathrm{DR} = \frac{\mathrm{M_t} - \mathrm{M_{t+\Delta t}}}{\Delta \mathrm{t}} \tag{3}$$

Where M_t and $M_{t+\Delta t}$ are the moisture content of the samples at time t and $t+\Delta t$ and t is the time in hours; the DR expresses the loss of water per unit dry matter (DM).

The color was measured with the Chroma Meter CR-400 (Minolta, Osaka, Japan). The averaged values of three different points per sample were used to describe the color change (ΔE) after seasoning and during drying with the following equation:

$$\Delta E = \sqrt{(L^*_i - L^*_t)^2 + (a^*_i - a^*_t)^2 + (b^*_i - b^*_t)^2} \tag{4}$$

with lightness/color before drying marked with $_i$ and with $_t$ at every time.

The samples were imaged with a visible near-infrared (VNIR) hyperspectral imaging system with an ImSpector V10E PFD camera combined to a linear translation stage (SPECIM Spectral Imaging Ltd., Finland). A 35 mm lens (Xenoplan 1.9/35, Schneider Optische Werke GmbH, Germany) was positioned with a 27 cm distance to the conveyor tray. The tray moved at 8 mm/s to produce a spatial area of 0.03 mm² per pixel. That enabled the capture of data of 400-1010 nm in 1.5 nm increments. Three 60 W halogen GU 10 bulbs acted as illumination source. A white tile of 200*24 mm (H*W) with a spatial resolution of 1700*1392 pixels was used as reference. For each captured image, a dark noise file was captured. After images were captured, they had to be pre-processed, by the initial estimation of the current dark noise in the image (MDN_λ), using a method previously explained in (Crichton et al., 2018, 2015; Retz et al., 2017). All image processing was carried out using Matlab 2013b.The relative reflectance spectrum for each pixel in the image ($R(\lambda_{xy})$) was calculated with the following equation with spatially averaged illumination spectrum $W(\lambda_{xy})$ and the irradiance spectrum $I(\lambda_{xy})$:

$$R(\lambda_{xy}) = \frac{I(\lambda_{xy}) - MDN_\lambda}{W(\lambda_{xy}) - MDN_\lambda} \quad (5)$$

The average relative reflectance spectrum for each sample was then calculated automatically. Partial least square regression (PLSR) was used to develop a prediction model from the average reflectance spectra for the determination of the feasibility of beef slice quality metric prediction in relation to moisture content, *a** and *b** values using the full spectral range. Afterwards, three different regression techniques according to the reduced spectral data were carried out. PLSR (Westad and Marten, 2000) is used to identify linear relationships between individual wavelengths in the reflectance spectra and specific qualitative measurement values such as moisture content in this present study. PLSR has been used to build predictive models with a small number of wavelengths previously (Pu et al., 2015; Wu and Sun, 2013b). Monte-Carlo uninformative variable elimination (MCUVE) (Cai et al., 2008; Centner et al., 1996) and the competitive adaptive reweighted sampling (CARS) (Li et al., 2009) are different wavelengths reduction methods, noted from here onwards as MCUVE-PLS and CARS-PLS respectively. In some cases better predictive models than PLSR models can be generated by these (Amigo et al., 2013). The performance of the models was evaluated using R^2 and RMSE, where R^2 needs to present high and RMSE small values.

All regression analysis was undertaken within a spectral range of 500-1010 nm, due to the low emission of the halogen bulbs between 400-500 nm. The dataset was divided into a calibration set (16 samples for each pre-treatment) and a validation set (eight samples for each pre-treatment) on an animal-by-animal basis. For example, bulls 1 and 2 could be the basis for the calibration model, and bull 3 for the test model. Models were built by randomizing the selection of animals for the calibration and test sets a total of 10 times, and results were then averaged.

3.4 Results and Discussion

3.1.1 *Drying behavior*

Drying behavior of different types of beef pre-treated with different marinades and of the blind samples are shown in Figure 3-1.

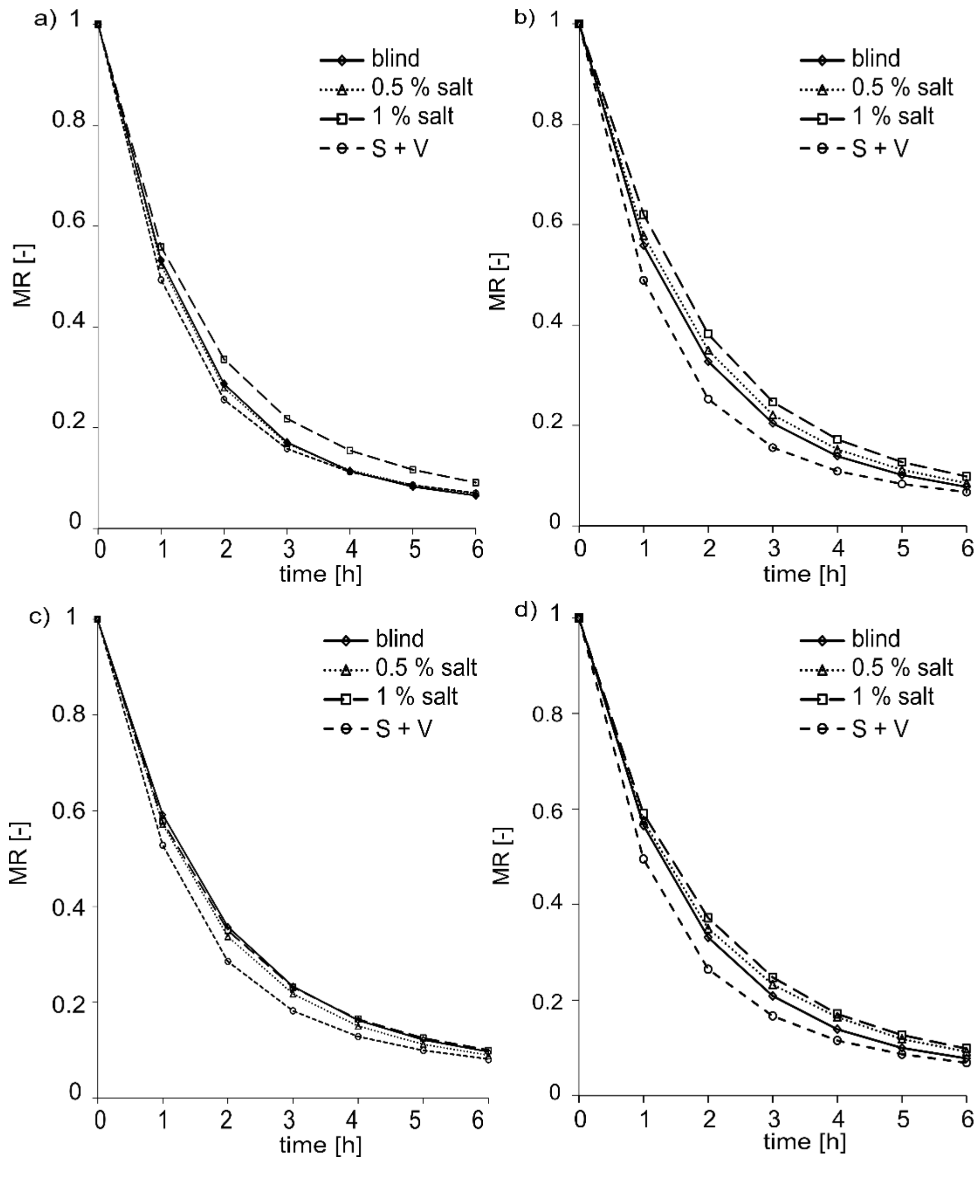

Figure 3-1: Drying curves of a) fresh, b) fresh frozen, c) matured and d) matured frozen beef slices

The samples had an average initial moisture content of 72 %. Samples treated with S+V dried the fastest compared to the blind and salted samples, whereas the salt concentration of 1 % led to a slower drying compared to 0.5 %. The effect of the pre-treatments was similar for all beef types but was increased by freezing-thawing treatments compared to the non-frozen samples. That corresponds with the results for a lower water holding capacity of frozen beef from Lagerstedt et al. (2008). Decreased drying times at increasing salt levels were described for pork jerky by Yang et al. (2012) and can be explained by an increased water holding capacity due to swelling of fibers which is induced by Na^+ and Cl^- ions of sodium chloride that leads to solubilization of functional myofibrillar proteins (Desmond, 2006). The higher water holding capacity leads to lower drip (Medyński et al., 2000) and cooking losses (Bess et al., 2013; Medyński et al., 2000) in beef, which has advantages for cooked and roasted meat by improving texture and juiciness, but leads to slower drying processes for meat. In the present study, an effect between 0.5 and 1 % was apparent. The beef in the present study dried the fastest when marinated with S+V.

An increased water holding capacity for meat induced by the addition of salt was investigated in many studies, but results differ when it comes to marination of meat with salt and acid. As the pH decreases below the isoelectric point of the meat (pH 5.2 for red meat), the water holding capacity increases (Gault, 1985). Oreskovich et al. (1992) found an increase in cooking losses, the closer the pH of beef was to the isoelectric point. Acids lead to swelling of muscle fibers and increase their lengths (Ke et al., 2009; Rao et al., 1989) which also leads to an increased water holding capacity and to reduced drip (Burke and Monahan, 2003; Medyński et al., 2000) and cooking losses (Medyński et al., 2000) for beef. Klinhom et al. (2015) described a decrease in water holding capacity for beef treated with acid, which might be due to a different fiber structure according to the origin of the beef or age of the bull before slaughter. The selected acid also influences the effect on water holding capacity. Lactic acid was investigated as more effective compared to citric acid, whereas the highest concentration that leads to the lowest pH was determined to always show the biggest effect (Aktas et al., 2003).

Ke et al. (2009) described a loss of beef muscle structure after acid marination depending on net positive charges and the resulting increasing repulsive forces at lower pH leading to a fusion of myofibrils and their solubilization by slackening of the meat (Goli et al., 2014). Marination of turkey meat in acidic solution was investigated to lead to an intake of liquid and, thus, to a decrease in protein content, and to losses of proteins from the meat.

In combination with salt the protein solubilization decreases (Goli et al., 2014). However, concomitant marinating with acid and salt led to higher cooking losses of muscle fibers in several studies. For minced pork and beef, a treatment with salt and lactic acid at the same time led to increasing drip and cooking losses (Medyński et al., 2000) that also occurred in turkey breast meat (Goli et al., 2014). This effect can be explained with both the osmotic and electrostatic theory of muscle swelling (Medyński et al., 2000). The adding of acid to take the pH below the isoelectric point induces the protonation of the carboxylic groups inside the muscle resulting in breakages of electrostatic bonds between the muscle proteins (Burke and Monahan, 2003) through an increase of net positive repulsions (Medyński et al., 2000). In combination with salt, those positive repulsions are screened by the negative chloride ions, so the effect of acid-induced swelling is reduced, meaning less water can be held by the muscle and hence a decreasing water-holding capacity.

It is also important to note the effects of freezing and maturing on the drying behavior when marinating. All four variations (fresh, fresh-frozen, mature, and matured-frozen), have the same trend of S+V drying fastest and 1 % salt slowest. However, when the beef had been frozen and thawed, the rates of drying for each treatment were further apart than those of the non-frozen. For the frozen samples, it is likely that the effect of each marinade is altered by the changes caused by freezing and thawing. In fresh beef, the water-holding capacity is known to be reduced compared to matured beef and only the 1 % salt marination led to an obvious decreasing drying effect in the present study. The water-holding capacity increases with increasing maturing time and the difference between the drying curves for blind and salted samples are close to each other, while the effect of S+V results in clearly separated curves. In the frozen samples, the ones salted with 1 % salt dried slower compared to the non-frozen samples due to the muscle swelling induced by salt as well as the water remaining after thawing being in a more strongly bound state (Thyholt and Isaksson, 1997). The effect of decreased dehydration has also been mentioned in the context of osmotic dehydration combined with convective drying. Salt pre-treatments decreased the initial moisture content but in the subsequent convective drying, the beef dried slower, explained by a salt induced formation of a crust (Chabbouh et al., 2011).

Figure 3-2 shows the drying rate DR vs. the moisture ratio (MR) and makes it once more clear that the margination effects the drying behavior of beef. For the beef marinated in S+V, the drying rate is roughly the same for non-frozen and frozen-thawed. This lack of change in rate can be explained by considering the antagonistic effect of acid and chloride ions as discussed, which prevents swelling and can even lead to shrinkage, squeezing out water and increasing the rate. Hence, this could counteract some of the damage caused by freezing by limiting the increase in water holding capacity that freezing can bring about.

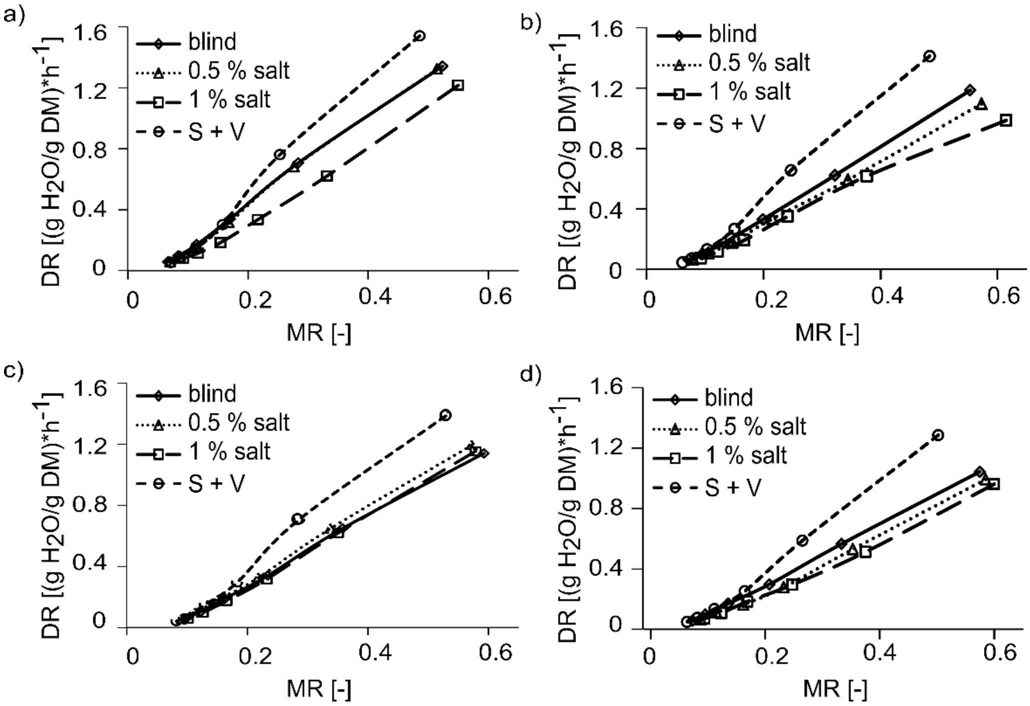

Figure 3-2: Drying rate (DR with DM for dry matter) vs. moisture ratio (MR) for a) fresh, b) fresh frozen, c) matured and d) matured frozen beef slices

3.1.2 *Color changes*

The results of color changes for different types of beef seasoned with different marinades are shown in Figures 3-3 – 3-6. The changes in color were similar alongside the different beef variations. Differences could be determined for both matured variances, that had lower a* and b* values from the beginning, which resulted in lower overall color changes after the drying process, while the final color was mainly the same as detected in the fresh variances. For matured samples, no effect of the salt concentration on lightness and yellowness was recognizable, while salt concentration affects these parameters in matured-frozen and fresh samples. The three other beef types all had lower L* and a* values for higher salt concentration, which correlates with other findings, which suggest an increase in salt decreases a* (Saricoban and Yilmaz, 2010). This is likely to be a result of the way in which the salt treatment brings about changes in the beef structure.

As previously discussed, salt increases the concentration of inorganic ions in the beef and, thus, can lead to repulsion and, hence, swelling. For the matured beef, the structural integrity of the beef is already lowered due to the enzymatic reactions, which take place during ageing. That leads to weakened myofibrils and drip channels becoming blocked over time (Farouk et al., 2012), which means that the damage from swelling may have less of an impact than in fresh, which was more intact initially. In addition, changes in salt concentration caused by water losses from thawing will make the effect of the marinade on the frozen samples more prominent by effectively increasing the concentration of salt as a result of higher thawing losses. It is interesting to note that the behavior of the beef marinated with S+V is most similar to the blind samples resulting in a shifted curve regarding the color change ΔE. This could be due to the stabilizing effect of the acid-ion combination, which prevents swelling and, thus, prevents water-holding capacity from increasing significantly relative to the blind, unlike the salted samples.

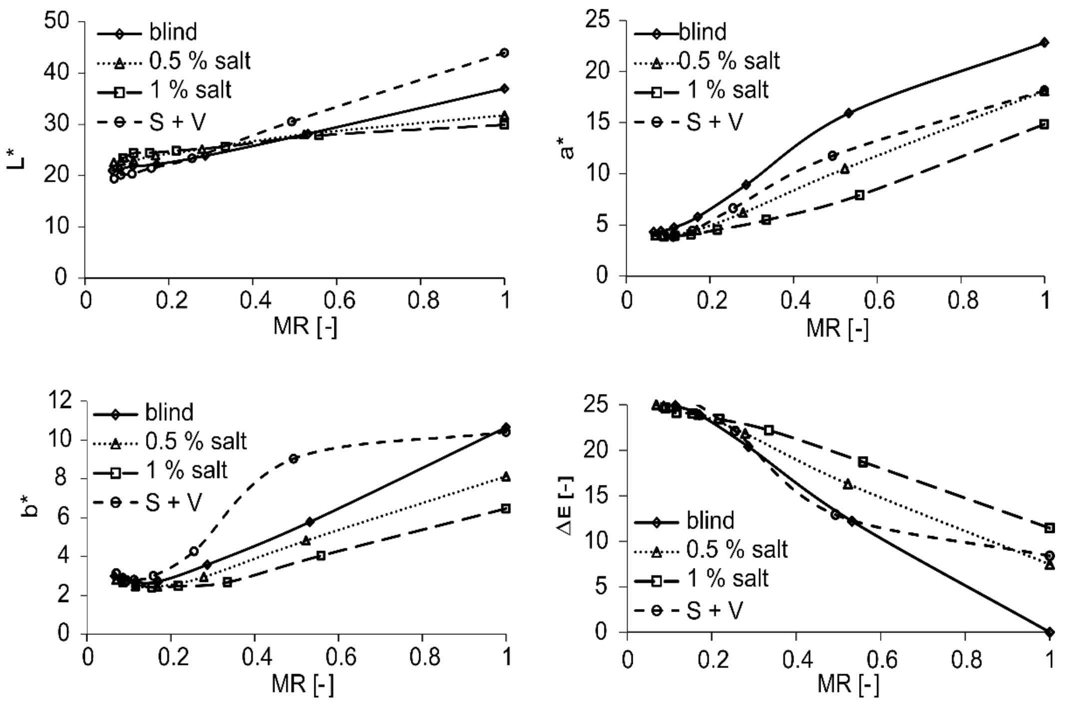

Figure 3-3: Development of L*, a* and b* values and color change ΔE during drying of fresh beef slices treated with four different pre-treatments

The changes in b* for the S+V show a significant trend for all beef types (fresh, fresh-frozen, mature and mature-frozen) which is not as pronounced for the other marinade pre-treatments. This again could be a result of the damage caused by the marinades. The stabilization of S+V may lead to it resisting changes caused by heat, in terms of color, for longer than salt or no marinade. Oxymyoglobin is the pigment which is primarily responsible for yellowness (Saricoban and Yilmaz, 2010), which implies that the structure and oxidation state of this pigment changes more slowly when the beef is marinated with S+V.

The different starting points for ΔE result from the color measurements before and after the pre-treatment and show that the samples marinated with 1 % salt were the most different. This resulted from low L*, a* and b* values at the start of the drying process for the samples salted

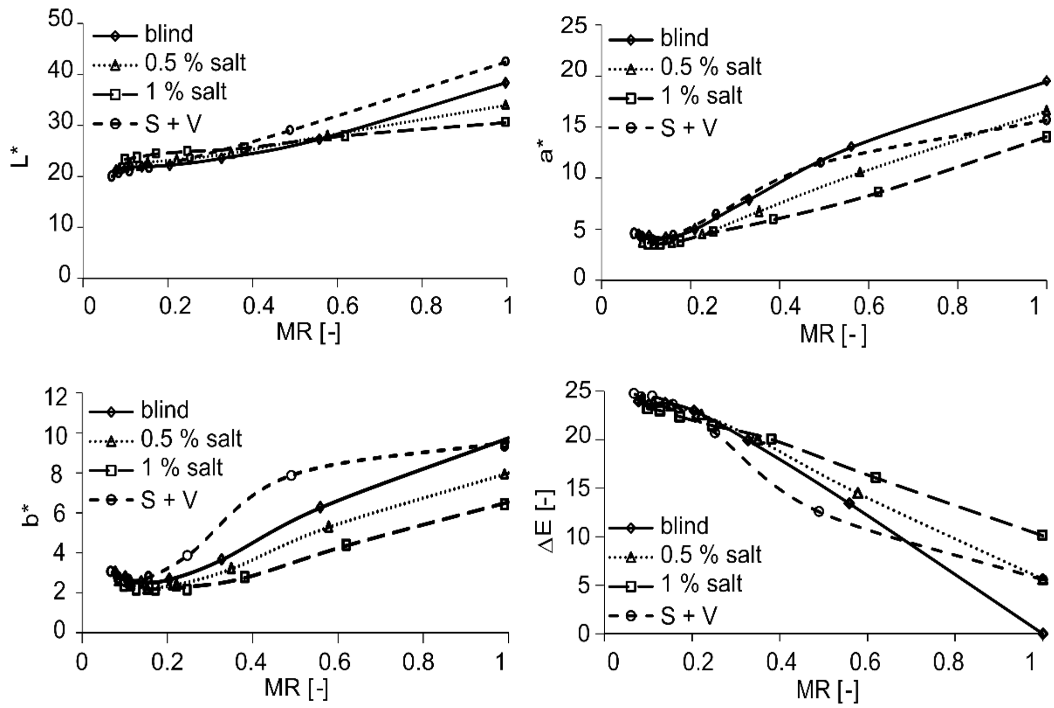

Figure 3-4: Development of L*, a* and b* values and color change ΔE during drying of fresh frozen beef slices treated with four different pre-treatments

with 1 % salt which correspond with the results from Teixeira et al. (2011), who found lower values particularly for a* and b* values in salted goat meat. Salt was found to have the ability to release iron from heme-binding molecules (Kanner et al., 1991) and, thus, acts as an oxidizer for myoglobin (reviewed by Seideman et al. (1984) that also leads to changes in color. The redness and yellowness was also lower for the samples treated with 0.5 % salt but to a lesser extent than observed for the higher salted samples. The highest lightness after pre-treatment was measured in S+V samples, which is likely a result of the acidity of the vinegar. Acids affect the structure of proteins and lead to more lightness and lower a* values, while b* values remained similar compared to the control samples (Stivarius et al., 2002). Arganosa and Marriott (1989) assumed a reaction from myoglobin to metmyoglobin induced by acids. In the present study, the samples appeared to fade after marination and could be differentiated by eye. This could be a result of proteins on the surface denaturing and becoming more opaque. As reviewed by King and Whyte (2006), myoglobin as well as other proteins in meat, begin to denature between 55 and 65 °C but are in general thermostable with denaturation temperatures between 65 and 80 °C (Martens et al., 1982). Furthermore, the decrease of water content leads to an increasing concentration of pigments in the beef slices and a change in color (Ferrini et al., 2012). However, it is important to point out that at the end of the drying process, the color was nearly the same for all beef variations and pre-treatments, which also resulted in almost the same ΔE at the end of the drying within each beef type.

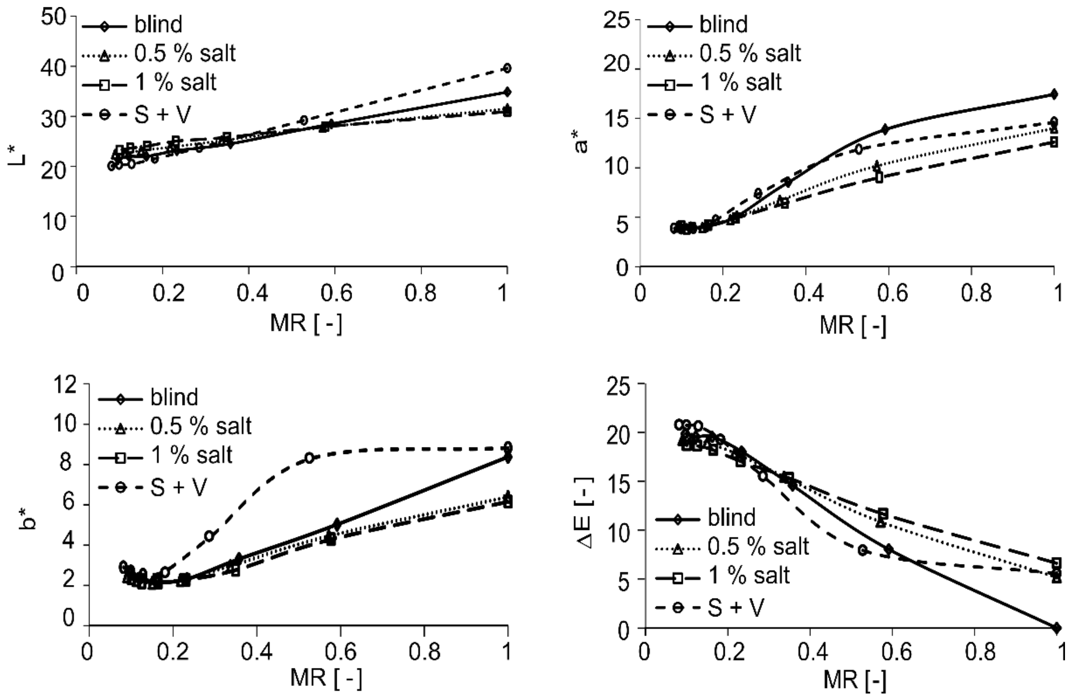

Figure 3-5: Development of L*, a* and b* values and color change ΔE during drying of matured beef slices treated with four different pre-treatments

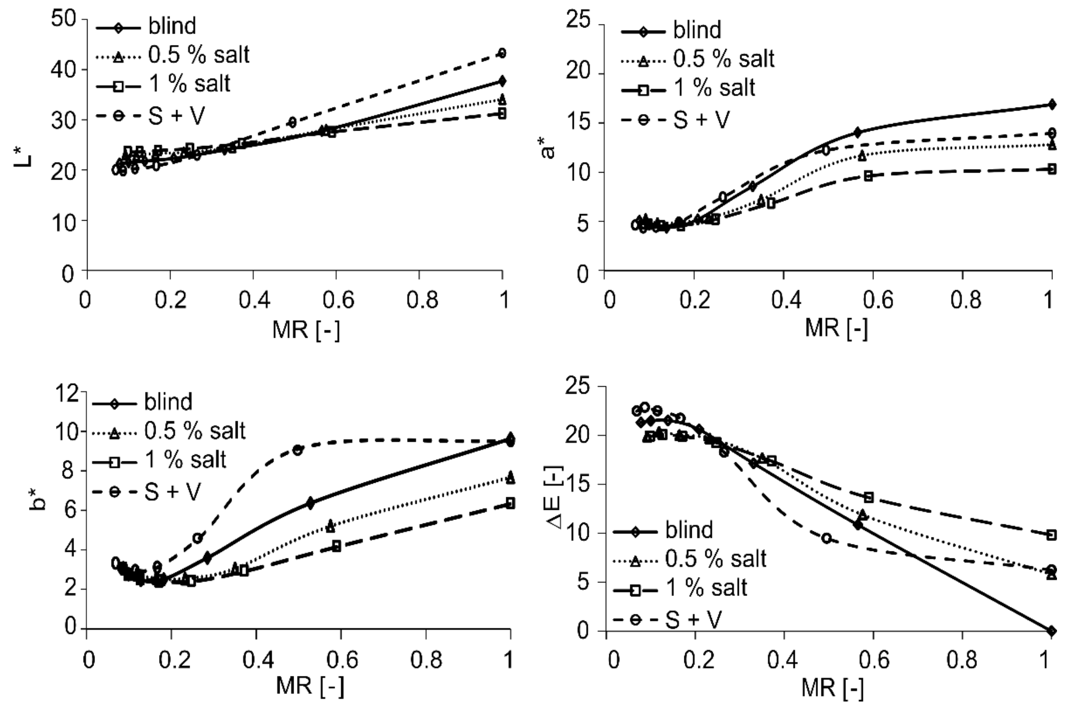

Figure 3-6: Development of L*, a* and b* values and color change ΔE during drying of matured frozen beef slices treated with four different pre-treatments

3.1.3 *Spectral Analysis*

Tables 3-1 – 3-4 show the results of the prediction models for moisture content, a* and b* value. While in fresh beef mainly the PLSR-model gives the best results, in the other beef types the MCUVE-PLS model shows a high performance, especially for the parameters moisture content and b*value. CARS-PLS is of less interest.

As previously stated, a number of different spectral pre-processing techniques were compared during the statistical modelling. Due to the large amount of results generated through these comparisons, Tables 3-1 – 3-4 contain only the information for the best performing method. Table 3-1 shows the results for the prediction of the quality metrics, moisture content and chromaticity, with the fresh beef. The full spectrum models can be seen to allow the prediction of the metrics with a very high level of accuracy. Using this information, reduced complexity models were then created with the wavelengths shown on the right hand side. The performance of these reduced dimensionality models can be seen to favor comparably with the more complex models. The marinades and beef pre-treatments can be seen to have an effect upon the wavelengths, which provide the ability to predict the metrics with a high level of accuracy. The performance of the moisture content models can be seen to allow decent prediction accuracy, although a smaller average RMSE would be preferable whilst maintaining the same R^2 level. Chromatic co-ordinate prediction (CIELAB a* and b*) is also at a high level of performance, with average errors being less than 1 perceptual unit of distance.

These performance characteristics repeat themselves across Tables 3-2 – 3-4 for fresh frozen, matured and matured frozen thawed beef samples, respectively. From these tables a number of conclusions can be made. The first is that the VNIR region is viable to predict moisture content and chromatic information during drying for all of the beef tested. Secondly, the important spectral effect of the beef storage conditions and pre-treatments is illustrated by the selection of different wavelengths to predict the quality metrics.

This shows that beef, and meat, is a more complex substance than other materials such as apples, where pre-treatments have been shown to have no effect upon wavelengths needed to predict the same metrics (Crichton et al., 2018). The creation of the spectral prediction models also allows pseudo color metric maps to be developed, to visualize and monitor the important features during drying.

Table 3-1: Performance of best PLS models for predicting moisture content (MC), a* and b* and selected wavelengths during drying for fresh beef slices treated with four different pre-treatments

Pre-treatment	Quality Metric	Full Wavelength Set: Calibration Set R^2	Full Wavelength Set: Calibration Set RMSE	Full Wavelength Set: Test Set R^2	Full Wavelength Set: Test Set RMSE	Reduced Wavelength Set: Calibration Set R^2	Reduced Wavelength Set: Calibration Set RMSE	Reduced Wavelength Set: Test Set R^2	Reduced Wavelength Set: Test Set RMSE	Best model	Selected wavelengths [nm]
blind	MC	0.97	0.16	0.96	0.17	0.97	0.16	0.97	0.15	MCUVE-PLS	539, 563, 589, 625, 847, 920, 967
	CIELAB a*	0.97	0.94	0.82	2.18	0.98	1.00	0.98	0.91	CARS-PLS	501, 545, 599, 600, 609, 620, 639, 876, 938, 959, 961, 977, 987, 995
	CIELAB b*	0.98	1.10	0.96	1.34	0.97	0.63	0.94	0.77	MCUVE-PLS	507, 528, 536, 580, 592
S+V	MC	1.00	0.04	0.95	0.19	0.98	0.14	0.97	0.17	PLSR	502, 768,812, 914, 962
	CIELAB a*	0.98	0.73	0.94	1.20	0.93	1.43	0.94	1.29	PLSR	582, 599, 687, 732, 777, 811, 959
	CIELAB b*	0.98	0.71	0.92	1.38	0.94	0.84	0.95	0.76	MCUVE-PLS	552, 582, 586, 622, 654, 806
0.5 % salt	MC	1.00	0.04	0.97	0.09	0.97	0.15	0.96	0.17	PLSR	599, 645, 705, 791, 855, 928, 967
	CIELAB a*	0.98	0.61	0.94	1.17	0.94	1.27	0.92	1.45	PLSR	565, 580, 662, 763
	CIELAB b*	0.98	0.37	0.93	0.52	0.96	0.45	0.93	0.54	PLSR	511, 575, 617, 648, 704, 739, 846, 906, 954
1 % salt	MC	1.00	0.05	0.96	0.13	0.95	0.19	0.93	0.22	PLSR	593, 673, 766, 807, 876, 982
	CIELAB a*	0.98	0.60	0.65	2.32	0.93	1.08	0.92	1.08	PLSR	599, 650, 703, 785, 833, 961
	CIELAB b*	0.97	0.29	0.85	0.52	0.91	0.56	0.83	0.61	PLSR	583, 594, 613, 678, 764, 936, 974

Table 3-2: Performance of best PLS models for predicting moisture content (MC), a* and b* and selected wavelengths during drying for fresh-frozen beef slices treated with four different pre-treatments

Pre-treatment	Quality Metric	Full Wavelength Set				Reduced Wavelength Set				Best model	Selected wavelengths [nm]
		Calibration Set		Test Set		Calibration Set		Test Set			
		R^2	RMSE	R^2	RMSE	R^2	RMSE	R^2	RMSE		
blind	MC	0.89	0.16	0.85	0.17	0.97	0.16	0.97	0.15	MCUVE-PLS	539, 563, 589, 625, 847, 920, 967
	CIELAB a*	0.99	0.48	0.62	2.70	0.91	1.33	0.94	1.16	MCUVE -PLS	505, 552, 575, 582, 809, 1007
	CIELAB b*	0.95	0.98	0.84	1.75	0.89	0.50	0.93	0.46	MCUVE-PLS	501, 517, 528, 537, 606, 654, 1003, 1005
S+V	MC	1.00	0.01	0.96	0.11	0.99	0.09	0.97	0.08	CARS-PLS	571, 582, 583, 912, 948
	CIELAB a*	0.99	0.30	0.96	0.71	0.96	0.80	0.93	0.76	MCUVE-PLS	540, 545, 546, 577, 930, 1005
	CIELAB b*	0.94	0.64	0.94	0.68	0.93	0.72	0.98	0.42	MCUVE-PLS	513, 552, 574, 575, 580, 583
0.5 % salt	MC	1.00	0.01	0.90	0.20	0.94	0.20	0.94	0.11	MCUVE-PLS	545, 560, 957, 977, 1002, 1008
	CIELAB a*	0.99	0.33	0.94	0.84	0.96	0.75	0.94	0.85	PLSR	514, 542, 583, 599, 634, 961
	CIELAB b*	0.99	0.18	0.94	0.44	0.98	0.31	0.96	0.30	MCUVE-PLS	501, 513, 575, 589, 592, 855, 918, 930
1 % salt	MC	1.00	0.02	0.68	0.31	0.96	0.15	0.89	0.16	CARS-PLS	626, 822, 857, 974, 975, 1003
	CIELAB a*	0.99	0.31	0.94	0.67	0.96	0.84	0.91	0.77	CARS-PLS	557, 559, 592, 673
	CIELAB b*	0.98	0.17	0.91	0.41	0.96	0.37	0.88	0.33	MCUVE-PLS	513, 516, 552, 556, 574, 585

Table 3-3: Performance of best PLS models for predicting moisture content (MC), a* and b* and selected wavelengths during drying for matured beef slices treated with four different pre-treatments

Pre-treatment	Quality Metric	Full Wavelength Set				Reduced Wavelength Set				Best model	Selected wavelengths [nm]
		Calibration Set		Test Set		Calibration Set		Test Set			
		R^2	RMSE	R^2	RMSE	R^2	RMSE	R^2	RMSE		
blind	MC	0.96	0.17	0.95	0.18	0.99	0.09	0.98	0.14	CARS-PLS	507, 516, 551, 577, 588, 650, 712, 828, 886, 893, 931, 985, 1002, 1008
	CIELAB a*	0.94	1.77	0.67	3.96	0.88	2.00	0.81	2.35	CARS-PLS	533, 557, 613, 667, 681, 697, 722, 738, 855, 876, 917, 947, 974, 997
	CIELAB b*	0.92	1.64	0.81	2.41	0.76	1.46	0.82	1.33	MCUVE-PLS	510, 519, 586, 969, 992, 1000
S+V	MC	1.00	0.02	0.95	0.15	0.95	0.19	0.94	0.23	CARS-PLS	667, 669, 692, 694, 701, 883, 910
	CIELAB a*	0.99	0.36	0.93	1.06	0.96	0.78	0.95	0.96	PLSR	586, 669, 768, 901, 971
	CIELAB b*	0.99	0.26	0.98	0.47	0.98	0.45	0.98	0.43	MCUVE-PLS	502, 508, 513, 516, 530, 563, 599, 602
0.5 % salt	MC	1.00	0.03	0.95	0.16	0.97	0.14	0.96	0.17	MCUVE-PLS	625, 667, 780, 828, 870, 920, 961, 989, 1002, 1005
	CIELAB a*	0.97	0.60	0.92	1.01	0.96	0.83	0.95	0.86	MCUVE-PLS	505, 511, 514, 516, 540, 585
	CIELAB b*	0.97	0.31	0.86	0.68	0.93	0.47	0.87	0.66	PLSR	501, 543, 605, 683, 766
1 % salt	MC	1.00	0.02	0.96	0.14	0.98	0.12	0.93	0.21	MCUVE-PLS	849, 851, 912, 917, 953, 956, 961, 972
	CIELAB a*	0.98	0.50	0.94	0.69	0.93	0.90	0.90	0.98	PLSR	580, 606, 689, 758, 851
	CIELAB b*	0.95	0.39	0.68	0.97	0.79	0.93	0.84	0.57	MCUVE-PLS	542, 545, 548, 569, 582, 596

Table 3-4: Performance of best PLS models for predicting moisture content (MC), a* and b* and selected wavelengths during drying for matured-frozen beef slices treated with four different pre-treatments

Pre-treatment	Quality Metric	Full Wavelength Set				Reduced Wavelength Set				Best model	Selected wavelengths [nm]
		Calibration Set		Test Set		Calibration Set		Test Set			
		R^2	RMSE	R^2	RMSE	R^2	RMSE	R^2	RMSE		
blind	MC	0.98	0.11	0.98	0.12	0.97	0.13	0.98	0.12	MCUVE-PLS	822, 961, 971, 974, 1005
	CIELAB a*	0.95	1.24	0.88	1.84	0.98	0.78	0.96	1.00	CARS-PLS	569, 599, 603, 606, 623, 645, 717, 747, 827, 828, 844, 883, 941, 966, 979
	CIELAB b*	0.98	0.71	0.97	0.94	0.94	0.65	0.94	0.62	CARS-PLS	502, 510, 557, 568, 747, 761, 804, 844, 846, 849, 899, 910, 956
S+V	MC	1.00	0.01	0.96	0.16	0.99	0.10	0.97	0.13	PLSR	501, 582, 676, 909, 969
	CIELAB a*	0.99	0.43	0.90	1.02	0.96	0.82	0.95	0.88	PLSR	523, 545, 562, 583, 599, 634, 662, 689
	CIELAB b*	0.99	0.32	0.94	0.56	0.97	0.48	0.93	0.80	PLSR	501, 520, 583, 650, 686, 742, 772
0.5 % salt	MC	1.00	0.02	0.99	0.06	0.99	0.09	0.97	0.13	MCUVE-PLS	508, 514, 562, 582, 825, 836, 838, 839, 851, 855
	CIELAB a*	0.98	0.49	0.85	1.18	0.94	0.87	0.89	1.16	PLSR	501, 591, 639, 709, 780
	CIELAB b*	0.98	0.29	0.91	0.54	0.94	0.54	0.94	0.54	MCUVE-PLS	516, 519, 557, 560, 563, 580, 603, 807
1 % salt	MC	1.00	0.02	0.99	0.07	0.98	0.12	0.99	0.08	MCUVE-PLS	504, 513, 514, 563, 565, 585, 708, 969
	CIELAB a*	0.98	0.34	0.84	0.91	0.86	0.96	0.95	0.57	MCUVE-PLS	551, 582, 606, 611, 656, 812, 1005
	CIELAB b*	0.99	0.19	0.83	0.55	0.93	0.49	0.86	0.39	MCUVE-PLS	549, 554, 583, 588, 614, 964, 967, 969

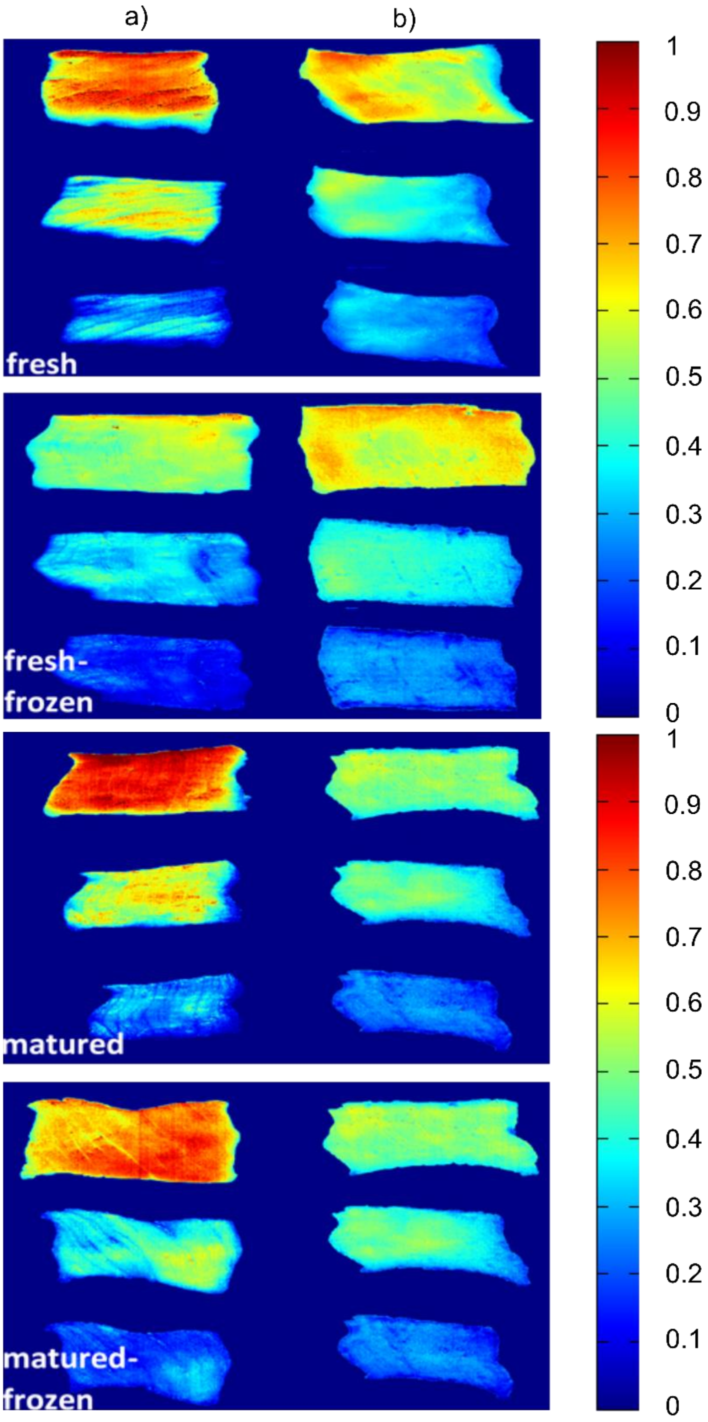

Figure 3-7: Visualized moisture content in fresh, fresh-frozen, matured and matured-frozen beef treated with a) S+V and b) 1 % salt before, after 1 and 2 hours of drying

Figure 3-7 shows the moisture distribution maps for the beef pre-treated with a) S+V, and b) a 1 % concentration of salt during the first 2 hours of drying (the images shown are for the same slice at 0 hours, 1, and 2 hours of drying (column-wise)). Whilst the differing spectral effect of pre-treatments has been noted through the selection of different important wavelengths this illustrates the difference in both, absolute moisture content and drying behavior of the samples. Across the four beef storage types, the S+V treated beef can generally be seen to have a higher initial moisture content. However, moisture content can be noted to be lost at a greater rate during the first 2 hours of drying for the S+V treated beef, which matches the results shown in Figure 3-1. These images allow for recognition of specific regions on the sample where moisture is being lost at a greater/ lesser rate. As such, they can also allow for a localized recognition of hot spots within a dryer, when a number of samples are imaged at the same time with assigned dryer bed locations.

3.5 Conclusions

The study shows that the drying behavior and color changes of beef are influenced by marinade and beef type. The drying rate increases when beef is pre-treated with S+V and decreases with increasing salt content. Fresh beef dries the fastest, previous freezing and thawing leads to slower drying in fresh beef whereas it increases drying of matured beef, that dries the slowest, resulting from changes in meat fibers during maturation. Although the color of beef slices changes during the marinating and drying process, the color does not present a quality parameter, because after the drying process, the color is nearly the same, irrespective of pre-treatment or beef type and the color before the drying process. VNIR hyperspectral imaging can be implemented in process monitoring for beef drying for chromaticity prediction as well as for moisture content prediction. PLSR and MCUVE-PLS models have been built successfully to predict moisture content and chromaticity during the drying of beef. A further step could be the transfer of selected wavelengths provided by selective lighting or filters to CCD cameras and implement that as a non-invasive monitoring system for beef-drying. This method would be much more cost efficient while the implementation of a hyperspectral imaging system is too expensive to use it in industry, especially for small scale production.

Further research in product stability might be interesting in terms of marinades and drying temperature, as well as product temperature. Since maturing is a time and energy consuming process in the meat industry, the consumer acceptance and sensory attributes for the different beef types should be evaluated in further research.

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4 Impact of drying temperature and salt pre-treatments on drying behavior and instrumental color and investigations on spectral product monitoring during drying of beef slices*

4.1 Abstract

Drying behavior and instrumental color development of beef slices untreated or pretreated with salt or salt and vinegar solutions were monitored by determining the moisture content and the color change by measuring CIELAB values during drying at 50, 60 and 70 °C. Time-series hyperspectral imaging (400 - 1000 nm) was applied with regard to the development of non-invasive measurement systems based on robust models to predict moisture and color independent of the pre-treatment and drying temperature. Samples pretreated with salt dried the slowest which became more prominent at increasing drying temperatures and the least color change ($\Delta E = 23$) was observed at 60 °C drying temperature. Robust prediction models for moisture content and CIELAB values irrespective of pre-treatment and processing conditions were developed successfully and improved by wavelengths selection with high R^2 (0.94 - 0.98) and low RMSEP (1.05 - 5.22) which will support the future development of simple and cost-effective applications regarding non-invasive product monitoring systems for beef drying processes.

Keywords: beef drying, visible near-infrared hyperspectral imaging, partial least square regression, wavelengths selection, smart drying

4.2 Introduction

The drying of meat is a well-known processing to preserve this highly perishable food product and enables an extended shelf life without additional cooling. Due to its nutritional value, dried meat is also a popular snack food and appreciated as a good supplementary for the diet of athletes and nutrition-conscious consumers. However, drying processes are usually taken at a sacrifice of very high energy consumption and the food drying sector faces low efficiencies of 35 - 45 % (Mujumdar, 2007). Low efficiencies can result from extended drying times where

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low drying temperatures were reached and/or moisture was removed insufficiently from the product e.g. due to low air velocities or inappropriate dryer constructions (Babu et al., 2019). This also affects the final product quality in terms of microbial stability, volatile compounds and appearance (Zhang et al., 2017). Additionally, inefficiencies can also result from an excessive length of drying, i.e. unnecessarily long-term heat treatments. Besides improvement of process efficiency and achieving shelf stable products, processing also has to satisfy the consumers' quality requirements for final products (Font-i-Furnols and Guerrero, 2014). Furthermore, drying processes are usually regarded to be static. Usually, only drying air temperature and potentially velocity are measured and/or controlled, which results in the finalization of the drying process after a certain time and not when the target product characteristics are reached (Sturm, 2018a).

To understand the dynamic development of changes inside a product during dehydration the monitoring of the products during drying becomes necessary. Analytical methods are often destructive and time-consuming and lead to interruption of the process, and, thus are not useful for real-time measurements (Liu et al., 2017). Conversely, non-invasive measurement systems are non-destructive, rapid in practice and/or applicable for real-time monitoring and online-applications. The gained information can directly feed back into control mechanisms of the system to adjust and finally terminate the process, which leads to individual drying processes dependent based on raw material characteristics and the individual changes during drying, respectively. This method is also known as *smart processing* or *smart drying* (Martynenko and Bück, 2018; Sturm, 2018b; Sturm et al., 2018).

Imaging technologies have been implicated to provide a solution for non-invasive product monitoring valuable compounds or moisture content (Su et al., 2015). In recent years, hyperspectral imaging (HSI) has gained more and more interest regarding monitoring of food properties (Ma et al., 2019; Qin et al., 2013). This technology combines spectroscopy with computer vision, which allows the capturing of 3-dimensional images (hypercubes) with one spectral and two spatial dimensions. This enables the measurement of product compounds and their spatial distribution. Based on the spectral range in question, HSI is differentiated into VNIR (visible and near infrared, 450 – 1000 nm) and NIR (near infrared, 900 – 1700 nm), which is important in terms of applicability with respect of cost efficiency since the detectors for VNIR-HSI are less expensive than for NIR-HSI (Qin et al., 2013). Furthermore, by wavelengths selection it might be possible to replace VNIR-HSI by low cost systems consisting of CCD cameras and filters or selective lighting (Chi et al., 2010). HSI has been applied for the measurement of different properties in fresh meat, such as pH, tenderness, water holding capacity, drip loss and color, which plays an important role regarding quality parameters and helps to classify different quality characteristics of muscle sections (Feng et al., 2018). The decision on VNIR or NIR spectral range has a significant influence on the prediction model accuracy and reliability, which depends on the parameter to be monitored (Cheng et al., 2017). Only few studies focused on application of time series HSI for meat drying processes, i.e. prediction of water distribution during dehydration at 40 °C (Wu et al., 2013), different maturation stages (Retz et al., 2017) and pre-treatments (von Gersdorff et al., 2018) at 70 °C drying air temperature and the developed of different prediction models and selected wavelengths for MC, CIELAB L*, a* and b* values related to each beef treatment.

In addition, drying process induced changes within a product need to be validated for the development of prediction models. However, for industrial applications it is necessary to develop models and select parameter-specific wavelengths that are valid for a wide range of raw materials. Regarding drying different drying conditions, pre-treatments and, thus, the changes inside the product need to be included into robust models. In a further step the results can be transferred to a multispectral imaging system, based only on the selected wavelengths, which results in decreased acquisition of data and less computation time and, therefore, in simpler and more cost efficient real-time applications (Kamruzzaman et al., 2016). Thus, the objective of the present study was to 1) investigate the drying behavior and development of instrumental color of beef slices pretreated with different solutions and dried at three temperatures. Further, 2) to develop one prediction model per parameter (MC, CIELAB L*, a* and b*) independent of the drying temperature or the pre-treatment and 3) to select specific wavelengths to develop simpler models with regard to simple practical applications.

4.3 Materials and Methods

4.3.1 Sample preparation

For the experiment 3 kg (± 0.35 kg) roast beef (*longissimus dorsi*) from four 26 month old heifers of the Uckermarker breed was used. They were raised on grassland and slaughtered according to the German law and the codes of good practices in a slaughter house in Northern Germany. Two days after slaughter, 1 kg portions were vacuumed, and shock frosted at -55 to -60 °C for 120 minutes. The beef was stored at -18 °C (Liebherr GP 1486 Premium, Germany) and thawed for 18 hours at 2 °C in a fridge (FKUv 1613 Premium, Liebherr, Germany) before slicing. For each drying experiment the beef was sliced in the direction of the fiber to 5 mm thickness with an electrical slicer (Graef, Allesschneider Vivo V 20, Germany) equipped with a smooth knife. Afterwards, 24 pieces (50 x 50 mm^2) were cut out of the slices with a sharp knife.

Two solutions with a salt content (NaCl) of 10 % (m/v) were prepared, one water based (S), the other one with apple vinegar as the solvent (S+V). For each treatment 8 pieces were dipped in the solution, the remaining 8 pieces stayed untreated as blind samples (b). The samples were then stored at 2 °C overnight and dabbed with a tissue before further measurements.

4.3.2 Drying and measurements

The samples were dried in a convective drier (HT mini, Innotech Ingenieursgesellschaft mbH, Germany) at 50, 60 or 70 °C, respectively, with a constant air velocity of 0.6 m/s. 4 repetitions were conducted for each pre-treatment and the respective drying temperature.

Weighing (lab scales, E2000D, Sartorius, Germany) and instrumental color measurement (CIELAB L*, a* and b* values (CIE, 2018)), with a Chroma Meter (CR-400, Minolta, Japan) by averaging the CIELAB values of three spots of each sample, were conducted before and during the trials. The instrumental color measurements was carried out with a D65 illuminant and the Chroma Meter was calibrated with a standard ceramic white plate (Konica Minolta) before each experiment. The measurements were done directly after slicing, after seasoning/storage and throughout the dehydration process applying intervals of every 20 min during the first hour, in two 30 minutes intervals during the second hour and hourly thereafter until the final MC of 14-20 % was reached. After drying, half of the samples were used to determine the final moisture content by oven drying (SLE 500, Memmert GmbH, Germany) at 105 °C for 24 hours (AOAC, 2016) until a constant weight was achieved. For the remaining samples the water activity ($a_{w)}$ (LabSwift, Novasina, Switzerland) was analyzed at a constant temperature of 23 °C.

The MC at each measurement point was used to calculate the moisture ratio (MR) during drying, according to Rayaguru and Routray (2012) with the simplified equation:

$$\mathrm{MR} = \frac{\mathrm{M}}{\mathrm{M_i}} \tag{1}$$

with M as moisture content at a given time, and M_i as the initial moisture content. The drying rate (DR) was expressed as

$$\mathrm{DR} = \frac{\mathrm{M_t} - \mathrm{M_{t+\Delta t}}}{\Delta \mathrm{t}} \tag{2}$$

where M_t and $M_{t+\Delta t}$ express the MC of the samples at time t and t+Δt.

The averaged instrumental color measurements of CIELAB L* (lightness), a* (redness) and b* (yellowness) values were used to calculate the color change ΔE during drying with the following equation:

$$\Delta E = \sqrt{(L^*_i - L^*_t)^2 + (a^*_i - a^*_t)^2 + (b^*_i - b^*_t)^2} \qquad (3)$$

with *i* expressing the initial L*, a* and b* values before drying and *t* expressing the values at every measuring point.

4.3.3 Hyperspectral imaging

VNIR-HSI measurements were taken in the spectral range of 400 - 1000 nm with an ImSpector V10E PFD camera combined with a linear translation stage (SPECIM Spectral Imaging Ltd., Finland). A 35 mm lens (Xenoplan 1.9/35, Schneider Optische Werke GmbH, Germany) was positioned with a distance of 27 cm to the conveyor tray. The detailed procedure and the image processing techniques to achieve the average reflectance of each beef slice (dark noise removal, segmentation etc.) performed with MATLAB 2013b software (TheMathwork, Nattick, USA) is described by Crichton et al. (2017), Retz et al. (2017) and von Gersdorff et al. (2018). Figure 4-1 shows an example of image segmentation with the RGB image, the NIR thresholded image and the final segmented image.

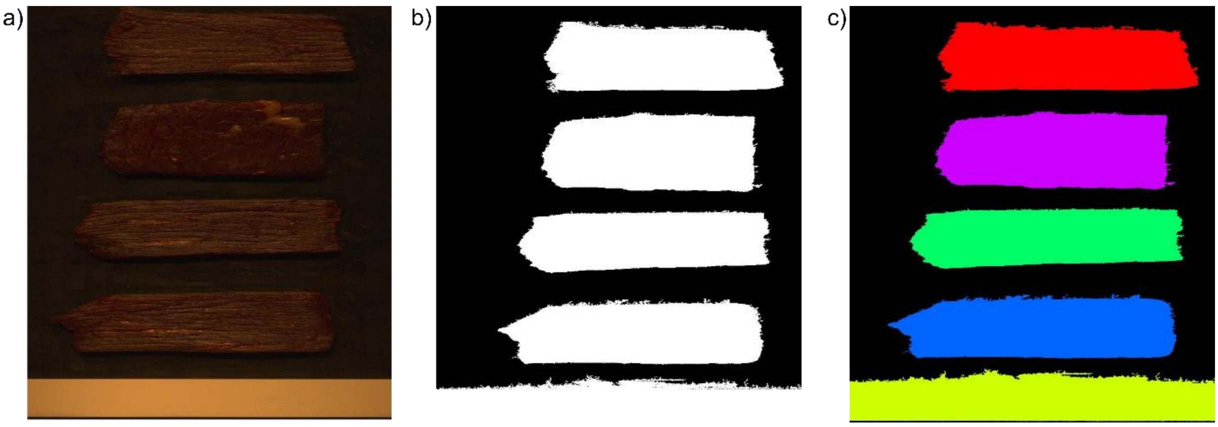

Figure 4-1: Image segmentation: a) RGB image, b) NIR threshold image, c) final segmented image with pseudo-colored shapes corresponding to beef slices and white reference

4.3.4 Prediction models

After the average relative reflectance spectrum of each slice was calculated, the spectral information and the data of the invasive measurements (weight, instrumental color) were used to develop PLSR prediction models with Rstudio software V.3.5.1, 2018, pls Package (Rstudio PBC, Boston, USA). The objective of the development of PLSR models was to find linear relationships between individual wavelengths in reflectance spectra and specific measured values (Westad and Marten, 2000). The data of all slices independent of the pre-treatment or drying temperature was combined to assess the likelihood to build one robust model per quality parameter based on measured and spectral data. The data set was split randomly to 70 % to build the PLSR model meanwhile to validate the model with the remaining 30 % of the data. In a first step, the model was built including 10 principal components. Afterwards the model was further developed into such a manner that 90 % of the variance could be explained with only 3 components for all parameters (MC, L*, a* and b* values). The coefficient of determination R^2 and the RMSEP (root mean square error of prediction) were thereafter used to evaluate the developed model. The selection of characterized wavelengths was carried out with the determination of the standardized regression coefficient at each wavelength, which was previously applied successfully in terms of non-invasive measurements in meat (ElMasry et al., 2012; Liu et al., 2013). The higher the regression coefficient, regardless of the sign, the more variations were explained by the wavelength for the related parameter.

4.3.5 Statistical analysis

To determine the effect of drying temperature and pre-treatment on final CIELAB color pattern and water activity a two-factorial Analysis of Variance (ANOVA) was calculated Rstudio software V.3.5.1, 2018 (Rstudio PBC, Boston, USA). The four animals were used as replicates for each drying temperature and pre-treatment including the values of each eight (instrumental color) and four slices (aw), respectively. The evaluation of the differences between the means were done using the Tukey post-hoc test and significance was considered as $P < 0.05$.

4.4 Results and Discussion

4.4.1 Drying behavior

Figure 4-2 a - c show the drying curves for beef slices with different pre-treatments at 50 °C, 60 °C and 70 °C and Figure 4-2 d - f the respective drying rates (DR), which clearly show the influence of temperature and pre-treatment on the drying behavior of beef. According to different studies the water holding capacity (WHC) (Bess et al., 2013; Chabbouh et al., 2011; Teixeira et al., 2011; von Gersdorff et al., 2018) is increasing after salt treatments during cooking or drying. Therefore, the samples pre-treated with the salt solution dried the slowest due to the ability of the myofibrils to swell and bind salt solutions which induced the solubilization of myofibrillar proteins by the Na^+- and Cl^--ions (Desmond, 2006). In the present study there is a slight indication that this effect becomes more prominent with increasing drying temperatures. However, the influence of salt on the WHC is impacted by many pre- and postmortem factors (Cheng and Sun, 2008). Sodium chloride is further known to release iron from sarcoplasmic proteins and, thus, is responsible for the denaturation of myoglobin

(Kristensen and Purslow, 2001) and, therefore, acts as a prooxidant for lipids which must be taken into account regarding the drying behavior and the shelf life of dried meat.

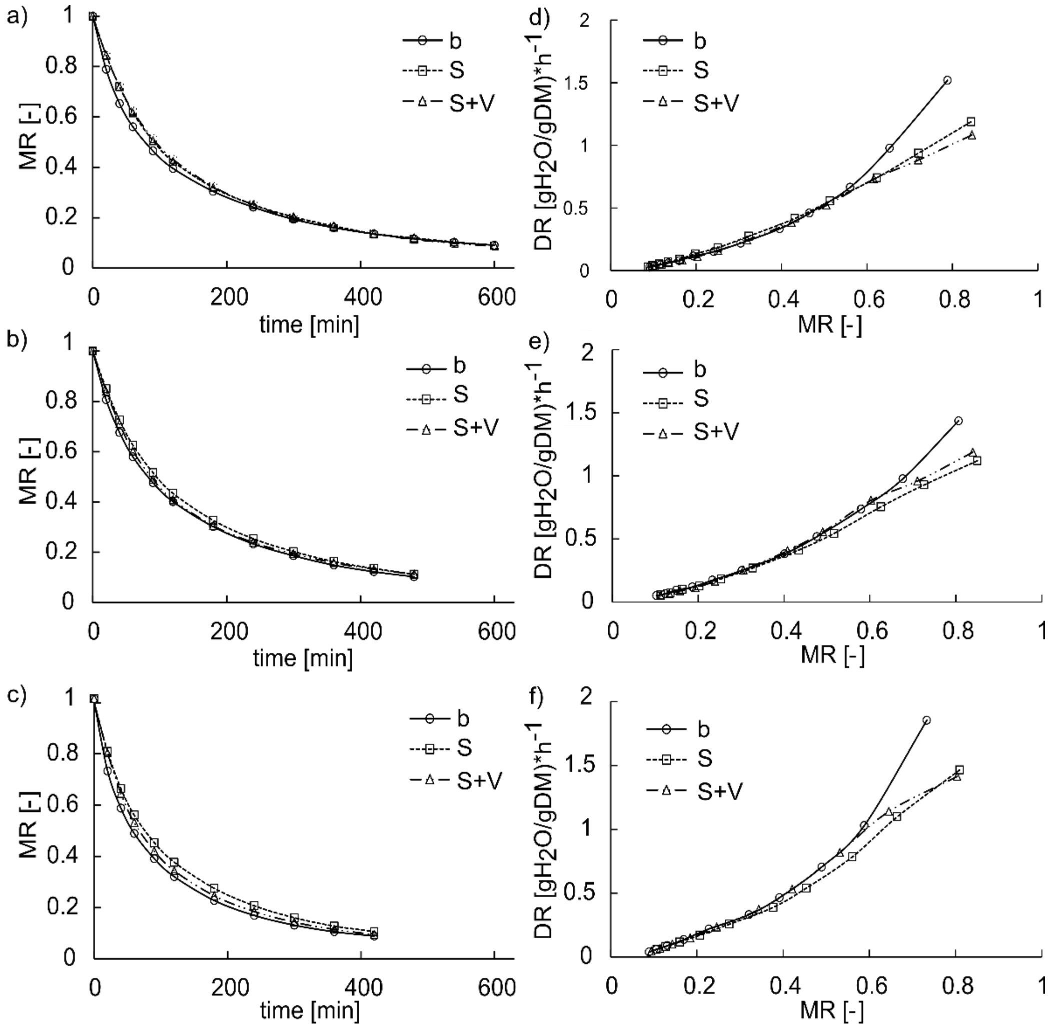

Figure 4-2: Drying curves and drying rates for blind samples (b), samples pre-treated with salt (S) and with salt and vinegar (S+V) dried at 50 °C (a), 60 °C (b) and 70 °C (c), d)-e) show the respective drying rates (DR)

The drying curves do not show visible differences between blind samples and those which were pretreated with S+V, which is contrary to the results in a previous study on dehydration on beef slices, where samples seasoned with S+V showed increased drying rates (von Gersdorff et al., 2018), compared to untreated samples. In the present study, for all drying temperatures, the blind samples showed the highest drying rate (DR) in the beginning of the drying process. However, for drying at 50 °C, the drying rate across the different pre-treatments developed similarly at a MR of 0.5, while drying at 60 and 70 °C resulted in similar DR only at a MR of 0.38. This can be explained by reduced heat and mass transfers at lower temperatures. The illustrated DRs and the development of MR are also not in accordance with the former study (von Gersdorff et al., 2018). Those different results regarding samples pretreated with S+V might be a result of the salt/acid concentration related to the beef, which is documented to influence the water holding capacity in different ways (Medyński et al., 2000). Medyński et al. (2000) showed that the drip loss is reduced for meat samples after acid treatment and above a certain salt concentration the drip increased for raw and cooked meat. In the present study, the applied salt concentration in the S+V treatment might have been too low and the effect of acid induced swelling of the muscle fibers might have led to an increased water binding during drying (Desmond, 2006; Teixeira et al., 2011). In addition, other factors such as the seasoning step influences the quality of the product as documented by Han et al. (2011). By comparing immersing and tumbling they found that curing time increases the transfer of the marinade into the meat. The dipping in the present study probably led to a reduced diffusion of the S+V solution into the beef samples compared to tumbling or immersing. Goli et al. (2014) described an approximation of the pH of the meat to the pH of the seasoning solution at longer immersion times. Additionally, different cattle breeds, sex and cut were used in the different trials, which could influence both the seasoning and the drying behavior, due to different characteristics and composition of the cut in question (Cafferky et al., 2019; Nassu et al., 2017).

4.4.2 Evolution of CIE color pattern

Although the seasoning influenced the instrumental color before drying, L*, a* and b* values were almost identical after drying for all pre-treatments with the samples appearing dark brown with almost identical ΔE with very low and nearly invisible temperature induced differences (Figure 4-3). ΔE was higher for samples dried at 50 °C and 70 °C than for samples dried at 60 °C. One reason might be that both, long drying times at low temperature as well as high temperatures negatively affected the quality, and, therefore, the instrumental color of the final product.

Table 4-1: CIELAB L*, a* and b* means and standard deviations after drying at 50 °C, 60 °C and 70 °C

		Drying temperature		
CIELAB	**Pre-treatment***	**50 °C**	**60 °C**	**70 °C**
L*	b	18.87^{c} (± 2.07)	19.56^{bc} (± 2.00)	21.06^{a} (± 1.23)
	S	19.16^{bc} (± 2.43)	20.17^{abc} (± 1.38)	21.04^{a} (± 0.92)
	S+V	20.26^{ab} (± 1.68)	20.20^{abc} (± 1.78)	21.12^{a} (± 1.37)
a*	b	3.96^{a} (± 1.55)	3.01^{bc} (± 0.32)	2.64^{c} (± 0.37)
	S	4.10^{a} (± 0.97)	2.91^{bc} (± 0.73)	2.67^{c} (± 0.31)
	S+V	4.07^{a} (± 1.10)	3.08^{bc} (± 0.61)	3.51^{ab} (± 0.59)
b*	b	2.43^{a} (± 0.96)	1.66^{c} (± 0.33)	1.86^{bc} (± 0.38)
	S	2.53^{a} (± 0.57)	1.61^{c} (± 0.50)	2.09^{abc} (± 0.38)
	S+V	2.54^{a} (± 0.80)	1.72^{c} (± 0.56)	2.34^{ab} (± 0.66)

*b, blind; S, salt pre-treatment; S+V, salt and vinegar pre-treatment, means with different superscripts ($^{a-c}$) are significantly different ($P < 0.05$ according to Tukey post-hoc test), standard deviations in brackets

The means and standard deviations of final CIELAB values are shown in Table 4-1. a* and b* values show a clear tendency that drying at 50 °C maintained redness and yellowness better than 60 and 70 °C. The development of the instrumental color in the present study might have been influenced by several factors: (i) A heat induced denaturation of myoglobin has been observed to be most dominant between 65-80 °C (Martens et al., 1982) which is in accordance with the highest final a* values achieved at 50 °C drying temperature compared to 60 °C or 70 °C. (ii) Heat treatments are known to increase the oxidization from oxi- and myoglobin to metmyoglobin (Bernofsky et al., 1959; Yin and Faustman, 1993) resulting in decreased a* values. The metmyoglobin formation decreases with the intensity of heat treatments, due to a shorter exposure time to oxygen which oxidizes the oxy- or deoxymyoglobin to metmyoglobin (Liu et al., 2018).

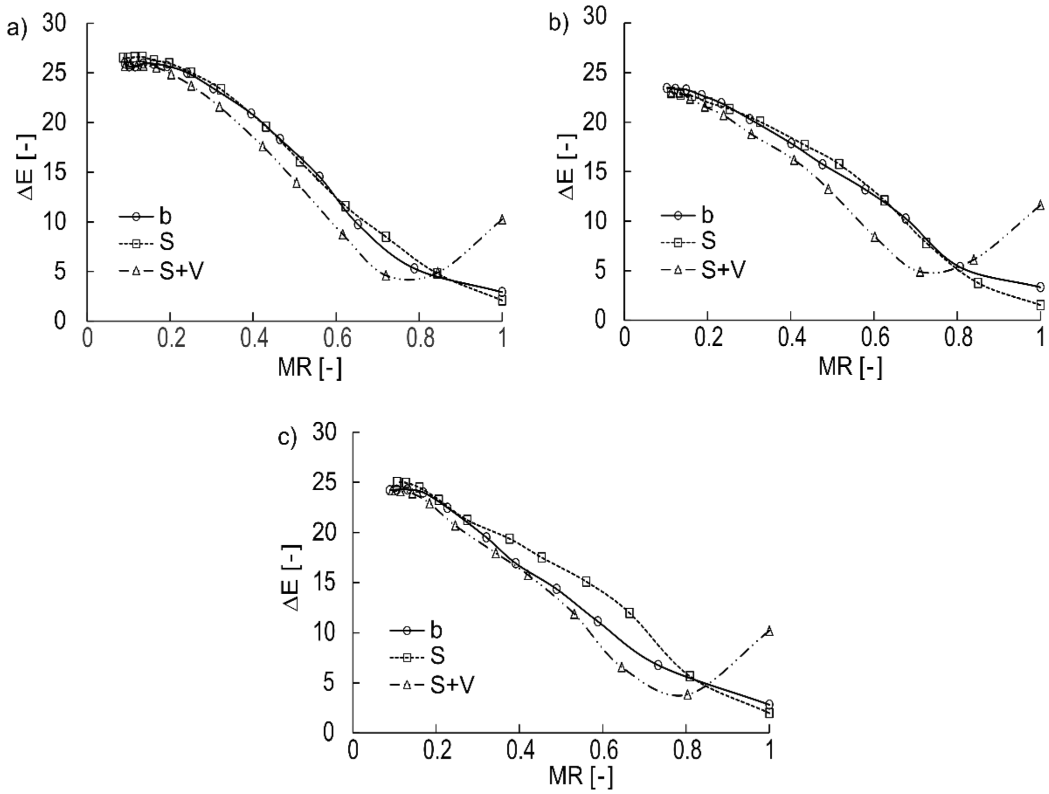

Figure 4-3: Development of the total color change ΔE for beef slices for blind samples (b), pre-treated with salt (S) or salt and vinegar (S+V) dried at 50 °C (a), 60 °C (b) and 70 °C (c)

Dehydration, therefore, results in a high level of formation of metmyoglobin due to relatively long processing times. (iii) It has been reported that impaired lightness was more likely occurring in such a case that the dehydration process greatly increased the concentration of pigments (i.e. myoglobin, oxymyoglobin, and metmyoglobin) (Ferrini et al., 2012). (iv) Lipid oxidation is caused by several factors during processing. Heat treatments have been shown to increase lipid oxidations in meat but have also been determined to be dependent on the duration of the heat treatment (Domínguez et al., 2014). Lipid oxidation and has been shown to be positively correlated to L* values (Luciano et al., 2009), which might explain the significantly higher L* values for samples dried at 70 °C in the present study. The salt induced oxidation of lipids and, thus, increased L* values did not show a significant influence regarding lightness in the present. (v) Acid treatments are known to induce myoglobin denaturation and, thus, to different relations of myoglobin forms inside the meat (Fernández-López et al., 2004). This effect is obviously in the present study regarding the high ΔE due to high L* and low a* values after seasoning with S+V, but low pH treatments did not significantly influence the final color. (vi) Shrinkage might have led to measuring mistakes of instrumental color due to resulting uneven surfaces, which could, at least partially, explain higher L* values for samples dried at 70 °C. Tornberg (2005) reviewed the influence of heat during cooking on sarcomeric, myofibrillar and connective tissue proteins of meat and summarized that increasing temperature increases the degree of shrinkage. In the present study, shrinkage was not taken into consideration and, therefore, the theory of a measuring errors is speculative.

The changes of physicochemical parameters like protein denaturation or oxidation processes that influence the instrumental color of beef during drying have not been investigated in the present study but should be regarded in a future study and linked to instrumental color to gain a deeper understanding of changes of quality parameters in terms of non-invasive product monitoring and process control.

4.4.3 Water activity

The results of the water activity a_w and the related standard deviations are shown in Table 4-2. According to Fontana´s, (2007) definition, all experiments led to stable products regarding bacterial growth ($a_w < 0.84$). The pre-treatment did not significantly ($p < 0.05$) influence the aw, but the results indicate that drying at 60 °C might lead to higher aw values compared to samples dried at 50 or 70 °C. However, due to a low number of samples analyzed in this study and different results regarding pH and a_w of a final meat product documented by Petit et al. (2014), it is unfeasible to deduce a clear connection, but might show that a_w is a function of several characteristics of the final product (chemical components, physicochemical state, porosity, temperature, and surface tension) (Rahman and Labuza, 2007).

Table 4-2: Water activity (a_w) means and standard deviations of blind beef slices (b), slices pre-treated with salt (S) and salt and vinegar (S+V) after drying at 50 °C, 60 °C and 70 °C

Drying temperature Pre-treatment*	50 °C	60 °C	70 °C
b	0.76^{ab} (± 0.04)	0.79^{a} (± 0.06)	0.76^{ab} (± 0.04)
S	0.73^{b} (± 0.06)	0.79^{a} (± 0.04)	0.76^{ab} (± 0.03)
S+V	0.74^{ab} (± 0.05)	0.77^{ab} (± 0.06)	0.73^{b} (± 0.03)

*b, blind; S, salt pre-treatment; S+V, salt and vinegar pre-treatment, means with different superscripts ($^{a-b}$) are significantly different (P < 0.05 according to Tukey post-hoc test), standard deviations in brackets

4.4.4 Spectral analysis

Applications of HSI regarding prediction of meat quality parameters already exist. However, investigations on time-series applications for beef drying are rare and limited to only a single drying condition (Retz et al., 2017; von Gersdorff et al., 2018; Wu et al., 2013). As seen in Figure 4-2 and 4-3, different pre-treatments and drying conditions have an impact on the development of product MC and ∆E during drying of beef slices. Figure 4-4a shows the averaged relative reflectance of beef slices prior to the pre-treatments and storage over night, after the pre-treatments with S or S+V or kept untreated as b and after drying at 50 °C, 60 °C and 70 °C (Figure 4-4b).

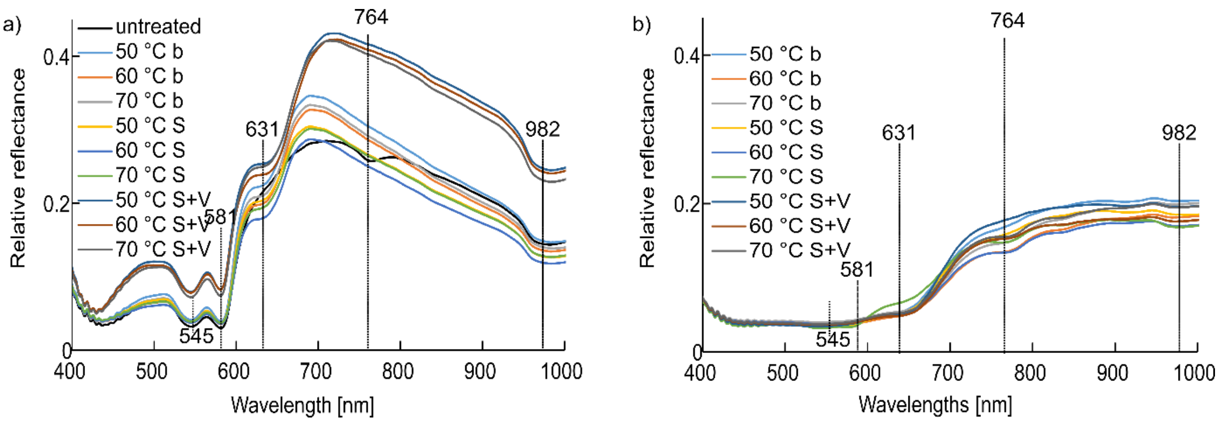

Figure 4-4: Averaged reflectance spectra of beef untreated and with different pre-treatments (b, blind; S, salted; S+V, salt and vinegar) before drying (a) and after drying (b) at three different temperatures. The meat specific reflectance wavelengths are highlighted

As shown in Figure 4-4a, the absorption peaks can be divided into two categories. Those wavelengths beyond 700 nm correspond to water absorption while in the visible area (lower than 700 nm) pigments are mostly dominant. The spectra of the untreated samples showed two absorption peaks at 760 and 970 nm which are related to the third and second overtones of O-H stretching vibrational modes, respectively (Yang et al., 2017). After storage overnight and different pre-treatments, the weak absorption peak at 760 nm disappeared probably due to the water insulation by the salt. Different muscle pigments lead to absorption maxima and dips in the reflectance spectra. Those appear at around 430 nm and 560 nm for deoxymyoglobin, for oxymyoglobin at 540 nm and 580 nm and for metmyoglobin around 410, 500 and 630 nm which was investigated and compared by Millar et al. (1996). While the dips for deoxymyoglobin and oxymyoglobin became visible, the dips related to metmyoglobin were not that prominent in the present study. The absorption at 630 nm might indicate the oxidation from oxymyoglobin and deoxymyoglobin to metmyoglobin, since it only occurred after storage overnight for all treatments. Similarly, Liu et al. (2018) observed absorption at 620 nm after 45 s of microwave heating, which showed in consistency with our claim the oxidation from deoxymyoglobin or oxymyoglobin to metmyoglobin during processing. Furthermore, it can be seen from Figure 4-4a that the samples pretreated with salt and vinegar showed the highest reflectance because decreasing pH values led to the increase of light reflectance (Serdaroğlu et al., 2007). It is worthwhile to note that a significant level of noise (visible in Figure 4-4a and b) was related to the range of 400-500nm, probably owing to the poor illumination provided by the halogen bulbs used. As a result, only the spectral range of 500-1000 nm was included into further analysis.

After the drying process, the reflectance spectra were almost identical independent of the pre-treatment (Figure 4-4b). This probably resulted from an increasing concentration of pigments accompanied by the decreased lightness values caused by the loss of moisture. Additionally, the chemical changes of myoglobin to met-, oxy- and deoxymyoglobin might also partially explain such effect (Liu et al., 2018).

The selection of wavelengths can improve the performance of a prediction model and further simplifies a model since it avoids the repetition of spectral information due to multi-collinearity of hyperspectral data (ElMasry et al., 2012) and, thus, reduces the high dimensionality of the hyperspectral data and the time for the acquisition of spectral and spatial data (Xing et al., 2006). Therefore, it saves computation time (Kamruzzaman et al., 2016), which makes it more applicable for real-time monitoring. For MC, five wavelengths were selected (512, 645, 745, 780 and 975), for lightness seven wavelengths (502, 544, 564, 580, 634, 699 and 929), for redness (a*) five wavelengths (532, 566, 590, 649, and 702) and for yellowness (b*) 4 wavelengths (546, 580, 642 and 709). The results show that the visible and the NIR range implies specific wavelengths for MC and CIELAB L* prediction, while for CIELAB a* and b* prediction they have been found only in the visible range.

The selected wavelengths were used to build predictive models, their computed results are shown in Figure 4-5 with key parameters describing a good model performance (R^2 and RMSEP) illustrated in the right hand side of Table 4-3.

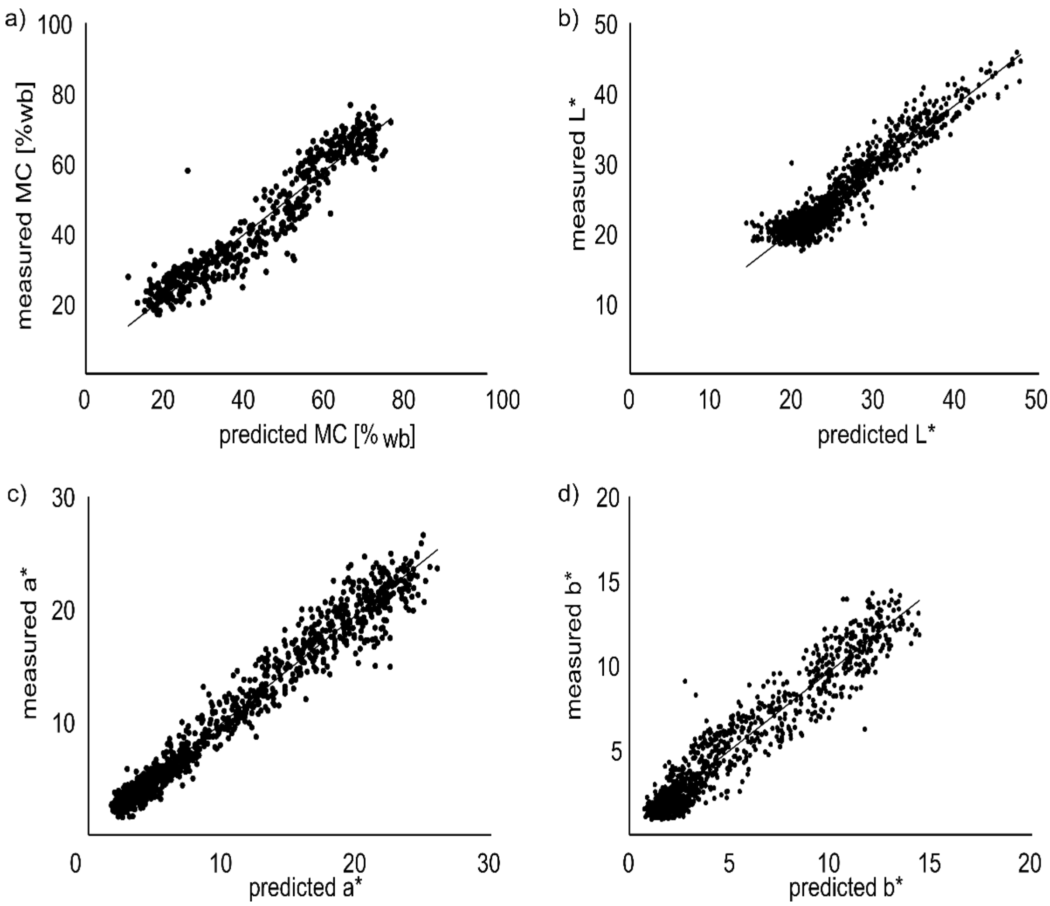

Figure 4-5: Measured vs. predicted MC, L*, a* and b*values using PLSR models with spectra from selected wavelengths from beef slices treated with different pre-treatments (b (blind), S (salt), S+V (salt and vinegar)) and dried at 50, 60 and 70 °C

The developed models for the different parameters show a good performance regarding high R^2s and low RMSEPs. Table 4-3 shows the slight improvement of the prediction models between the usage of the full spectral range and the reduced sets of wavelengths. Regardless of drying temperature or pre-treatment, the models can predict the quality parameters with a high accuracy. Thus, unlike using selected sets of wavelengths depending on the pre-treatment before beef drying (Retz et al., 2017; von Gersdorff et al., 2018), the results of the present study enable the usage of spectral imaging to monitor MC and instrumental color in real-time during drying process.

Table 4-3: Comparison of performance indicators of PLSR models developed from full and reduced sets of wavelengths and independent of pre-treatment and drying temperature for prediction of moisture content (MC), CIELAB L*, a* and b* values of beef slices during drying

Parameter	Full set of wavelengths		Reduced set of wavelengths	
	R^2	RMSEP	R^2	RMSEP
MC	0.944	5.758	0.953	5.263
L*	0.947	1.998	0.948	1.975
a*	0.978	1.605	0.982	1.475
b*	0.959	1.1	0.963	1.05

R^2 = coefficient of determination, RMSEP = root mean square error of prediction

The results will support the development of monitoring systems and to use the data gained from non-invasive MC and color measurements to link them to physiochemical changes to be investigated in future work to improve drying strategies for high quality dried beef. The results further offer the potential to be used regarding the development of a multispectral imaging system. Regarding cost-effective monitoring solutions the outcome of the present study can be transferred to a simpler application, consisting of selective lighting combined with CCD cameras and, thus, enable also small scale processors to use non-invasive measurement systems in processing.

4.5 Conclusions

In this study, pre-treatment and drying temperature showed a joint influence on the drying behavior and instrumental color changes during the dehydration of beef. The samples pre-treated with salt solution dried the slowest, followed by salt and vinegar treatment, while blind samples dried the fastest. This effect increased slightly with increasing drying temperature. ΔE was the lowest for samples dried at 60 °C and did not visible differ across the pre-treatments. Although the drying behavior and the instrumental color difference varied between the experiments, the hyperspectral data acquired during drying allowed the development of robust PLSR prediction models for MC, CIELAB L*, a* and b* values independent of drying temperature or pre-treatment. An exclusion of uninformative wavelengths by selection of parameter specific wavelengths led to simpler models and, therefore, the ability to acquire smaller amounts of data while maintaining the accuracy of prediction. Further, an inclusion of other beef cuts and, in general, meat varieties as well as other quality parameters could be of future interest to predict product parameters during drying by varying prediction models in an industrial significance and in regard to product controlled drying for beef

4.6 Acknowledgement

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4.7 References

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5 Method comparison between real-time spectral and laboratory based measurements of moisture content and CIELAB color pattern during dehydration of beef slices*

5.1 Abstract

In this study, partial least square (PLS) regression models were developed to predict moisture content (MC) (model 1), CIELAB color (model 2) or all four parameters (model 3) of beef slices during drying. Model development was based on data from two measurement campaigns of MC (%), CIELAB L*, a* and b*values and hyperspectral data in the range of 500 – 1009 nm. To increase the robustness of the models, the beef samples varied dependent on cattle breed, cut and pre-treatment. With low-cost, non-invasive continuous monitoring systems in mind, the models were simplified by wavelengths selection. The Deming and Passing-Bablok regression and the Bland-Altman plot revealed high model performances. Mean differences (full/reduced model) of -0.64/-0.64 for MC, -0.14/-0.15 for CIELAB L*, 0.05/0.04 for a* and 0.08/0.06 for b* values were achieved for model 3, which shows the high potential for simple real-time monitoring applications combining all investigated factors and parameters.

Key words: beef drying, hyperspectral imaging, method comparison, non-invasive measurement, real-time measurements, PLSR

5.2 Introduction

Drying is one of the oldest preservation techniques used for food stuffs. Recently, smart drying processes have gained increasing interest in the drying community. Smart drying has the potential to generate tailored and, thus, efficient solutions in terms of increasing process efficiency and final product quality, reducing the environmental impact and post-harvest losses (Martynenko and Bück, 2018; Sturm, 2018a, 2018b). A core aspect of smart drying is the utilization of information on dynamic changes occurring inside the product during the process for optimized control of the process. To do so requires continuous monitoring of the product during processing. To avoid an interruption of the process, and to achieve a quick

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determination of product parameters, non-invasive measurements are imperative for real-time monitoring and, thus, for smart drying applications based on individual characteristics of the product at hand (Su et al., 2015).

Hyperspectral imaging (HSI) has gained increased interest over recent years regarding the development of non-invasive technologies and techniques. HSI provides a great amount of information which can be used for the development of simpler practical applications in terms of wave length reduction. HSI has successfully been applied to measure quality in raw and processed meat products, such as the prediction of protein and water content in cooked ham (Talens et al., 2013), the characterization of intramuscular fat distribution in beef (Lohumi et al., 2016), intramuscular fat content prediction in pork (Liu and Ngadi, 2014), real-time monitoring of chicken (Kamruzzaman et al., 2016a) or horse meat adulteration (Kamruzzaman et al., 2015) in minced beef was realized and water injection into beef was detected successfully by HSI (Liu et al., 2016). Crichton et al. (2017a) were able to differentiate maturation stages (e.g. 7 and 21 days) and freezing pre-treatments of beef, as well as unusually high post-slaughter pH (Crichton et al., 2017b) by visible and near-infrared (VNIR-) HSI. Freshness is always a concern regarding fresh meat and is expressed by specific compounds, which were successfully evaluated non-invasively for frozen-thawed pork (Wu et al., 2016), refrigerated chicken (Feng et al., 2018) and even for the microbial contamination of pork (Barbin et al., 2013; Tao and Peng, 2014). Time-series VNIR HSI has been investigated for salting of pork patties in terms of salt content and prediction of water activity (a_w) (Liu et al., 2013). Liu et al. (2018) used VNIR-HSI to monitor the moisture content and color development during microwave heating of beef. However, Images acquired by HSI require significant computation time in terms of image processing and, thus, HSI is difficult to implement in continuous real-time measurement applications. The utilization of specific wavelengths reduces the computation time (ElMasry et al., 2011), and promotes the robustness of the prediction models (Xiong et al., 2015), which enables simpler and quicker data collection and ultimately leads to more cost-efficient systems.

As HSI provides a great number of independent, collinear predictors (wavelengths), the development of partial least square regression (PLSR) models is a common technique to describe the maximum correlation between the wavelengths and the observations (Wold et al., 2001) by reducing the predictors to non-collinear components. Often, the correlation coefficient *r* is used to evaluate the accordance of predicted data and data gained from the reference method. However, *r* simply describes the linear association between pairs of values and it is hardly surprising that two methods that measure the same variable usually show a good correlation (Giavarina, 2015). Thus, other parameters are required to evaluate if the differences between two measurements are at a tolerable level. Ordinary least square regression (OLS) assumes that there is no measurement error in the X direction (reference method) but only in the Y direction (“new” method), which leads to biased slope estimates and misleading assumption of hypotheses regarding the accordance of methods (Linnet, 1998). Therefore, if regression is used to compare two methods, measurement errors should be assumed for both variables. Two common regression techniques that fulfill the requirements in terms of measurement uncertainties in both directions are the Passing-Bablok (PBR) (Passing and Bablok, 1983) and the Deming regression (DR) (Deming, 1943). Regression analyses can further be supported by a difference plot, e.g. Bland-Altman (Bland and Altman, 1986) to interpret the obtained results.

The moisture content (MC) of a product represents an important parameter to avoid over drying or insufficient drying of beef. The color of meat products is related to internal quality parameters of meat products (Ni et al., 2020) and represents an important attribute for consumers’ perception of quality (Font-i-Furnols and Guerrero, 2014). The understanding of changes with regard to MC and color parameters during drying necessitates continuous product monitoring and enables the integration of the observations into the development of drying strategies. Alternatively, data gained from monitoring can be used directly as control parameters to adjust the drying parameters (smart drying). Several studies exist regarding time series HSI for beef during drying in terms of MC or CIELAB color prediction, respectively (Retz et al., 2017; von Gersdorff et al., 2018, Wu et al., 2013). Little is, however, reported with regard to increasing model robustness which can be achieved by the inclusion of different sample varieties or drying conditions as was shown for prediction models for moisture content during tuber (potato and sweet potato) drying (Su et al., 2020). In beef drying the inclusion of different raw materials (breed, gender, cut and cold storage) and treatments (seasoning, drying temperature) can be assumed to increase the model robustness for MC and color prediction. Furthermore, to our knowledge, no methods comparison has yet been applied to prove the applicability of spectral time-series measurements as a continuous monitoring device of MC and CIELAB color pattern during dehydration of beef slices. Based on the extensive datasets of two previous beef drying studies (von Gersdorff et al., 2018, unpublished results), the objective of the present study was to 1) develop prediction models for either MC or CIELAB L*, a* and b* values or for MC and CIELAB color pattern of beef slices during drying based on HSI data independent of random and systematic variance components (raw material, pre-treatment etc.), to 2) reduce the models by wavelengths selection to enable simpler and cost effective, but robust applications and to 3) apply methods comparisons to evaluate the performance of the models (full and reduced sets of wavelengths) and their potential in terms of continuous, non-invasive real-time applications for determination of MC and CIELAB L*, a* and b* values of beef during drying.

5.3 Materials and methods

5.3.1 Raw material

For the experiment, the beef of seven cattle was used. To increase the variance components and the according robustness of the prediction models, beef of different origins (breed, gender, cut, freezing treatments) was utilized.

2 kg fresh (stored for one week at 4 °C after slaughter) and 2 kg matured (maturation at 4 °C for 21 days) (± 0.25 kg) upper shell of three two-years old crossbreed bulls (Limousin x Simmental) were used. 1 kg of the fresh and matured beef, respectively, was used immediately after purchase for the experiment, the remaining part was frozen at -18 °C and stored for two weeks in a freezer (Liebherr GP 1486 Premium, Germany). From a second cattle breed (Uckermarker) 3 x 1 kg portions (± 0.35 kg) of roast beef of four two-year old heifers were used. The beef was vacuumed and immediately shock frozen two days after slaughter at -55 to -60 °C for 120 minutes with cold air in a cabinet and stored after purchase in the freezing unit at -18 °C.

5.3.2 Pre-treatments

Before each drying experiment, the 1 kg portions of beef were sliced in the direction of the fiber with a slicing machine (Graef, 116 Allesschneider Vivo V 20, Germany) to a thickness of 5 mm and cut to samples of 50 mm x 50 mm with a sharp knife. The frozen beef was thawed for 18 hours at 2 °C in a fridge (FKUv 114 1613 Premium, Liebherr, Germany) before slicing. From the crossbreed beef, 32 samples were cut out of each 1 kg, the Uckermarker beef resulted in 24 samples of which each 8 samples were pre-treated differently with four (crossbreed) and three pre-treatments (Uckermarker), respectively. 2 different salt concentrations were applied to the crossbreed beef samples (0.5 % and 1 % m/m) by sprinkling it directly on the slices. The samples of the Uckermarker were dipped in a 10 % (m/v) salt solution. Regarding the salt and vinegar pre-treatment, the samples were dipped in a solution prepared with salt and apple vinegar (10 % m/v). As a reference 8 samples per trial were kept untreated as blind samples. The samples were stored overnight in the fridge at 2 °C. Each animal represented a repetition which resulted in 48 data sets regarding the crossbreed cattle and 36 data sets regarding the Uckermarker.

5.3.3 Drying and measurements

The samples were dried in a convection dryer (HT mini, Innotech Ingenieursgesellschaft mbH, Germany) at 70 °C (crossbreed) and 50, 60 and 70 °C (Uckermarker), respectively, and an air velocity of 0.6 m/s. The beef samples were weighed before and during drying until an estimated MC of 14-20 % wet basis (MC_{wb}) was reached. After the drying process, the samples were dried in an oven at 105 °C for 24 hours (AOAC, 2016) to calculate the MC_{wb} at every measuring point. Further, the CIELAB color pattern (CIE, 2018) was measured with a Chroma Meter (CR-400, Minolta, Japan) at three points of each sample with a D65 illuminant, which was calibrated with a standard ceramic plate. The three CIELAB L*, a* and b* values were averaged for each sample.

The measurements of the crossbreed beef started before drying and continued hourly during drying. The Uckermarker beef samples were measured directly after slicing, before drying and every 20 min in the first hour of drying, every 30 min in the second hour and hourly afterwards. From the measured data, the MC_{wb} at every time interval was calculated according to Bradley (2010) using equation 1, referred to as MC onwards:

$$\mathrm{MC_{wb}} = \frac{\mathrm{m_w}}{\mathrm{m_t}} \text{ x } 100 \tag{1}$$

where m_w is the mass of water inside the sample at every time and m_t is the total mass of the sample at every time. Due to the experimental set up and the related measurement of water activity (a_w) of the dried beef samples, only 4 of each 8 samples were available for final MC estimation.

The samples were imaged with a hyperspectral camera combined with a linear translation stage (ImSpector V10E PFD, SPECIM Spectral Imaging Ltd., Finland) to collect the spectral data. Three 60 W halogen bulbs provided the illumination source to capture the wavelengths in the region of 500 - 1009 nm in increments of 1.5 nm which results in a full set of 323 wavelengths. A 35 mm lens was positioned at a distance of 27 cm to the samples, which were moved by a conveyor tray with a speed of 8 mm/s. The images were calibrated with a white and a dark reference and images were processed with MATLAB 2013b software at a Dell Precision T5500 workstation with 32 GB main memory (Dell Technologies, Round Rock, Texas, USA) to gain the average reflectance of each sample, which is described in detail by von Gersdorff et al. (2018).

5.3.4 Development of PLSR models

The data was transferred to JMP® pro 15.0.0 software to develop the PLSR models. Due to the experimental setup, fewer data points were available for MC (four slices per pre-treatment) than for CIELAB L, a* and b* values (eight slices per pre-treatment). Therefore, PLSR models

1 and 2 were developed to predict only MC or only CIELAB color pattern, respectively. However, as the measured parameters are correlated, it was worthwhile developing PLSR model 3 predicting MC, CIELAB L*, a* and b* values based on the data containing MC and CIELAB of only 4 beef slices per pre-treatment.

The data of the four or eight samples was averaged for further analyses. Subsequently, the data was split into a training, a validation and a test set (70/20/10), replacing a cross validation. The randomization/stratification of the data to divide the data into the different sets was based on the factors ‘animal’, ‘breed’, ‘aging/storage’, ‘pre-treatment’, ‘drying temperature’ and ‘drying time’.

The development of the PLSR models was based on the NIPALS algorithm (Nonlinear Iterative Partial Least Squares) that reduces the number of predictors (wavelengths) by extracting components that explain most of the variance of all variables. Initially, 1 - 15 components were used. The PRESS (PRedictive Error Sum of Squares) was used to decide on the final number of principal components to be included in the models (Sawatsky et al., 2015).

5.3.5 Wavelengths selection

Important wavelengths of MC and CIELAB L*, a* and b* values were selected by the VIP (Variable Importance in the Projection) method, which evaluates which wavelengths have the most influence on the model and usually only VIP with a score of at least 1 are considered (Wold et al., 2001). However, according to Andersen and Bro (2010), values below 1 should not simply be removed. Thus, in the present study, the selection was made regarding the centers of the peaks (Stellacci et al., 2016) with scores of at least 0.8. The reduced sets of wavelengths were used to build simpler PLSR models (1r - 3r) according to the three data sets.

5.3.6 Methods comparison

The trained and validated PLSR models from the full and reduced wavelengths sets were tested with the test subset of data. The null hypothesis of both measurements to be equivalent was evaluated by calculating PBR and DR. The agreement of both methods is displayed by the regression line that expresses the regression equation with an intercept close to 0 and a slope close to 1. The bias between both methods is visualized as the deviation between the regression line and the equality line (intercept=0, slope=1). The residues are regarded as normally distributed and independent. The measurement error ratio is assumed to be constant in the DR, while for PBR no specific deviation of single observations and residues is assumed and slope and intercept are calculated on the basis of the median of all slope triangles for all possible pairs of values. This leads to a more robust regression against outliers compared to

DR. Both, DR and PBR scatter plots enable an evaluation of the linearity and systematic measurement errors through intercept, slope and the spread of data pairs around the regression line. The Bland-Altman (BA) plot represents the difference between the two measurements as a function of the mean of two measurements, which enables an evaluation of the behavior of the difference (Giavarina, 2015). This plot allows a quantification of agreement by showing the limits of agreement (LOA) which is the mean difference ± 1.96 standard deviations (SD) and, thus, shows the limits in that 95 % of the differences between two methods can be expected (Bland and Altman, 1986). The statistical significance of the deviations of intercept (DR, PBR) and mean difference (BA) from 0 and of the slope from 1 (DR, BPR), respectively, is proven by confidence intervals which become narrower with increasing test sets, which will, however, not affect the LOA (Bland and Altman, 1986).

5.4 Results and Discussion

5.4.1 Moisture content

The measured MC ranged from 74.78 % in fresh to 13.34 % in dried samples. As an example, Figure 5-1 representatively shows the averaged MC at each measuring interval point of beef slices treated with S+V related to the two experiments with two different maturation durations and their respective frozen version or three different drying temperatures, respectively. The MC decreased throughout the drying, however, it is obvious that cut, cold storage after slaughter and drying temperature, but also the different amount of measurement intervals during the first two hours of drying impacted the drying behavior.

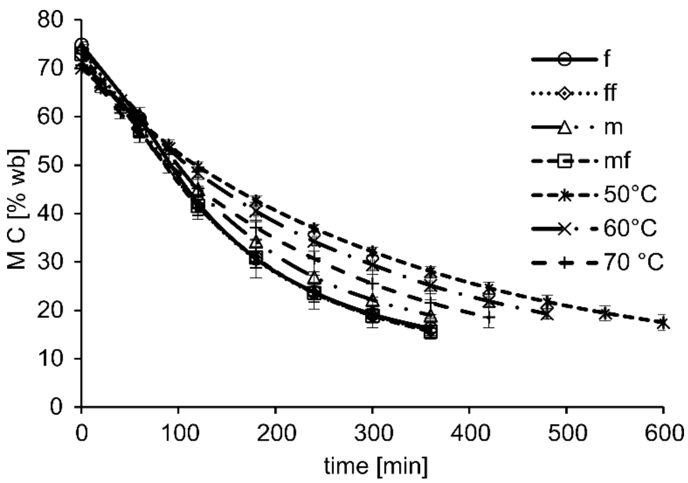

Figure 5-1: Averaged moisture contents (MC) ± standard deviation at each measurement point of beef slices seasoned with salt and vinegar (S+V) for fresh (f), fresh frozen-thawed (ff), matured (m) and matured frozen-thawed (mf) samples of crossbreed beef dried at 70 °C (n = 3) and for samples of Uckermarker beef dried at 50 °C, 60 °C and 70 °C (n = 4)

5.4.2 Wavelengths selection

Related to the least root mean PRESS the PLSR models for the full wavelengths sets were built of 11 components (models 1 + 2) and 13 components (PLSR model 3).

The VIP score of PLSR model 1 led to the selection of eight wavelengths (511, 546, 564, 581, 604, 690, 780, 975 nm) developing PLSR model 1r for the prediction of MC, as well as for model 2 (500, 543, 564, 581, 654, 656, 557, 983 nm) to develop PLSR model 2r to predict CIELAB L*, a* and b* values. Figure 5-2 shows the VIPs for prediction of MC, CIELAB L*, a* and b* values related to PLSR model 3 with ten wavelengths (500, 518, 543, 564, 581, 606, 628, 666, 780, 978 nm) marked according to the peak VIP scores that were selected for the development of a simplified model (PLSR model 3r). Some of the selected wavelengths correspond to absorption peaks that are related to specific bonds, e.g. around 760 nm and 970 nm, for the O-H stretching overtones (Yang et al., 2017), and related to the myoglobin derivates like oxymyoglobin (540 nm and 580 nm), deoxymyoglobin (430 nm and 560 nm) and metmyoglobin (410, 500 and 630 nm) (Millar et al., 1996). However, when comparing the selected wavelengths according to the data sets, a repetition of some specific wavelengths, which might be due to the correlation of the parameters, is evident and might already indicate the potential of a single PLSR model to predict MC, CIELAB L*, a* and b* values.

Evaluated by the PRESS PLSR model 1r required eight, PLSR model 2r six and PLSR model 3r nine components.

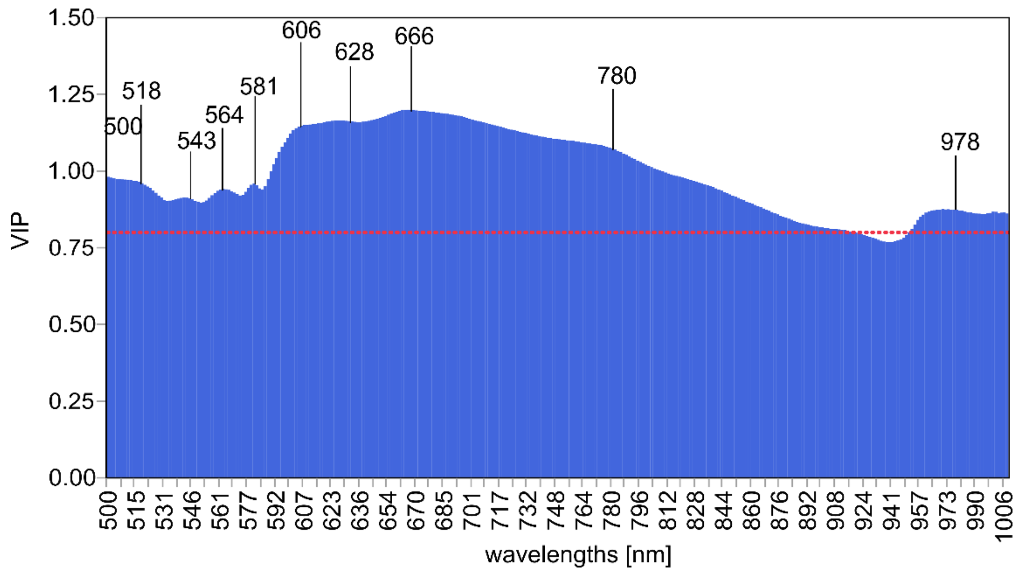

Figure 5-2: Variable Importance of Protection (VIP) plot of the full range of wavelengths of PLSR model 3 for prediction of MC, CIELAB L, a* and b* values. The centers of the peaks with scores >0.8 are marked according to the related wavelengths to be selected for a reduced PLSR model of data set 3

5.4.3 Methods comparison

The prediction results gained from the full and reduced set of wavelengths were used to compare the spectral measurement with the reference method for MC and CIELAB, respectively, utilizing the DR and PBR (Figure 5-3 – 5-6). The closer the intercept is to 0 and the slope to 1, the better the accordance of both methods can be regarded. The results of the DR are further displayed in a modified way in a BA plot (Figure 5-3 – 5-6). This plot further describes the mean difference and displays the LOAs represented by the mean difference ± 1.96 (SD) to evaluate the significance of differences between two methods by studying the differences and not their agreements (Giavarina, 2015). DR, PBR and BA plot were already used to clearly interpret the results of spectral and laboratory measured parameters in the context of apple drying (Shrestha et al., 2019, 2020). The relevant parameters regarding DR, PBR and BA are shown in Tables 5-1 – 5-4 to enable a precise interpretation of the graphs.

5.4.3.1 Moisture content prediction

Figure 5-3 presents the DR and PBR results of predicted and conventionally measured MC related to each developed PLSR model. The prediction results of PLSR model 3 with the full range of wavelengths predicts the MC slightly better than the PLSR model 1 expressed in an intercept not significantly different from 0 for the PBR and a slope not significantly different from 1, while for the results of data set 1 the intercept and the slope are just significantly different from 0 and 1, respectively. The model performance decreased slightly for both data sets at reduced wavelengths, however, intercept and slope for DR and PBR are not significantly different regarding PLSR model 3r, while for PLSR model 1r only the intercept related to the PBR is not significantly different from 0, while the slope at both regression techniques is just significantly different from 1. However, the null hypothesis that both methods for measuring MC are equivalent is rated as still acceptable related to PLSR models 1 and 3 and the reduced models 1r and 3r, respectively. Further, the correlation coefficient r from the DR is very close to 1, independent of the data sets used for model development, which shows a good linear relationship between measured and predicted values. The proof of accordance of both methods is strengthened by the BA plots. The mean difference between measured and predicted MC is not confidentially different from 0 for the results of PLSR models 1 and 3, however, the reference method measures on average higher MCs, which led to a negative mean difference. Further, no trend is visible and the plotted values do not show any curvature or irregular patterns, which indicates no systematic error for the reference or prediction method.

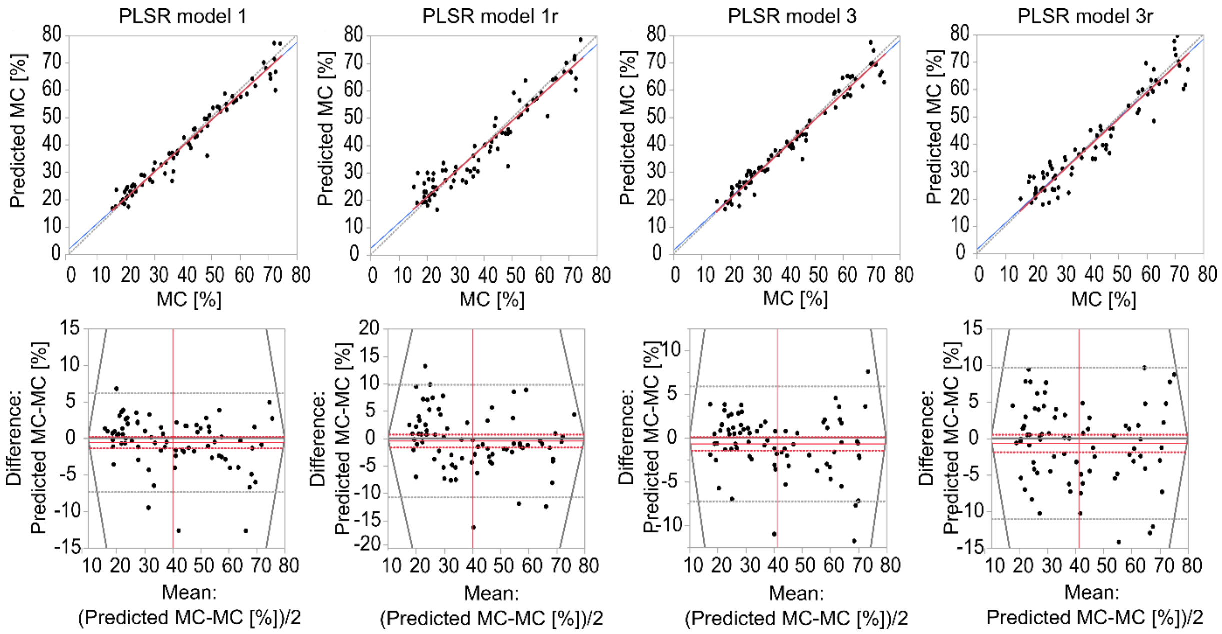

Figure 5-3: Results of methods comparison for wet based moisture content (MC % wb) predicted with PLSR models of full (1 and 3) and reduced sets of wavelengths (1r and 3r) vs. conventionally measured.
Above: Deming regression (DR) red line, Passing Bablok regression (PBR) blue line. Dashed grey line: line of equality. The respective Bland-Altman plots underneath show the difference between both measurements vs the mean value of predicted and conventionally measured values. The grey line shows the line of equality, the red line the mean difference (bias) with the dashed red lines representing the 95 % confidence interval (CI) around the mean difference and the dashed grey lines representing the limits of agreement (LOA) which is the mean difference ± 1.96 SD

The mean difference regarding PLSR models 1r and 3r led to increased SDs, which widened the LOAs. The LOAs do not specifically allow to evaluate two methods to be equal or not, but define the limits in that 95 % of the differences of both methods lie, but needs an a priori definition, on how much difference is tolerable.In the present study, the majority of the measured data by spectral measurements are expected to differ in a range of 5.91 % MC and -7.19 % MC when using the full range wavelengths and 9.72 and -11 % MC for PLSR model 3, respectively. The LOA of the reduced models (1r and 3r) are slightly wider than for the full wavelengths models (Table 5-1).

Table 5-1: Key parameters of methods comparison of moisture content (MC) measurements (conventionally vs. predicted by PLSR model) of beef slices

Method comparison		PLSR model			
		1r	1r	3	3r
Deming Regression	Intercept	1.54	2.62	0.80	0.76
	Slope	0.95	0.92	0.97	0.97
	Lower/Upper CI	0.91/0.99	0.86/0.99	0.92/1.00	0.90/1.04
	r	0.98	0.96	0.98	0.96
	Orthogonal fit ratio	0.90	0.85	0.93	0.93
Passing-Bablok Regression	Intercept	1.76	2.27	1.25	1.40
	Lower/Upper CI	0.34/2.71	-0.36/4.76	-0.57/2.91	-1.22/3.71
	Slope	0.94	0.93	0.95	0.96
	Lower/Upper CI	0.91/0.99	0.87/0.99	0.92/0.96	0.89/1.04
Bland-Altman	Mean Difference	-0.57	-0.43	-0.64	-0.64
	Lower/Upper CI	-1.35/0.22	-1.62/0.76	-1.40/0.12	-1.84/0.56
	SD	3.45	5.24	3.34	5.29
	Lower/Upper CI	2.90/4.00	4.41/6.08	2.81/3.87	4.45/6.13
	Upper LOA	6.20	9.85	5.91	9.72
	Lower/Upper CI	4.86/7.54	7.81/11.88	4.61/7.20	7.67/11.77
	Lower LOA	-7.33	-10.71	-7.19	-11.00
	Lower/Upper CI	-8.67/-5.99	-12.75/-8.68	-8.49/-5.89	-13.05/-8.95

CI = confidence interval 95 %, r = correlation coefficient, LOA = limit of agreement, SD = standard deviation, PLSR model 1 and 3 = full models, PLSR models 1r and 3r = reduced models

5.4.3.2 CIELAB color pattern prediction

The laboratory measurements ranged from 16.64 – 47.35 for L*, 2.17 – 24 for a* and 0.95 – 13.63 for b*. Figure 5-3 - 5-5 show a high correlation between measured and predicted values regarding CIELAB L*, a* and b* values with intercepts not confidentially different or close to not significantly different from 0. This was similar for the slopes of DR and PBR expressed in *r*s close to 1 for the DR (Tables 5-2 – 5-4). Only small differences between conventionally measured and predicted data of PLSR models 2 and 3 and 2r and 3r, respectively were observed. Thus, based on the statistical results, the null hypotheses that both methods are equivalent can be accepted regarding CIELAB color measurements. The Bland-Altman plots for the comparison of reference and spectral method to measure CIELAB L*, a* and b* values do not show any slopes, curvatures or irregular patterns, which indicates no systematic measurement error. The according mean difference is not confidentially different from zero, irrespective of the applied prediction model, and the LOAs display a high accordance between reference and spectral measurements.

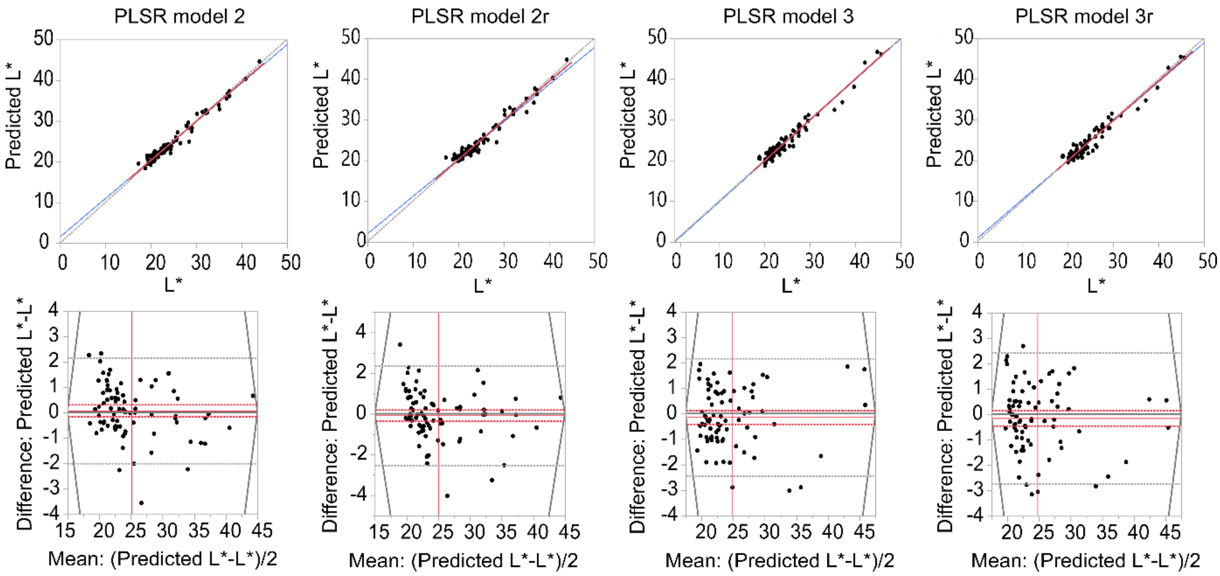

Figure 5-4: Results of methods comparison for CIELAB L* predicted with PLSR models of full (2 and 3) and reduced sets of wavelengths (2r and 3r) vs. conventionally measured.
Above: Deming regression (DR) red line, Passing Bablok regression (PBR) blue line. Dashed grey line: line of equality. The respective Bland-Altman plots underneath show the difference between both measurements vs the mean value of predicted and conventionally measured value. The grey line shows the line of equality, the red line the mean difference (bias) with the dashed red lines representing the 95 % confidence interval (CI) around the mean difference and the dashed grey lines representing the limits of agreement (LOA) which is the mean difference ± 1.96 SD

Table 5-2: Key parameters of methods comparison of CIELAB L* measurements (chromameter vs. predicted by PLSR model) of beef slices

Method comparison		PLSR model 2	2r	3	3r
Deming Regression	Intercept	1.19	0.96	-0.29	0.45
	Slope	0.96	0.96	1.01	0.98
	Lower/Upper CI	1.00/0.92	1.01/0.91	0.96/1.06	0.92/1.03
	r	0.98	0.98	0.98	0.97
	Orthogonal fit ratio	0.91	0.92	1.01	0.95
Passing-Bablok Regression	Intercept	1.49	1.94	0.27	0.80
	Lower/Upper CI	0.47/2.85	0.56/3.55	-1.74/1.71	-0.90/2.70
	Slope	0.94	0.91	1.00	0.96
	Lower/Upper CI	0.89/0.98	0.85/0.97	0.87/1.00	0.88/1.03
Bland-Altman	Mean Difference	0.08	-0.08	-0.14	-0.15
	Lower/Upper CI	0.31/-0.16	0.20/-0.36	-0.40/0.13	-0.45/0.15
	SD	1.06	1.25	1.18	1.31
	Lower/Upper CI	0.89/1.22	1.05/1.44	0.99/1.37	1.10/1.52
	Upper LOA	2.15	2.36	2.18	2.42
	Lower/Upper CI	1.75/2.56	1.89/2.84	1.71/2.63	1.91/2.93
	Lower LOA	-2.00	-2.52	-2.45	-2.72
	Lower/Upper CI	-2.40/-1.59	-3.00/-2.05	-2.91/-1.99	-3.23/-2.21

CI = confidence interval 95 %, r = correlation coefficient, LOA = limit of agreement, SD = standard deviation, PLSR model 2 and 3 = full models, PLSR models 2r and 3r = reduced models

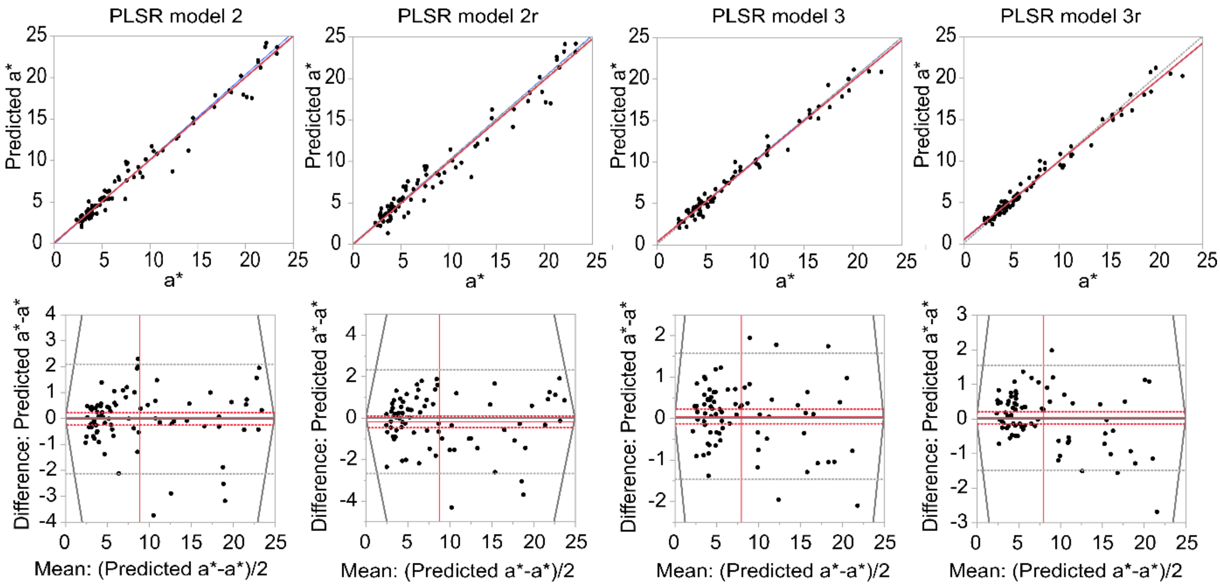

Figure 5-5: Results of methods comparison for CIELAB a* predicted with PLSR models of full (2 and 3) and reduced sets of wavelengths (2r and 3r) vs. conventionally measured.
Above: Deming regression (DR) red line, Passing Bablok regression (PBR) blue line. Dashed grey line: line of equality. The respective Bland-Altman plots underneath show the difference between both measurements vs the mean value of predicted and conventionally measured value. The grey line shows the line of equality, the red line the mean difference (bias) with the dashed red lines representing the 95 % confidence interval (CI) around the mean difference and the dashed grey lines representing the limits of agreement (LOA) which is the mean difference ± 1.96 SD

Table 5-3: Key parameters of methods comparison of CIELAB a* measurements (chromameter vs. predicted by PLSR model) of beef slices

Method comparison		PLSR model 2	PLSR model 2r	PLSR model 3	PLSR model 3r
Deming Regression	Intercept	0.01	0.089	0.26	0.45
	Slope	1.00	0.99	0.97	0.94
	Lower/Upper CI	0.96/1.04	0.95/1.04	0.94/1.01	0.92/0.98
	Correlation r	0.99	0.98	0.99	0.99
	Orthogonal fit ratio	0.99	0.98	0.95	0.90
Passing-Bablok Regression	Intercept	-0.14	0.00	0.27	0.33
	Lower/Upper CI	-0.43/0.09	-0.34/0.31	-0.03/0.48	0.12/0.62
	Slope	1.02	1.01	0.99	0.95
	Lower/Upper CI	0.99/1.06	0.95/1.06	0.95/1.03	0.92/0.99
Bland-Altman	Mean Difference	-0.02	-0.17	0.05	0.04
	Lower/Upper CI	-0.26/0.22	-0.46/0.11	-0.12/0.23	-0.14/0.21
	SD	1.08	1.27	0.77	0.77
	Lower/Upper CI	0.91/1.24	1.07/1.47	0.65/0.90	0.65/0.90
	Upper LOA	2.09	2.32	1.57	1.55
	Lower/Upper CI	1.68/2.50	1.84/2.80	1.27/1.87	1.25/1.85
	Lower LOA	-2.13	-2.67	-1.46	-1.48
	Lower/Upper CI	-2.54/-1.72	-3.15/-2.18	-1.76/-1.16	-1.78/-1.18

CI = confidence interval 95 %, r = correlation coefficient, LOA = limit of agreement, SD = standard deviation, PLSR model 2 and 3 = full models, PLSR models 2r and 3r = reduced models

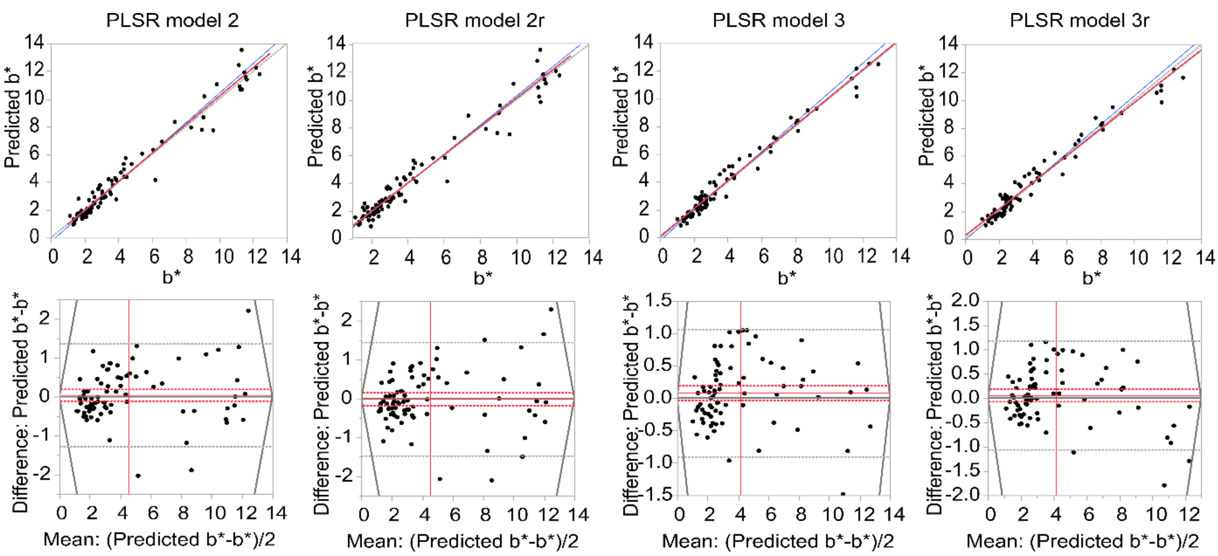

Figure 5-6: Results of methods comparison for CIELAB b* predicted with PLSR models of full (2 and 3) and reduced sets of wavelengths (2r and 3r) vs. conventionally measured.
Above: Deming regression (DR) red line, Passing Bablok regression (PBR) blue line. Dashed grey line: line of equality. The respective Bland-Altman plots underneath show the difference between both measurements vs the mean value of predicted and conventionally measured value. The grey line shows the line of equality, the red line the mean difference (bias) with the dashed red lines representing the 95 % confidence interval (CI) around the mean difference and the dashed grey lines representing the limits of agreement (LOA) which is the mean difference ± 1.96 SD

Table 5-4: Key parameters of methods comparison of CIELAB b* measurements (chromameter vs. predicted by PLSR model) of beef slices

Method comparison		PLSR model 2	2r	3	3r
Deming Regression	Intercept	-0.08	-0.12	0.09	0.24
	Slope	1.03	1.02	1.00	0.96
	Lower/Upper CI	0.98/1.07	0.97/1.08	0.96/1.03	0.91/1.00
	r	0.98	0.98	0.98	0.98
	Orthogonal fit ratio	1.06	1.05	1.00	0.91
Passing-Bablok Regression	Intercept	-0.29	-0.23	-0.20	0.07
	Lower/Upper CI	-0.55/-0.08	-0.51/-0.06	-0.39/0.02	-0.31/0.14
	Slope	1.07	1.05	1.06	1.04
	Lower/Upper CI	1.01/1.15	0.99/1.15	1.01/1.12	0.96/1.11
Bland-Altman	Mean Difference	0.05	-0.01	0.08	0.06
	Lower/Upper CI	-0.10/0.19	-0.18/0.16	-0.03/0.19	-0.07/0.19
	SD	0.67	0.74/	0.50	0.57
	Lower/Upper CI	0.57/0.78	0.63/0.86	0.42/0.58	0.48/0.66
	Upper LOA	1.37	1.45	1.06	1.18
	Lower/Upper CI	1.11/1.62	1.17/1.73	0.87/1.26	0.96/1.40
	Lower LOA	-1.28	-1.47	-0.90	-1.06
	Lower/Upper CI	-1.53/-1.02	-1.75/-1.19	-1.10/-0.71	-1.28/-0.84

CI = confidence interval 95 %, r = correlation coefficient, LOA = limit of agreement, SD = standard deviation, PLSR model 2 and 3 = full models, PLSR models 2r and 3r = reduced models

5.4.4 Practical aspects

The BA plots for all measured parameters clearly show that the evaluation of whether the spectral measurements could replace the reference measuring methods for MC and CIELAB color should not be done only on the basis of the statistical values. The limits of the maximum acceptable difference need to be defined *a priori* (Giavarina, 2015) and should be inside the LOAs of the BA plot. The results in the present study show that single spectral measurements pose the risk to over- or underestimate the investigated parameters. Conversely, the mean differences (BA) related to all models predicting MC and CIELAB values are not confidentially different from 0, which indicates that continuous spectral measurements would lead to valid measurement results and, thus, can replace the respective reference method. Outliers can be expected related to real-time measurements, but on average the predicted values will meet the ones measured with the reference method. Further, decreasing measurements intervals related to non-invasive measurements will allow proper measurements compared to the conventional MC and CIELAB color determination, respectively. Further, the linear equation of DR and PBR offers the possibility to correct the predicted data as it is utilized to calibrate the measurement system (Dietrich and Schulze, 2017). It should be noted that the equation parameters gained from the present study are highly related to the experimental set up and, thus, are not generally valid. However, the results clearly show that the relations of the factors to one another are appropriate.

In the present study, the image acquisition per four beef slices took one minute and the processing took ten min, which is significantly different from real-time measurements. However, the utilization of reduced sets of wavelengths will lead to decreased data acquisition, data acquisition times and decreased computation times and, thus, will lead to simpler and more cost effective applications compared to the usage of full wavelengths models (Kamruzzaman et al., 2016b). Reduced models that were comparable or even better than full models have also been developed for color and moisture prediction during drying of potato (Xiao et al., 2020). In this context, the results in the present study regarding reduced models (1r - 3r) indicate a great potential for practical applications. Due to collinearity of the parameters measured, PLSR model 3r is the most relevant for further investigations as it predicts MC and CIELAB color pattern with only ten wavelengths. According to Qiao et al. (2019) often models are developed that predict only one parameter, resulting in several models for different parameters, which make practical application too complex with regard to low data processing times. The results of the present study will support further work regarding real-time monitoring of beef during drying and the development of product related control strategies regarding individual and, thus, smart drying processes.

The results clearly show the successful development of robust PLSR models for the spectral measurements of MC and CIELAB color pattern during drying of beef slices independent of cut, breed and pre-treatment. Regarding industrial application, more data should be included into the model development of training and validation sets to achieve more robust measurements. However, the present study can be regarded as a successful “proof of concept” and the inclusion of a consistent population in the impact matrix would be worthwhile.

5.5 Conclusions

The current study shows that it is possible to develop robust algorithms, which are valid for MC and CIELAB measurement during drying of beef slices, irrespective of pre-treatment, raw material status, breed, muscle or drying temperature. Besides the development of models predicting either MC or CIELAB L*, a* and b* values, the development of a single model to predict all four parameters was successful and the model was simplified by selecting specific wavelengths. The Deming and Passing-Bablok regression and a Bland-Altman plot to evaluate the differences between spectral and laboratory based measurements were conducted. The study revealed a good relationship between the factors included in the development of prediction models and indicate a high potential for the implication of spectral systems for continuous non-invasive monitoring of moisture content and color parameters during beef drying. This will lead to a deeper understanding of changes inside the product to develop drying strategies or to directly integrate the product data into control strategies. Therefore, the value of increasing the impact matrix to gain a general model to predict MC and CIELAB color pattern of beef slices during dehydration processes is estimated to be high.

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6 General discussion

With regard to individualized, product related drying strategies knowledge on the impact of influencing factors on the drying behavior and the color development could allow more efficient processes. The studies included in this thesis investigated the impact of fresh, matured, frozen and unfrozen beef state, of pre-treatments with salt or salt/vinegar and of the drying air temperature on the drying behavior and color development of beef slices during convective drying. The influence of environmental variables was minimized by purchasing beef of that the origin in terms of herd, raising, gender, race, age and cut was exactly known. Concerning the development of non-invasive real-time monitoring applications, additionally to laboratory measurements HSI was applied. Prediction algorithms for MC and CIELAB color pattern were developed and with regard non-invasive real-time monitoring systems required, e.g. for smart drying applications, the potential of spectral measurements to replace invasive laboratory measurements was investigated.

This chapter discusses the main outcomes from chapters 3-5 and gives a critical view and outlook on the conducted work.

6.1 Impact of pre-drying storage and preparation

In terms of post-slaughter handling, 21 days of maturation led to decreasing drying rates compared to beef stored only for seven days at 4 °C as the muscle structure is more degraded by maturation processes and less drip channels support the removal of moisture during drying (Retz et al., 2017). In the context of economic aspects and the fact that maturation is a time and energy consuming factor, the utilization of matured beef needs to be considered against the consumer´s willingness to pay in terms of overall processing costs, as well as possible changes in sensory attributes, which are known to be increased by maturation for cooked or roasted meat (Ouali et al., 2006).

The freezing and thawing of matured beef prior to drying decreased the drying rates due to damages induced by ice crystal formation occurring as an increased drip after thawing (Leygonie et al., 2012). The low water binding ability post-slaughter (Honikel, 1986) for slices of Uckermarker breed, which was frozen only three days post slaughter, is not expressed in higher drying rates compared to fresh beef slices of the crossbreed, which might result from the freezing that is known to lead to stronger bound water compared to unfrozen meat (Thyholt and Isaksson, 1997). This might further explain the decreased drying rates of fresh frozen beef in comparison to fresh beef of the crossbreed. Shock freezing, like applied to the Uckermarker beef, is known to cause less damage due to the formation of smaller ice crystals induced by a higher freezing rate (Mortensen et al., 2006). This might have led to lower drying rates compared to the slices cut from crossbreed beef, which was frozen only at 18 °C and, thus, can be assumed to have been more damaged by ice crystal formation.

The results included in this study clearly show that the salt application (sprinkled (chapter 3) or dipped in salt/water solution (chapter 4)) increased the drying rates compared to blind and S+V samples independent of the beef origin. This underlines the salt induced water binding due to swelling and solubilization of myofibrilar proteins (Desmond, 2006), which is also known from pork jerky pre-treated with different salt concentrations (Yang et al., 2012). The effect of S+V solutions is contradictory and showed higher drying rates compared to the blind samples for beef slices cut from crossbreed upper shell (chapter 3), while for beef slices cut from Uckermarker roast beef it was the other way around expressed in higher drying rates for blind samples. Thus, both the maturation duration as well as freezing treatments might have influenced the impact of the S+V pre-treatment on the drying behavior. Besides, the impact of different cut, breed and gender might also have influenced the effect of S+V treatments, which requires further research. However, the results indicate that seasonings of low pH in combination with salt increase the drying rates compared to salted samples and, therefore, lead to decreased drying times. The results show that the post-slaughter handling of the beef (maturation, freezing), as well as the seasoning affects the drying behavior, which needs to be taken into account with regard to batch processing to avoid over- or insufficient drying throughout the batch.

Although the seasoning affected the color of the raw beef, especially the pre-treatments with salt and vinegar, the samples showed similar values for CIELAB L*, a* and b* values after drying. The inhomogeneity of meat caused by natural variations in color is shown in the investigations on the same raw material state (chapter 4), expressed in varying degrees of ΔE within the same pre-treatment after storage overnight and prior to drying at different temperatures.

In the present study, neither beef state nor pre-treatment resulted in a salient retention of redness (CIELAB a*) during drying. That shows that it was not possible to avoid a heat-induced oxidation from myoglobin derivates to metmyoglobin (Yin and Faustman, 1993) during dehydration. This is in accordance with low values for redness observed for unseasoned beef slices dried at 50 °C and 60 °C (Mewa et al., 2018), aged and salted goat meat dried at ambient conditions (Teixeira et al., 2011) or for charqui after sun drying (Youssef et al., 2003). In industrial processes, redness is often maintained by pre-treatments of sodium nitrite, which was also observed to result in higher a* values for beef dried in the sun (Youssef et al., 2003) or in an industrial dryer (Yong et al., 2019). Due to increasing concerns of chemical ingredients, vegetable powders were investigated to replace sodium nitrite in meat processing (Sebranek and Bacus, 2007). Similar results were found for the preservation of color in dried meat, while after 56 days of storage, the "cured color" of dried meat treated with sodium nitrite was preserved to a greater extent (Sindelar et al., 2010). Moreover, as vegetable powders could risk to be disadvantageous due to undesirable flavor and increasing processing costs, novel pre-treatments like e.g. atmospheric pressure plasma processing could offer an alternative (Yong et al., 2019). Redness is a main quality parameter of meat products and is associated with freshness by consumers, while brown color with a lack of freshness (Carpenter et al., 2001). However, when considering how red or similar to the color of fresh meat dried meat should be, especially in the area of natural or organic processing, consumer perceptions should be taken into account by preference tests to finally decide on the utilization of color preventing or coloring food additives and techniques.

Concerning flavor and shelf life of the final product, the correlation between changes in CIELAB color pattern and increased lipid oxidation needs further investigation. It has already been shown for pork and beef jerky that the cut can influence the degree of lipid oxidation during drying due to different fat contents and differences in fatty acid compositions (Yang et al., 2009).

6.2 Impact of drying parameter settings

Due to the highest heat and mass transfer related to the highest temperature applied, the most efficient drying temperature in the present thesis is 70 °C. However, there is a slight indication of an increased redness for drying at 50 °C compared to beef dried at 60 °C or 70 °C, which might present a conflict of interest between process efficiency and product quality (Mujumdar 2007). The decrease in yellowness (CIELAB b*), potentially impacted by drying temperature and lipid oxidation, needs future research to develop appropriate beef drying strategies. Although Karabacak et al. (2014) demonstrated increasing drying rates of beef at increasing air temperature and air velocity, little is known about the impact of high drying rates on the final quality of dried beef. In this context, Pask et al. (2017) defined the knowledge on the impact of air temperature and velocity on product quality as a key gap related to food dehydration processes. Sturm (2010) observed differences in shrinkage and surface roughness related to different drying rates for drying of apple slices. Accordingly, different drying rates for beef can also be assumed to cause structural changes of the final product, as it was already shown for different salt concentrations for pork jerky (Yang et al., 2012). Structural changes can influence the product surface and in case of surface enhancement could lead to increased oxidation processes of lipids and proteins and, thus, the development of decreasing sensory parameters during shelf life.

There is further a big need for dynamic, product related process control throughout the drying process as, e.g. the moisture loss is higher at the beginning of the drying process, which has also been observed in the present thesis expressed in higher drying rates. To achieve a reliable removal of surface moisture in this phase, the airflow rate (specific volume flow) needs to be increased (Sturm and Esper, 2018). The increased moisture loss in this phase further leads to significantly lower product temperatures compared to the drying air temperature applied (Sturm et al., 2014), which needs to be considered and justifies drying temperatures above critical product temperatures in the beginning of a drying process to increase the process efficiency.

6.3 Spectral measurements

In the present thesis, HSI in the range of 500 nm - 1000 nm was used to collect as much spectral information as possible during the drying of beef slices to build prediction models based on laboratory measured MC, CIELAB L, a* and b* values. The successful selection of specific wavelengths simplified the models and showed promising results in terms of the acquisition of convenient data sets by MSI in view of real-time measurements.

The results of the individual models concerning each measured parameter and each pre-treatment (Chapter 3), showed a high accuracy for models built from full or reduced sets of wavelengths and have further shown that it is worth testing different PLSR prediction models to gain the highest accuracy. However, the sets of different wavelengths selected for each parameter clearly show that the factors ‘cut’, ‘maturation’ and ‘pre-treatment’ influenced the spectral reflectance, which was expressed in different sets of selected wavelengths and, therefore, differs from observations in terms of pre-treatments for apple drying (Crichton et al., 2017). The models for predicting MC or color parameters independent of drying air temperature or marinade pre-treatment (Chapter 4) were successfully developed. These models can be assumed to be less accurate than those developed in chapter 3, where only a minimum of influencing factors were considered for each model. However, the robustness against the factors ‘pre-treatment’ and ‘drying temperature’ was increased and the accuracy between predicted and measured MC, CIELAB L*, a* and b* values is still on an acceptable level confirmed by R^2 values > 0.94.

In order to increase the robustness of the models against beef origin and pre-processing handling (breed, gender, cut, maturation, freezing/thawing pre-treatments) and against marinade pre-treatments, the data from both conducted studies was used to develop one model for MC prediction and one for CIELAB color pattern prediction, respectively (chapter 5). With regard to collinearity between moisture content and CIELAB color pattern, additionally, an entire model was developed that predicts the focused parameters simultaneously. The Deming and Passing-Bablok regression, based on the slope and intercept not significantly different from 1 and 0, respectively (Deming, 1943; Passing and Bablok, 1983), revealed no significant lower performance compared to the models predicting either MC or CIELAB L*, a* and b* values. With regard to practical applications, the entire model is of an increased importance as it predicts MC and CIELAB values simultaneously, allowing the use of low-complexity detectors as opposed to using two different wavelength sets. The Bland-Altman plot (Altman and Bland, 1983) shows very low mean differences between spectral and laboratory measured values of -0.64 % for MC (% wb), -0.15 for L*, 0.04 for a* and 0.06 for b* values, whereas the plotted values are randomly scattered and do not show any curves or irregular patterns so that a systematic measurement error can be excluded. However, the observed limits of agreement (LOA), caused by a pronounced scattering, indicate to risk unacceptable over- or underestimations, which would be challenging, if the spectral measurements were used as single measurements. However, the investigations included in this thesis aimed to find approaches regarding continuous monitoring approaches for online applications so that single outliers will hardly impact the overall monitoring results. Against this background, the results provide a sound basis as it has been shown that it is possible to predict four parameters simultaneously with only one model. The selection of only ten wavelengths to meet multispectral data acquisition and, thus, low computation times for real-time measurements offers promising results in view of embedded systems and low-cost solutions. After implementation of the multispectral monitoring system into a beef drying device, the regression equation can be used for a first calibration of the measurement system by shifting the intercept (Dietrich and Schulze, 2017).

As an example, according to intercept and slope obtained from the DR regarding model 3r (see Table 5-1), the equation for MC is $y = 0.76 + 0.97x$.

By inserting predicted values ‘x’, obtained by the multispectral system and the related prediction model, ‘y’ is calculated to present the corrected values for MC measurements. As with any measuring instrument, the calibration requires continuous evaluation at specific time intervals, which is achieved by cross checking the measured values during operation with invasive laboratory measurements to consequently readjust the regression equation and, thus, the measurement system.

6.4 Critical review and Outlook

The research questions that led to the preparation of this study (see page 3) can be answered as follows:

(i) Maturation and freezing/thawing lead to decreased drying rates of beef slices, while for matured beef the freezing and thawing increased the drying rates. The drying rates were further decreased by salt pre-treatments compared to blind samples or samples pre-treated with salt and vinegar. The highest drying rates were achieved by the highest drying air temperatures. Contrary, the final color of beef slices after drying is only affected to a barely perceptible degree by post-slaughter and pre-processing factors or product temperature, respectively.

(ii) Data acquired by HSI and laboratory measurements can be used to develop successfully prediction models for non-invasive MC and color monitoring of beef slices during drying.

(iii) The robustness of the prediction models can be increased by the inclusion of different post-slaughter and pre-processing handlings into the model development. The most relevant wavelengths were identified for each parameter to develop simplified prediction models for MC and CIELAB color pattern of beef slices during drying and even for an entire model to predict the parameters simultaneously. Therefore, the high potential of MSI to be utilized for simple non-invasive real-time monitoring techniques as, e.g. required for smart drying applications for beef drying, has been successfully proven.

The deeper understanding of changes in beef slices during drying obtained from invasive monitoring in the present study and gained through future studies based on invasive and non-invasive measurements will support the development of improved beef drying strategies with regard to product and process efficiency and economic aspects, respectively. Robust and simplified prediction models further developed from the current ones will be utilized to develop non-invasive real-time monitoring systems, which could offer feedback control parameters related to the changes occurring into the product in view of smart beef drying applications.

However, the results of this study can be assumed to be impacted by the repeated interruption of the drying process due to the invasive measurements conducted for data collection. The laboratory measurements led to cooling down and re-heating of the samples, which might have influenced the drying behavior and color development negatively. Although the decreased measurement intervals during the first two hours of drying of Uckermarker beef (Chapter 4) allowed a closer monitoring of product parameters compared to the experiments conducted with crossbreed cattle (Chapter 3), it makes a direct comparison of the drying behavior and color development of the two studies difficult.

Further, the present study focused only on different drying temperatures, but also the effects of relative humidity and air velocity must be considered with regard to improved drying strategies. In this context, the thesis emphasizes the need of real-time online monitoring systems to enable continuous measurements to evaluate the effect of different combinations of drying parameters and raw material states of beef. Based on the monitored data this will further improve the understanding of the drying behavior and color development of beef slices and, thus, to support the development of efficient drying strategies in terms of product and process quality.

With regard to the development of non-invasive monitoring systems, the robustness of the developed prediction models could be increased to a certain degree in the course of work carried out for this thesis. However, the models cannot be assumed to be generally valid, as, based on the resources available for this study, only a small part of the influencing variables matrix could be filled (two breeds, two cuts, three maturation stages, two freezing techniques and three pre-treatments prior to drying). Therefore, measurements would risk predicting insufficient values, if beef slices with attributes beyond the influencing factors included in the model development were measured. Therefore, the regression equation mentioned in the previous section (6.3) serves only to illustrate the calibration principle and should not be used to correct insufficient prediction results caused by a model of insufficient robustness and permanent readjustment of the system, respectively. Thus, a future approach will be to increase the robustness of the model by a homogeneous filling of the influencing variable matrix. That means the inclusion of as many as possible breeds, ages (with a homogenous distribution related to the gender), cuts, post-slaughter handlings and seasonings, drying strategies and beyond that eventually meat from other species to develop a generally valid model. According to the investigations and evaluation of different prediction models (MUCVE, CARS) in chapter 3, it is also worth further investigating additional prediction models according to the selected wavelengths of the entire model. The mapping of MC as shown in chapter 3 to observe the moisture distribution of beef during drying might further help to understand the dynamics of water inside the product related to the applied drying strategy.

For the implementation in food dryers, the results of a generally valid model and the according selected wavelengths can be transferred to practical MSI systems including, e.g. a camera with a CCD sensor (charge coupled device) to be positioned inside a dryer above the product. Combined either with halogen lamps and specific filters or with LEDs according to the selected wavelength, this will enable the acquisition of most important product information. Depending on the focal length of the lens and the distance to the product, single or several samples can be monitored simultaneously. Thus, a cost-effective monitoring system can be provided that is applicable to small, medium and large processors as well as to research dryers in terms of product monitoring and development of improved drying strategies.

Future work might further include the monitoring of parameters relevant for processed meat products like lipid oxidation, for which spectral prediction models already were successfully applied regarding raw and processed meat products (Feng et al., 2018), during cold storage (Cheng et al., 2019) and during dehydration processes (Yang et al., 2017).

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7 Summary

Convection drying is widely used for industrial food preservation processes and is required to provide high process efficiencies and high product qualities, which can be contradictory. Thus, it is important to understand the changes that occur inside the product to develop drying strategies that consider process and product efficiency to an acceptable level. In this context, it is of a great importance to further gain an understanding of the impact of the raw material on the product during dehydration to achieve sustainable processes.

The dehydration of meat has a long tradition all over the world and technically dried meat is increasingly valued as a low-fat snack or high-protein food product. Meat is an inhomogeneous material that is impacted by species and cut, but also by maturation, cold-storage and seasoning treatments. It can be assumed that these factors also affect the meat during drying and influence the drying behavior or the properties of the final product. Unlike product monitoring by conventional invasive measurements during drying, the development of non-invasive measurement systems can avoid interrupting the process and provide product related information in real time throughout the drying process. In this context, prediction algorithms that connect spectral and laboratory data have already shown a big potential in food quality assurance. In future, these could be extended to continuous monitoring applications and product related, so called “smart” control systems.

The present study investigated the cumulative impact of maturation, freezing/thawing pre-treatments, seasoning pre-treatments and drying air temperature on the drying behavior and color development of beef slices. The first experiment included the observations on slices cut from fresh (stored for seven days post-slaughter) and matured (21 days post-slaughter) upper shell (*m. semimembranosus*) of three crossbreed bulls. Further, beef of those two maturation stages was utilized after freezing and thawing. Three pre-treatments were applied (0.5 % and 1 % salt w/w and 10 % (m/v) salt/vinegar solution) and the drying behavior and color development was compared to blind samples during drying at 70 °C. The impact of the drying air temperature (50, 60 and 70 °C) was investigated in a second experiment for slices cut from fresh roast beef (*l. dorsi*) of four Uckermarker heifers that was shock frozen prior to cold storage (after storage for three days post-slaughter). Prior to drying, the samples were dipped in salt (S) or salt/vinegar (S+V) solution (10 % m/v) or kept as blind samples. During the drying process, the moisture content (MC) and CIELAB color pattern was monitored invasively by laboratory measurements, which were supplemented by the acquisition of hyperspectral data in the visible to near infrared (VNIR) range of 500 - 1009 nm. The MC data was standardized by moisture ratio (MR) calculations to evaluate the drying behavior. The CIELAB L*, a* and b*

values were used to evaluate the development of color parameters and to calculate the color change ΔE of beef slice during dehydration. Based on the laboratory measurements of MC and CIELAB color pattern and hyperspectral data of the samples, partial least square regression (PLSR) prediction models were developed. The models were further simplified by wavelengths selection. With regard to an applicability as a spectral real-time monitoring technique, method comparisons were performed to evaluate the agreement between laboratory and spectral measurements.

The results revealed increasing drying rates at increasing drying air temperature and clearly showed an influence of post-slaughter and pre-processing treatments prior to drying. Compared to beef slices from fresh beef, slices of matured beef were observed to show decreased drying rates, while freezing and thawing of beef prior to slicing decreased the drying rates for fresh but increased ones for matured beef. Regarding the seasoning, the drying rates were reduced compared to blind samples or samples treated with salt/vinegar solutions for beef slices treated with salt and drying rates decreased with increasing salt concentrations. This is due to a salt induced swelling and solubilization of myofibrillar proteins and, hence, an increased water binding ability. The treatment with salt/vinegar solutions decreased the water binding ability compared to salted samples while the effect led to similar drying rates as of blind samples. With regard to beef drying processes, these investigations indicate the necessity of utilization of similar raw materials and seasonings per batch to achieve homogenous drying results and to avoid partial over or insufficient drying throughout the batch.

The investigations on the color during drying showed different developments of CIELAB L^*, a^* and b^* values resulting in different development of ΔE according to the drying condition, beef origin, post-slaughter and pre-drying treatments with final color changes of $\Delta E = 19\text{-}25$ after dehydration. However, no significant differences were found in the individual color parameters after drying with respect to the raw material or the drying conditions after the drying process. This is due to the drying induced increased concentration of pigments, resulting in a low lightness ($L^* = 18 - 23$). Furthermore, this is related to the oxidation of myoglobin derivatives to metmyoglobin, which was expressed in low values for redness ($a^* = 3 - 5$). For yellowness, overall low values of only $b^* = 2 - 3$ were observed after drying.

The development of single PLSR models to predict MC or CIELAB a^* and b^* values during dehydration of beef slices of the first experiment revealed that based on the models of reduced wavelengths the best model evaluated by the lowest root mean square error (RMSE), is affected by the influence of beef state and seasoning pre-treatment. The CARS-PLS played only a tangential role, while PLSR and MCUVE-PLS were most dominant present. Further, the wavelengths sets selected to simplify the models differed throughout the factor combinations, which indicated a low robustness against influencing factors of each model. In the second experiment, PLSR models were developed that predicted MC, CIELAB L*, a*, or b* values independent of the pre-treatment applied, resulting in increased model robustness. The high accuracies were maintained for models developed by reduced sets of wavelengths with R^2 = 0.953 for MC, R^2 = 0.948 for L*, R^2 = 0.982 for a* and R^2 = 0.963 for b*. By including data of both experiments into the development of PLSR models (experiment 3) to predict either MC (model 1) or CIELAB color pattern (model 2) or to predict all parameters simultaneously with an entire model (model 3), the impact matrix (breed, cut, gender) and, hence, the model robustness was increased. The selection of wavelengths led to the development of PLSR models for MC or CIELAB L*, a* and b* values with only eight wavelengths and to the selection of only ten wavelengths for the entire model predicting MC and CIELAB color pattern simultaneously. Methods comparisons (Deming (DR) and Passing-Bablok regression (PBR)) showed no inferiority for the entire model 3 compared to model 1 and 2. The high accuracy between laboratory and spectral measurements was expressed with intercepts not significantly different from 0 and slopes not significantly different from 1, respectively. The Bland-Altman plot confirmed the high accuracy for the entire model expressed in low mean differences calculated between laboratory and spectral measurements (MC = -0.64, L* = -0.14, a* = 0.05, b* = 0.08), which were maintained after wavelengths selection (MC = -0.64), slightly increased (L* = -0.15) or even decreased (a* = 0.04, b* = 0.06). Based on the confidence intervals (0.95 %) the mean differences was evaluated as not significantly different from 0. Nor the regression lines (DR/PBR), neither the BA plot showed a curvature or irregular pattern, which excludes a potential systematic error for both methods. Thus, the studies show the high potential to use spectral measurements as a non-invasive technique and even for simultaneous measurements of MC and CIELAB color pattern during beef drying, independent of the used beef state or pre-treatment applied. With regard to real-time monitoring, the use of selected wavelengths can allow a quick data acquisition and processing and, hence, the utilization of multispectral measurements in embedded control systems as required, e.g. for smart drying systems.

Thus, this work demonstrated that the raw material influences the drying behavior of beef slices and should be considered in the development of individual drying strategies with respect to increasing the efficiency of beef drying processes. An increased knowledge about drying behavior as well as color development during drying can thereby be utilized for future development of improved and individualized drying processes. MC and color values have further been shown to be capable to be monitored non-invasively and in real-time by spectral measurements and, hence, to integrate MSI systems in beef drying devices.

Zusammenfassung

Die Konvektionstrocknung ist in der industriellen Konservierung von Lebensmitteln weit verbreitet und muss hohe Prozesseffizienz und hohe Produktqualitäten bieten, welche widersprüchlich zueinander sein können. Daher ist es wichtig, die Veränderungen, die im Produkt auftreten, zu verstehen um Trocknungsstrategien zu entwickeln, die Prozess- und Produkteffizienz auf einem akzeptablen Niveau berücksichtigen. In diesem Zusammenhang ist es von großer Bedeutung, auch ein Verständnis für den Einfluss des Rohmaterials auf das Produkt während der Trocknung zu erlangen um nachhaltige Prozesse zu erzielen.

Die Trocknung von Fleisch hat weltweit eine lange Tradition und technisch getrocknetes Fleisch wird zunehmend als fettarmer Snack bzw. eiweißreiches Lebensmittelprodukt geschätzt. Fleisch stellt ein inhomogenes Material dar, das durch Art und Teilstück, aber auch durch Reifung, Kühllagerung und Marinieren beeinflusst wird. Es ist davon auszugehen, dass diese Faktoren sich auch während der Trocknung auf das Fleisch auswirken und das Trocknungsverhalten bzw. die Eigenschaften des Endprodukts beeinflussen. Im Gegensatz zur Produktüberwachung durch herkömmliche invasive Messungen während der Trocknung kann die Entwicklung von nicht-invasiven Messsystemen eine Unterbrechung des Prozesses vermeiden und produktbezogene Informationen während des gesamten Trocknungsprozesses in Echtzeit liefern. In diesem Zusammenhang haben Vorhersagealgorithmen, die z. B. Spektral- und Labordaten miteinander verbinden, bereits ein großes Potenzial in der Qualitätssicherung von Lebensmitteln gezeigt. Diese könnten zukünftig auf kontinuierliche Überwachungsanwendungen und produktbezogene, so genannte "intelligente" Kontrollsysteme ausgeweitet werden.

In der vorliegenden Studie wurde der kumulative Einfluss von Reifung, Gefrieren/Auftauen, Marinieren und Trocknungslufttemperatur auf das Trocknungsverhalten und die Farbentwicklung von Rindfleischscheiben untersucht. Der erste Versuch umfasste die Untersuchung von im frischen (sieben Tage nach der Schlachtung gelagert) und gereiften Zustand (21 Tage nach der Schlachtung) aus der Oberschale (*m. semimembranosus*) von drei Kreuzungsbullen geschnittenem Fleisch. Ebenso wurde Fleisch dieser beiden Reifezustände nach dem Einfrieren und Auftauen in die Versuche einbezogen. Drei Vorbehandlungen wurden angewendet (0,5 % und 1 % Salz w/w, 10 % (m/v) Salz/Essig-Lösung) und das Trocknungsverhalten und die Entwicklung der Farbe mit dem von unbehandelten Proben während der Trocknung bei 70 °C verglichen. Der Einfluss der Trocknungslufttemperatur (50, 60 und 70 °C) wurde in einem zweiten Experiment für aus frischem Roastbeef (*l. dorsi*) geschnittenen Proben von vier Uckermarker Färsen untersucht, welches vor der Kühllagerung

(drei Tage nach der Schlachtung) schockgefroren wurde. Vor der Trocknung wurden die Proben in Salz- (S) oder Salz/Essig-Lösung (S+V) (10 % m/v) getaucht oder unbehandelt als Blindproben verwendet. Während des Trocknungsprozesses wurde der Feuchtigkeitsgehalt (MC) und die CIELAB-Farbwerte invasiv durch Labormessungen überwacht, die durch die Erfassung von Hyperspektraldaten im sichtbaren bis nahinfrarot Bereich (VNIR) von 500 - 1009 nm ergänzt wurden. Die MC-Daten wurden durch Berechnungen des Feuchteverhältnisses (MR) standardisiert, um das Trocknungsverhalten zu bewerten. Die CIELAB L*-, a*- und b*- Werte wurden genutzt, um die Entwicklung der Farbparameter und die Farbveränderung ΔE der Proben während der Trocknung zu bewerten. Basierend auf den Labormessungen der MC-, CIELAB-Farbwerten und den Hyperspektraldaten wurden Regressionsmodelle der partiell kleinsten Quadrate (PLSR) entwickelt. Die Modelle wurden durch die Selektion von Wellenlängen vereinfacht. In Hinblick einer Anwendbarkeit zur spektrale Echtzeitüberwachung wurde ein Methodenvergleich durchgeführt um die Übereinstimmung zwischen Labor- und Spektralmessungen zu bewerten.

Die Ergebnisse zeigten steigende Trocknungsraten bei zunehmender Trocknungslufttemperatur und einen deutlichen Einfluss der Behandlung des Rindfleischs nach der Schlachtung bzw. vor der Trocknung. Im Vergleich zu Rindfleischscheiben aus frischem Rindfleisch wurde bei Scheiben aus gereiftem Rindfleisch eine Erhöhung der Trocknungsraten beobachtet, während das Einfrieren und Auftauen von Rindfleisch vor dem Aufschneiden die Trocknungsraten bei frischem Fleisch erhöhte, bei gereiftem aber verringerte. Hinsichtlich der Marinaden waren die Trocknungsraten bei den mit Salz behandelten Rindfleischscheiben im Vergleich zu Blind- oder mit Salz/Essig-Lösung behandelten Proben reduziert und die Trocknungsraten nahmen mit steigender Salzkonzentration ab. Dies ist auf eine salzinduzierte Quellung und Solubilisierung der myofibrillären Proteine und damit auf eine erhöhte Wasserbindungsfähigkeit zurückzuführen. Die Behandlung mit Salz/Essig-Lösungen verringerte das Wasserbindungsvermögen im Vergleich zu gesalzenen Proben, während der Effekt zu ähnlichen Trocknungsraten wie bei Blindproben führte. Im Hinblick auf Rindfleischtrocknungsprozesse weisen diese Untersuchungen auf die Notwendigkeit der Verwendung ähnlicher Rohstoffe und Marinaden pro Charge hin, um homogene Trocknungsergebnisse zu erzielen und eine partielle Über- oder Untertrocknung innerhalb der Charge zu vermeiden.

Die Untersuchungen zur Farbe während der Trocknung zeigten unterschiedliche Entwicklungen der CIELAB L*-, a*- und b*-Werte, die je nach Trocknungsbedingung, Rindfleischherkunft, der Behandlung nach der Schlachtung und Vorbehandlungen zu einer unterschiedlichen Entwicklung von ΔE, mit insgesamten Farbveränderungen von ΔE = 19-25 nach der Trocknung führten. Bei den einzelnen Farbparametern wurden nach der Trocknung hinsichtlich der einzelnen Farbparameter jedoch insgesamt keine nennenswerten Unterschiede in Bezug auf das Rohmaterial oder die Trocknungsbedingungen nach dem Trocknungsprozess festgestellt. Dies ist auf eine durch die Trocknung hervorgerufene erhöhte Konzentration von Pigmenten zurückzuführen, was zu einer geringen Helligkeit (L* = 18-23) führte. Weiterhin hängt dies mit der Oxidation von Myoglobinderivaten zu Metmyoglobin zusammen, was sich in niedrigen Werten für die Röte (a* = 3 - 5) ausdrückte. Für die Gelbfärbung wurden nach der Trocknung insgesamt niedrige Werte von nur b* = 2 - 3 beobachtet.

Die Berechnung einzelner PLSR-Modelle zur Vorhersage von MC- oder CIELAB a*- und b*-Werten während der Dehydratation von Rindfleischscheiben ergab, dass das beste Modell, bewertet nach dem niedrigsten mittleren quadratischen Fehler (RMSE), durch den Einfluss des Rindfleischzustandes und der Marinade beeinflusst wird (erstes Experiment). Das CARS-PLS Modell spielte nur eine untergeordnete Rolle, während die PLSR und MCUVE-PLS Modelle am dominantesten präsent waren. Außerdem unterschieden sich die zur Vereinfachung der Modelle gewählten Wellenlängensets über die Faktorkombinationen hinweg, was auf eine geringe Robustheit gegenüber Einflussfaktoren der einzelnen entwickelten Modelle hindeutet. Im zweiten Experiment wurde PLSR-Modelle berechnet, die MC-, CIELAB L*-, a*- oder b*-Werte unabhängig von der angewandten Reifevorbehandlung vorhersagten, und somit eine erhöhte Robustheit erzielt. Die hohen Vorhersagegenauigkeiten mit R^2 = 0,953 für MC, R^2 = 0,948 für L*, R^2 = 0,982 für a* und R^2 = 0,963 für b* wurden auch für Modelle beibehalten, die mit einer reduzierten Anzahl von Wellenlängen berechnet wurden. Durch die Einbeziehung der Daten beider Experimente in die Berechnung von PLSR-Modellen (Experiment 3) zur Vorhersage von entweder MC (Modell 1) oder der CIELAB-Farbwerte (Modell 2) oder zur gleichzeitigen Vorhersage aller Parameter mit nur einem Gesamtmodell (Modell 3), wurde die Einflussgrößenmatrix (Rasse, Schnitt, Geschlecht) und somit die Robustheit der Modelle weiter erhöht. Die Auswahl der Wellenlängen führte zur Entwicklung von PLSR-Modellen für MC- oder CIELAB-L*-, a*- und b*-Werte mit nur acht Wellenlängen und zur Auswahl von nur zehn Wellenlängen für das gesamte Modell, welches MC und CIELAB-Farbwerte gleichzeitig vorhersagt. Methodenvergleiche (Deming (DR) und Passing-Bablok-Regression (PBR)) zeigten keine Unterlegenheit für das Gesamtmodell im Vergleich zu Modell 1 und 2. Die hohe Genauigkeit zwischen Labor- und Spektralmessungen drückte sich in Achsenschnittpunkten, die sich nicht signifikant von 0, und Steigungen, die sich nicht signifikant von 1 unterschieden, aus. Der Bland-Altman-Plot bestätigte die hohe Genauigkeit für das Gesamtmodell, die sich in niedrigen Differenzmitteln zwischen Labor- und Spektralmessungen ausdrückte (MC = -0. 64, L* = -0,14, a* = 0,05, b* = 0,08) und sich nach der Selektion der Wellenlängen gleichbleibend (MC = -0,64), leicht erhöht (L* = -0,15) oder sogar verringert (a* = 0,04, b* = 0,06) zeigten. Anhand der Konfidenzintervalle (0,95 %) konnten die Differenzmittel als nicht signifikant verschieden von 0 bewertet werden. Weder die Datenpunkte zur Berechnung der Regressionsgeraden (DR/PBR) noch jene im BA-Plot zeigten eine Krümmung oder ein unregelmäßiges Muster, was einen möglichen systematischen Fehler für beide Methoden ausschließt. Somit zeigen die Untersuchungen das hohe Potenzial, spektrale Messungen als nicht-invasive Technik und sogar zur gleichzeitigen Messung von MC und CIELAB-Farbwerten während der Trocknung, unabhängig vom verwendeten Rindfleisch oder der angewandten Vorbehandlung, einzusetzen. Im Hinblick auf die Echtzeitüberwachung kann die Verwendung ausgewählter Wellenlängen eine schnelle Datenerfassung und -verarbeitung ermöglichen und damit die Nutzung von Multispektralmessungen in eingebetteten Kontrollsystemen, die z.B. für intelligente Trocknungssysteme notwendig sind, ermöglichen.

Somit konnte in dieser Arbeit gezeigt werden, dass das Rohmaterial das Trocknungsverhalten von Rindfleischscheiben beeinflusst und bei der Entwicklung von individuellen Trocknungsstrategien hinsichtlich der Steigerung der Effizienz von Rindfleisch-Trocknungsprozessen Berücksichtigung finden sollte. Ein erhöhtes Wissen über das Trocknungsverhalten sowie über die Farbentwicklung während der Trocknung kann dabei für die zukünftige Entwicklung verbesserter und individualisierter Trocknungsprozesse genutzt werden. Des Weiteren hat sich gezeigt, dass MC- und CIELAB Farbwerte durch spektrale Messungen nicht-invasiv und in Echtzeit überwacht werden können und somit eine Integration von multispektralen Systeme in Fleischtrocknungsanlagen möglich ist.

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