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“Desarrollo de una tecnología óptica de control del grado de emulsificación en emulsiones cárnicas”

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INFORMAN

Que el trabajo de investigación titulado: “Desarrollo de una tecnología óptica de control del grado de emulsificación en emulsiones cárnicas” ha sido realizado, bajo su supervisión y tutela, por la Srta. Verónica Irene Torres Flores dentro del Máster en Calidad de Alimentos de Origen Animal de la Universidad Autónoma de Barcelona.

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ABSTRACT

The main challenge to obtain a stable meat emulsion such as frankfurters and bolognas is the control of the emulsification process as a result of the lack of warning signs for emulsion breakdown during chopping. The chopping process is designed to reduce meat and fat particle sizes, which results in better protein extraction and fat-water holding capacity. Under-chopping results in minimal binding and over-chopping causes a massive fat and water separation during the cooking process. In both cases the emulsion stability is inadequate and the emulsion breakdown is only evident during the heating process, but at this point it is too late to introduce corrective actions. Results of previous studies suggest that light extinction spectroscopy could provide information about emulsion stability. In this study six different treatments were analyzed corresponding to the industrially-generated meat emulsions and frankfurters made with two variables: a) formula at two levels -high quality (no starch) and low quality (starch at 2% in the meat emulsion)- and b) chopping speed at three levels -low, medium and high-. The most appropriate optical configuration to monitor industrial emulsion samples was identified and optical predictors containing useful information on the stability of emulsions were generated from the optical spectra. In order to identify the best optical predictors for the meat emulsion stability/quality indexes evaluated (emulsions: chemical composition, cooking losses, color and rheology; frankfurters: chemical composition, rheology and texture), Pearson correlations between these physical-chemical variables and the optical predictors were evaluated. The results showed that there exist high correlations between physical-chemical properties of both meat emulsions and frankfurters and the optical predictors found. Therefore, light backscatter has potential as an early predictor of emulsion stability during finely comminuted meat products manufacturing.

Keywords: Meat emulsions, frankfurters, starch, chopping speed, light extinction spectroscopy, light transmission, light backscatter, optical predictors.

RESUMEN

El principal desafío para obtener una emulsión cárnica estable tales como salchichas y mortadelas requiere un mejor control del proceso de emulsificación debido a la falta de señales de advertencia de rotura de la emulsión durante el picado. El proceso de picado está diseñado para reducir el tamaño de la carne y de las partículas de grasa, dando como resultado una mejor extracción de proteínas y mayor capacidad de retención de agua y grasa. Un tiempo de picado insuficiente resulta en una unión mínima mientras que el exceso de picado causa una separación masiva de la grasa y el agua durante el proceso de cocción. En ambos casos, la estabilidad de la emulsión es inadecuada y la rotura de la emulsión es sólo evidente durante el proceso de calentamiento, pero en este momento ya es demasiado tarde para introducir acciones correctivas. Los resultados de estudios previos sugieren que la espectroscopía de extinción de la luz podría proporcionar información sobre la estabilidad de la emulsión. En este estudio seis tratamientos diferentes fueron analizados correspondientes a las emulsiones de carne y salchichas procesadas industrialmente con dos variables: a) fórmula en dos niveles -alta calidad (sin almidón) y baja calidad (almidón al 2% en emulsión de carne)- y b) velocidad de picado en tres niveles -: bajo, medio y alto. Se identificó la configuración óptica más apropiada para monitorear las muestras industriales, y se generaron predictores ópticos con información útil sobre la estabilidad de las emulsiones. A fin de identificar los mejores predictores ópticos para los índices de estabilidad/calidad de las emulsiones evaluados (emulsiones: composición química, pérdidas por cocción, color y reología; salchichas: composición química, reología y textura), se obtuvieron y evaluaron las correlaciones de Pearson entre dichas variables físico-químicas y los predictores ópticos generados. Los resultados mostraron que existen numerosas correlaciones significativas entre las propiedades físico-químicas tanto de las emulsiones cárnicas como de los *frankfurts* y los predictores encontrados, por lo tanto la dispersión de luz tiene un fuerte potencial como predictor de la estabilidad de las emulsiones durante la fabricación de productos cárnicos finamente picados.

Palabras clave: Emulsiones cárnicas, salchichas, almidón, velocidad de picado, espectroscopía de extinción de la luz, transmisión de luz, dispersión de la luz, predictores ópticos.

ABBREVIATIONS

C: Collagen

CF: Crude Fat

CP: Crude Protein

IT: Integration Time

L: Losses

M: Moisture

S: Salt

N: Newton

Pred: Predictors

1. Introduction

Production and consumption of meat continues growing worldwide and according to the Agricultural Outlook 2015-2024 (OECD-FAO, 2015) in the coming years it will continue to grow. The reason lies in the population growth and economic development in many countries. Specifically, world meat production is expected to increase from 297 million tons in 2011 to 350 million in 2021. The expected annual world growth production for beef and pork is 1.8% and 1.4%, respectively (Emphasis Alimentation, 2015).

Frankfurters and bolognas are the most popular comminuted products. In the US, they account for the 25% of all sausages sold (NHDSC, 2006). In 2007, consumers spent more than 4.1 billion dollars on hot dogs and sausages in US supermarkets, which equals more than 0.68 billion kilos of hot dogs and sausages bought at retail stores alone (Álvarez et al., 2009).

Meat emulsions are finely chopped and cooked products composed of water, protein, fat, salt, and non-meat ingredients where meat proteins serve as the natural emulsifier. Before cooking, proteins must surround fat particles to allow proper fat emulsification. The chopping process is designed to reduce meat and fat particle sizes, which results in better protein extraction and fat-water holding capacity (Xiong, 2000).

Under-chopping results in minimal binding because fat particles are too large to yield a stable product. Over-chopping triggers a massive fat and water separation during the cooking process due to very small fat particles with highly increased surface area requiring more protein to emulsify the fat. In both cases the emulsion stability is inadequate. Indeed, obtaining a stable emulsion requires improved control of the emulsification process as a result of the lack of warning signs for emulsion breakdown during chopping (Barbut, 1998).

Based on an average cooking loss of 2.64% (w) under optimum chopping conditions, the estimated economic loss resulting from non-optimum emulsion stability during the cooking

process was estimated to range between 0.2 and 1.65 billion dollars per year in USA and 5-40 million euros in Spain (Nieto et al., 2014).

The three main requirements for optimum emulsion stability and final product quality are directly dependent on the chopping process. The first requirement is an extensive extraction and dispersion of myofibrillar proteins from the cellular structures. The second requirement is the optimum reduction of particle sizes and the third requirement is to keep the degree of myofibrillar proteins denaturation to a minimum during chopping to ensure optimum coating of the fat particles. In summary, obtaining a high quality finely comminuted meat product requires adequate gelation of myofibrillar proteins during cooking. In general, loss of emulsion stability leads to a low quality product (Jones and Mandingo, 1982; Allais et al., 2004).

Improving process control and automation of the meat emulsification process would reduce the economic impact of emulsion breakdown in meat industry worldwide. To date, only empirical and subjective methods based on experience and process time and temperatures are being used to control meat emulsion stability during meat chopping at production scale. There are no effective inline alternative technologies (Nieto et al, 2014). Emulsification defects result in weight product losses of 5 to 20%. Thus, meat processors are keen on introducing technological innovations that would allow them to improve their competitiveness by increasing their productivity and, consequently, their profitability.

Several authors have observed the correlation between emulsion color parameters and fat and water losses induced by the emulsion heat treatment. Cooking losses can be predicted from the change on the raw emulsion lightness (L^*) during the chopping process (Serdaroglu, 2006; Álvarez, et al., 2007). These evidences suggest that the intensity of backscattered light could also be correlated with physical-chemical properties changing during emulsification and impacting emulsion stability.

The application of light scatter sensors for process control and optimization has already been deeply studied in the dairy industry (Mateo et al., 2010). Few years ago, this

technology has aroused great interest in the meat sector. Álvarez et al. (2009 and 2010) made a study of the optical properties of beef emulsions having different fat/lean ratios at various chopping durations and at several distances between the emitting and detecting optical fibers in order to detect changes in comminuted meats that may be correlated with technological parameters associated with emulsion stability (e.g., cooking losses, water, and fat separation, etc.). The results of these studies suggested that light extinction spectroscopy could provide information about emulsion stability.

In addition, Nieto et al. (2014 and 2015) worked on establishing whether light scatter measured at several radial distances from the light source in fresh pork emulsions having a range of lipid oxidation, and presence or not of hydrolyzed potato protein and emulsion stability tendencies could be used to predict important final product stability indices such as textural parameters, susceptibility of the emulsion to phase separation during cooking and lipid oxidation during subsequent refrigerated storage and the results showed that light backscatter response measured during meat emulsification has potential as an early predictor of emulsion stability during finely comminuted meat products manufacturing.

The Objective of the present study is included into a global project objective, which addresses optimization of the process of meat emulsion manufacture in order to reduce the cooking losses and, concomitantly, increase the final product yield, using an optical inline sensor technology. Within the frame of this work, the first specific objective was to identify the most appropriate optical configuration for real-time stability monitoring of industrial meat emulsion samples. The second objective was to identify the optical predictors containing useful information on the stability of emulsions through the analytical determination of optical and physical-chemical parameters of pork meat emulsions and the subsequent analysis of correlations between those optical and physical-chemical parameters.

2. Setting up the optical measurement system and methodology

Previous studies were performed at the Food Engineering Lab (Department of Biosystems and Agricultural Engineering, University of Kentucky), which aimed the measurement of light scatter in laboratory-generated comminuted meats at different fiber optic distances in order to identify and detect physical-chemical changes occurring during chopping that may be correlated to emulsion stability. Based on these previous studies, an improved optical laboratory system and data acquisition methodology has been designed, set up and implemented in the Optical Sensor Laboratory at UAB (Department of Animal and Food Science) in order to optimize the acquisition of optical data in industrially-generated emulsion samples.

2.1. Differences between the previous and the current optical measuring systems

A laboratory optical sensor prototype was designed, built and tested in the Food Engineering Lab at the University of Kentucky. Two small plastic probes were built and configured such that light scatter from the sample could be detected using a High-Resolution Fiber Optic Spectrometer (Model HR4000, Ocean Optics, Inc., Dunedin, FL, USA). The light source utilized was a tungsten halogen (300-1100 nm) bulb (LS-1, Ocean Optics, Inc.).

Fiber optic cables were manufactured using 600 μm diameter fibers (Spectran Specialty Optics, Avon, CN, USA). The terminating (i.e., measuring) ends of the two fibers were built into the plastic probes while the other two ends were connected, using an SMA connector, to the spectrometer and light source. The data acquisition system consisted of a PC connected by a USB port to the spectrometer and programmed for data acquisition with SPECTRA SUIT Spectroscopy Platform Software (Ocean Optics, Inc.). Before each measurement, the terminating ends of the fibers were aligned vertically and horizontally to the same level.

Emulsion samples were placed in a double-jacketed sample holder. The fiber tips were immersed into the emulsion sample up to a final depth of approximately 12.7 mm from the surface of the sample. The temperature of the sample was controlled by means of connecting the sample holder to a water bath (Lauda Ecoline RE220. Brinkman Instruments Inc. NY. USA; ± 0.01 °C of accuracy). Light scatter intensity of the samples was measured at the target radial distances of 2, 2.5, and 3 mm from the emitting light source, and at an integration time (IT) ranging from 19 to 60 s where IT was the detector light exposure time.

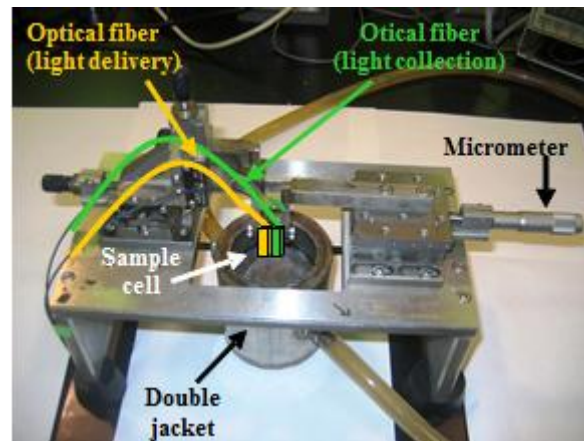


Figure 1. Initial optical device to determine the light scattering in meat emulsions at different radial distances.

Based on these previous studies, a portable measuring optical system was designed and assembled at Autonomous University of Barcelona. A halogen light source sends visible and infrared light to a measuring cuvette containing the meat emulsion samples having different levels of emulsion stability. A portable spectrophotometer measures light scatter generated by the industrially-generated samples revealing microstructural/compositional characteristics of the samples.

The new and considerably simplified control system consists of two commercial optical fibers with a diameter of 600 μm (Ocean Optics, Inc., Dunedin, FL, USA). The first one connected at one end with a miniature fiber optic spectrophotometer (High-Resolution Fiber Optic Spectrometer, model HR4000, Ocean Optics, Inc., Dunedin, FL, USA) and the other with a light scattering probe (Reflectronics Inc, Lexington, Kentucky, USA)

constituted by two separated optical fibers at a distance of 700 μm . The second fiber connected the probe to a halogen source (LS-1, Ocean Optics, inc.).

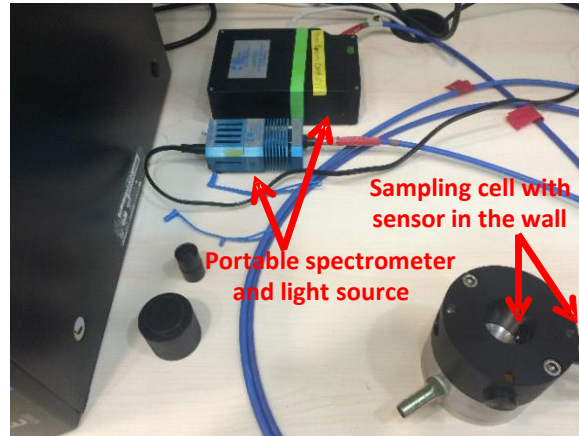


Figure 2. New portable optical device with a specific probe adapted to a distance between fibers of 700 μm .

The probe is housed in the wall of a small measuring cell (Figure 2) where the emulsion sample is placed. This cell has four holes arranged at 90° where probes (or plugs where no probe is used) can be introduced in various measurement configurations: transmission (two probes), dispersion at 90° (two probes) or backscatter at 180° (one probe) (Figure 3). An IT of three seconds was used for data acquisition.

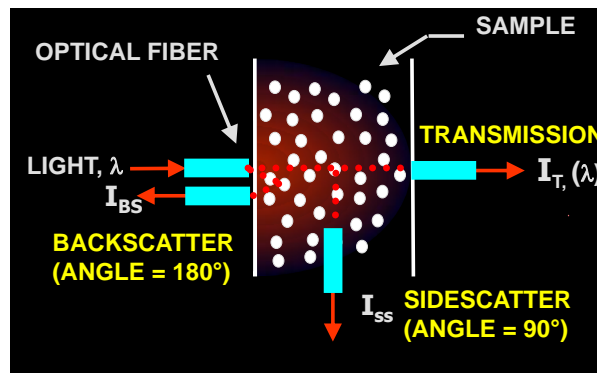


Figure 3. Alternative optical measurement configurations using fiber optic.

Light backscatter intensity of the samples was measured at a constant radial distance of 700 μm from the emitting light source, and at an integration time (IT) of 3 s, where IT was the detector light exposure time.

2.2. Optical Configuration

According to previous results, transmission configuration was evaluated in first place. However, transmission configuration was too difficult to work with. During setting up of the transmission configuration, it was observed that the shape of the transmission spectrum was inconveniently sensitive to minimal alignment deviations, which discouraged the application of this methodology using the current probes (note that the current probes contain two optic fibers). Taking in account the assays made with transmission configuration, similar difficulties were anticipated for side scatter configuration (90°), and subsequently this configuration was dismissed directly.

Experimental assays were made many times and they allowed the optimum selection of measurement settings, determining light scattering at 180° as the more suitable configuration as this configuration used only one optical unit and did not present the problems that the others configurations did. The backscatter configuration also showed the maximum reproducibility between analyses therefore was selected as the best one for determining meat emulsions information by a simple, dedicated spectrophotometric method.

2.3. Light backscatter measurement procedure

Industrially-generated meat emulsion samples were delivered by Grupo Alimentario ARGAL. Samples were delivered chilled and vacuum packed. Once samples were received, the optical analysis was immediately performed. The introduction of the sample in the measurement cell was another problem as the degree of compactness and homogeneity of the sample was highly dependent on the procedure to introduce the sample on the sampling cell. As a result, the reproducibility of the optical response varied widely between samples. As a result, a trial-error procedure was used to optimize the measurement procedure maximizing the reproducibility. After this trial-error procedure was concluded, a number of exploratory assays were performed using samples with extreme degrees of emulsion stability generated by Grupo Alimentario Argal to fine-tune the optical measurement

methodology in order to optimize differences in the spectra with a maximum response to meat emulsion stability.

2.4. Improvements obtained in the optical spectrum

By comparing the shape of the spectra resulting from previous studies with those obtained from the new optical configuration, it was found that:

a) the shape of the two spectra types was quite similar. In both cases, an absolute maximum peak was observed at ~627 nm and two relative maxima peaks at ~500 and ~560 nm were detected. In the previous optical configuration, the peaks were observed at ~493, ~560 (relative maximum peaks 1 and 2), and ~636 nm (maximum peak);

b) even when the appearance of the two relative peaks were less evident in the current spectra than in those from the previous configuration, the intensity of the maximum peak (i.e., the intensity of the optical response) was substantially much higher (4-5 times higher as an average) than in previous experiments, even though in the current configuration the IT used was substantially smaller (3 s versus ITs ranging from 20 to 60 s previously used). The increase of optical response obtained was attributed to the proximity between the optical fibers used, which seems to allow improved sensitivity and, in turn, higher accuracy and precision. On the other hand, the decrease in the IT introduced in the current configuration seems to represent a clear advantage for the industrial implementation of the proposed optical control system.

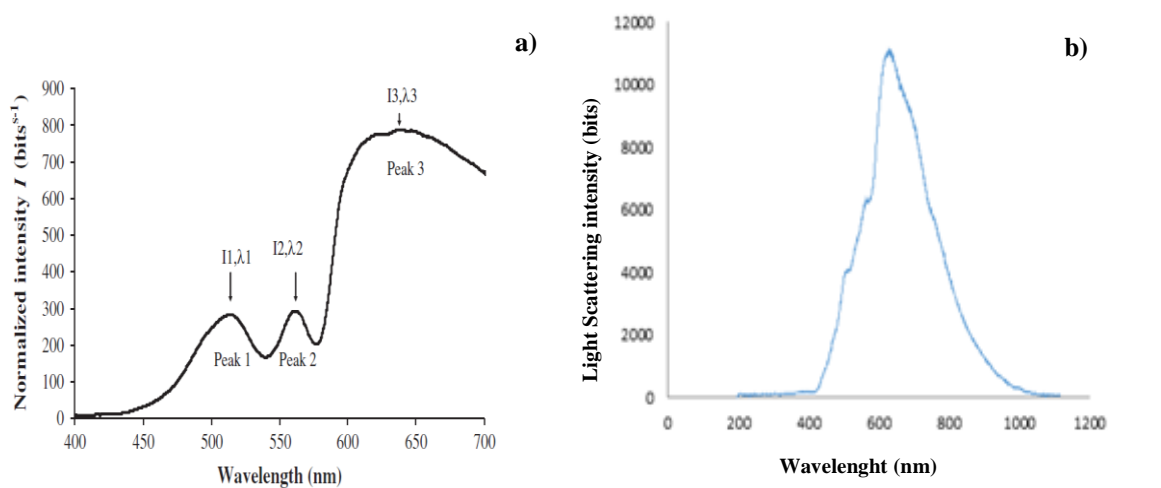


Figure 4. Typical optical spectrum scan using (a) previous configurations and (b) current configuration.

3. Experiment

3.1. Materials and methods

The first part of the study (section 2) identified the most appropriate optical configuration for real-time monitoring in meat emulsions samples while the second part addressed the identification of the optical predictors, which contained useful information not only about the stability degree of the meat emulsions, but also about several other physical-chemical emulsion properties such as color, cooking losses, chemical composition and rheological indexes as well as textural and rheological parameters in final frankfurters. The generated information was obtained and analyzed with the aim of finding correlations between the optical predictors and all the measured emulsion/frankfurter quality parameters. A total of six different treatments were analyzed corresponding to the meat emulsions and frankfurters made with two types of formula: high quality (no starch) and low quality (starch) and three different chopping speeds: low, medium and high.

3.1.1. Meat emulsion processing, chemical composition and cooking losses

Meat emulsions were prepared by Grupo Alimentario Argal, a company with a history of 25 years in the Spanish market and one of the pioneers -in its day- in the production of meat

products, using an industrial crusher/mixer INOTEC (Model IM-4500, Reutlingen Germany), where meat, salt, spices and other ingredients were mixed. After that, the mixture went through a mill homogenizer INOTEC (Model - I175CDVM-90D, Reutlingen Germany). During this second processing step, different times of chopping were obtained by changing the screw speed of the homogenizer. Homogenized mix at different speeds of chopping were stuffed in an edible collagen casing intended for human consumption for the production of fresh, cured, cooked and/or smoked sausages.

Meat emulsions and Frankfurters were delivered cold and under vacuum by Grupo Alimentario ARGAL. The company also performed a basic composition analysis of protein, moisture, fat, salt, and collagen in meat emulsions and frankfurters using a Food Scan NIR Meat Analyzer (DK-3400, FOSS, Hillerød, Denmark). The previously calibrated equipment used performed the measurement in the range of 850-1050 nm, with a precision wavelength < 0.5 nm and a wavelength accuracy < 0.01 nm. Analyses of cooking losses were made by difference of weights between fresh and cooked frankfurters.

3.1.2. Light backscatter measurement of meat emulsions

Meat emulsion samples (~5 g) were placed in the double jacked sample holder of the laboratory optical sensor prototype described in the section 2 of this study (Figure 2). The tip of the fiber probe was introduced through one of the wall holes on the side of the holder and immersed into the meat emulsion up to a depth of ~12.7 mm from the surface of the sample. Delrin caps were introduced on the remaining wall holes of the sample holder to close them and properly retain the sample inside the holder. An opaque delrin enclosure was used on the top side of the holder to isolate the sample from ambient light interference. Nine measurements were performed at room temperature using an IT of 3 s for each meat emulsion. The results were plotted in a graph and the six closest corresponding measurements were used for statistical analyses.

3.1.3. Color of meat emulsions

Color of meat emulsions were measured with a portable Hunter Lab spectrophotometer (MiniScan™ XETM, Hunter Associates Laboratory INC., Reston, Virginia, USA). Meat emulsions were measured under D65 illuminant, with a 2° observer angle. L* value describes lightness-darkness (ranges between 100-0), a* value indicates greenness (negative values) to redness (positive values) and b* values reflects blueness (negative values) to yellowness (positive values). Five measurements were made per sample at constant room temperature (~21 °C).

3.1.4. Rheology of meat emulsions and frankfurters

Rheology analyses of meat emulsions were made with a rotational rheometer (Rheo Stress 1, Haake, Thermo Electron Corporation, Karlsruhe, Germany), coupled to a thermostatic bath (Phoneix C25P, Haake, Thermo Electron Corporation). Meat emulsion samples were cautiously transferred from the bag to the flat serrated base plate MPC/DC60 and the upper flat serrated probe PP60 was slowly lowered down onto the sample until a distance of 1 mm from the base plate, and any excess of meat was carefully removed with a spatula. Dynamic oscillatory test was performed at 21 °C with a frequency sweep range of 1-100 Hz at a maximum strain of 0.01%. Rheowin software (Rheo Stress 1, Haake, Thermo Electron Corporation, Karlsruhe, Germany) was used to obtain storage (G'), loss (G'') and shear (G^*) moduli at 10 Hz. These rheology parameters were determined by quadruplicate tests by sample.

Rheology analyses of frankfurters were made with the same equipment and conditions as for meat emulsions. Four frankfurters were analyzed per treatment and they were sliced with a blade in four cylindrical pieces of ~6 mm of height. An upper flat serrated probe PP35 was slowly lowered down onto the sample until a distance of 3 mm from the base plate. Frequency sweep was carried out over the range 1-100 Hz at a maximum strain of 0.02%. The rheology analyses of frankfurters were made sixteen times by sample.

3.1.5. Texture of frankfurters

A uniaxial compression test was carried out with a TA-XT2 texture analyzer (State Microsystem, Surrey, UK). Four frankfurters were analyzed per treatment and they were sliced with a blade in four cylindrical pieces of ~1.9 mm of height. Frankfurters were compressed to 85% of their original height using a compression cylinder probe of 50 mm of diameter at a crosshead speed of 2 mm s⁻¹. Fracture force, distance and work parameters were obtained with Exponent program (Stable Micro System, Surrey, UK). The texture analyses of frankfurters were performed sixteen times per treatment.

3.1.6. Statistical analyses

The whole experiment was repeated in 3 independent occasions. Data was processed to evaluate differences between chopping speeds of the two different formulas by multifactor analysis of variance (MANOVA) and one-way (ANOVA) using the general linear models procedure of Statgraphics (Statgraphics Inc., Chicago, IL, USA) taking into account both chopping speed and production factors, as well as their interaction and the replica (i.e., the batch). LSD test was used for comparison of sample data, and evaluations were based on a significance level of $P < 0.05$. In addition, data was ordered and processed to evaluate correlations of physical-chemical characteristics –chemical composition, cooking losses, color and rheology of meat emulsions, chemical composition and rheology and texture of frankfurters– with the optical predictors by multiple-variable analysis and Pearson lineal correlation test using Statgraphics software (Statgraphics Inc.).

3.2. Results and discussion

3.2.1. Composition and cooking losses

Table 1 shows the basic composition and cooking losses of meat emulsions for both formulas at the three chopping speeds. It was found that there were no statistically

significant differences between values at any speed by formula. However there was a clear difference between formulas, having the high quality the greater values for all the analyses.

Referring to cooking losses, high quality meat emulsion reached around 3% more than low quality formula. According to Foegeding, Lanier, and Hultin (2000), improving fat coating of comminuted products, usually requires starch and non-meat proteins addition to enhance the textural properties of the products. Starch gelatinizes during cooking, increasing the emulsion viscosity and reducing fat globule mobility. Chopping operation and addition of starch are factors that influence in final product properties, resulting in smaller cooking losses and harder gels.

Similar values were found in previous studies by Allais et al. (2004), Álvarez et al. (2007) and Bañón et al. (2008). The increase of cooking losses also could be related to the speed of chopping, increased of temperature, and chemical composition of the meat emulsions (ratio fat/protein). Over-chopping and subsequent over-heating negatively affected further gelation, increasing cooking losses and diminishing gel strength (Bañón et al., 2008).

Table 1. Composition and cooking losses of meat emulsions per chopping speed for both formulas[†].

MEAT EMULSIONS								
SPEED	M (%)	CP (%)	CF (%)	S (%)	C %	L(%)		
Low quality								
Low	63.25 ± 1.09	12.38 ± 0.74	13.04 ± 1.57	2.10 ± 0.22	2.11 ± 0.43	7.67 ± 1.53		
Standard	63.50 ± 1.10	12.50 ± 0.67	13.15 ± 1.29	2.25 ± 0.21	1.59 ± 0.37	7.05 ± 0.29		
High	63.19 ± 1.05	12.55 ± 0.91	13.23 ± 1.18	2.24 ± 0.24	1.85 ± 0.38	6.73 ± 1.57		
High quality								
Low	65.11 ± 0.34	13.61 ± 1.07	14.60 ± 1.83	2.29 ± 0.05	2.21 ± 0.55	10.49 ± 2.75		
Standard	64.89 ± 0.68	13.39 ± 0.36	14.87 ± 2.05	2.29 ± 0.11	2.01 ± 0.19	10.45 ± 2.55		
High	65.02 ± 0.60	13.16 ± 0.81	14.82 ± 1.80	2.29 ± 0.13	2.15 ± 0.56	11.51 ± 2.37		

[†] Mean value ± s.d.; n=9; values per formula were not significantly different ($P<0.05$). M: Moisture; CP; Crude Protein, CF; Crude Fat; S; Salt, C: Collagen; L: Loses

As for frankfurters was concerned (Table 2), there were no statistically significant differences between values of composition at any speed by formula. Although, in general, large changes in composition between the meat emulsions and the final product were not observed, the moisture content decreased in frankfurters as compared to emulsions. This is what was expected since water is separated from the emulsion during cooking and the

surface therefore becomes dehydrated, corresponding the largest decrease to the high quality formula (Tables 1 and 2) as it does not contain starch.

In contrast, the fat values increased in frankfurters, being the larger increase observed for high quality formula. However, the difference of protein and salts contents between emulsions and frankfurters remained mostly unchanged between formulas. Collagen also increased for frankfurters, due to the collagen casing that was used when stuffing the cooked product.

Table 2. Composition of frankfurters per chopping for both formulas [†].

		FRANKFURTERS					
	SPEED	M (%)	CP (%)	CF (%)	S (%)	C %	
Low quality							
	Low	61.19 ± 1.20	15.78 ± 6.14	14.98 ± 1.46	2.17 ± 0.21	2.95 ± 0.51	
	Standard	61.22 ± 1.16	12.45 ± 0.95	14.99 ± 1.43	2.19 ± 0.19	2.89 ± 0.53	
	High	61.41 ± 0.98	12.49 ± 0.75	14.87 ± 1.45	2.14 ± 0.20	2.87 ± 0.48	
High quality							
	Low	61.51 ± 0.98	14.27 ± 0.30	17.52 ± 2.74	2.20 ± 0.11	3.36 ± 0.26	
	Standard	61.55 ± 2.84	14.32 ± 0.25	17.62 ± 2.54	2.26 ± 0.09	2.78 ± 0.65	
	High	61.16 ± 1.64	14.53 ± 0.20	17.75 ± 2.39	2.23 ± 0.06	3.01 ± 0.18	

[†] Mean value ± s.d.; n=9; values per formula were not significantly different ($P<0.05$). M: Moisture; CP; Crude Protein, CF; Crude Fat; S: Salt, C: Collagen.

Values obtained for frankfurters composition are within the usual commercial ranges as indicated by Denmark National Food Institute.

3.2.2. Optical Predictors

The letters presented in Table 3 corresponded to 58 optical predictors found by the analysis performed with the optical sensor, but for confidentiality reasons the description/definition of each predictor and the mean values have not been presented in the table but only the classification obtained by the statistical method MANOVA LSD.

The values of all predictors indicated in the table were found to be significantly different depending on the speed for the two formulas or at least for one of them. Predictors 10 and 15 for both formulas showed significantly different values for the three speeds. This means

that these two predictors were able to categorize meat emulsion processing speeds independently of the formula. However, a wide range of predictors was able to categorize meat emulsions processing speeds depending on the formula. Predictors showing different values for the three different speeds were predictor 20 for low quality formula, and predictors 9, 11, 14, 16 in the case of high quality.

The rest of predictors showed significantly different values in one or both formulas but they were not able to categorize the three speeds since at least two of the latter were found not to be statistically different.

Table 3. Ranges of different optical predictors per chopping speed for both formulas.

	Low quality			High quality		
	Low speed	Standard speed	High speed	Low speed	Standard speed	High speed
Predictor 1	a	b	b	a	a	a
Predictor 3	c	c	b	b	b	b
Predictor 4	b	a	a	a	a	a
Predictor 5	a	a	a	a,b	a	b
Predictor 6	a	a	a	a,b	a	b
Predictor 9	a	b	a	c	b	a
Predictor 10	a	c	b	c	b	a
Predictor 11	b	b	a	c	b	a
Predictor 12	a	a	a	b	a,b	a
Predictor 13	a,b	b	a	a	a	a
Predictor 14	a	b	a	c	b	a
Predictor 15	a	c	b	c	b	a
Predictor 16	a	b	a	c	b	a
Predictor 17	b	a	a	a	a	a
Predictor 18	a	b	b	a	a	a
Predictor 19	a	b	b	a	a	a
Predictor 20	c	a	b	a	a	a
Predictor 21	a	b	b	a	a	a
Predictor 22	b	a	a	a	a	a
Predictor 23	a	a	a	b	a	a,b
Predictor 24	a	b	b	b	a	b
Predictor 25	b	a	a	b	a	b
Predictor 26	a	a	a	a	b	a,b
Predictor 27	b	a	a	a	b	a
Predictor 28	a	b	b	a	b	a
Predictor 33	b	a	a	a	a	a
Predictor 34	a	a	a,b	a	a	a
Predictor 35	b	a	a,b	a	a	a
Predictor 36	a	a	a,b	a	a	a
Predictor 39	b	a	a,b	a	a,b	b
Predictor 40	a	b	a,b	b	a,b	a
Predictor 41	a	a	a	a	a,b	b
Predictor 42	a	a	a	b	a,b	a
Predictor 43	b	a	a	a	a	a
Predictor 44	a	b	b	a	a	a
Predictor 47	a	b	b	a	a	a
Predictor 48	b	a	a	a	a	a
Predictor 49	b	a	a,b	a	a	a
Predictor 50	a	a	a	a,b	a	b
Predictor 51	a	a	a	b	a	a,b
Predictor 52	a	b	a	b	a	a,b
Predictor 53	a	a	a	b	a	b
Predictor 54	a	b	a,b	a	a	a
Predictor 55	a	a	a	a,b	b	a
Predictor 56	a	a	a	a	b	a,b
Predictor 57	b	a	b	a	b	a,b
Predictor 58	a	a	a	a	b	a

n=54; values per formula without common characters were significantly different ($P<0.05$).

3.2.3 Color

Color of meat emulsions was significantly affected by chopping speed (Table 4), production and the interaction between both of them. Considerable changes in light reflection were observed between formulas. Lightness (L^*) for high quality formula obtained higher values against low quality formula. In contrast, a^* and b^* values were larger for low quality formula.

Different trends were observed taking into account the formula. For example, in the case of L^* value, significantly higher values were observed for low speed emulsions in low quality formula whereas in high quality L^* value significantly decreased compared with the other two speeds. In the case of a^* values, for low quality formula, the value increased as the speed increased; on the contrary, for high quality formula, a^* value decreased as the speed increased. Finally, the behavior of b^* value did not show a clear trend. Higher b^* values were observed for standard speed in the low quality formula, which corresponded to the lowest b^* value in high quality emulsions.

As for chopping speed, similar trend values for L^* in low and high quality formula were found by Álvarez et al. (2007) and Bañón et al. (2008). According to Barbut (1998) and Álvarez et al. (2007), the maximum L^* value corresponds to the maximum degree of meat emulsion stability (i.e., minimum cooking losses) during chopping and could provide useful information to predict cooking losses.

The increase in redness (a^*) for low quality emulsions means that these samples reflected more light and could be explained by a reduction of exudation, i.e., quantity of liquid in the meat emulsion surface was likely smaller, since starch improves the water-binding capacity (Bañón, 2008). Indeed, color changes in meat batters during chopping have been attributed to a combination of physical-chemical phenomena, probably associated with fat particle size variations, the presence of air bubbles and/or protein-fat interactions (Álvarez et al., 2007). In fact, both the reduction in fat particle size and the presence of air bubbles entrapped in the meat emulsion may act as light scattering agents (Palombo et al., 1994).

Table 4. Color of meat emulsions per chopping speed for both formulas [†].

	L*	a*	b*
Low quality SPEED			
Low	59.90 ± 0.89 ^a	18.53 ± 0.65 ^b	21.74 ± 0.76 ^b
Standard	59.08 ± 0.89 ^b	18.68 ± 0.88 ^b	22.43 ± 0.60 ^a
High	58.94 ± 0.85 ^b	19.54 ± 0.45 ^a	21.69 ± 0.69 ^b
High quality			
Low	62.07 ± 0.84 ^b	16.86 ± 0.53 ^a	20.72 ± 0.22 ^{a,b}
Standard	63.28 ± 0.60 ^a	16.46 ± 0.45 ^b	20.44 ± 0.10 ^b
High	63.09 ± 0.21 ^a	16.39 ± 0.12 ^b	20.87 ± 0.25 ^a

^{a-b} Mean value ± s.e.; n=45; values per formula without common superscripts were significantly different ($P<0.05$). [†] L*: lightness; a*: green-red component; b*: blue-yellow component.

3.2.4. Rheology of meat emulsions and frankfurters

The storage modulus (G') of tested meat emulsions were always higher than the loss modulus (G''), which demonstrates that meat emulsions have an elastic characteristic rather than a viscous one (Table 5). Therefore, meat emulsions can be described as a weak gel or to have gel-like behavior (Flores et al., 2007; Kara-man et al., 2011; Dogan et al., 2013; Hernandez-Marín et al., 2013; Li et al., 2013; Savadkoohi et al., 2013).

Moreover, the rheological parameters studied (G' , G'' and G^*) were significantly influenced by both factors speed and production and their interaction, only for high quality formula. For low quality formula, their values decreased with increasing chopping speed although no statistical differences were found between low and standard speeds for the two formulas.

In general the values of the three moduli for high quality formula were lower than those for low quality formula, which could mean that the starch addition increased these figures and contributed to the elastic properties of the low quality meat emulsion.

Table 5. Rheology of meat emulsions per chopping speed for both formulas †.

MEAT EMULSIONS						
SPEED	G' (kPa)		G'' (kPa)		G^* (kPa)	
Low quality						
Low	8.88 ±	0.28 ^a	1.90 ±	0.06 ^a	9.08 ±	0.29 ^a
Standard	8.55 ±	0.26 ^a	1.82 ±	0.05 ^a	8.75 ±	0.27 ^a
High	8.43 ±	0.48 ^a	1.82 ±	0.11 ^a	8.62 ±	0.49 ^a
High quality						
Low	8.33 ±	0.20 ^b	1.76 ±	0.03 ^{a,b}	8.51 ±	0.20 ^b
Standard	8.36 ±	0.18 ^b	1.73 ±	0.05 ^b	8.53 ±	0.19 ^b
High	8.98 ±	0.13 ^a	1.84 ±	0.04 ^a	9.17 ±	0.13 ^a

^{a-b} Mean value ± s.e.; n=36 values per formula without common superscripts were significantly different ($P<0.05$). † G' : storage modulus; G'' : loss modulus; G^* : shear modulus.

As for high quality formula meat emulsions, the values of the three moduli in cooked frankfurters were significantly influenced by both factors (speed and production) and their interaction. In general, high quality formula showed greater G' , G'' and G^* than low quality. Standard speed yielded the highest values for the three parameters in both low and high quality formulas.

Rheology in frankfurters has not been as studied as texture but in our study we can make comparisons with the raw emulsions. For both low and high quality formulas, the three moduli increased in frankfurters. However, it can be observed that for low quality formula meat emulsions there were no significant differences between speeds whereas in frankfurters the differences between speeds in low quality formula were clear. Additionally, for frankfurters there was a clear difference between low and standard speed irrespectively of the formula considered.

Table 6. Rheology of frankfurters per chopping speed for both formulas †.

FRANKFURTERS						
SPEED	G' (kPa)		G'' (kPa)		G^* (kPa)	
Low quality						
Low	18.75 ±	0.81 ^b	3.97 ±	0.14 ^b	19.17 ±	0.82 ^b
Standard	21.48 ±	0.60 ^a	4.46 ±	0.11 ^a	21.94 ±	0.61 ^a
High	20.11 ±	0.70 ^{a,b}	3.93 ±	0.11 ^b	20.50 ±	0.71 ^{a,b}
High quality						
Low	25.81 ±	0.82 ^b	5.02 ±	0.16 ^b	26.30 ±	0.84 ^b
Standard	28.20 ±	1.31 ^a	5.33 ±	0.25 ^a	28.70 ±	1.33 ^a
High	27.09 ±	0.86 ^{a,b}	5.28 ±	0.14 ^{a,b}	27.61 ±	0.86 ^{a,b}

^{a-b} Mean value ± s.e.; n=144; values per formula without common superscripts were significantly different ($P<0.05$).

† G' : storage modulus; G'' : loss modulus; G^* : shear modulus.

3.2.5. Texture of frankfurters

Meat emulsions such as e.g., sausage and pate are emulsions in which the continuous phase is a complex colloidal system of gelatin, proteins, minerals and vitamins, were fat globules are dispersed (Yada, 2004). In solid food emulsions, the texture is determined by the composition of the food, the homogenization conditions and post-processing operations such as heating or freezing (Fellows, 2000).

To measure the texture of frankfurters, a Uniaxial Compression Test was performed in which two peaks corresponding to two successive fractures were observed. The first fracture was internal, which was not visually observed (i.e., inside meat) while the second one represented a larger resistance by the cooked collagen membrane.

As per the rheology, texture profile of frankfurters was affected by both factors (chopping speed and formulation) and their interaction. Frankfurters made with low quality formula and chopped at standard speed required less force to start breaking (Table 7). Equally, frankfurters made with the mentioned formula and speed required the minimum force to get finally broken. In frankfurters made with high quality formula, the minimum force to start braking was obtained with meat emulsion chopped at high speed.

Values for distance remained similar for low and high quality formula, which means that frankfurters showed similar percentage of plasticity despite of the quantity of added starch. Since our results showed that low quality formula obtained the lowest values for force this could mean that the percentage of added starch did not affected the final structure of the frankfurters.

In high quality formula, the mentioned decrease of firmness observed as chopping speed increased might have been due to improper fat emulsification as fat size decreased (i.e., the globule surface to volume ratio increased) and/or to the local denaturation of meat emulsifying proteins. This lack of consistency was typically accompanied by a significant increase of cooking losses (Barbut, 1998).

The texture of foods is mostly determined by the moisture and fat contents, and the types, amounts and interactions of structural carbohydrates (cellulose, starches and pectic materials) and proteins that are present. Changes in texture are caused by loss of moisture or fat, formation or breakdown of emulsions and gels, hydrolysis of polymeric carbohydrates, and coagulation or hydrolysis of proteins (Felloes, 2000).

Bourne (2002) and Bañón et al. (2008) described the variables force and distance as the mechanical parameter of hardness and springiness. The figures presented in Table 7 agree with Allais et al. (2004), which found that hardness decreased as speed increased. Bañón et al. (2008) found that with less chopping time the value of hardness and springiness decreased despite of the added percentage of starch according with our previous conclusion about starch.

Table 7. Texture of frankfurters per chopping speed for both formulas)[†].

FRANKFURTERS					
SPEED	Force 1 (N)	Distance 1 (mm)	Force 2 (N)	Distance 2 (mm)	
Low quality					
Low	64.49 ± 1.42 ^b	9.50 ± 0.07 ^a	80.17 ± 1.28 ^a	12.31 ± 0.12 ^a	
Standard	62.23 ± 1.97 ^b	9.47 ± 0.09 ^a	75.39 ± 1.45 ^b	11.94 ± 0.06 ^b	
High	63.74 ± 2.24 ^b	9.22 ± 0.10 ^b	79.35 ± 1.89 ^a	12.01 ± 0.09 ^b	
High quality					
Low	81.41 ± 1.75 ^a	9.56 ± 0.11 ^a	95.07 ± 2.55 ^b	11.76 ± 0.15 ^b	
Standard	77.23 ± 1.58 ^b	9.22 ± 0.12 ^b	96.04 ± 1.39 ^b	11.76 ± 0.08 ^b	
High	75.93 ± 2.11 ^b	8.94 ± 0.12 ^b	96.48 ± 1.51 ^b	11.72 ± 0.06 ^b	

^{a-b} Mean value ± s.e.; n=169 and 126 for low quality formula and high quality formula respectively; values per formula without common superscripts were significantly different ($P<0.05$).

[†] Force 1 and Distance 1: force and distance

3.2.6. Correlations

As shown in Table 8, there is a positive correlation between predictors 54 to 58 and moisture and cooking losses; however predictors 9 and 10 showed the highest correlations with cooking losses. Predictor 18 showed a high correlation with moisture and protein. , As show in Table 9, predictors 19 and 28 showed a high negative correlation with salt content in frankfurters whereas predictors 20 and 24 showed the highest positive correlation with the same additive.

Additionally, when comparing tables 8 and 9, it can be observed that a number of the correlations obtained in the meat emulsions were not observed in the frankfurters. As expected, many predictors were correlated with color attributes as a result of the optical nature of both types of measurements.

Table 8. Correlation matrix with Pearson values between the variables (optical predictors vs. physical-chemical parameters) with significant correlations – Physical-chemical parameters considered: basic composition of meat emulsions and cooking losses.

Predictors	MEAT EMULSIONS					
	L (%)	M (%)	CP (%)	CF (%)	S (%)	C (%)
Pred. 1		-0.62	-0.71	-0.60		
Pred. 3					0.73	-0.05
Pred. 9	-0.70	-0.53		0.34	0.73	-0.05
Pred. 10	0.68			0.75		0.67
Pred. 11	0.53			0.61		
Pred. 17	-0.49	-0.87	-0.21			
Pred. 18	0.52	0.87	0.74			
Pred. 19		-0.68	-0.70		0.88	
Pred. 20		0.64	0.67		0.89	-0.47
Pred. 21						0.71
Pred. 22				-0.47		-0.71
Pred. 23	-0.65	-0.55		-0.57		-0.55
Pred. 24					0.57	
Pred. 25				-0.55	0.64	-0.70
Pred. 26	0.63	0.59		0.52		0.53
Pred. 27			-0.50		-0.65	
Pred. 28				0.50	-0.70	0.67
Pred. 54	0.56	0.55				
Pred. 55	0.56	0.57				
Pred. 57	0.56	0.54				
Pred. 58	0.54	0.51				

n=54; values per formula without common characters were significantly different ($P < 0.05$). M: Moisture; CP; Crude Protein, CF; Crude Fat; S: Salt, C: Collagen; L: Loses

For color, predictors 19 to 28 showed correlations with the three color attributes: L*, a* and b*. The highest positive and negative correlations for L* and a* values corresponded to predictor 10 (r values of 0.92 and -0.89, respectively). As per b* value, predictors 24, 25, 27 and 28 had the highest correlation values ($|r| > 0.85$). As a consequence of the intrinsic adaptability of light scatter measurements for inline implementation, the existing correlation between color attributes and numerous light backscatter predictors represents an

advantage, which shows potential for industrial meat emulsion processing control improvement.

Table 9. Correlation matrix with Pearson values between the variables (optical predictors vs. physical-chemical parameters) with significant correlations – Physical-chemical parameters considered: composition of cooked frankfurters and color properties of meat emulsions.

Predictors	Meat Emulsions			Frankfurters			
	L*	a*	b*	M (%)	CF (%)	S (%)	C (%)
Pred. 1	-0.63	0.56	0.60		-0.68		
Pred. 9	-0.79	-0.78	-0.73	-0.58		-0.71	
Pred. 10	0.92	-0.89	-0.66	-0.49	0.75		
Pred. 11	0.75	-0.72	-0.45	-0.60	0.61		
Pred. 17						-0.59	
Pred. 18						0.53	
Pred. 19	0.49	-0.52	-0.67			-0.81	0.50
Pred. 20	-0.51	0.53	0.67			0.79	-0.50
Pred. 21	0.59	-0.57	-0.56				
Pred. 22	-0.60	0.59	0.57		-0.48		
Pred. 23	-0.62	0.58	0.65		-0.62		
Pred. 24	-0.61	0.59	0.85			0.75	-0.65
Pred. 25	0.76	0.74	0.88		-0.52	0.57	-0.62
Pred. 26	0.55	-0.51	-0.60		0.58		
Pred. 27	0.59	-0.58	-0.85			-0.81	0.65
Pred. 28	0.73	-0.72	-0.89			-0.64	0.62
Pred. 54	0.57	-0.48					
Pred. 55	0.56	-0.47					
Pred. 56	0.58	-0.49					
Pred. 57	0.59	-0.50					
Pred. 58	0.53	-0.44					

n=54; values per formula without common characters were significantly different ($P<0.05$). M: Moisture; CP; Crude Protein, CF; Crude Fat; S: Salt, C: Collagen; L: Loses, L*: lightness; a*: green-red component; b*: blue-yellow component.

Values observed in table 8 and 9 agree with previous studies of Álvarez et al., (2009, 2010 and 2014), which showed that many of the optical parameter (predictors) studied were highly correlated with cooking losses and color parameters in lab-scale generated emulsions.

Data presented in Table 10 shows that predictors 27 and 33 had a high negative correlation with G' , G'' and G^* values for meat emulsions while predictors 24 and 34 showed the highest positive correlation for these variables.

Table 10. Correlation matrix with Pearson values between the variables (optical predictors vs. physical-chemical parameters) with significant correlations – Physical-chemical parameters considered: rheological properties of meat emulsions and frankfurters.

Predictors	Meat Emulsions			Frankfurters		
	G' (kPa)	G'' (kPa)	G^* (kPa)	G' (kPa)	G'' (kPa)	G^* (kPa)
Pred. 1		0.53				
Pred. 3	0.52	0.48		0.48		
Pred. 5				-0.53		-0.52
Pred. 8	0.58	0.53	0.57			
Pred. 9	-0.50	-0.50	-0.50			
Pred. 12	-0.68	0.75	-0.69			
Pred. 13	-0.68	-0.64	-0.68			
Pred. 14	-0.59	-0.60	-0.59			
Pred. 17				-0.57	-0.47	-0.57
Pred. 18				0.54		0.54
Pred. 19	-0.65	-0.54	-0.65	-0.59	-0.55	-0.59
Pred. 20	0.64	0.54	0.64	0.57	0.54	0.57
Pred. 23		0.56				
Pred. 24	0.84	0.85	0.84			
Pred. 25	0.71	0.73	0.71			
Pred. 26		-0.53				
Pred. 27	-0.86	-0.84	-0.86			
Pred. 28	-0.75	-0.75	-0.75			
Pred. 33	-0.86	-0.84	-0.86			
Pred. 34	0.84	0.85	0.84			
Pred. 35		0.48				
Pred. 37					-0.47	-0.57
Pred. 38						0.54
Pred. 39	-0.65	-0.54	-0.65		-0.55	-0.59
Pred. 40	0.64	0.54	0.64	0.57	0.54	0.57
Pred. 42		-0.51				
Pred. 45	0.72	0.80	0.72			
Pred. 46	-0.74	-0.80	-0.74			
Pred. 47	0.63	0.69	0.63			
Pred. 48	-0.65	-0.70	-0.65			
Pred. 49				-0.50		-0.49
Pred. 50						-0.53
Pred. 55				0.48		0.47
Pred. 56				0.48		0.47
Pred. 58				0.51		0.51

n=54; values per formula without common characters were significantly different ($P<0,05$). G' : storage modulus; G'' : loss modulus; G^* : shear modulus.

As observed in composition correlations, rheology of meat emulsion presented more significant correlations than rheology for frankfurters. This could be because the structure of frankfurters changed with the cooking process, which presumably modified the existing

correlations. Therefore according to table 10, predictors have more potential to predict successfully rheological properties in meat emulsions than in frankfurters.

According to Table 11, predictors 19 and 39 obtained the highest negative correlations for force 1 and predictors 17, 19 and 37 showed the highest negative correlations for force 2. Predictors 17 to 20 were highly correlated with the three of the four textural attributes tested: forces 1 and 2, and distance 2.

Generally, Table 11 shows that many of the predictors obtained are correlated with texture parameters, which suggests that the use of an optical sensor to study light backscatter in finely comminuted meat emulsions may have potential for predicting the texture of the final product.

Values observed in table 11 agree with previous studies of Álvarez et al. (2014), which showed that many of the optical parameter (predictors) studied were highly correlated with hardness texture parameter. Additionally, the results point in a similar direction to those obtained by Allais et al. (2004) who suggested that prediction found a correlation between final texture parameters of meat emulsions and fluorescence spectroscopy measurements.

Table 11. Correlation matrix with Pearson values between the variables (optical predictors vs. physical-chemical parameters) with significant correlations – Physical-chemical parameters considered: texture properties of frankfurters.

Frankfurters			
Predictors	Force 1 (N)	Force 2 (N)	Distance 2 (mm)
Pred. 5	-0.55	-0.67	0.54
Pred. 6		-0.48	
Pred. 7		0.57	
Pred. 8	0.55		
Pred. 9	-0.51		
Pred. 13	-0.67	-0.61	0.48
Pred. 14	-0.53		
Pred. 17	-0.69	-0.74	0.53
Pred. 18	0.64	0.70	-0.53
Pred. 19	-0.76	-0.70	0.57
Pred. 20	0.73	0.67	-0.56
Pred. 24	0.48		
Pred. 27	-0.56	-0.47	
Pred. 29			-0.51
Pred. 30			0.50
Pred. 31		-0.49	
Pred. 32			
Pred. 33	-0.59	-0.50	0.22
Pred. 34	0.48		
Pred. 37	-0.69	-0.70	0.57
Pred. 38	0.64	0.70	
Pred. 39	-0.76		
Pred. 40	0.73	0.67	-0.56
Pred. 49		-0.48	
Pred. 50		-0.51	
Pred. 55		0.47	
Pred. 58		0.49	

n=54; values per formula without common characters were significantly different ($P<0.05$).

4. Conclusions

The numerous correlations found strongly suggest the feasibility of these optical parameters not only as potential predictors of meat emulsification degree and potential exudates but also of relevant rheological and textural attributes of meal emulsions and frankfurters at industrial level. The information reported by light backscattering configuration could have potential in the development of a new inline optical sensor technology to select the optimum end-point of chopping that would be able to minimize cooking losses and maximize the yield.

Successful development of this technology could be implemented at industrial level since the samples used for the experiment were made and delivered by an important meat process plant and the results obtained were similar to those found in previous studies named in this work where the experiment was made at lab scale or in a pilot plant.

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