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REVIEW ARTICLE

Application of Spectroscopic Techniques to Evaluate Heat Treatments in Milk and Dairy Products: an Overview of the Last Decade

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Abstract

In the food industry, thermal treatments are generally an essential step to increase the shelf life of the products. This is especially true for milk and dairy products in which heat treatments help to eliminate pathogenic organisms, minimize microbiological development, and improve some sensory properties. However, they can also induce biochemical, physico-chemical, and sensory changes in foods, and then adversely affect the final quality of the products. To assess the quality of milk and dairy products during heating, some non-destructive techniques exist. In this article, the application of spectroscopic non-destructive techniques (fluorescence, infrared, NMR) is analyzed to point out the pertinence by using them as tools to monitor milk and dairy product quality changes during heating. An overview of the last studies on the effect of different conventional and emerging methods of milk and dairy product heating on biochemical, physico-chemical, and sensory quality is also presented, as well as the perspectives of research in this topic.

Keywords Milk · Dairy products · Heating · Sensory · Quality · Spectroscopy techniques

Introduction

Milk and dairy products are important food components in the human diet. They offer a number of nutritional compounds (e.g., protein, fat, salts, vitamins) and are useful as ingredients in the food industry, especially milk that can provide a great diversity of subproducts (e.g., milk powder, cheese, butter, cream, etc.) (Dominguez-Salas et al., 2018).

The evaluation of milk and dairy product qualities (biochemical, sensory, physico-chemical, and microbiological) is very important in the dairy industry because it influences several subsequent processes as well as the safety of the final products. Since thermal treatments are widely employed in this sector and because they are directly implicated in changes of these mentioned qualities, it is important to monitor their effects on milk and dairy products. Currently, heat treatments of milk are primarily applied to inactivate pathogenic microorganisms and enzymes, to develop stability of products during storage, and subsequently to extend shelf life. They are also used in order to modify the structure of specific molecules and by consequence influence positively the texture of the final products (e.g., cheese, yoghurt) or change the performance of subsequent technological operations (Lyck et al., 2007; Miloradovic et al., 2018; Sfakianakis and Tzia, 2014). For example, heat treatments lead to the denaturation of specific proteins, improving the texture of the cheeses and the technological yield. Thermal processing can also enhance color, taste, and flavor of milk and dairy products (e.g., cheese browning, cheese melting). However, heat treatments can also affect the physico-chemical, technological, sensory, and/or nutritional characteristics of products. For example, the Maillard reaction can appear

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in heat-treated products and be undesirable by its involvement in the degradation of the lysine amino acid and the development of unpleasant flavors (Aktağ et al., 2019). The pre-heat treatment applied to the liquid skim milk before spray drying typically controls the degree of whey protein denaturation, but in turn affects its functional properties (Patel and Patel, 2015; Sharma et al., 2012). The extent of these changes depends on the method of heating used, the time-temperature factor applied, the processing facilities affecting the energy transfer (i.e., direct or indirect heating), the component ratios, the humidity content, and the nature of the product.

Different types of heat treatments can be used in the milk and dairy industry. Conventional methods such as thermization, pasteurization, cooking, and sterilization are well-established and known techniques (Fig. 1), but present some disadvantages linked to the temperature gradient and reduction of the heating rate, as well as to some damages in the structure and/or components of the raw material. Alternative methods such as ohmic heating, ultrasonication, microwaves, and high-intensity pulsed electric fields (Fig. 1) have been studied and developed in the last years (Aboud et al., 2019; Cappato et al., 2017; Martins et al., 2019), but they also present disadvantages.

In any case, heat treatments when applied to a food product must take into account the preservation of its organoleptic, nutritional, and functional qualities. To control the effect of these treatments on material changes, industry and laboratories mostly use techniques like sensory analysis, microbiological enumeration, biochemical procedures, and physico-chemical measurements. These methods are useful, but they are often time-consuming and destructive and induce a time lag in relation to the application time of the heat process. For these reasons, it is difficult to use these techniques in-line and they are not always adapted to the technical and regulatory requirements of the modern food industry.

To overcome these constraints, several spectroscopic techniques in different ranges of the wavelength (UV-VIS, IR, fluorescence, Raman) have been studied in milk and dairy products in the last years. Their objectives are to procure information about authentication, adulteration, biochemical characteristics of the products, and changes in process and/or to obtain a useful process analytical technology (PAT). The use of these spectroscopic techniques is based on their generally reported advantages, i.e., fast, low cost, non-destructive, and accurate, as well as the possibility of being implemented for monitoring processes in real time. This makes it possible to control and optimize the production line, while guaranteeing high-quality production that complies with technical and regulatory requirements (Hassoun et al., 2020). Figure 2 shows that there is an increase in the use of these techniques as PAT in the milk and dairy industry in general, but also in the monitoring of heat treatment in particular. Several review articles on non-destructive techniques for monitoring food processing have been published recently (Loudiyi et al., 2020; Lei and Sun, 2019; Hassoun et al., 2020), but no review focusing on the potential of spectral techniques to assess changes in the quality of milk and dairy products during heat treatments has been found.

Therefore, the aims of this review are to summarize the original studies published over the past decade about the influence of heat treatments on the biochemical, physico-chemical, and sensory changes of milk and dairy products and to highlight the potential of the different types of spectroscopic techniques to monitor these changes in these kinds of foods. In this manner, an overview of conventional and emergent methods of heating usually used in the milk and dairy industry is presented. Then, an exposition of the most relevant studies on the effect of heating on the biochemical, physico-chemical, and sensory qualities is shown. Finally, the characteristics and applications of different spectroscopic techniques (Table 1),

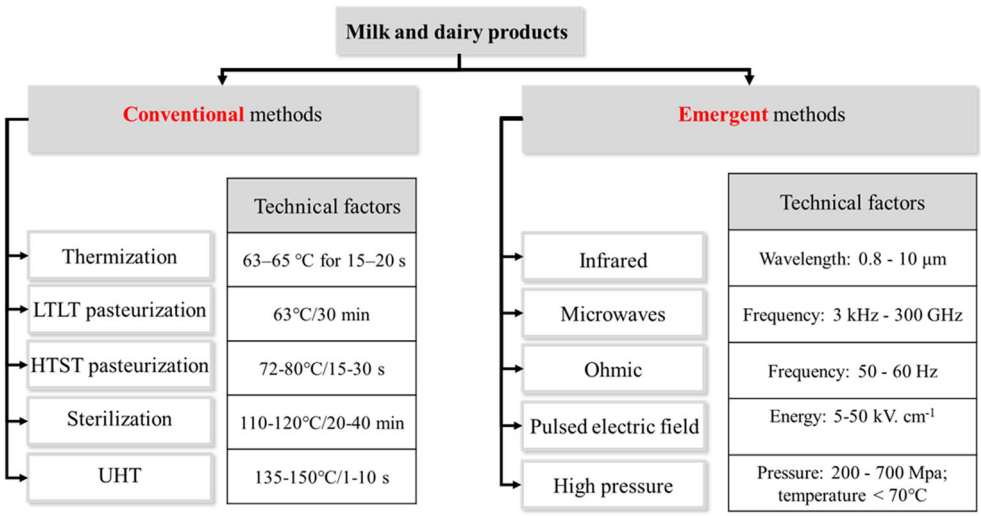

Fig. 1 Schematic diagram presenting technical factors of different methods of heat treatment applied to milk and dairy products

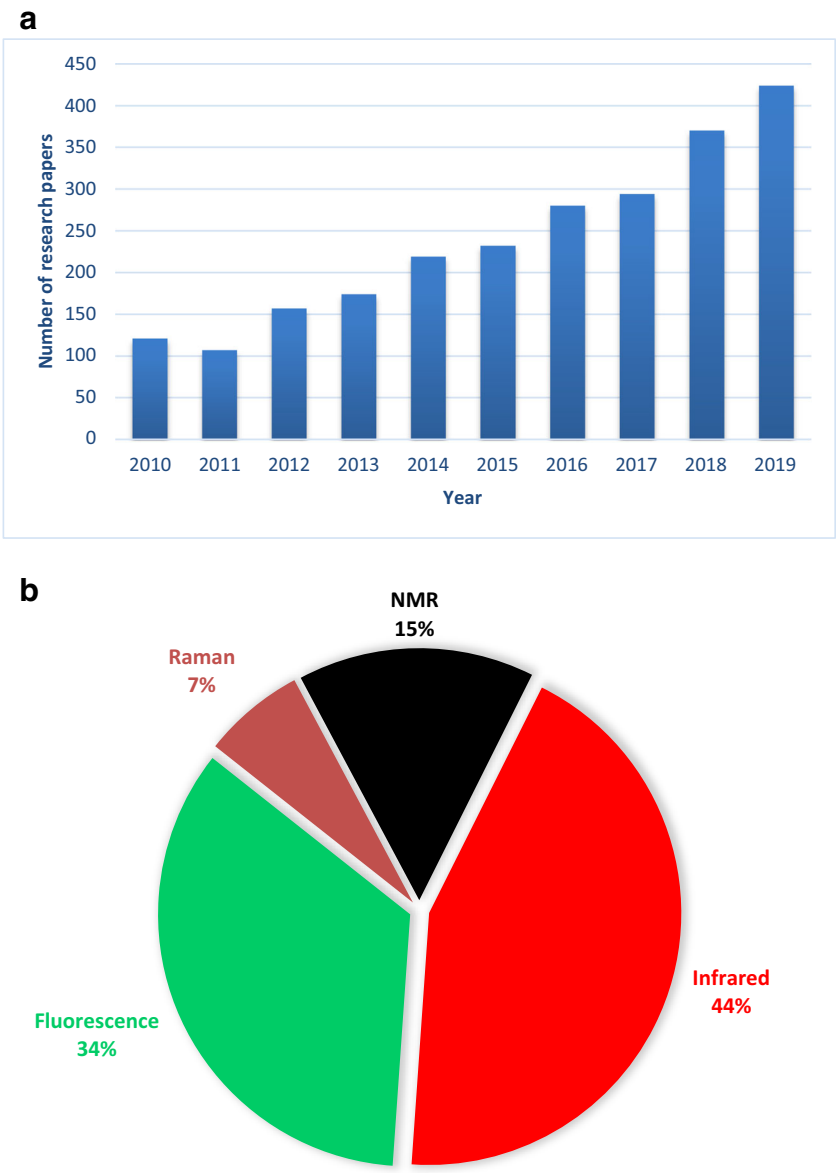

Fig. 2 **a** Total number of scientific articles published on the application of spectroscopic techniques for monitoring thermal treatments in milk. The data was obtained from the database of ScienceDirect the 23rd of July 2020 [*search criteria: Title-abstract-keywords (Thermal treatment) AND Article title-Abstract-Keywords (Spectroscopy) AND Article title-Abstract-Keywords (Milk)*]. **b** The proportions of scientific articles published from 2010 to 2019 based on the spectral techniques for monitoring thermal treatments in milk [*search criteria: Title-abstract-keywords (Thermal treatment) AND Article title-Abstract-Keywords (Spectroscopy) AND Article title-Abstract-Keywords (Milk) AND Article title-Abstract-Keywords (Infrared) OR Article title-Abstract-Keywords (Fluorescence) OR Article title-Abstract-Keywords (Raman)*]

their advantages and disadvantages, and the most research trends existing in this topic are described and discussed in order to complete this review.

Different Methods of Heat Treatment of Milk and Dairy Products

Conventional Methods of Heating

Milk generally needs to be heated to remove harmful microorganisms and enzymes, to be transformed (e.g., to produce powdered milk), or to produce different types of dairy products (e.g., cheese, yoghurt, cream). Conventional heat treatments at different temperature-time conditions have been widely used in the milk and dairy industry: thermization, pasteurization, ultra-pasteurization, sterilization, and extended shelf life (ESL). The choice of the treatment generally depends on consumer preferences, the target market, and the shelf life desired for the final products. For example, the use of thermization prevents deterioration in the quality of milk when it is necessary for the milk to be cooled for a period before being processed. The temperatures at which this is usually done are between 57 and 78 °C for 10–20 s, with the aim of reducing growth of psychotropic bacteria which may release heat-resistant proteases and lipases into the milk and then alter its quality. Pasteurization and sterilization techniques are certainly the most used treatments in the industry of milk and dairy products. Pasteurization is the thermal process in which the temperatures and holding times range between 63 °C for 30 min (low temperature–long time, LTLT) and 71 °C for 15 s (high temperature–short time, HTST). The

Table 1 Examples of studies that evaluated the potential of spectroscopic techniques to monitor thermal treatments in milk and dairy products

Product	Thermal treatment	Spectroscopic technique	Main results	Reference
Cheddar cheese	Different storage temperatures	MIR + Raman	Lower storage temperatures delay oxidative processes	(Firmani et al., 2020)
Milk	UHT	ATR-IR	ATR-IR can be exploited to assess proteolysis in UHT milk	(Ranvir et al., 2020)
Milk	Heat treatment (25–80 °C)	FTIR + NMR	Mineral behaviors are affected by thermal heating	(Boiani et al., 2018)
Milk powders	Heat treatment (25–100 °C)	FTIR	The main changes in the secondary structure of proteins appear at a temperature higher than 70 °C in whole milk and in infant formula at 50 °C. The β-structures (β-sheet and β-turn) in the whole milk powder both decreased in the range of 70 to 85 °C, whereas α-helix structures increased.	(Ye et al., 2017)
Milk	Vat pasteurization (63–64.5 °C, 30 min)	^{1}H-NMR, GC-MS, UPLCQ-ToF/MS	Pasteurization was a mild process that affects nutritional compounds at a minimum, while significant differences were obtained after 8 d of storage	(Zhu et al., 2020a)
Mozzarella cheese	Heat treatment (20 to 65 °C)	NMR	Heating of cheese resulted in an increase in the free water content and an increase in the mobility of cheese fat	(Smith et al., 2017)
Cantal cheese	Heat treatment (20 to 60 °C)	FFSFS	FFSFS combined with ICA can be used to monitor fat melting	(Loudiyi et al., 2018a)
Tilsit cheese	Heat treatment (25 to 75 °C)	FFFS	Fluorescence spectroscopy has the potential for accurate, non-destructive, and rapid prediction of cheese melting temperature ($0.98 \leq R^2 \leq 0.99$)	(Ozbekova and Kulmyrzaev, 2017)
Milk	Pasteurization, sterilization, and UHT	FFFS	Excellent results in the discrimination of the milk samples according to the heat treatment	(Mungkarndee et al., 2016)
Buffalo milk	Raw, UHT, boiled, pasteurized	FFSFS	FFSFS technique coupled with PCA could be used to classify the four types according to the 410 nm peak	(Ali et al., 2019)
Milk	Thermal treatments (70, 80, and 90 °C) at five heating times (0, 5, 10, 15, and 30 min)	FFFS	Prediction models of retinol concentrations were established and showed good fitting (R^2 = 0.87)	(Liu et al., 2018)

traditional sterilization employs temperatures ranging from 110 to 120 °C and times ranging between 20 and 40 min (Ali et al., 2019; Birlouez-Aragon et al., 2002; Ritota et al., 2017) while the ultra-high temperature technique (UHT) applies high temperatures (135–150 °C) during a short time (1–10 s). Hence, food products subjected to one of these two treatments can be stored at room temperature during several weeks or months, while pasteurization extends the shelf life of the products for a few weeks or days. Other variations of pasteurization, sterilization, and/or UHT methods exist, but they remain used occasionally. It is the case of the intensive pasteurization (85 °C/30 s) and in-bottle sterilization (120 °C/10 min) (Birlouez-Aragon et al., 2002; Hougaard et al., 2013).

Most of the conventional heat treatments mentioned above have several limitations. For example, the overheating of milk can change the sensory characteristics (flavor, odor, color, texture, etc.) and destroy thermosensitive nutrients in the final products. Those effects are associated with the temperature transfer phenomenon from the heating source to the food by convection or conduction. In fact, heat is usually obtained by an electric field or by water vapor. The contact between the food and the source of heating can be direct (e.g., water steam) or indirect (e.g., plate or tube exchangers), and a gradient of temperature is generally formed between the surface and the center of the food. Therefore, a long time may be required to observe temperature equilibrium throughout the food during heating. The determination of the time of heating varies depending on the product characteristics (e.g., liquid without particles, liquid with particles, solid, volume, density), the type of heat transfer applied (e.g., conduction or convection), and the heating system used (e.g., indirect or direct). However, in recent years, new technologies such as microwaves (Martins et al., 2019), infrared (Aboud et al., 2019), ohmic (Cappato et al., 2017), and radio frequency (Di Rosa et al., 2018) have

been proposed to heat foods and minimize the disadvantages of the conventional heat treatments.

Emerging Methods of Heating

Microwaves

The use of microwaves is one of the new alternative methods to conventional heat treatments. It uses microwave energy from 300 MHz to 3 GHz, ranging between infrared and radio frequencies in the electromagnetic spectrum. Briefly, the principle of microwave generation consists of two perpendicular fields, one electric field and other magnetic. When microwave irradiates a product, the electric field of the radiation is partially absorbed and converted into heat. The increase in food temperature is due to intermolecular friction generated by the rotation of water dipoles and other polar substances present in the food matrix (Falciglia et al., 2018). For conventional heating technologies, heat is transferred from the surface to the center of the product whereas microwaves penetrate directly into the food matrix, depositing energy throughout the volume of the material (Falciglia et al., 2018). This unique reverse heating process offers many advantages such as great uniformity of heating, rapid energy transfer, and considerable reduction in processing time. Besides, Jiang et al. (2018) and Tang (2015) have recently reviewed the mechanism of conversion of electrical into thermal energy inside microwave ovens, as well as the interaction between the heated material and the microwaves, and presented other fundamental characteristics of microwave heating. This technology is very beneficial to improve microbial safety, to extend shelf life, and to enhance the functional properties of different food products (Chizoba Ekezie et al., 2017; Gulzar et al., 2020; Tang, 2015). Due to its reduced total cumulative heat, microwave heating retains some important components of food quality such as aromas, vitamins, and pigments (Mudgett, 1986). Since the early 1960s, microwave heating has found common applications in the process of cooking, baking, thawing, roasting, drying, and extracting bioactive compounds. One of the main disadvantages of using microwaves is the possible inhomogeneous distribution of temperature across the product and the occurrence of cold spots inside the food (Jiang et al., 2018; Martins et al., 2019; Sobral et al., 2018). Another disadvantage is the efficiency of the energy transfer to the food material that is limited ($\leq 65\%$) (Salengke, 2000) compared for example to ohmic heating in which about 100% of the energy is transferred (Jun and Sastry, 2005). However, many microwave-assisted food processing techniques have been studied in recent years, showing promising results (Chizoba Ekezie et al., 2017). In a recent review, Martins et al. (2019) presented a comprehensive overview of the application of different microwave technologies in the dairy industry and its impact on the physico-chemical and microbiological aspects of products.

Radio Frequency

As microwaves, radio frequency is another dielectric heating technique, but it covers a lower frequency range, from 1 to 200 MHz. Therefore, radio frequency heating has higher material penetration and more uniform heating than microwaves (Han et al., 2018; Nunes and Tavares, 2019). Due to the lack of temperature gradients, it is known that the sterilization performed by radio frequency is effective at lower temperatures (15–20 °C) than conventional sterilization (Moejes and Van Boxtel, 2017). This can be extremely beneficial for the sensory and nutritional qualities of foods, especially for liquid ones like milk. Radio frequency heating in industrial installations is potentially twenty-five times faster than conventional heat exchangers. The heating process becomes more flexible and can be performed in smaller-sized equipment (Moejes and Van Boxtel, 2017). Despite these advantages, the use of radio frequency heating is still limited to only a few industrial applications, such as thawing of frozen products or use in combination with other heating techniques. For example, in a recent study, raw cow's milk was sterilized by a combination of steam and radio frequencies at different temperatures (Di Rosa et al., 2018). The results showed that the radio frequency–heated milk remained safe and retained good sensory and nutritional attributes for up to 40–45 days.

Ohmic

Another alternative to traditional heat treatments is ohmic heating. The concept of ohmic heating is not new; it was proposed at the beginning of the twentieth century to pasteurize milk and other liquid products using plates with a voltage difference (De Alwis and Fryer, 1990). Due to various technological improvements in recent years and the possibility of its implementation in a continuous heating process, ohmic heating is gaining force in the food industry. In summary, according to its principle, ohmic heating occurs when an alternating electric current passes through food, producing internal heat by Joule effect (i.e., electrical resistance) (Cappato et al., 2017; Jaeger et al., 2016; Kubo et al., 2020). This characteristic has earned it the name of Joule heating or electric resistance heating. Compared to conventional heat treatments, ohmic heating is advantageous because it allows faster rise in temperature and more uniform heating. It is also more efficient and "environmentally friendly." Moreover, compared to the conventional heating process, several studies reported a limited thermal impact of ohmic heating on nutrients and sensory attributes of foods (Cappato et al., 2017; Kubo et al., 2020).

Infrared

Infrared heating involves exposing foods to infrared electromagnetic radiation, which is radiation between the visible and microwave ranges. The infrared wavelength range has three spectral regions called near infrared (0.75 to 2.5 μm), mid-infrared (2.5 to 25 μm), and far infrared (25 to 1000 μm). The radiation used is generally between 1.8 and 3.4 μm. Most food components absorb this kind of radiation, and the absorbed energy causes intermolecular vibrations and friction between water molecules and other food components, resulting in heat generation. Therefore, this technique is characterized by uniform heating in a short time, which results in energy savings and a high degree of process control (Aboud et al., 2019; Chen et al., 2020; Rastogi, 2012). However, the use of infrared radiation as a heating source for milk and dairy products is still limited (De Sousa et al., 2017; Krishnamurthy et al., 2008).

Other promising new technologies, such as pulsed electric field, high-pressure treatment, and obstacle technology, have appeared in recent years (Alirezalu et al., 2020; Pasha et al., 2014). They seem to have effects on reducing the microbial load, increasing shelf life, and preserving protein structures, the functionality of fat globules, and vitamin contents by inactivating certain unwanted enzymes, but investigations are very few and more data are necessary to confirm these trends.

Effects of Heating on Milk and Dairy Product Characteristics

It is well documented and known that different biochemical, physico-chemical, and sensory changes can occur in milk and dairy products during heat treatments. The extent of these changes varies depending on the product considered (e.g., milk, cheese, cream), the technology used (e.g., thermization, pasteurization, sterilization, UHT), and the intensity of the heating process (temperature and time). An overview of some recent studies on this topic is presented below.

Biochemical and Physico-chemical Changes

The biochemical and physico-chemical changes generally reported in milk and dairy products during conventional heat treatments include a decrease in pH, partial precipitation of calcium phosphate, isomerization of lactose, denaturation of whey proteins and their interaction with casein, Maillard browning, and modifications of casein micelles (Fox et al., 2015; Walstra and Jenness, 1984). These thermal changes and others such as the production of hydroxymethylfurfural (HMF), carboxymethyllisine, and glycoxidation products are often used as indicators to assess the intensity of heat treatments.

Two important factors implicated in the quality and processing properties of milk are pH and Ca^{2+} concentrations. These two indicators usually decrease at high temperatures (On-Nom et al., 2010; Ul Haq et al., 2013). Therefore, these two indicators are linked: increasing the temperature of milk induces the reduction in pH and then in the solubility of calcium phosphate. According to On-Nom et al. (2012), the pH of milk in a holding tube was as low as 5.6 at 140 °C. Concerning the calcium, it could be measured at elevated temperatures using electrodes directly in milk up to 60 °C (Chandrapala et al., 2010), in ultrafiltered (UF) permeates up to 80 °C and in dialysates up to 120 °C (On-Nom et al., 2010). Ultrafiltration performed in milk up to 140 °C by placing the UF module directly in the holding tube of an ultra-high-temperature plant is also possible. The dialysates and the UF permeates collected at high temperatures showed no changes in pH and Ca^{2+} contents when cooled at room temperature (On-Nom et al., 2012).

Hattem et al. (2011) have also studied the effect of temperature on camel milk and showed that the modification of different components occurred after pasteurization. They reported that the total protein, ash, and total solid contents increased slightly with the increase of temperature (63, 80, and 90 °C for 30 min and 72 °C for 15 s). These results were in agreement with those of Ul Haq et al. (2013) on skimmed buffalo milk. They compared three treatments: thermization (60 °C for few seconds), pasteurization (65 °C for 30 min), and sterilization (110 °C for 10 min), and underlined that a higher content of fat was observed in sterilized than in pasteurized and thermized skimmed milk. Concerning lactose and ash, some differences were observed after pasteurization and sterilization, but not after thermization of milk. For total proteins, pasteurized skimmed milk presented a higher concentration than the other treatments. Hattem et al. (2011) also reported that non-protein nitrogen (NPN), non-casein nitrogen (NCN), and whey protein nitrogen (WPN) gradually decreased as temperature and time of thermal treatments increased. However, no change in the total nitrogen content (TN) was observed. In another approach, Sakkas et al. (2014) monitored the effect of heating on β-lactoglobulin and α-lactalbumin, lactulose and furosine of raw milk, reconstituted condensed milk, and three types of reconstituted powdered milk. The samples were heated at 80 °C and 140 °C for 4 s. They observed no reliable amount of β-lactoglobulin at temperatures above 100 °C, while residual α-lactalbumin was found at 130 °C. For lactulose, levels of 51 to 1549 mg/L were detected at temperatures ≥ 100 °C. For furosine, contents of 1.9 and 126.5 mg/L were reported in raw milk and milks subjected to 140 °C, respectively. The behavior of the components of the reconstituted condensed milk was similar to that of reconstituted powdered milks after heating, except for the

concentration of furosine. Actually, reconstituted powdered milk contained high amounts of lactulose and furosine.

Various studies have been carried out to increase the shelf life of pasteurized milks and to avoid the development of cooked taste, generally associated to UHT milk. The results obtained have strongly contributed to the development of milk with ESL. For example, Mayer et al. (2010) compared the effect of different heat treatments (raw, pasteurized, ESL, and UHT) in 128 milk samples. Two molecules, β-lactoglobulin and acid-soluble furosine, were used as indicators of the intensity of heat treatments. Reverse-phase high-performance liquid chromatography (RP-HPLC) was used to quantify these two molecules. In addition to RP-HPLC, electrophoresis was employed as a cost-effective high-throughput screening method to assess the impact of heating on milk and to distinguish the different methods used. The authors reported that 45% of the samples of ESL milk presented furosine contents below 40 mg 100 g^{-1} protein, and acid-soluble β-lactoglobulin contents greater than 1800 mg L^{-1} milk. The remaining 55% of the ESL milk samples tested had low levels of acid-soluble β-lactoglobuline (< 500 mg L^{-1}) and high levels of furosine (> 40 mg 100 g^{-1} protein), results comparable to those obtained in UHT milk samples. These data suggest an urgent need in order to define precise limits for ESL milk to better control nutritional and sensory properties of such products.

Recently, Jo et al. (2019) studied the effect of heat treatments on volatile sulfur compounds. They compared the effects of high temperature short time (HTST) (72 °C for 15 s) and ultra-pasteurization (UP at 140 °C for 2.3 s) by direct steam injection (DSI-UP) and indirect heat (IND-UP) on three formulations of skimmed milks containing different ratios of casein (95, 80, and 60%) and soluble proteins (5, 20, and 40%). The authors reported that volatile sulfur compounds were affected by serum protein ratios. Skim milks formulated with a higher serum protein isolate had higher concentrations of hydrogen sulfide and carbon disulfide than natural skim milk or skim milk formulated with lower proportions of serum protein isolate. Hydrogen sulfide and carbon disulfide appeared to contribute to eggy and sulfur/burnt flavors, respectively.

In cheeses, Miloradovic et al. (2018) reported that heat treatment (80 °C/5 min and 90 °C/5 min) had a mainly significant influence on whey protein levels of Quark-type cheeses produced with cow's and goat's milk. These authors pointed out higher levels of β-lactoglobulin and α-lactalbumin when higher temperature was used, regardless of the type of milk. In another study, Cilliers et al. (2014) investigated the effect of three different methods of heating: (i) HTST (73.5 °C/15 s), (ii) ultraviolet (UV), and (iii) a combination of UV and HTST that they called UV pasteurization or UVP, which corresponds to the use of UV light below 6 °C + HTST at 73.5 °C/15 s. The analysis was performed on whole cream used for making Cheddar, and no significant difference was found in the composition of macronutrients. Nevertheless, the authors pointed out lower cholesterol levels in creams when UV was used compared to HTST. On the other hand, when using UVP, slight differences in the fatty acid profile were observed, in particular in terms of oleic and stearic acid amounts in comparison to control (raw milk). In other studies, it was reported that heat treatment also affects the profile of volatile compounds of different types of cheeses made with raw, pasteurized, or microfiltered milk (Demarigny et al., 1997; Fernández-García et al., 2002). For example, cheeses produced from raw milk contained a greater amount and variety of volatile compounds than cheeses produced from pasteurized milk (Rodríguez-Alonso et al., 2009). More recently, Pappa et al. (2019) reported that cheeses made from raw sheep's milk showed significant differences in their biochemical composition compared to those from pasteurized sheep's milk (63 °C/30 min). Actually, the pasteurization affected lipolysis, volatile fatty acid profiles, and free fatty acid profiles, but did not significantly ($p > 0.05$) affect physico-chemical characteristics, as well as the degree of proteolysis of the cheeses.

Heating can also affect the clotting time of rennet in milk. Hattem et al. (2011) reported that the coagulation time of rennet increased depending on the temperature of the milk (63, 80, and 90 °C for 30 min and 72 °C for 15 s) regardless of the amount of $CaCl_2$ used (0 to 20 mg per 100 ml). However, the greater the quantity of $CaCl_2$, the shorter the clotting time of the rennet, regardless of the heat treatment used. In addition, incubation of the milk with a yogurt culture at 40 °C for 12 h revealed a significant increase in acidity and a decrease in pH, independently of the heat treatment method used.

With regard to emerging methods of heat treatment, microwaves appear to provide dairy products with superior nutritional quality and extended shelf life compared to pasteurization, sterilization, and UHT methods (Chandrasekaran et al., 2013; Martins et al., 2019). Depending on the treatment and the conditions applied (duration, form, frequency, and intensity), microwave heating promotes the denaturation of proteins by modifying the quaternary and tertiary structures. It can also generate protein aggregation, Maillard reaction, lactose isomeration, and changes in enzyme activities (Bi et al., 2015; Bohr and Bohr, 2000; Clare et al., 2005; Mecherfi et al., 2011). According to Mounir et al. (2019), high-power microwave treatment (e.g., 60 kW) deactivated enzymes by denaturing them. On the other hand, controlled irradiation with low-power microwaves (e.g., 30 W) improves enzyme activities.

Few studies reported the effects of ohmic treatment on milk and dairy products. Compared with conventional heat treatment methods, the preservation of the nutritional characteristics of dairy products can be obtained by ohmic heating, as reported by Cappato et al. (2017, 2018). In another study,

Roux et al. (2009) explored the feasibility of an ohmic laboratory system (5 kW AC–50 Hz, electric field 0.1 to 3 kV/m, 100 ml) in stabilizing milk infant formula. According to them, prediction of the effects of this technology on the quality of dairy products was possible based on the modification of five quality indicators: Fluorescence of Advanced Maillard products and Soluble Tryptophan (FAST) index, soluble proteins, furosine, carboxymethyllisine, and color of products (CIE Lab). Roux et al. (2016) conducted a similar study at pilot scale (25 kHz, 15 kW; 300 from 4000 V) and reported comparable results. In addition, the effects of UHT steam injection were compared to those of ohmic heating and the authors observed equivalent levels of the same nutritional markers cited above. However, products obtained after ohmic heating exhibited higher values for the *K* factor in the FAST index, furosine, and carboxymethyllysine than the other methods of heating analyzed. The authors also described higher values of vitamin C contents and Lab parameters of color in ohmic samples than the other ones. A higher concentration of bioactive compounds (e.g., phenolic compounds) and an increase in the antioxidant capacity (DPPH free radical method and fluorescence recovery after photoblanching, FRAP) of the milk-based product (whey acerola–flavored drink) were also demonstrated in another study (Cappato et al., 2018). Cappato et al. (2017) reported on the preservation of nutritional compounds and the reduction of fouling in dairy products using ohmic heating. Ferreira et al. (2019) described an increase in in vitro antidiabetic properties, while Costa et al. (2018) and Pires et al. (2020) found an increased number of volatile compounds using this method. Despite previous studies, Shivmurti et al. (2014) reported that fat, solids non-fat, protein, and total solids of buffalo milk treated with ohmic heating (50 Hz; 0.6 A; 8.38 V/cm) were comparable to those found in conventional heating (72 °C/15 s). However, the authors did not perform statistical analysis to support their findings. In contrast, they showed that the processing time was reduced to around 18% when ohmic heating was used. In a different approach, Pereira et al. (2016) investigated the formation of protein aggregates in whey protein isolates under the influence of mild electric fields during ohmic heating. The authors reported that ohmic heating reduced clumping and increased solubility of protein in the early stages of heating. Treatment using 12 V/cm per 10 s induced major protein retention, suggesting that rapid come up time (CUT) led to a material of better quality. Metal contamination of milk samples treated with conventional pasteurization and by ohmic heating was also assessed (Suebsiri et al. 2019). The results showed that milk processed by the ohmic method, using stainless steel electrodes, contained a relatively higher level of iron and chromium than the control.

Regarding cheese, Rocha et al. (2020) demonstrated the positive effect of the ohmic technology on different properties of Minas Frescal cheese (soft, fresh white Brazilian cheese). They compared two methods of thermal treatments (conventional: 72–75 °C for 15 s and ohmic: 4, 8, and 12 V/cm at 60 Hz, 72–75 °C for 15 s). Ohmic heating (weak and intermediate electric fields; 4 and 8 V/cm) had a positive effect on the formation of bioactive compounds and a negative impact on the free acid profile of cheeses. Nevertheless, the microbiological quality of the samples was the same in both heat treatments.

Nowadays, ohmic heating is considered as a promising technology for the dairy industry, with great commercial interest (Jermann et al., 2015). The lack of studies on ohmic heating on dairy products (yogurt, cheese, butter, fermented milk) is still important, opening a range of research opportunities in this area.

Sensory Changes

Sensory analysis includes a wide variety of tests to measure human responses to stimuli. Appropriate application of these tests allows product characterization and consumer preferences to be assessed (Drake, 2007). Many studies have investigated the impact of different heat treatments on the sensory properties of milk and dairy products (Jo et al., 2018; Li et al., 2018; Ul Haq et al., 2014; Zamberlin and Samaržija, 2017). Heat treatments can, for example, damage the flavor and/or texture of milk, as reported by Israr ul Haq et al. (2014) on skimmed buffalo milk. These authors reported that the appearance/color of pasteurized (65 °C for 30 min) and sterilized milk (110 °C for 10 min) was often considered non-acceptable compared to thermized skimmed milk (60 °C for a few seconds) and raw milk. Concerning sterilization, it can improve the taste/flavor and body/texture of skimmed milk during room temperature and cold storage. Differences between milk processed by ultra pasteurization (UP) and HTST were revealed by different authors (Chapman and Boor, 2001; Jo et al., 2018; Valero et al., 2001). The flavor of UP-treated samples (140 °C/2.3 s) was characterized by higher overall, cooked, and sulfur/eggy aromas than HTST-treated samples (78 °C/15 s). Lee et al. (2017) confirmed these findings when comparing three methods of milk heating: (i) direct steam injection UP (DSI-UP), (ii) indirect steam injection UP (IND-UP) at 140 °C for 2.3 s, and (iii) HTST (78 °C/15 s). Milks treated by HTST had lower cooked flavor than UP samples. More specifically, DSI-UP milk was characterized by sulfur, eggy and cooked flavors. Moreover, the consumer test revealed that sulfur and eggy flavors played a negative role on consumer preferences. In fact, the consumers preferred HTST to UP-treated milk. Another significant point of difference between HTST and UP samples concerned viscosity or thickness. The average viscosity rate of UP samples was higher than samples treated by HTST. Other studies comparing different techniques of heating on sensory characteristics can be reported. For example, Clare et al.

(2005) studied the impact of the UHT method on flavor in comparing it with microwaves. UHT samples of milk were evaluated darker, higher in caramelized and stale/fatty flavors, and more astringent than microwaved samples of milk.

In cheese, heat treatment can influence sensory properties depending on the heating method and cheese technology used. Generally, cheeses made from raw milk develop a more intense and specific flavor than cheeses processed from pasteurized milk (Awad, 2006; Montel et al., 2014). This was observed in hard-cooked cheeses (Swiss-type, Emmental, Reggianito), hard uncooked cow's milk cheeses (Cheddar, Gouda, Raclette, Morbier-type, Cantal-type), and sheep or goat's milk cheeses (Idiazabal, Roncal, Canestrato Pugliese cheeses) (Alonso et al., 2013; Vélez et al., 2010). Rynne et al. (2004) showed an effect of milk pasteurization (72 °C, 77 °C, 82 °C, or 87 °C for 26 s) on texture and heat-induced functionality of half-fat Cheddar. They reported that raising the pasteurization temperature reduced the flowability and stretchability of the heated cheese and increased its apparent viscosity. In contrast, increasing pasteurization temperature from 72 to 77 °C did not significantly affect these properties (Rynne et al. 2004). Nonetheless, Miloradovic et al. (2018) reported no influence of milk heat treatments (80 °C/5 min and 90 °C/5 min) on composition, texture, and sensory properties of cows' and goats' Quark-type cheeses.

Numerous studies have been carried out to better understand the properties of melted cheeses (e.g., meltability, oiling-off, or stretchability) using different instrumental and/or empirical methods (Guinee et al., 2000; Wang et al., 2018). Bord et al. (2016) showed differences in cooking properties of several French blue cheeses (Fourme d'Ambert, Bleu d'Auvergne, and Fourme de Montbrison) during and after heating (200 °C/2 min in an oven). They observed that some heated blue cheeses exhibited useful culinary properties such as "meltability," "stretchability," and weak "oiling-off," while being well discriminated in their taste profiles during the sensory tests. By applying a sensory dynamic method (TDS: temporal dominance of sensations) to evaluate the temporal dimension of different gustatory attributes during food consumption, the dominance of specific attributes of heated cheeses (e.g., sour, bitter, and salty tastes) during the swallowing and at the end of the tasting in comparison with the conventional sensory profiles was observed. These temporal elements are useful to understand the evolution of taste perceptions of cheeses, particularly heated cheeses in which the evolution of taste perceptions is more complex than non-heated cheeses due to the presence of a cooling phase (Bord et al., 2016, 2019).

Little research describes the impact of ohmic treatment on the sensory quality of cheese. Nevertheless, Rocha et al. (2020) recently showed that cheeses manufactured with milk subjected to ohmic treatment (4, 8, and 12 V/cm at 60 Hz, 72–75 °C/15 s) had similar or even better sensory properties than those obtained with milk heated by a conventional method (72–75 °C/15 s).

Evaluation of Milk and Dairy Product Changes by Spectroscopic Techniques

Visible and Infrared-Based Spectroscopic Techniques

Visible (Vis) and infrared (IR) spectroscopies combined with data fusion techniques have been widely used to assess the quality of dairy products (Biancolillo et al., 2019; Karoui and Debaerdemaeker, 2007). Several studies on the application of Vis and IR in controlling the quality of milk and dairy products exist, mainly because tracing dairies is an important challenge in the dairy industry. An example is provided by Pillonel et al. (2003) who analyzed Emmental cheese by using four different instruments working in the MIR or NIR regions. The spectra were firstly analyzed by principal components analysis (PCA), and then a linear discriminant analysis (LDA) was calculated on the PCA scores. The objective was to classify the cheeses according to their origin. The classification model built on MIR spectra collected in transmission mode led to a correct classification rate of 100% when Swiss Emmental was discriminated. Vis and IR spectroscopies have also been used to predict changes in organoleptic characteristics of cheeses during ripening (Currò et al., 2017) as well as their shelf life (Cattaneo et al., 2005). However, few articles are available on the exploitation of these techniques for the evaluation of product quality after the use of different heat treatment conditions. In this regard, several applications have been developed, involving on-, at-, or in-line analysis.

Baum et al. (2016) investigated the possibility of quantifying casein in subcritical heated skimmed milk by Fourier-transformed (FT-IR) spectroscopy. These authors used different milk samples heated at lower than 42 °C, presenting diverse casein and total protein contents, and analyzed by a MilkoScan FT2. This device allows on-line measurements of samples under controlled conditions and provides a three-way data structure (first mode: samples; second mode: wavelengths; third mode: time). The cube was centered along with the first two modes, and then parallel factor analysis (PARAFAC) (Bro, 1997) and N-partial least squares (N-PLS) (Bro, 1996) were used to quantify casein on-line in the presence of calcium, whey, and cream. The tensor decomposition approaches led to an accuracy 0.12 whereas precision was 0.07%. Similarly, Al-Qadiri et al. (2008) conducted another interesting work on skimmed milk. These authors investigated the applicability of Vis-NIR spectroscopy (600 to 1100 nm) in determining and controlling the spoiling of milk stored at different conditions. In particular, pasteurized milk aliquots were stocked at different temperatures (6, 21, and 37 °C) for various time shifts (between 3 and 30 h). Total aerobic

bacteria counts and pH were also monitored for each sample. Eventually, PCA and soft independent modeling by class analogy (SIMCA) (Cocchi et al., 2018; Wold and Sjöström, 1977) were exploited to inspect grouping tendencies among samples, and PLS used to predict pH and bacteria counts. PCA highlighted the presence of clusters among samples, confirmed by the classification model (SIMCA). In fact, the class-modeling approach correctly accepted 55 (over 60) control samples. The accurate coefficients of determination (R^2) obtained by both PLS models (R^2 = 0.99) indicated that the proposed approach was suitable for its aim. The same spectroscopic techniques were used to investigate the effects of different temperatures on cheeses. For instance, Loudiyi and Aït-Kaddour (2018) investigated the impact of gentle heating on Cantal-type cheeses prepared with diverse salt compositions and exposed to different ripening times (5 and 15 days). To pursue their study, five different recipes were used to prepare samples having different salt concentrations (three recipes included bare NaCl: 0.5%, 1%, and 2%, and two recipes contained both NaCl and KCl: 1.5%/0.5% and 1%/1%). Mid-infrared (MIR) spectra (3800 and 900 cm^{-1}) were directly collected on cheeses (by means of an attenuated total reflectance—ATR—device) after 5 and 15 days of ripening. MIR signal collection took place during a heating ramp (5 °C steps) spanning from 20 up to 60 °C. Additionally, they assessed the chemical composition of samples. Firstly, a two-way analysis of variance (ANOVA) was used to test whether NaCl substitution had an effect on the physico-chemical characteristics of cheeses. The outcome of this analysis is that when ripening time is the same, replacing NaCl by KCl affects all the investigated properties except proteins, proteolysis, and water activity. As expected, heating affected the intensity of MIR spectra. The absorption peaks between 3800 cm^{-1} and 3000 cm^{-1} and 1700 cm^{-1} and 1500 cm^{-1} increased with the temperature. On the other hand, the absorption intensity between 3000 and 2800 cm^{-1} increased when measured up to 35 °C, and then decreased between 35 and 60 °C (Fig. 3). The authors employed independent component analysis (ICA) to inspect changes in cheeses during heating. Among other things, they concluded that between 20 and 40 °C, the most significant effect was linked to the fusion of triglycerides, whereas, when the temperature was between 40 and 60 °C, it was cheese sample resistance that changed the most, mainly due to the stability of proteins. Eventually, the authors concluded that ICA represents a suitable approach for monitoring molecular alterations in cheese. These results agree with those reported in previous studies (Boubellouta and Dufour, 2012; Loudiyi et al., 2017a). Additionally, Loudiyi and Aït-Kaddour (2018) have also observed that the reduction of NaCl slightly modified the comportment of the cheeses, probably due to different interactions created between proteins themselves or proteins and water. In a similar scenario, Boubellouta and Dufour (2012) investigated different characteristics of cheeses when the temperature goes from 20 to 80 °C. They heated Comté and Raclette cheeses, applying an increasing ramp of steps of 5 °C, and collected MIR and front-face fluorescence (FFFS) at any temperature of the ramp. In addition to spectroscopic techniques, they conducted rheological measurements in cheese samples. The investigation of MIR measurements led to several findings: as expected, the spectra collected at different temperatures differ from each other. In particular, as the temperature increased, shifts at higher wavelengths occurred. For instance, the absorption peak of the CH_2 stretching has shifted to lower wavenumbers when temperature increased from 20 to 50 °C. PCA and common components and specific weights analysis (CCSWA) (Biancolillo et al., 2020; Mazerolles et al., 2006; Qannari et al., 2000) were performed on spectra collected, leading to similar results. Looking at the score plot from PCA, the authors observed a clear distribution of the objects along with the first PC: a "colder" sample presented low PC1 values, whereas the "warmer" fell at a higher value of this component. In addition, PC2 allows samples to be discriminated at higher temperatures. The MIR spectra (collected at 1634 cm^{-1}) were used to predict the melting temperature, and the results obtained were entirely in agreement with those of rheological measurements. The same achievement was obtained by inspecting the similarity maps from CCSWA. Additionally, the authors observed that the loading for the first common component (explained variance > 80%) is dominated by absorptions at approximately 2853 and 2922 cm^{-1}, associated with physical modifications of fat due to the increase of the temperature (Boubellouta and Dufour, 2012).

Raman Spectroscopy

Raman spectroscopy is also a vibrational spectroscopy technique, but its great advantage is the low interference of the water signal due to a weak O–H stretching vibration, which allows sensitive analysis of milk products. Raman measurements are fast, non-destructive, and therefore easily applicable for PAT to adjust and to redirect the product during transformation (Mazurek et al., 2015). Concerning the use of Raman spectroscopy for monitoring thermal treatments applied to milk and dairy products, two recent studies are distinguished. The study of Reiner et al. (2020) evaluated the potential of this technique in discriminating samples of milk (full cream and skim milk) according to the heat treatment used during production. More precisely, milk samples treated by ultra-high temperature (UHT) and two types of extended shelf life (ESL) methods were compared. The authors tested whether it was possible to distinguish samples according to the method used. They used ESL higher-heat shorter time (ESL-HHST) by heating the milk at 125–127 °C for 4–2 s, and ESL microfiltration (ESL-MF) by using lower temperatures (72–75 °C) for a longer time (15–30 s).

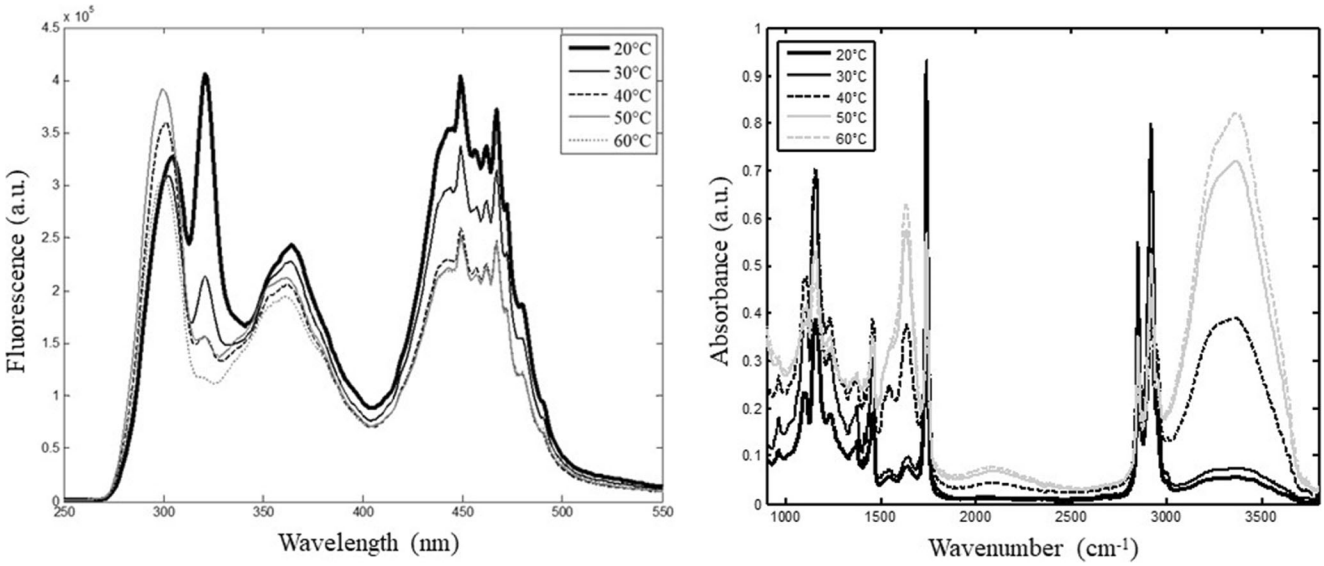

Fig. 3 Effect of heating on molecular synchronous fluorescence and ATR-MIR spectra of cheese

To pursue this study, products with different fat and lactose contents and from different dairies were used to ensure statistical representativeness. They collected Raman measurements (laser excitation wavelength of 784.98 nm, spectral range of 65–3290 cm^{-1}) on pre-processed samples at different positions to consider sample inhomogeneity. Then, PCA was performed on unsmoothed spectra after baseline correction. Regardless of the classification problem investigated (i.e., UHT vs. ESL or ESL-HHST vs. ESL-MF), two clusters were formed along PC1, showing a clear distinction between heat treatments. Further inspection of data highlighted that the UHT method caused an increase of the Maillard reaction compared to the ESL processes, producing spectral changes in protein and fat regions (500–1225 cm^{-1}). Comparing the two ESL processes, differences were related to the Maillard reaction (as mentioned earlier), but the rearrangement and denaturation of the whey proteins occurred to a different extent according to the temperature-time applied. The other mentioned study on this subject, published by Yazgan et al. (2020), discriminated raw (94 samples) and pasteurized (94 samples, at 65 °C for 30 min in a water bath) milk samples, despite the use of different milk species (cow, goat, ewe, and mixture). Raw and heat-treated samples (1 mL of milk) were analyzed in the range 200–2000 cm^{-1} with a 785 nm (limited interference of fluorescence) laser source and an integration time of 25 s. Minimal variation in intensities and shape was observed between samples for the bands at 476 (C–C stretching or skeletal vibration), 607 (phospholipid group), 1232 (deformation of in-plane *cis* double bond), and 1361 (methylene twisting deformation) cm^{-1}. Minimal variation was also observed in 343–386 (C–O–C, stretching and bending vibrations), 913–921 (ν(C–C), stretching vibrations), and 1470–1520 (methylene scissoring deformation) cm^{-1} ranges. The collected Raman spectra were subjected to baseline correction and mean centering, and eventually equally divided into a calibration (129 samples) and a validation (59 samples) set by using Kennard-Stone-type selection, to develop a partial least squares-discriminant analysis (PLS-DA) model able to classify samples according to heat treatment. A threshold was defined for both classes as the value where false negatives and false positives are minimized. A model calculated using six latent variables was chosen through a cross-validation procedure (venetian blinds, ten splits, and one sample per split) to obtain the least standard error. Predictions showed high sensitivity (0.897 and 0.933 for raw and pasteurized, respectively) and specificity (0.933 and 0.897 for raw and pasteurized samples, respectively), providing a successful classification.

Raman spectroscopy can also be a suitable tool to investigate whey protein modifications in response to thermal treatments. For example, Wang et al. (2013) used surface-enhanced Raman spectroscopy (SERS) in combination with PCA for the structural characterization of partially glycosylated (through Maillard-induced glycosylation) whey protein (PGWP) in whey protein-based beverages under the influence of pH and heat (75 °C for 30 min in a water bath). PGWP samples were prepared by mixing whey protein isolate (WPI) with dextran and purified with hydrophobic interaction chromatography (HIC). SERS spectra from 3000 to 200 cm^{-1} were acquired in quadruplicate using 4 mW laser power on PGWP samples, WPI controls (before and after heat treatment), and dextran. Spectra were then subjected to secondary derivative transformation with a Norris derivative filter and standard normal variate for PCA. Clusters according to the heat treatment were seen along PC2, whereas PC1 showed a clear distinction between WPI and PGWP samples. Smith et al. (2017) have proposed another valuable research work in this context. They exploited Raman spectroscopy coupled with PCA to study the modification during production and

storage of the lactose state of whey protein concentrate (WPC) because of thermization (65 °C/15 s) or pasteurization (72 °C/15 s). The different treatment conditions showed their effect on phase change from amorphous into crystalline lactose, reflecting in spectral changes near 2900 cm^{-1} and in 1200–800 cm^{-1} range. In the same vein, Blanpain-Avet et al. (2012) inspected the thermal denaturation of the fouling solution of a heat exchanger (a severe issue in the dairy industry) by micro-Raman spectroscopy (MRS) in the 800–1800 cm^{-1} range.

With the same purpose, Raman spectroscopy has also been applied to other dried dairy ingredients. For instance, Wang et al. (2018) investigated the potentiality of this technique, together with MIR, in the discrimination of pre-heat treatments applied on skim milk powder (SMP), demineralized whey protein (DWP) powder, and whey protein concentrate, as well as in the differentiation of each dried ingredient. The authors pre-heated each reconstituted product at three temperatures (72 °C, 95 °C, 115 °C), and Raman spectra were collected in the range 23–3397 cm^{-1}. PCA was then performed to explore sample behavior, followed by the comparison of PLS1-DA and PLS2-DA models to discriminate each pre-heat temperature from the others. For that, the three selected whole Raman spectral ranges were used as well as a selection of wavenumbers. The best performances were achieved by PLS1-DA models built on both selected MIR and Raman variables; in fact, this model provided 100% accuracy (i.e., all samples were correctly classified).

NMR Spectroscopy

Questions about nutritional and quality changes in milk due to the application of different heat treatments first arose in milk almost 50 years ago (Ashton, 1972). The effect of heating, pasteurization, freezing, and thawing on the hydrolysis of triglycerides and subsequent changes in triglyceride availability were among the main interest topics (Walstra and Jenness, 1984; Wardell et al., 1981). The changes in the conformation of milk proteins and the states of aggregation and unfolding processes of whey proteins induced by thermal effects were monitored by NMR in different studies (Belloque and Ramos, 1999; Belloque and Smith, 1998; Lambelet et al., 1992; Rollema and Brinkhuis, 1989).

At the beginning of the use of NMR, it was difficult to obtain well-resolved liquid ^{1}H NMR spectra in non-homogeneous foods like milk and cheese without pre-treatment of samples. Extraction, pH adjustment, and the use of spin filters were some of the most frequently used pre-treatments. Nonetheless, thanks to the increasing advances in technology and the improvements in specifications of NMR spectrometers, it is nowadays possible to acquire solid and liquid-like relaxation data for complex food matrices such as yogurt, ice cream, and cheese (Mariette, 2009). Hence, several NMR-based techniques have been developed for monitoring changes in milk and dairy products. As a non-destructive and structure-sensitive method, NMR finds an expanding number of usage in milk and dairy research.

In a very recent study, Zhu et al. (2020a) used high-resolution ^{1}H-NMR, gas chromatography-mass spectroscopy (GC-MS), and ultra-performance liquid chromatography–quadrupole time-of-flight mass spectrometry (UPLCQ-ToF/MS) to monitor changes in cows' milk metabolites throughout vat pasteurization process and storage at 4 °C up to 12 days. Minor changes were observed in the metabolites detected by these three methods during processing. On the other hand, significant differences were obtained after 8 days of storage for 24 different metabolites determined by NMR and MS techniques. The metabolites were mainly organic acids, amino acids and peptides, vitamins, carbohydrate derivatives, carboxylic ester, ketones, and fatty acids (Zhu et al., 2020a). Using a similar experimental approach, the authors investigated the effect of freeze-drying and the subsequent storage up to 224 days at three different temperatures on milk metabolites. The stability of the metabolome of milk powders underwent minor changes after freeze-drying as well as after storage at 4 °C and − 20 °C, while significant changes occurred in samples stored at 20 °C. The study has revealed the importance of the prior storage temperature of milk when applying metabolomics measurements in the verification of the origin or of the authenticity of milk samples because the changes in metabolites sensitive to storage are very important (Zhu et al., 2020b).

Cheese matrix is widely analyzed by using NMR-based methods, but there are limited studies in the literature monitoring the effect of thermal treatments on the structure of the cheese matrix. In the study of Vogt et al. (2015), structural changes and changes in the mobility of some components of mozzarella and Cheddar cheese during heating were determined by using NMR. The importance of the changes in water mobility was emphasized since it affected the cheese matrix behavior and the cheese rheology during heating. T_2 relaxation time values, which consist of shorter relaxation time T_{2A} and longer relaxation time T_{2B}, were determined. T_{2A} and T_{2B} were ascribed to the casein-water association and free water that does not interact with protein, respectively. The applied heating procedure increased the T_2 relaxation values of fat for all cheese samples. This result was explained by the passage of the cheese fat from solid/liquid state to the liquid state, which has a longer relaxation time compared to solid fat. Similarly, the heating caused an increase in the free water content of all cheese samples. T_{2B} values increased with heating from 20 to 65 °C, which also indicated the increased mobility of free water in the structure of the cheese (Vogt et al., 2015). In their further study, Smith et al. (2017) investigated changes in water mobility as a function of temperature and age in Mozzarella cheese during maturation and subsequent heating (20 to 65 °C). They showed that the free water content decreased, and the water associated with proteins

increased as maturation progressed. They also found an increase in the free water content when cheeses were heated. They explained this change by the contraction of the casein matrix due to the enhanced hydrophobic interactions. On the other hand, heating affected the T_{2A} values in different ways throughout maturation. These inconsistent changes were explained by the presence of thermally transient water, reabsorption of the associated water, and proteolysis, which directly affected thermally induced water mobility. Through the formation of diffusion relaxation correlation (D-T_2) contour plots, the authors stated that heating increased the rotational mobility of water. D-T_2 contour plots also revealed that heating caused the melting of cheese fat and increased its mobility (Smith et al., 2017).

Fluorescence Spectroscopy

Fluorescence concerns the emission of lower-energy light by a fluorescent molecule after the absorption of UV or VIS lights. Food products contain molecules (called fluorophores) such as tryptophan, vitamin A, and riboflavin, which generally possess one or more conjugated bonds, giving rise to the phenomenon of fluorescence. Fluorescence spectroscopy is nowadays recognized as a powerful technique. It offers several advantages for the characterization of food products (e.g., authentication, evaluation of biochemical content) due to its sensitivity to molecular interactions, to chemical reactions, and to changes in the environment of molecules (Locquet et al., 2018; Loudiyi and Aït-Kaddour, 2018). Consequently, this method has been used to investigate changes in the structure of milk components and their interactions during heating (Boubellouta and Dufour, 2008, 2012; Loudiyi and Aït-Kaddour, 2018). Most of the studies reporting the use of fluorescence spectroscopy have been performed by using the so-called classical front-face fluorescence spectroscopy (FFFS) and front-face synchronous fluorescence spectroscopy (FFSFS). As reported by different authors, the FFSFS presents an attractive advantage compared with FFFS. Indeed, compared with an FFFS spectroscopy emission or excitation spectrum that is mainly specific to one fluorophore, a FFSFS spectrum generally depicts bands related to multiple fluorophores.

In milk, many fluorophores (e.g., aromatic amino acids, vitamin A, riboflavin, NADH, oxidation products, and Maillard reaction products) have been used. They allow (i) detection of changes induced in milk following the application of thermal treatments, (ii) classification of milk samples according to their thermal treatments, or (iii) prediction of changes in concentrations of certain constituents in milk during the application of thermal treatments (Andersen and Mortensen, 2008; Ritota et al., 2017; Shaikh and O'Donnell, 2017; Sikorska et al., 2019).

Most of the fluorescence spectroscopic studies investigating heat-induced changes in milk were based on the measurements of emission spectra of tryptophan, acquired in the range 305–450 nm after using an excitation wavelength of 290 nm, and the emission spectra of fluorescent Maillard compounds, recorded in the range 380–600 nm after using an excitation wavelength of 360 nm. A method called fluorescence of advanced Maillard products and soluble tryptophan (FAST) was developed to quantify the protein denaturation and the relationship with the fluorescence resulting from Maillard reaction products (Birlouez-Aragon et al., 2002). The FAST method is rapid in analyzing the effects of heat treatments on dairy products, but it was firstly developed using the traditional right-angle fluorescence procedure. Later, Schamberger and Labuza (2006) conducted an experiment to improve the FAST method and adapt it to FFFS instead of the right-angle configuration. Among the different methods investigated (FFFS, Hunter L*, a*, b*, hydroxymethylfurfural—HMF, tryptophan, and optical density), the authors obtained best results using the FFFS and the chemical analysis of HMF, which presented high correlations (R^2 greater than 0.95 in the emission spectrum region between 394 and 447 nm).

Classification of milk samples according to their thermal treatments is another promising application of fluorescence spectroscopy. For example, the emission spectra measured in the range of 400–600 nm using the excitation wavelength at 375 nm were used to discriminate milk samples according to both heat treatment (pasteurized, sterilized, UHT, and recombined milk) and different types of milk and milk-like beverages (e.g., fermented, soy, and corn "milk") (Mungkarndee et al., 2016). The results of PCA applied to the fluorescence data showed a good separation between the different groups. Besides, LDA performed on the same data displayed excellent results in the discrimination of milk samples according to the heat treatment used. An accurancy of 100% was obtained after cross-validation with the leave-one-out method. Recently, fluorescence spectroscopy, based on chlorophyll, vitamins, and other fluorophores, was able to elucidate the effect of severe heat treatments on the molecular composition of desi ghee obtained from buffalo milk. It also allows classifying heated samples according to different temperature treatments (Ahmad and Saleem, 2018). More recently, tryptophan fluorescence was successfully used to discriminate reconstituted skim milk powder samples according to their thermal loads (Ayala et al., 2020).

Fluorescence spectroscopy has also been used in predicting individual constituents such as lactulose, furosine, β-lactoglobulin, and retinol in heat-treated milk (Ayala et al., 2017; Kulmyrzaev and Dufour, 2002; Kulmyrzaev et al., 2005; Liu et al., 2018). In a recent study, the potential of FFFS was investigated to predict retinol degradation during thermal treatments at three different temperatures (70, 80, and 90 °C) and five heating times (0, 5, 10, 15, and 30 min). Prediction models of retinol concentrations were established by

considering the variation of the maximum emission intensities during heating of milk native fluorescent markers (such as tryptophan, dityrosine, Maillard reaction products, and riboflavin). Three predictive equations taking into account the variation in intensity of the different bands were proposed and the best of them (the model III) gave a correlation coefficient of 0.92 and a RMSECV of 5.37% (Liu et al., 2018). In another study, lactulose kinetics in heat-treated reconstituted skim milk powder was studied using FFFS based on a large number of fluorescent markers (Ayala et al., 2017). The best model to predict lactulose contents used tryptophan emission at 307 nm and a combination of two ratios: the ratio of fluorescence of tryptophan and dityrosine, emitted at 324 nm and 377 nm, respectively, and the ratio of dityrosine fluorescence and Maillard compounds, emitted at 377 nm and 425 nm, respectively (R^2 of 0.91). This study demonstrated that the combination of different fluorescent indicators was an effective method to determine the lactulose concentrations in thermally treated dairy products. Formerly, FFFS was used to determine lactulose and furosine in milk treated with different heat treatments, namely overheated semi-skimmed UHT, semi-skimmed UHT, and pasteurized milks (Kulmyrzaev and Dufour, 2002). In this study, the application of principal component regression (PCR) on tryptophan fluorescence intensity (measured at a wavelength range of 305–450 nm with an excitation at 290 nm) among other fluorescence data was correlated to the results obtained by referenced methods (HPLC furosine content, $R^2 = 0.956$; enzymatic lactulose determination, $R^2 = 0.987$).

Most of the previously reviewed studies were conducted using only one excitation or emission wavelength, targeting a specific fluorophore. Although this procedure is fast, it can limit the potential of fluorescence spectroscopy in investigating several fluorophores simultaneously. On the other hand, fluorescence excitation-emission matrices (EEM) and FFSFS take longer times but enable scanning over a wide range of excitation and emission wavelengths.

Contrary to FFFS, few studies were found in the literature describing the use of FFSFS or EEM for monitoring thermal treatments of milk. Blecker et al. (2012) investigated, by using FFSFS, the effect of different heating treatments (60 and 80 °C during 20 min) and rennet-induced coagulation temperatures (30 and 40 °C) on the modification of milk coagulation properties. After chemometric analysis of spectral data, a clear discrimination was highlighted between (i) raw milks and heated ones and (ii) milks renneted at 30 °C from those renneted at 40 °C. Hougaard et al. (2013) confirmed these results by reporting that FFFS coupled with PARAFAC is able to distinguish milk samples according to heating intensity (72–120 °C).

In a study conducted on buffalo milk, the authors reported that the FFSFS technique coupled with PCA was used to classify four types of the milk (UHT, domestic boiled at 93 °C, pasteurized, and raw) as a function of their thermal treatments. This classification was possible according to changes in the fluorescence emission peak at 410 nm, which were indirectly attributed to vitamin A (Ali et al., 2019). The UHT milk showed a distinct clustering due to the formation of Maillard reaction products and irreversible changes in protein structures when compared to the other heating treatments.

Concerning EEM, the modeling of obtained fluorescence data by PARAFAC was able to characterize milk during mild heating and acidification (Boubellouta and Dufour, 2008), and to distinguish milk samples according to heat treatments (instant infusion pasteurization at 72–120 °C/0.2 s, HTST at 75 °C/15 s, and a more intensive pasteurization at 85 °C/30s) (Hougaard et al., 2013). The discrimination between both heat treatments was highlighted by the fluorescence of vitamin A.

In recent years, another fluorometric technique, called time-resolved fluorescence, has emerged and acquired increasing attention due to its desirable features. Indeed, this technique has several advantages compared with the above-mentioned techniques. For example, it allows measuring changes in the picosecond or nanosecond time range, making it a useful technique in biomolecular structure and dynamics analysis (Brandao et al., 2019). The time-resolved fluorescence or fluorescence lifetime has been used for different classification and discrimination purposes (Brandao et al., 2017a, 2017b), while only one study has been found regarding the application of this technique to evaluate the impact of mild heat treatments in bovine milk (Brandao et al., 2019). The results of this study showed that the fluorescence lifetime decreased linearly from room temperature until 40 °C, and it increased slowly for temperatures higher than 40 °C (Brandao et al., 2019).

All the above-discussed studies indicate that fluorescence spectroscopy has an extensive potential to be used as an online tool for monitoring thermal treatments of milk through the detection of heat-induced changes and classification/prediction purposes.

Concerning cheese, studies generally focus on the evaluation of the properties linked to the melting (Boubellouta and Dufour, 2012; Karoui et al., 2003; Loudiyi et al., 2017a,b; Loudiyi et al., 2018a,b). Karoui et al. (2003) evaluated for the first time the potential of classical FFFS to monitor the thermal behavior of Comté, Emmental, and Raclette cheeses during gentle heating from 5 to 60 °C. The analysis by PCA and FDA of protein tryptophan emission spectra and vitamin A recorded on cheese samples showed a clear discrimination of cheese samples as a function of temperature. In addition, they reported that the analysis of the variation of the maximum intensity of vitamin A fluorescence spectra during heating described two distinct linear regions. In addition, the intersection point of these two linear regions could be used to identify the melting temperature of fat in cheese. Finally, canonical correlation analysis (CCA) applied to dynamic rheological

measurements of cheeses and spectral data (tryptophan and vitamin A fluorescence spectra) highlighted high correlation values between the 1st and 2nd components, 0.94 and 0.49, respectively. This revealed that rheological data and spectral measurements described the same melting phenomenon but at different scales, macroscopic (rheology) and molecular (fluorescence) (Karoui and Dufour, 2003). Boubellouta and Dufour (2012), on Raclette and Comté cheeses heated from 20 to 80 °C, confirmed those conclusions. Contrary to the previous study, these authors used synchronous fluorescence spectra recorded between 250 and 500 nm with a $\Delta\lambda = 80$ nm. The melting temperature of the fat in cheese was also evaluated by monitoring the maximum intensity variation of the vitamin A fluorescence band, located at 320 nm. The authors reported that FFSFS could be a valuable tool to identify the melting properties of the cheese matrix. The evaluation was performed by drawing the intensity variation of the tryptophan band (at 295 nm) during heating and identifying the melting temperature of the cheese matrix as the intersection point of the two distinct linear regions (Boubellouta and Dufour, 2012). These results corroborated with the results of Loudiyi et al. (2017b). They demonstrated that the melting temperatures of fat and cheese matrix could be evaluated after the analysis of synchronous fluorescence spectra recorded during heating (i.e., 20 to 60 °C). These authors proposed to analyze the independent component (IC) scores variation obtained after ICA of fluorescence spectra. The analysis of the IC related to the vitamin A and tryptophan presented two distinct linear regions depending on the temperature, and the intersection point was identified as the melting temperature of the fat and the cheese matrix, respectively. Nonetheless, they reported that a slight decrease in the melting temperatures of the cheese matrix was obtained with the fluorescence compared to those calculated with the reference method (i.e., viscoelastic measurement) (Loudiyi et al., 2017b). They also reported the suitability of FFSFS to characterize the structural changes at a molecular level in cheeses during gentle heating (20 to 60 °C) and subsequent cooling (to 20 °C) regardless of the salt contents of cheeses. Compared to the control, the reduction of NaCl and its substitution by KCl induced changes in synchronous fluorescence spectra at 20 °C before heating, while small changes were observed after subsequent cooling at 20 °C. In addition, the joint analysis of the rheology data and the IC scores obtained after analysis of synchronous fluorescence spectra demonstrated a great link between observed spectral modifications and modifications in cheese texture (Fig. 4).

In another study, Ozbekova and Kulmyrzaev (2017) evaluated the potential of classical FFFS to predict the melting temperature of Tilsit cheese, a Kyrgyzstan cheese. PCA and PLS regression were applied to the fluorescence spectra to extract information on melting temperatures of the cheese matrix during heating from 25 to 75 °C. The results of PCA proved the close relationship between the spectral data and temperature conditions and, consequently, between the spectral data and viscoelastic characteristics of the cheeses. By using PLS regression, the authors demonstrated that fluorescence spectroscopy can potentially be used to obtain accurate, non-destructive, and rapid prediction of cheese melting temperature. The highest correlation was obtained with the tryptophan emission spectra ($R^2 = 0.99$, LV = 6), followed by the vitamin A emission spectra ($R^2 = 0.98$, LV = 9) and the vitamin A excitation spectra ($R^2 = 0.89$, LV = 6). The above-discussed studies were carried out on a limited number of samples and cheese varieties. It is, therefore, essential to perform further studies on a larger number of cheeses to test the robustness of the methods. On the other hand, the cheeses studied were hard or semi-hard cheeses, which present a certain isotropy in terms of structure and texture, unlike blue cheeses which are highly anisotropic. Studies on higher temperature ranges (> 100 °C) would also be necessary to define the potential of fluorescence spectroscopy to predict cheese behavior during cooking.

Advantages and Limitations of Spectroscopy Techniques

Conventional biochemical, physico-chemical, and sensory analyses, as well as spectroscopic techniques such as fluorescence, MIR, NIR, Raman, and NMR, have been successfully used to evaluate heating treatments on milk and dairy products. Table 2 presents some advantages and limitations of these techniques. Undoubtedly, the advantage of devices based on spectroscopy compared to conventional ones is the possibility to realize rapid measurements after and during processing with reduced time of preparation of samples. Nonetheless, depending on the spectral technique used, some differences in the versatility of spectroscopic techniques can be highlighted. Due to its high-quality spectra, measurement simplicity, high repeatability, and sensitivity, MIR spectroscopy is certainly one of the most used techniques. MIR spectroscopy has the advantage of being suitable for analyzing both diluted and highly concentrated samples especially when the equipment include an attenuated total reflectance (ATR) cell. The high specificity of spectral pics and bands makes chemical assignment and structural modification relatively easy to analyze. However, for extracting in-depth information on molecular structure and components concentration, mathematical modeling is generally necessary (e.g., chemometric tools, band deconvolution methods). In addition, this method is highly sensitive to water vibration, which can lead to a problematic interpretation of chemical bands, especially for the analysis of secondary structure of proteins. Moreover, its in-line implementation is quite challenging. This technique is also unable to determine substances that are present in very low concentrations in milk and dairy products.

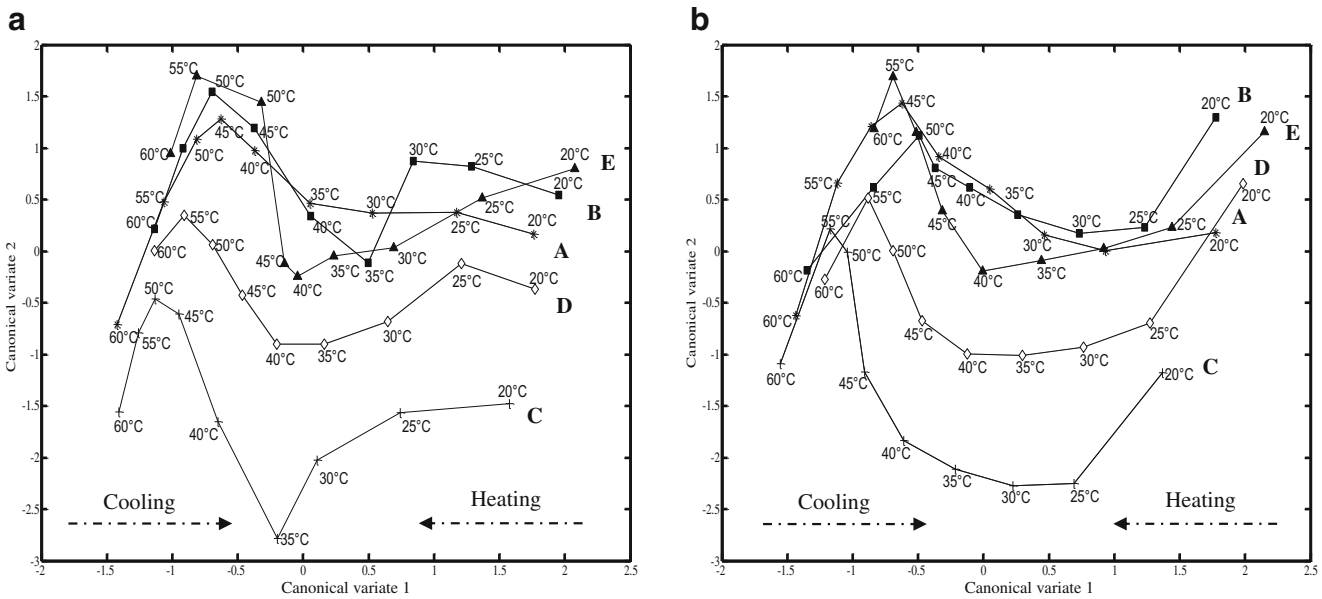

Fig. 4 Results of canonical components analysis applied jointly to spectral data and rheology data recorded on Cantal-type cheeses during heating and subsequent cooling. **a** Similarity map defined by canonical variates 1 and 2 for the proportions obtained after ICA on Synchronous Fluorescence spectra recorded on five cheese with different salt contents (A, 0.5% NaCl (asterisks); B, 1% NaCl (black squares); C, 2% NaCl (crosses); D, 1.5/0.5% NaCl/KCl (diamonds); E, 1/1% NaCl/KCl (black triangles)). **b** Similarity map defined by canonical variates 1 and 2 for the rheology data of the same cheeses A, B, C, D, and E

Concerning NIR spectroscopy, it is gaining popularity in the monitoring of different milk and dairy industrial operations due to its low running costs. It is rapid, reliable, environmentally friendly, and simple to perform real-time measurements in various online applications. However, the on-line monitoring of the processes is challenging due to factors such as variation in temperature and sample motion. In addition, NIR spectroscopy cannot directly determine the concentrations of components that are below 0.1% (w/w). The low structural selectivity of NIR is due to the overlap of different overtones and combination bands as compared to MIR radiation, through which many fundamentals can be observed at distinct positions. Such overlapping of signals makes the interpretation difficult in the conventional NIR spectrum. The high amount of moisture in the samples is also a disadvantage for the interpretation of NIR signals (Williams and Norris, 1987). Furthermore, the complex food matrix and the heterogeneity of the sample make it challenging to obtain accurate results.

Fluorescence spectroscopy coupled with chemometric tools is also a valuable method to monitor milk and dairy products chemical changes during processes and predict their sensory, rheological, and melting properties. In comparison with light absorption measurements, fluorescence spectroscopy is more sensitive (i.e., 1000 times more sensitive and specific than absorption techniques), because the measured signal has, in principle, zero background. In addition, fluorescent compounds are susceptible to their environment and offer the possibility to characterize molecular interactions and reactions. Despite the exciting potential of this technique and its high sensitivity for the characterization of milk and dairy products during processing, the interpretation of fluorescence spectra is still challenging, likely due to the complexity of the food product matrix (composition and anisotropy). Indeed, the use of single-pair emission-excitation wavelengths can be severely limited by the overlap of spectra and inner filter effects (in which non-fluorescence compounds absorb the excitation radiation or that emitted by the target fluorophore). Moreover, reduction in the fluorescence intensity can occur due to chemical or environmental influences (Lourenço et al., 2012). These phenomena create difficulty in associating a clear and specific physico-chemical phenomenon to the variation of the bands, especially when using classical FFFS. Using the SFS and EEM as multidimensional fluorescence can provide a better overview of the biochemical phenomenon occurring during processing. In the future, efforts need to be made by the spectrometer designers in terms of spectral acquisition times in order to make this technique more compatible with the requirement of the industry. Fluorescence imaging using a multispectral device can also be a powerful strategy to give a clearer picture of the undergoing chemical modification during food processing due to its ability to gather both spectral and spatial information.

While most of the studies have used NIR, MIR, and fluorescence spectroscopies for the characterization of milk and dairy products (e.g., composition, quality, geographic origin), the NMR spectroscopic technique has also demonstrated its high potential and its versatility for studying opaque heterogeneous samples. NMR can be considered as a promising method for the

Table 2 Advantages and disadvantages of different techniques that can be used to monitor the quality of milk and dairy products during or after heat treatments

Techniques	Concept	Advantages	Disadvantages
Sensory analysis	Description of sensory properties and/or identification of consumers' preferences. It uses human senses (sight, smell, taste, touch, and hearing)	• Wide range of tests and methods to meet specific characteristics • Closely linked to real consumer perceptions • Detailed description of sensory qualities and deficiencies	• Long, can be fastidious and expensive to implement according to sensory tests used • It needs standard conditions (a trained panel and individual test booths) • Not suitable for on-line measurements • Recruitment of a large number of consumers (in the case of hedonic test)
Microbiology	Development of microorganisms on agar medium (or other media) adapted to the desired microbial population	• Usually standards-based method • Provides microbiological quality	• Long to implement • Not suitable for on-line measurements
Wet chemistry	Use of different chemical reagents and solvents	• Provides multicomponent concentrations • Usually standards-based method	• Long to implement • Not suitable for on-line measurements • Needs skilled operators • Uses chemicals that can be hazardous to the operator and environment
HPLC/GC	Difference in the affinity between the solvent and the column	• Provides multicomponent concentrations • Usually standards-based method • Highly sensitive	• Needs skilled operators • Not suitable for on-line measurements • Long and expensive to implement
Raman spectroscopy	Vibrational spectroscopy technique after laser excitation (generally 785 and 1050 nm)	• Suitable for on-line measurements • Highly sensitive (especially in SERS configuration) • Provides multicomponent structural, quantitative, and qualitative information • Low interference of water	• Interference from biological fluorescence • Background signals • Requires complex data analysis (chemometrics) • Small part of the sample is irradiated (laser spot) • Low sensitivity and complex instrumentation
Fluorescence spectroscopy	Emission of low-energy light after absorption of UV and VIS light	• Suitable for on-line measurements • Highly sensitive • Provides multicomponent structural, quantitative, and qualitative information	• Can be affected by Raman scattering • Requires complex data analysis (chemometrics) particularly in 3D fluorescence configuration • Limited to fluorescent molecules • Sensitive only to surface measurement • Needs skilled operators
NMR spectroscopy	Absorption of electromagnetic radiation Observe local magnetic field around atomic nuclei	• Simple to operate • Presence of spatial information (if coupled with the imaging) • Provides multicomponent information	• Requires complex data analysis (chemometrics) • Expensive equipment
MIR spectroscopy	Fundamental vibration of organic molecules	• Simple to operate • Provides multicomponent information • Provides multicomponent structural, quantitative, and qualitative information	• Not suitable for on-line measurements • Requires complex data analysis (chemometrics) • High interference of water • Sensitive only to surface measurement • Difficult to handle sample anisotropy
NIR spectroscopy	Overtones and combinations of fundamental vibrational of organic molecules	• Suitable for on-line measurements • Insensitive to direct detection of low-concentration components • Deep penetration of the radiation into the sample • Provides multicomponent quantitative and qualitative information	• Requires complex data analysis (chemometrics) • Difficult to interpret spectra due to the overtones and combination of fundamental vibrations, and the interference of water absorption • Difficult to handle sample anisotropy • Redundant information of the spectral bands

classification of dairy product quality and monitoring their chemical modifications during milk and dairy product processing, especially due to its sensitivity to relaxation of water. It can also provide information that cannot be gathered via other spectral techniques known. However, this technique is less sensitive as compared to MIR or fluorescence and a number of parameters that need to be optimized. For example, the wrong selection of pulse sequence provides aberrant results. Newer devices are in development, which will pave the way for experiments and use of NMR outside research laboratories.

Conclusions and Future Trends

This review reported and discussed some of the recent studies on the applications of spectroscopic techniques to monitor and/or predict the effect of heat treatments on the qualities of milk and dairy products. Most of the studies reported are in relation to infrared (approx. 44%) and fluorescence spectroscopy (approx. 34%). Applications of these techniques deal with the direct interpretation of spectra as a response to thermal treatments, or the interpretation of more complex interactions (e.g., calibration development, classification) using chemometric analysis (e.g., principal components analysis, partial least squares regression, etc.). Most of the applications analyzed here focused on the classification and/or calibration of samples analyzed in the laboratory. Minority of the studies have used these techniques for online and inline analyses. However, the number of online applications of these analytical methods is expected to increase as new instruments become available (e.g., handheld and portable NIR spectrophotometers) and as new algorithms for processing data appear.

This review provides evidence that spectral analytical techniques mentioned above have vast advantages over other analytical approaches to monitor heat treatment in milk and dairy products. Despite this, some challenges persist: the application and implementation of these technologies in industries require extensive knowledge of the field of spectral analysis. This involves training new researchers and industrial practitioners, as well as transferring knowledge and technology between academic and industrial researches.

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