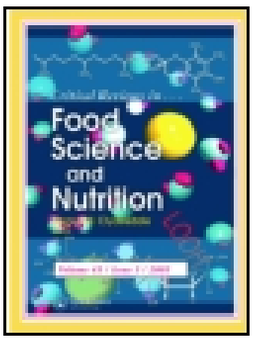

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Recent developments in the food quality detected by non-invasive nuclear magnetic resonance technology

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Abstract

Nuclear magnetic resonance (NMR) is a rapid, accurate and non-invasive technology and widely used to detect the quality of food, particularly to fruits and vegetables, meat and aquatic products. This review is a survey of recent developments in experimental results for the quality of food on various NMR technologies in processing and storage over the past decade. Following a discussion of the quality discrimination and classification of food, analysis of food compositions and detection of physical, chemical, structural and microbiological properties of food are outlined. Owing to high cost, low detection limit and sensitivity, the professional knowledge involved and the safety issues related to the maintenance of the magnetic field, so far the practical applications are limited to detect small range of food. In order to promote applications for a broader range of foods further research and development efforts are needed to overcome the limitations of NMR in the detection process. The needs and opportunities for future research and developments are outlined.

Keywords Nuclear magnetic resonance, Relaxation, Composition, Quality, Food

Introduction

Nuclear magnetic resonance (NMR) is a phenomenon of energy exchange with an alternating magnetic field by nuclei of fixed magnetic moments such as ^{1}H, ^{13}C, ^{23}Na, ^{31}P, ^{15}N (Laghi et al., 2014; Gudjonsdottir et al., 2015). NMR in food science was first used to determine moisture of food in the 1950s (Zhu, 2017). With the development and improvement of NMR technology in food, NMR is applied to different food fields including quantitative analysis (Chen et al., 2010; del Campo et al., 2010), conformational analysis (Vilén et al., 2010; Youssouf et al., 2017), nutritional or functional aspects (Caligiani et al., 2010a; Lee et al., 2010), quality detection (Alonsosalces et al., 2010; Di et al., 2011) and process control (Gudjonsdottir et al., 2010). The advantages of NMR are that there is no need to separate different food ingredients, a small amount of sample pretreatment and preparation, as well as repeatability, non-destructive and quantitative (Kirtil and Oztop, 2015). However, the detection limits and sensitivity of NMR need to be further improved compared to gas chromatography, liquid chromatograph mass spectrometer, inductively coupled plasma mass spectrometry, liquid chromatography electrospray ionization and mass spectrometry technology (Marcone et al., 2013; Herrero et al., 2010; Le Bourse et al., 2010).

NMR technology has two types of high resolution NMR (HR-NMR) and low field NMR (LF-NMR) basing on NMR spectrum and magnetic resonance imaging (MRI) techniques (Kirtil et al., 2017). NMR technology was widely applied for food science in recent years because of the need for effective quality control analysis and the growing demand for technological and product innovation in the food industry (Spyros and Dais, 2012; Marcone et al., 2013). NMR spectroscopy was proposed for the determination of relaxation time. NMR relaxation time is used not only to measure chemical compositions, but also to evaluate the quality of food (Mariette, 2009). MRI is also a powerful tool to observe the interior structure information of food and monitor physical and chemical properties in food processing and storage (Marcone et al., 2013; Lagnika et al., 2013; Jiang et al., 2013; Trimigno et al., 2015; Xu et al., 2015). Therefore, NMR relaxation and NMI technology are very popular in food-related applications.

The advances in the application of NMR have been reviewed in food including wine (Ogrinc et al., 2003), dairy products (Karoui and Debaerdemaeker, 2007) or other applications such as food traceability (Mannina et al., 2012), food authenticity (Moore et al., 2012), study on the relationship between water distribution and the quality parameters in fruits and vegetables and meat (Pearce et al., 2011; Butz et al., 2010; Islam et al., 2015; Su et al., 2017). However, these discussions had a certain limited coverage in NMR application. Much progress in

recent years has been made to study NMR application in the food science. Therefore, the aims of this paper were to present an overview of the recent developments in the quality of food detected by NMR and prospects further research.

Quality discrimination and classification by NMR

The properties of food quality generally concerned by consumers including flavor, type, origin, year and so on (Consonni et al., 2011; Consonni et al., 2008; de Oliveira et al., 2014). These are important properties for evaluating food quality. NMR technology has been a valid method for molecular structure identification and research of food components, and plays an important role in quantitative analysis (Corsaro et al., 2016; Ribeiro et al., 2014a; Panarese et al., 2012). NMR combined with common multivariate methods including principal component analysis (PCA) and partial least square discriminant analysis (PLS-DA) was applied to identify and characterize food components by the collection and analysis of one-dimension (1D), two-dimensional (2D) and multidimensional NMR spectrum (Deborde et al., 2017; Kirtil et al., 2017; Panarese et al., 2012). Recently, NMR technology is the most common method in the field of food classification and discrimination applications (Ribeiro et al., 2014b; Santos et al., 2016; Yang et al., 2009; Boffo et al., 2009; Hachem et al., 2016; Zhang et al., 2012b; Shao and

Li, 2010). According to the specific standards of different foods, NMR can identify type, origin, date of manufacture, preservation time of fruit juice, meat and aquatic products, as shown in Table 1.

The nutrients and sensory attributes from different kinds of fruits are different. The NMR technique can be applied to identify the fruit juice and classify fruits cultivars, thus guaranteeing authenticity of fruit juice consumed by the large number of the consumers (Ogrinc et al., 2003). Cuny et al. (2008) presented that ^{1}H NMR spectroscopy combined with different chemometric analyses were used to discriminate orange juice from grapefruit juice. They obtained that independent-component analysis (ICA) as a useful chemometric tool gave better classification results than PCA basing on ^{1}H NMR spectra information. As reported by Clausen et al. (2011), juices from seven sour cherry clones/cultivars were classified by sensory evaluation and high-resolution ^{1}H NMR spectroscopy combined PCA and PLS. They obtained that malic acid was the most important metabolite and highly correlated with sweetness and sourness by PCA and PLS combined with 2D NMR spectra such as ^{1}H-^{1}H COSY and ^{1}H-^{13}C HSQC. On the other hand, Koda et al. (2012) presented that band-selective excited low-field ^{1}H NMR spectra combined with PCA was used to identify five mango cultivars (a total of 55). They found that arginine, histidine, phenylalanine, glutamine, shikimic acid and trigonelline were important components for five mango cultivars classified by F2-selective 2D NMR spectra including TOCSY, DQF-COSY, NOESY, ^{1}H-^{13}C HSQC and ^{1}H-^{13}C CT-HMBC. Similarly, ^{1}H NMR technology combined with PCA and PLS was used not

only to discriminate orange varieties but also to evaluate degradation of fresh orange juice with the changes of storage temperature and time for 24 h (de Oliveira et al., 2014). Results showed that formic, fumaric and acetic acids were produced in orange juice after storage 24 h, and succinic and lactic acids and ethanol in orange juice increased at the early stage of storage. Moreover, five orange juice varieties can be effectively identified basing on PCA analysis. Therefore, NMR coupled with chemometric techniques can be used to distinguished different varieties of fruit juice.

NMR technology has been explored to discriminate traceability and geographical origin of meat products such as beef. As reported by Jung et al. (2010), ^{1}H NMR assisted with PCA and orthogonal projection to latent structure-discriminant analysis (OPLS-DA) was applied to discriminate geographical origin. They found that beef extracts originated from Australia, Korea, New Zealand and United States were significantly difference. In addition, the succinate and amino acids were major metabolites, among which amino acids included isoleucine, leucine, methionine, tyrosine, and valine for discriminating the origin of beef according to ^{1}H NMR spectra and OPLS-DA analysis. Zanardi et al. (2013) presented that ^{1}H NMR combined with stepwise linear discriminant analysis (sLDA) and artificial neural network (ANN) was applied for classifying 90 irradiated or 30 non-irradiated beef samples. They obtained that both irradiated and non-irradiated beef can be classified by sLDA and ANN analyses. Thus, NMR could be suitable for identifying the irradiation dose in meat. On the other hand, time-domain ^{1}H Nuclear

Magnetic Resonance (TD-NMR) using CPMG and CWFP sequences was applied to discriminate 99 beef samples basing on race and sex of various bull (Santos et al., 2014). They obtained that TD-NMR combined with PLS-DA can provide a reliable identification for tracing beef samples. Therefore, NMR combined reliable analysis methods can be suitable to discriminate meat sample.

NMR technology has also been extended to discriminate authenticity and origin of aquatic products such as salmon, hake and prawn. Masoum et al. (2007) presented that the origin of salmon was classified by using ^{1}H NMR and support vector machines (SVMs). Results showed that ^{1}H NMR spectroscopy combined with SVMs can provide a validated method to discriminate wild and farmed salmon and reduce the possibility of fraud from salmon origins. As reported by Greiff et al. (2014), low-field NMR transverse relaxation time (T_2) was suitable to distinguish 0-3.0% salt hake basing on PCA. They obtained that ^{1}H LF-NMR can be used to study low-salt products. On the other hand, Li et al. (2018) investigated that LF-NMR spectroscopy and MRI was used to detect the hydrocolloids injected prawns. They found that T_2 fitting curves can be successfully applied for distinguishing adulterated prawns and formal ones with PCA. Therefore, NMR is a powerful technique to distinguish aquatic products.

NMR technology has been used to discriminate authenticity and origin of honey. Schievano et al. (2010) presented that the botanical origin of unifloral and polyfloral honey was identified by using ^{1}H NMR. They obtained that the time of NMR analysis required only 30 min and the yield of correct honey identification was 100%. On the other hand, honey adulteration increased with adding different commercial sugar syrups by producers because of limited production of honey. Ribeiro et al. (2014a) reported that the possibility of differentiating the botanical origin of honeys was evaluated by using LF-NMR. They established the relationship between T_2 parameters and physicochemical data of honey from 8 botanical sources including water content, water activity, pH and color. Results showed that good linear correlations were obtained between the T_2 and T_{21} parameters and physicochemical data. Similarly, Ribeiro et al. (2014b) investigated honey adulteration by high fructose corn syrup concentration from 0% to 100% using LF-NMR. Results showed that NMR T_2 parameters increased with increasing high fructose syrup concentration in adulterated honey. Linear correlations were observed between the T_2, T_{21} and T_{22} parameters and physicochemical data. Therefore, LF-NMR can be used to discriminate and classify honeys by their botanical origin.

Composition analyzed by NMR

The energy and nutrition for people are mainly originated from food. All kinds of food have complex compositions including water, fat, protein, carbohydrates, cellulose and inorganic salts, which contain rich protons (Shao and Li, 2011; Kovrlija and Rondeau-Mouro, 2017; Li et al., 2012b; Petrov et al., 2008; Nowacka et al., 2014; Aursand et al., 2010; Gudjonsdottir et al., 2015; McDonnell et al., 2013; Gudjónsdóttir et al., 2011a; Wang et al., 2016). NMR technology can rapidly and accurately analyze the change of components without changing the original state of foods. NMR technology in recent years has been widely applied to analyze water, fat and protein from food (Petrov et al., 2008; Sekiyama et al., 2012; Zhang and McCarthy, 2013; Tao and Ngadi, 2017; Niu et al., 2014; Khan et al., 2016; Castell-Palou et al., 2011; Mati et al., 2015; Yang et al., 2018).

Water analysis

Water distribution and water content are closely related to the quality of food in production, processing, storage and transportation (Halder et al., 2011; Mikac et al., 2015; Hong et al., 2009; Xu et al., 2016; Otero and Préstamo, 2009; Fundo et al., 2016; Aguiló-Aguayo et al., 2014; Santagapita et al., 2012; Geng et al., 2015; Sanchez-Alonso et al., 2014; Lv et al., 2016). Water distribution and water content in food can be measured by using NMR technology. According to NMR parameters including longitudinal relaxation time (T_1) and transverse relaxation

time (T_2) and MRI, water signal and can reflect the quantification of water content and water distribution in food such as fruits and vegetables, meat and aquatic products, as shown in Table 2.

NMR was widely used to study water distribution of fruits and vegetables. In terms of fruits, Cheng et al. (2014) investigated that LF-NMR and MRI were applied to analyze water status in osmotically dehydrated strawberry treated by ultrasound and pulsed vacuum. They found that the vacuolar space decreased and cytoplasm intercellular space increased basing on T_2 and MRI analysis. As reported by Tylewicz et al. (2016), TD-NMR was applied to assess water distribution of freeze-dried apple treated by pulsed electric fields at 0.3-1.2 kV cm^{-1} for 5, 10 and 15 pulses. Results showed that water molecules had high T_1 values in vacuoles and extracellular spaces. Similarly, Traffano-Schiffo et al. (2017) studied that water distribution in osmotic dehydrated kiwifruit pretreated by pulsed electric fields at 100-400 V/cm for 60 pulse was observed by using TD-NMR. They obtained that TD-NMR can quantify water molecules basing on water state of kiwifruit tissue, and obtain sorption isotherms of adsorbed water by applying BET model. In terms of vegetables, Xin et al. (2013) reported that LF-NMR and MRI was used to analyze water state in broccoli treated by ultrasound-assisted osmotic dehydration using trehalose. Results showed that less mobile water in cytoplasm (T_{21}) and high mobile water in vacuole (T_{22}) were decreased by ultrasound-assisted osmotic dehydration within 30 min. Meanwhile, MRI signals would present a bright "water strip". Similarly, as reported by Xu et al. (2017), whose studied water distribution by LF-NMR and

MRI techniques in the broccoli during hot-air drying. On the other hand, Lv et al. (2017) investigated that LF-NMR was used to measure online water status of six kinds of vegetables dried by microwave vacuum drying. Results showed that there is no single empirical model fitting for signal amplitude (A_2) and moisture content (M_1) of mushroom, carrot, potato, lotus, edamame and corn. However, there was a significant linear relationship between A_2 and M_1 in different microwave vacuum drying stages for each vegetable. Therefore, NMR can was used to quantify and observe water status and distribution.

NMR as a fast noninvasive tool has been applied to understand water mobility and distribution in meat and aquatic products. For example, Carneiro et al. (2016) investigated that water mobility of salted sardines muscle was detected by using LF-NMR during storage at 30 °C. Results suggested that T_2 data displayed three components: pool I (11.2-17.5 ms), pool II (35.3-44.7 ms) and pool III (161.7-256.5 ms). On the other hand, Shao et al. (2016) studied that protein hydration states were measured for mobility water in meat batters by added water and fat. They found that there were three relaxation components (T_{2b}, T_{21} and T_{22}) in meat and fat groups by using LF-NMR. T_{2b} and T_{21} (0-10 ms) were protons in macromolecules and combination with macromolecules, respectively. T_{22} (40-70 ms) was water or fat combined with sol matrix of meat batters. After meat batters were heated, T_{23} (200-300 ms) was observed. In addition, T_{22} of meat added fat was higher than that of meat added water. However, T_{23} of meat added water was higher than that of meat added fat. Similarly, as reported by Ojha et al. (2017), LF-NMR

was used for evaluating the changes of water distribution in the drying of beef jerky by ultrasound pretreatment at 25, 33 and 45 kHz for 30 min. Results showed that there were three distinct peaks, which were respectively bound water (T_{2b} with the relaxation time of 0-10 ms), water from dense myofibrillar protein matrix (T_{21} with the relaxation time of 10-100 ms) and free water (T_{22} with the relaxation time of >100 ms). Therefore, LF-NMR can be used to detect water mobility in meat and aquatic products.

Fat analysis

Fat for the human body plays an important role in food composition (Elmsjo et al., 2015). On the one hand, fat is an essential component of the cell membrane and maintenance of body moisture, which is also a lot of hormone-generating material in the human bodies (Blair et al., 2016). On the other hand, the high intake of fat can cause obesity, coronary heart disease, and other health problems (Sun et al., 2017). Therefore, it is very important to detect the content and composition of fat. NMR spectroscopy can be used to analyze the content and composition of fat in foods (Youssef and Barbut, 2009; Fang et al., 2013; Colnago et al., 2011; Nascimento et al., 2017; Castell-Palou et al., 2012; Fry et al., 2017; Tao and Ngadi, 2017; Miklos et al., 2014; Ritota et al., 2010; Pereira et al., 2013a). Recently, some researchers have applied NMR to measure the content and fatty acid composition of the fat in animal products. For instance, Nestor et al. (2010) investigated

that high resolution magic angle spinning (HR-MAS) NMR was applied for quantifying small metabolites in Arctic char muscle. They found that HR-MAS NMR can obtain total n-3 fatty acid, EPA and DHA contents. In addition, they also observed the most abundant creatine/phosphocreatine, anserine and taurine. Similarly, Manzano Maria et al. (2010) studied that conjugated linoleic acids (CLA) in beef was measured by ^{1}H NMR spectra. Results showed that ^{1}H NMR was at least 10 times faster than GC for measurement of CLA content. Moreover, NMR can also detect CLA content in dairy and other meats products. Stefanova et al. (2011) reported that ^{1}H-NMR spectroscopy was used for examining fatty acid compositions of chicken meat irradiated by gamma-ray at 0.5-15.0 kGy. They found that there was a dose-effect relationship of irradiation by ^{1}H-NMR. As reported by Miklos et al. (2015), fat mobility in gels adding lard, diacylglycerols or sunflower oil was measured by LF-NMR. Results found that there was no significance in fat mobility between lard and diacylglycerols. However, sunflower oil has a large influence in fat mobility. On the other hand, Zang et al. (2017) investigated that fat content of small yellow croaker was measured during drying according to LF-NMR and multivariate analyses including PCA and PLS. They obtained that fat content was 0.43-1.72 g and accurately predicted by LF-NMR combined with PLS. Therefore, NMR can be effectively used to quantify fat content and analyze fatty acid compositions.

Protein analysis

Protein is an important component of food. NMR has been used for the qualitative and quantitative analysis of protein and amino acids in food (Consonni et al., 2011; Carneiro et al., 2016; Lee et al., 2010; Ritota et al., 2010; Gudjónsdóttir et al., 2011b). For example, Gudjónsdóttir et al. (2011a) presented that the relationship between NMR parameters and protein content was analyzed in rehydrated cod. Results showed that T_1 and T_{21} were the most significant for changes and denaturation of protein. Additionally, T_1 and T_{21} had significantly correlated with the non-protein nitrogen content (free amino acids, peptides, trimethylamine oxide, and trimethylamine). As reported by Sanchez-Alonso et al. (2014), LF-NMR was performed to analyze quality of hake muscle in ice for 3 and 14 days prior to different freezing methods (nitrogen, air blast and walk-in) and frozen storage temperature (-10 and -20 °C for 5 days, 8 and 18 weeks). Results found that T_{21} time constant decreased in hake muscle at -10 °C for 18 weeks due to protein denaturation and aggregation. Similarly, Carneiro et al. (2016) investigated that LF-NMR was used to measure the quality changes of salted sardines induced by water mobility during storage at 30 °C. They found that decarboxylase enzymes increased in salted sardines due to protein fraction denatures and degrades causing an increase in free amino acids. On the other hand, NMR combined with PCA was used to evaluate the changes of amino acids from orange juice treated by atmospheric cold plasma (ACP) and ozone (Alves Filho et al., 2016). Results showed that ^{1}H NMR spectrum for orange juice after ACP processing displayed high concentration of several amino acids including proline at 2.06 and 2.34 ppm, dimethylproline at 2.06 ppm, glutamic acid at 2.18 ppm, and of arginine at 1.94

ppm. ^{1}H NMR spectrum for orange juice after ozone processing presented high amount of tyrosine at 6.96 ppm and 7.24 ppm. Therefore, NMR is an effective tool to obtain qualitative and quantitative information on proteins.

Quality detected by NMR

The quality changes of food would take place in processing and storage (Gudjonsdottir et al., 2015; Carneiro et al., 2016). In order to evaluate physical, chemical, structural and microbiological properties changes, some researchers have used NMR to detect the quality of different food (del Campo et al., 2010; Gudjónsdóttir et al., 2011c; Zhang et al., 2012a; Caligiani et al., 2010b; Sekiyama et al., 2012; Fundo et al., 2016; Geng et al., 2015; Bizzani et al., 2017; Pereira et al., 2013b; Li et al., 2014).

Physical properties

Physical properties including color, water activity, texture, pH and water holding capacity are very important indicators for the quality of food in processing and storage (Pearce et al., 2011; Corsaro et al., 2016). Some researchers have used NMR to detect physical parameters (Li et al., 2012a; Pereira et al., 2013a; Geng et al., 2015; Li et al., 2014).

The color is an essential physical indicator from the consumer point of view. Li et al. (2012a) studied that the relationship between T_{21} and brightness (L^* value) was analyzed for thawed pork in Group A (38.68 ms for T_{21}), B (42.32 ms for T_{21}) and C (44.53 ms for T_{21}) by LF-NMR. Results presented that T_{21} was positive and significant correlation with L^* value ($P<0.05$). As presented by Carneiro et al. (2013), the color parameters (L^*, a^* and b^*) of frozen shrimp with added sodium polyphosphate was investigated by using LF-NMR. They found that T_{21} was significant correlation with the L^* ($r = -0.82$), a^* ($r = -0.82$) and b^* ($r = -0.79$). Additionally, T_{21} were correlated with a lower intensity of a^* and b^*. Similarly, Li et al. (2017) presented that color changes in frozen shrimps were analyzed by LF-NMR. They observed that T_2 (T_{2b} and T_{21}) with exception for T_{22} was significantly correlated with L^* and b^*. However, T_2 was insignificant correlation with a^*. On the other hand, Fundo et al. (2016) investigated that a relationship between T_2 and total color difference (TCD) was established. Results showed that TCD decreased with an increase in T_2 because free water in cells can promote the biochemical reaction causing color changes. Therefore, NMR can reflect color change of food.

Water activity is important parameter for the microbiological stability of food. Aursand et al. (2008) presented the relationship between T_2 parameters and water activity during salted cod and salmon. They found that T_{21} has a good linear correlation ($F\leq0.05$) with water activity basing on LF-NMR T_{21} parameter. Similarly, Gudjónsdóttir et al. (2011a) also analyzed LF-NMR parameters (T_1, T_{21} and T_{22}) correlation with

water activity in the injected and brined pre-salted cod muscle. Results showed that T_1, T_{21} and T_{22} presented significantly correlation with water activity. On the other hand, As presented by Fundo et al. (2016), the relationship between NMR parameters and water activity was established for fresh-cut 'Rocha' pear during 7 days of refrigerated storage. Results showed that an increase in maximum value for T_2 was with an increase in water activity. Therefore, NMR can be used to predict water activity in food processing and storage.

Another relevant physical parameter of food quality is texture. Sanchez-Alonso et al. (2012) investigated that shear stress was assessed by LF-NMR in hake during frozen storage at -10 °C for 6 months. They found that the shear stress values showed significant correlation with T_{21} by bi-exponential or CONTIN analysis. However, there was poor correlation with shear stress. Therefore, authors considered that T_{22} could be more heterogeneous than T_{21}. In addition, relaxation time of other water populations could also be closer to that of free water. Similarly, Li et al. (2014) presented that shear force of chicken jerky was measured during drying process by LF-NMR. They obtained that the signal area of the immobilized water per mass (A_2) was notable correlations with the shear force. The signal area of total water content per mass (A) and the shear force had significantly correlation. On the other hand, As presented by Geng et al. (2015), the correlations of NMR T_2 parameters and texture parameters (hardness, chewiness, cohesiveness and springiness) was showed in dried sea cucumber during rehydration process. Results demonstrated that both hardness and chewiness had significant correlations with T_2 parameters. However, there was a good

correction between cohesiveness and T_{21}. The springiness had insignificantly correlated with T_2 parameters. Li et al. (2017) also presented that the correlations of NMR T_2 data and texture parameters were established in frozen shrimp muscle. They also observed that the hardness, gumminess, and chewiness were significant correlations with T_2 data with exception of T_{22} ($p<0.01$ or $p<0.05$). Therefore, NMR can be used to evaluate the texture change of food.

The change of pH can reflect the quality changes of food. Gudjónsdóttir et al. (2011a) presented that the correlation was studied between the NMR relaxation parameters and the muscle pH in cod during the salting and rehydration processes. They observed that the muscle pH had significantly correlated with T_1. The transversal water distribution (A_{21}/A_{22}) was also significantly correlated with muscle pH. The similar results were reported by Gudjónsdóttir et al. (2011c), they found that the NMR T_{22} data was significant correlation with the muscle pH in brine injected cod loins during superchilled storage. The NMR T_1, T_{21} and T_{22} data had also significant correlation with the muscle pH in low salt cod loins. As presented by Li et al. (2012a), They investigated the correlation of NMR T_{21} component and the muscle pH in thawed pork. Results showed that NMR T_{21} was significantly correlated with muscle pH ($p<0.05$). On the other hand, Carneiro et al. (2013) studied that the correlation of NMR T_2 data and muscle pH was described in frozen shrimp treated by sodium tripolyphosphate. They observed that T_2

relaxation time (r=0.87 for T_{21}; r=0.70 for T_{22}) had a good correlation with muscle pH. Therefore, NMR was a good way to indicate pH change of food.

Water holding capacity (WHC) can reflect the water and affect economic value of food. Gudjónsdóttir et al. (2011a) investigated that the relationship between NMR relaxation parameters and WHC was analyzed in cod during salting and rehydration process. They found that T_1, T_{21} and T_{22} were significant correlations with WHC in cod muscle. The similar results were reported by Gudjónsdóttir et al. (2011c), They also observed that the relaxation times T_{21} and T_{22} had significant correlations with the WHC in brine injected and high salt cod loins during superchilled storage. T_1, T_{21} and transversal water distribution (A_{21}/A_{22}) had also significant correlations with the WHC in modified atmosphere packed cod. There was significantly correlation between T_{21} and the WHC in brined cod. According to Sanchez-Alonso et al. (2012), they studied the relationship between LF-NMR parameters and the WHC in hake during frozen storage at -10 °C for 6 months. Results showed that T_{21} and T_{22} had significant correlations with the WHC by bi-exponential analysis ($p<0.10$). On the other hand, Carneiro et al. (2013) studied that the relationship of LF-NMR relaxation parameters and the WHC was established in frozen shrimp muscle treated by sodium tripolyphosphate. They obtained that T_{21} and T_{22} showed good correlations with the WHC (r=0.90 for T_{21}; r=0.81 for T_{22}). As presented by Gudjonsdottir et al.

(2015), they investigated the correlation of LF-NMR relaxation times and the WHC in cod during the salting process. The relaxation time T_{21} was significant correlation with the WHC ($p<0.05$). Therefore, NMR can be suitable to estimate the water holding capacity of food.

Chemical properties

Chemical compounds from food could be changed in processing and storage. Some researchers have used NMR to detect the chemical compounds of food (Alves Filho et al., 2016; Bizzani et al., 2017; Ritota et al., 2010). For instance, Otero and Préstamo (2009) presented that HR-MAS NMR was employed to analyze sugars and content and organic acids in control and pressurized strawberry at low pressures (100-200 MPa). They observed that there were no significant changes of organic acids in pressurized strawberries in comparison to the control ones basing on ^{1}H HR-MAS NMR spectrum region of 0-3 ppm. However, there was a significant hydrolysis of sucrose detected by ^{1}H HR-MAS NMR spectrum. The major sugars content of strawberries decreased in the region of 3-5 ppm causing an increase in glucose and fructose. Similarly, Alves Filho et al. (2016) also presented that HR-NMR combined with PCA was applied for evaluating composition changes of orange juice treated by atmospheric cold plasma and ozone. Results showed that there were no significant changes on sucrose, β-glucose, fructose and citric acid in the orange juice by quantitative analysis. As reported by Soares et al. (2017), ^{1}H HR-NMR spectroscopy coupled with PCA and

PLS-DA was used to estimate chemical composition changes in passion fruit juice at 85 and 140 °C for 4, 15, 30, and 60 s. Results showed that the degradation of sucrose in passion fruit juice occurred causing an increase in glucose and fructose and the formation of 5-(hydroxymethyl)-2-furfural (HMF) basing on ^{1}H HR-NMR spectroscopy combined with chemometric analysis at 140 °C for 60 s. Furthermore, according to the concentration change of HMF monitored by ^{1}H NMR profile thermally treated in fruit juice, the possibility of overheat damages was easily detected. Francini et al. (2017) investigated that quantitative ^{1}H HR-NMR (^{1}H HR-qNMR) spectroscopy with PCA was used to identified and quantified several polyphenols including catechin, epicathechin and chlorogenic acid in different apple varieties after drying. Results showed that the signals of several polyphenol compounds appeared in the NMR spectrum region of 6.0-8.5 ppm. ^{1}H HR-qNMR spectra combined with PCA provided the phenolic compounds information of different dried apple. Thus, the phenolic contents of different apple products can be detected to improve the nutraceutical value. Therefore, NMR was a valid way to evaluate chemical properties of food.

Structural properties

The tissue structure changes of food were induced in processing and storage. MRI was widely used to visualize the difference in tissue structure. Some researchers have applied MRI to observe water status and water distribution related to internal tissue structure changes in

food processing and storage (Otero and Préstamo, 2009; Gonzalez et al., 2010; Zhang and McCarthy, 2012; Xu et al., 2017). For instance, Zhang and McCarthy (2012) presented that the tissue structure differences of healthy pomegranate and pomegranate with black heart were reflect by using MRI during postharvest storage. They found that the T_2 weighted MRI image can visualize all arils within the rind in healthy pomegranate. However, the arils region of pomegranate with black heart had some void areas and a decrease in signal intensity for portions. Thus, MRI can recognize black heart region in pomegranate tissues to ensure postharvest quality of fruit. As reported by Xin et al. (2013), water-selective transverse images showed different water distribution and migration in untreated and osmotically treated broccoli by using MRI. They found that a bright image signal was obtained in untreated broccoli. There was a 'water strip' in broccoli osmotically treated by ultrasound before 30 min. However, 'water strip' disappeared and MRI images darkened in broccoli osmotically treated by ultrasound after 30 min. MRI results suggested that the ultrasound treatment for long time destroyed cell structure in broccoli causing water and solid loss. The similar results were presented by Cheng et al. (2014), MRI provide visual images on water distribution and status in the cellular and intercellular spaces of untreated and osmotically dehydrated strawberry. Therefore, MRI can reflect water distribution and status related to cell structure change of strawberry during processing. Geng et al. (2015) studied that MRI was used for analyzing interior structure of dried sea cucumber during the rehydration. Results showed that T_1 and T_2 weighted MRI images visualize interior structure changes of lightly dried sea

cucumber during presoaking process for 48 h and rehydration for 144 h. T_1 and T_2 weighted MRI images respectively showed bound water distribution and free water distribution in rehydrated sea cucumber. They found that water distribution was relatively uniform at presoaking time for 24h, and free water population was sufficient at rehydration time for 96 h. Therefore, MRI can provide internal tissue structure information to monitor the quality of food.

Microbiological properties

The product safety is the major factor which determines the product's suitability and market value. The microbiological growth is responsible for many deteriorative processes. Thus, the control of microbiological quality of foods is very critical. Some researchers have used NMR to detect microbiological quality of foods in processing and storage (Duarte et al., 2006; Gudjónsdóttir et al., 2011c). For example, Gudjónsdóttir et al. (2011c) investigated that the correlation of LF-NMR T_2 relaxation times and total viable counts (TVC) of H_2S-producing bacteria was established in brine injected and air-stored cod loins during superchilled storage. They found that the relaxation times (T_1, T_{21} and T_{22}) were significant correlations with TVC in the brine injected cod loins. T_1 was also significant correlations with TVC of H_2S-producing bacteria in the air-stored cod loins. Therefore, fish quality deterioration can be monitored by using NMR technology. On the other hand, water

dynamics can affect microbiological quality of food. Fundo et al. (2016) studied T_2 parameter of fresh-cut pears during 7 days of refrigerated storage by solid state HR-NMR. Results showed that the T_2 maximum values changed to low values at 3 days. This demonstrated that the subcellular structure was disrupted through the wounding and a decrease in water activity led to water being use for microbiological growth. Thus, microbiological quality of food was evaluated basing on food water dynamics studied by NMR technology.

Potential application of online monitoring by NMR

The traditional analytical methods were difficult to be used for online monitoring in food processing because of long time and sample damage. Many non-invasive detection methods such as light transmission, X-ray transmission and ultrasonic have been applied to assess the quality of food. However, these methods were difficult to provide specific information about complex properties of food (Marcone et al., 2013). Recently, NMR was applied to achieve food online processing. For Example, Gudjonsdottir et al. (2011) presented that online control during shrimp processed by prebrining with polyphosphates and freezing was investigated by LF-NMR. They found that LF-NMR can rapidly detect physicochemical properties changes of shrimp including muscle pH, protein content, phosphate levels, moisture content and water-holding capacity. They also found that measurement settings, number of sample replicates, size of analyzing surface and suitable probe

selection need to be further optimized in shrimp processing by using NMR online control. On the other hand, Lv et al. (2017) presented that moisture contents of vegetables were measured online during microwave vacuum drying by LF-NMR method. They found that the relationship between signal amplitude A_{21}, A_{22}, A_{23} and A_2 and moisture content of fresh vegetables (mushroom, carrot, potato, lotus, edamame and corn) could not be fitted by using a single mathematical model. This method can measure moisture content of six kinds of vegetables online. However, the results from NMR could not be predicted all fresh vegetables. They also pointed out that online measuring efficiency should be improved needing further research to industrial application. Therefore, NMR is very potential to achieve online monitoring in food processing.

Conclusion and future research

Basing on the literature, it is clear that NMR technology is an effective and non-invasive tool to detect the authenticity, composition and quality of food such as fruits and vegetables, meat and aquatic products in processing and storage. NMR combined with multivariate analysis can rapidly identify and classify foods. NMR can be also used to analyze water, fat and protein composition in different food. Moreover, NMR can detect physical properties including color, water activity, texture, pH and WHC changes of foods. Meanwhile, NMR can provide quantitative information on chemical composition of foods. MRI can visualize internal tissue structure change of foods. From microbiological

quality analysis, the relationship between NMR parameters and microbiological growth was established to evaluated quality deterioration of food. However, there are still some drawbacks needing further research to successfully apply NMR in the quality detection of the industry. The future research and developments for NMR technology in food analysis area can be as follows: (1) NMR should hyphenated other spectroscopy methods such as NIR spectroscopy, size exclusion chromatographic, ultrasound, mass spectrometry, scattering techniques and rheometry to improve more accuracy, sensitivity and stability in quality detection process. (2) The developments of NMR hardware and user-friendly software are helpful to achieve simple operation of equipment and ensure the reliability of the obtaining data for food researchers. (3) A comprehensive database for NMR should be established for specific food products considering richness and diversity of food types. (4) Novel NMR sequences and automated data processing procedures should be created to achieve high throughput.

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List of Tables

Table 1 Recent studies on discrimination and classification of food by NMR

Sample	NMR type	Analytical method	Research aim	Reference
Sour cherry juice	High-resolution ^{1}H NMR, 600.13 MHz	1D ^{1}H NMR spectra, 2D NMR (^{1}H-^{1}H COSY and ^{1}H-^{13}C HSQC) spectra, sensory evaluation, PCA and PLS	To classify juices from seven sour cherry clones/cultivars	Clausen et al. (2011)
Mango	1D and 2D 1H NMR, 500 MHz	Low-field 1D 1H NMR spectra, 2D NMR (TOCSY, DQF-COSY, NOESY, ^{1}H-^{13}C HSQC and ^{1}H-^{13}C CT-HMBC) spectra, PCA	To discriminate five mango cultivars	Koda et al. (2012)
Orange juice	^{1}H NMR, 400 MHz	^{1}H NMR spectrum, PCA and PLS	To identify five different orange varieties	de Oliveira et al. (2014)
Beef	^{1}H NMR, 600 MHz	^{1}H NMR spectroscopy, 2D NMR (^{1}H-^{1}H TOCSY, ^{1}H-^{13}C HMBC and ^{1}H-^{13}C HSQC) spectra, PCA and OPLS-DA	To differentiate geographical origin of beef	Jung et al. (2010)

	^{1}H NMR, 600 MHz	1H NMR spectra, sLDA and ANN	To classify irradiated or non-irradiated beef	Zanardi et al. (2013)
	Time-domain ^{1}H NMR, 85 MHz	TD-NMR, CPMG and CWFP pulse sequences, SIMCA, KNN and PLS-DA	To discriminate beef basing on bull race and sex	Santos et al. (2014)
Hake	Low-field ^{1}H NMR, 20 MHz	Transverse relaxation time (T_2), PCA	To distinguish 0-3% salt hake	Greiff et al. (2014)
Prawn	Low-field ^{1}H NMR, MRI, 21.16 MHz	Spin-spin relaxation time (T_2), PCA	To detect adulterated prawns	Li et al. (2018)

Table 2 Recent studies on application of NMR in water analysis

Sample	NMR type	Main results	Reference
Strawberry	^{1}H HR-MAS NMR, 500 MHz; MRI, 200 MHz	T_1, T_2 data and MRI maps clearly indicated the changes of water molecules caused by 100-200 MPa pressure in strawberry.	Otero and Préstamo (2009)
Pear	Solid state NMR, 300 MHz	There are 3 kinds of water in the cells after processing of fresh-cut pear. T_2 values are respectively 10 ms, 187 ms and 3 s for cellular wall, cytoplasm and vacuole.	Fundo et al. (2016)
Kiwifruit	TD-NMR, 20 MHz	T_2 signals by TD-NMR can quantify water distribution in osmotic dehydrated kiwifruit treated by pulsed electric fields.	Traffano-Schiffo et al. (2017)
Broccoli	LF-NMR, 23.2 MHz	T_2 with three water fractions and MRI provide the changes of water status and distribution in broccoli during hot-air drying, which showed water mobility and water loss.	Xu et al. (2017)
Pork	LF-NMR, 23.4 MHz	T_2 can identify three water components (7–13 ms for T_{2b},	McDonnell et al. (2013)

		39-54 ms for T_{21} and 133-177 ms for T_{22}), all of which were significantly affected by NaCl concentration.	
Beef	LF-NMR, 23.2 MHz	Water distribution presented three peaks (T_{2b}, T_{21} and T_{22}) during drying of beef by T_2 analysis.	Ojha et al. (2017)
Salmon	LF ^{1}H NMR, 20 MHz	Water distribution of salmon has significant difference by T_2 analysis in ante-mortem handling, fillet location and brine salting.	Aursand et al. (2010)
Cod	LF-NMR, 20 MHz	Two water populations (23.1-94.2 ms for T_{21} and 126-210 ms for T_{22}) were observed according to T_2 data	Gudjónsdóttir et al. (2011a)
	LF-NMR, 20 MHz	A major band (T_{21}, 47-60 ms) of fresh cod muscle was presented basing on T_2 data. T_{21} of freezing cod muscle had no change at -20 °C and decreased at -18 °C during frozen storage.	Sanchez-Alonso et al. (2014)
	LF ^{1}H NMR, 20 MHz	Water distribution was the most homogeneous in cod with injection of salt and phosphates by T_2 analysis.	Gudjonsdottir et al. (2015)
Sea cucumber	LF-NMR, 23.2 MHz	Water distribution basing on T_2 data revealed the water uptake process during rehydration of dried sea cucumber.	Geng et al. (2015)