Effects of milk concentration and freshness on microwave dielectric properties

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ABSTRACT

The knowledge of dielectric properties may hold a potential to develop a new technique for quality evaluation of milk. The dielectric properties of water-diluted cow's milk with milk concentrations from 70% to 100% stored during 36 h storage at 22 °C and 144 h at 5 °C were measured at room temperature for frequencies ranging from 10 to 4500 MHz using open-ended coaxial-line probe technology, along with electrical conductivity and pH value. The raw milk had the lowest dielectric constant when the frequency was higher than about 20 MHz, and had the highest loss factor at each frequency. The highest linear coefficient of determination, 0.995, between the milk concentration and the loss factor at 915 MHz was observed. The change tendency of the loss factor was inversed to pH during milk storage with the best linear correlation ($R^2 = 0.983$) at 1100 MHz. The loss factor can be an indicator in predicting milk concentration and freshness.

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1. Introduction

Milk is one of the most important foods in daily life providing rich nutrients. But milk adulteration is a common phenomenon, especially in certain areas of the world where water, starch solutions, industrial alkalis, and nitrite are common materials added in milk. Milk adulteration leads to economic losses, deterioration of the quality of end products, and a risk to consumers' safety (Mabrook and Petty, 2003b). Therefore, it is important for the milk industry to confirm the quality of raw milk supplied by dairy farmers and for consumer agencies to verify the quality of fresh milk purchased from the market.

Since traditional methods for evaluating milk quality are lengthy, labor-intensive, and expensive (Harding, 1995), several analytical and electrical methodologies have been developed to detect milk adulteration. Sato and Kawano (1990) used visible/near infrared (NIR) spectroscopy to detect foreign fat adulteration of milk. Cozzolino et al. (2001) reported identification of milk adulteration using matrix-assisted laser desorption/ionization time-of-flight mass spectrometry. Recently, electrical conductivity of milk has been studied as a means to detect freshness and adulteration of milk (Mabrook and Petty, 2003b; Winquist et al., 1998). Up to now, there has been little study to develop a new technique for quality control of milk based on dielectric properties measurements.

Open-ended coaxial-line probe method associated with network analyzers or impedance analyzers is an useful technique to determine dielectric properties, i.e. dielectric constant, dielectric loss factor, and loss tangent, of materials, especially for liquid foods, according to the reflection coefficient at the material-probe interface. Nunes et al. (2006) reported dielectric spectroscopy of whole milk at various dilutions at room temperature over the frequency range of 1–20 GHz, while the spectroscopy at low frequencies (<1000 MHz) was not included and they did not study how to predict water content or milk concentration based on permittivity. A novel method to detect the added water to full fat milk has been developed at 100 kHz (Mabrook and Petty, 2002, 2003b), but permittivity at microwave (MW) range was not studied. Therefore, the knowledge on dielectric properties over a wide range of frequency is needed to establish complete relationships of concentrations of milk in water-diluted solution.

On the other hand, milk is a food product that easily deteriorates during storage. The contained microorganisms make the flavor, sensory, and pH of milk change during storage. Therefore, pasteurization is an important process in packaging milk. Because of fouling problems in a plate heat exchanger (Changani et al., 1997), there is an increased interest in developing advanced pasteurization and sterilization processes for milk products using MW and radio frequency (RF) energy. Dielectric properties, or permittivities, are intrinsic properties that determine the interaction of electromagnetic energy with milk when subjected to dielectric heating. Knowledge of dielectric properties is essential

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to determine suitable heating rates and to design optimum treatment load thickness for milk. Dielectric properties of milk as affected by protein, lactose and fat contents have been reported, but only at 2450 MHz (Kudra et al., 1992). Nunes et al. (2006) reported dielectric properties of whole, low fat, and skim milk over a period of two weeks while the samples were allowed to spoil at room temperature. But they didn't monitor the pH value, which is an important milk quality indicator used to evaluate milk freshness. Moreover, the adulteration of natural milk with synthetic milk using alternating current (AC) conductance measurement from 20 Hz to 1 MHz was determined (Sadat et al., 2006). The contributions of lactose, fat, sodium caseinate in cow's milk to milk electrical conductivity were also measured only at lower frequency band (<100 kHz) by Mabrook and Petty (2003a). The temperature profiles of milk after heating in a continuous-flow tubular microwave system based on dielectric properties of milk at three fat levels at 915 MHz were studied (Coronel et al., 2008). However, there is little knowledge of the relationship between MW dielectric properties and pH value of raw milk during storage, which can be used as an indicator of spoilage.

The objectives of this research were (1) to study the influence of added water content or milk concentration and freshness during 22 and 5 °C storage on the dielectric spectroscopy from 10 to 4500 MHz with the open-ended coaxial-line probe technology, (2) to determine useful frequencies where the dielectric properties are sensitive to milk concentration and freshness, (3) to establish the relationship between permittivities and milk concentration and between permittivities and pH during storage, and (4) to estimate the penetration depth for providing general guidance in design of the treatment bed thickness for uniform MW and RF heating.

2. Materials and methods

2.1. Sample

The fresh untreated raw cow milk (raw milk) was obtained from a local stock farm half hour before experiment in the morning, and was filled in two plastic bottles of 2500 ml sterilized with boiling water. The temperature outside was about 7 °C when the samples were transported to the laboratory. The milk had 2.90% fat, 2.37% protein, and 8.42% non-fat solids in mass.

2.2. Dielectric properties measurement

The dielectric properties were measured with an Agilent Technologies E5071C vector network analyzer and Agilent Technologies 85070B open-ended coaxial-line probe (Agilent Technologies, Penang, Malaysia). Dielectric constant and loss factor were calculated with Agilent Technologies 85070D dielectric probe kit software according to the reflection coefficient of the material in contact with the active tip of the probe. Settings were made to provide 101 measurements on a logarithmic scale from 10 to 4500 MHz, which was the upper frequency limit of the measurement system. The port of the network analyzer used in the experiment was calibrated with open, short, and matched 50 Ω load in sequence followed by calibrating the coaxial probe with open (air), short-circuit and deionized water at 25 °C. A personal computer was used to control the system. During the experiment, the rigid cable which connected the analyzer and the probe was fixed to avoid the effect of cable position and shape changes on the measurement accuracy.

2.3. Procedure

Predetermined amounts of deionized water were added to 70 ml of raw milk to prepare milk solutions with different milk

concentrations of 70%, 75%, 80%, 85%, 90%, and 95% as a ratio of the mass of milk to the mass of milk solution at room temperature. The masses were measured by an electronic balance (FA2104A, Shanghai Precise Scientific Instrument Co., Shanghai, China) with a precision of 0.0001 g. The raw milk obtained from the farm, was stored in two covered bottles in an incubator at 5 °C and at room temperature, 22 °C, respectively. The milk samples were used for measurements every 12 h for storage at 5 °C and every 2 h for storage at 22 °C until the sample was deteriorated to study the influence of milk spoilage on dielectric properties. Before each measurement, the milk in bottles was shaken evenly. For the milk stored at 5 °C, it was warmed to room temperature by circulating warm water at 25 °C for 5 min around the beakers, to reach equilibrium before permittivity measurement. The temperature of samples was determined with a high accuracy mercury laboratory thermometer. Care was taken in sample temperatures as the dielectric properties of food materials were found to be dependent on temperature (Guo et al., 2008; Nelson, 2003; Wang et al., 2005, 2008). Each beaker filled with milk solution in 15 ml was placed on a platform of 50 mm in diameter, and was raised up until the downward open-ended coaxial-line probe was completely immersed in the sample. Care was also taken to eliminate bubbles between the probe and the sample during measurements, since air bubbles might interfere with proper permittivity determinations. Dielectric properties measurements were repeated three times for each sample. Between replications, the probe was washed with water and wiped dry. Then electrical conductivity was determined using a conductivity meter (DDSJ308A, Shanghai Leici Instrument Co., Shanghai, China), and pH value was measured by a pH meter (pHS-2C, Shanghai Precise Scientific Instrument Co. Ltd., Shanghai, China).

Nine readings in three replications were recorded for permittivities, conductivity and pH measurements. The average of the three replicates was used in the analysis. In order to detect milk deterioration by high temperature flocculation, the milk from the three replicates was collected in a steel cup that was heated to about 107 °C, which is the boiling point, at normal pressure to see whether floccules appeared. The milk was regarded as deteriorated if floccules appeared in heated milk.

2.4. Dielectric properties, electrical conductivity and penetration depth

At RF and MW heating, loss mechanisms of foods and agricultural products with a high moisture content are dominated by ionic conduction and dipole polarization (Ryynänen, 1995). This can be mathematically expressed as

$$\varepsilon'' = \varepsilon_d'' + \varepsilon_\sigma'' \tag{1}$$

where ε_d'' and ε_σ'' are dielectric loss due to dipole rotation and ionic conduction, respectively. ε_σ'' is further expressed as:

$$\varepsilon_\sigma'' = \frac{\sigma}{2\pi f \varepsilon_0} \tag{2}$$

By taking the logarithm on both sides of Eq. (2), it becomes:

$$\log \varepsilon_\sigma'' = \log \frac{\sigma}{2\pi \varepsilon_0} - \log f \tag{3}$$

where σ is ionic conductivity of a material in S/m; f is frequency in Hz; and ε_0 is the permittivity of free space (8.854×10^{-12} F/m). Eq. (3) expresses a negative linear relationship between the dielectric loss factor contributed by ionic conductance and the frequency in a log–log plot. To evaluate the influence of ionic conductivity on the loss factor, the electrical conductivity of fresh raw milk was used to calculate ε_σ'' according to Eq. (2).

Penetration depth of RF and MW power is defined as the depth where the power is reduced to $1/e$ ($e = 2.718$) of the power entering

the surface. The penetration depth d_p in m of RF and MW energy in milk was calculated according to von Hippel (1954):

$$d_p = \frac{c}{2\pi f \sqrt{2\varepsilon' \left[\sqrt{1 + \left(\frac{\varepsilon''}{\varepsilon'}\right)^2} - 1 \right]}} \qquad (4)$$

where c is the speed of light in free space (3×10^8 m/s). After obtaining the dielectric properties at 22 °C, the penetration was calculated as a function of frequency for milk concentrations of 70% and 100%, and in raw milk at storage periods of 0, 18, and 36 h at 22 °C.

3. Results and discussion

3.1. The influence of milk concentrations on permittivities

The frequency dependent permittivities of fresh raw milk (milk concentration of 100%) and diluted milk with the milk concentration of 70% from 10 to 4500 MHz at 22 °C are shown in Fig. 1. The dielectric constant of the raw milk and diluted milk decreased with increasing frequency, especially at lower frequencies. The dielectric constants of raw milk decreased faster than that of the diluted one. When the frequencies were higher than about 20 MHz, the raw milk had lower dielectric constant than the diluted milk (Fig. 1a). For example, it was 97.7, 68.1, and 65.9 for raw milk, while 93.3, 70.9 and 69.1 at 10, 915, and 2450 MHz, respectively, for 70% milk solution.
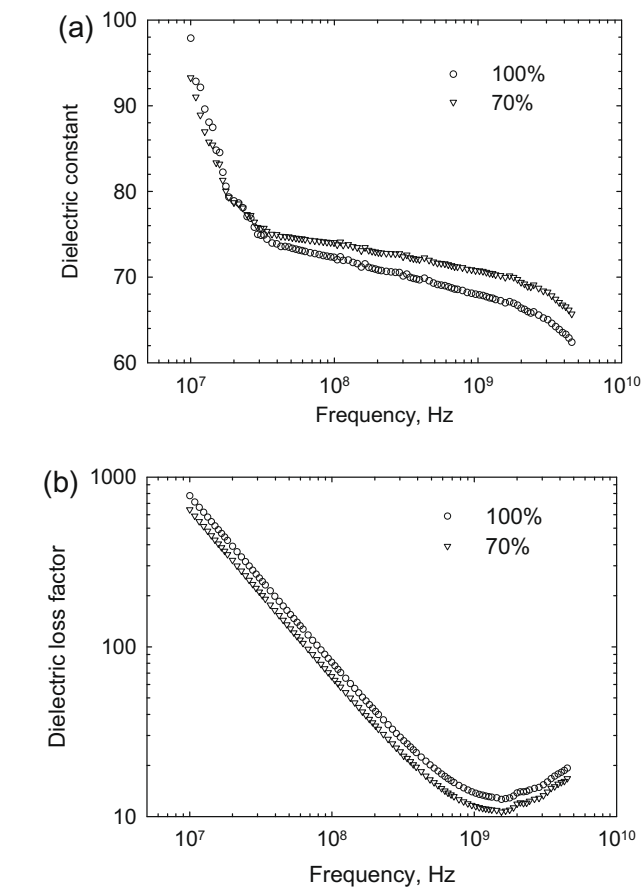

Fig. 1. The frequency dependent permittivities of fresh raw milk (100%) and diluted milk with milk concentration of 70% over the frequency from 10 to 4500 MHz at 22 °C.

The dielectric loss factor against frequency from 10 to 4500 MHz is shown in a log–log plot (Fig. 1b). The loss factor looked similar for raw milk and diluted milk, and reached a minimum at about 1700 MHz. Moreover, there was a good linear relationship between $\log f$ and $\log \varepsilon''$ at the lower frequency range (e.g. <600 MHz), which further confirmed the negative relationship indicated in Eq. (3). Because the added water diluted the ionic concentration in milk, the loss factor decreased with increasing water content, that is, with decreasing milk concentration. The dielectric constants and loss factors of milk solutions with different milk concentrations at 22 °C at several selected frequencies are listed in Table 1. Among them, 27, 915 and 2450 MHz are allocated by the US Federal Communications Commission (FCC) for MW and RF heating applications.

3.2. Correlation between electrical conductivity and loss factor

Fig. 2 shows the comparison of measured ε'' and ε''_σ of raw milk calculated from measured electrical conductivity from 10 to 4500 MHz at 22 °C. The close data of measured ε'' and the estimated ε''_σ were observed when frequency was lower than about 300 MHz together with the negative linear relationship between ε'' and frequency in log–log plot, suggesting that ionic conduction dominated the loss mechanism at the RF range (Wang et al., 2005, 2008). The difference between ε'' and ε''_σ was ε''_d, which became greater with increasing frequencies. That is the dipole polarization increased with the increasing frequency and contributed greater than ionic conduction at the MW range. This phenomenon has also been observed in fruits (Guo et al., 2007a; Nelson et al., 1994) and eggs (Guo et al., 2007b), and been demonstrated by Wang et al. (2008) in salmon fillets, and by Wang et al. (2005) in fruits.

3.3. The correlation between permittivities and milk concentration

Fig. 3 shows the good linear regression between the measured electrical conductivity and milk concentration with coefficient of determination of 0.972. Therefore, the electrical conductivity could be used in predicting milk concentration.

Linear regressions between permittivities and milk concentration (Mc, in %) were carried out at 101 measured frequencies from 10 to 4500 MHz. The coefficient of determination, R^2, between the dielectric constant ε' and Mc at all detected frequencies was lower than 0.75 (not shown), but reached the highest ($R^2 = 0.995$) for the

Table 1
Dielectric constant (ε') and loss factor (ε'') of milk solutions with different milk concentrations at 22 °C at four selected frequencies.

| Milk concentration	Permittivity	Frequency (MHz)			
		27	915	2450	4500
70%	ε'	76.4 ± 0.1	70.9 ± 0.0	69.1 ± 0.0	65.7 ± 0.1
	ε''	233.8 ± 1.2	11.9 ± 0.0	12.4 ± 0.1	16.8 ± 0.2
75%	ε'	76.3 ± 0.1	70.4 ± 0.1	68.5 ± 0.1	65.1 ± 0.1
	ε''	245.3 ± 3.2	12.4 ± 0.0	12.6 ± 0.1	17.0 ± 0.2
80%	ε'	76.4 ± 0.1	69.9 ± 0.1	68.0 ± 0.1	64.7 ± 0.1
	ε''	255.7 ± 1.9	12.8 ± 0.0	12.9 ± 0.1	17.3 ± 0.1
85%	ε'	76.3 ± 0.1	69.5 ± 0.1	67.5 ± 0.1	64.3 ± 0.1
	ε''	266.4 ± 1.4	13.3 ± 0.0	13.2 ± 0.1	17.6 ± 0.2
90%	ε'	76.3 ± 0.7	69.0 ± 0.7	66.9 ± 0.7	63.8 ± 0.7
	ε''	277.1 ± 2.9	13.7 ± 0.0	13.4 ± 0.1	18.1 ± 0.2
95%	ε'	75.1 ± 0.3	67.7 ± 0.3	65.6 ± 0.2	62.2 ± 0.3
	ε''	291.1 ± 2.1	14.2 ± 0.0	13.5 ± 0.1	17.8 ± 0.1
100%	ε'	75.8 ± 0.3	68.1 ± 0.0	65.9 ± 0.0	62.4 ± 0.0
	ε''	282.1 ± 3.1	14.3 ± 0.2	14.3 ± 0.1	19.3 ± 0.3
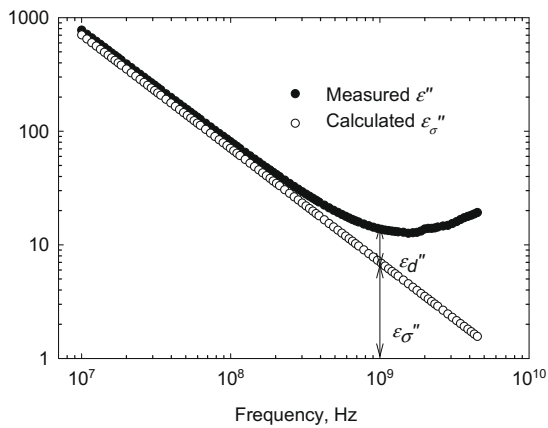
Fig. 2. Measured ε'' and ε_σ'' of fresh raw milk calculated from measured electrical conductivity over the frequency range from 10 to 4500 MHz at 22 °C. The difference between ε'' and ε_σ'' represents the contribution from the dipole rotation of polar molecules (ε_d'').
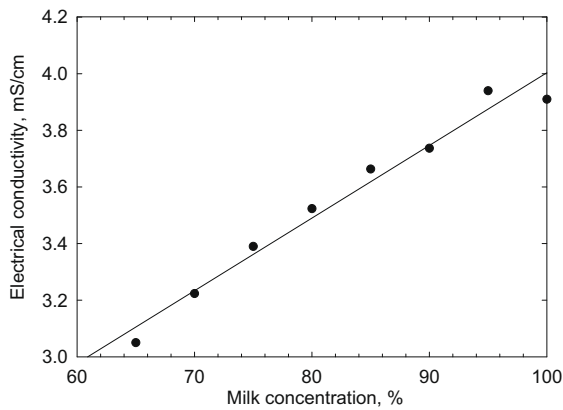

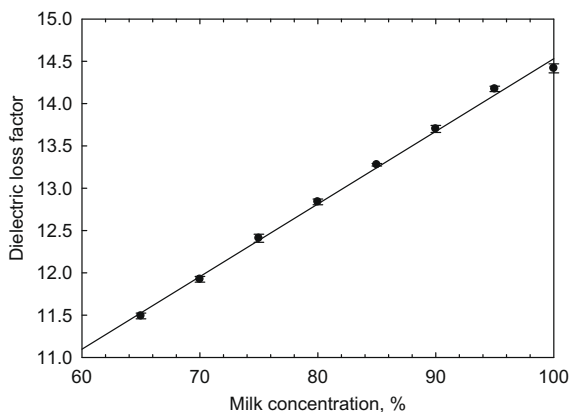**Fig. 3.** The linear regression between electrical conductivity and milk concentration with R^2 = 0.972.

Fig. 4. The linear regression between dielectric loss factor and milk concentration at 915 MHz with R^2 = 0.995.

loss factor at 915 MHz as shown in Fig. 4. The linear regression equation was:

$$\varepsilon'' = 0.084Mc + 6.083, \quad R^2 = 0.995 \tag{5}$$

then,

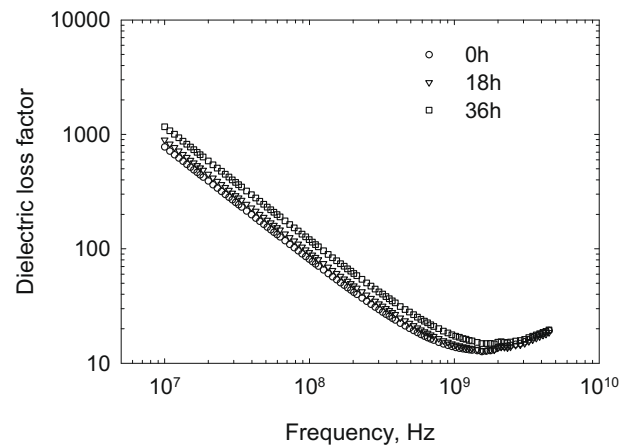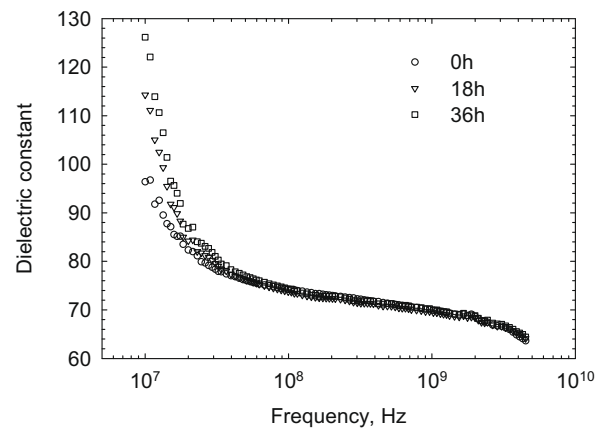$$Mc = 11.905\varepsilon'' - 72.420 \tag{6}$$

Fig. 5. Dielectric properties of raw milk from 10 to 4500 MHz at the indicated storage time at 22 °C.

If the loss factor of milk solutions at 915 MHz is known, milk concentration can be predicted from Eq. (6).

3.4. The influence of freshness on permittivity

Fig. 5 shows the dielectric properties of raw milk at frequencies ranging from 10 to 4500 MHz at storage times of 0, 18 and 36 h at 22 °C. The fresh milk had the lowest dielectric constant at lower frequencies and the lowest loss factor when frequencies were lower than about 1700 MHz. The dielectric properties of raw milk at 22 °C during the cold storages at 5 °C for 0, 72 and 144 h are shown in Fig. 6. The milk kept for 72 h had higher dielectric constant at all frequencies and lower loss factor than fresh milk, while the milk kept for 144 h had lower dielectric constant and higher loss factor than fresh milk, when frequency was lower than about 1700 MHz.

The loss factors at 915 MHz and pH values of raw milk as a function of the storage time at 22 and 5 °C, respectively, are presented in Fig. 7. Leaving milk at room temperature showed a small decrease in pH and small increase in loss factor for the first 12 h, followed by quick decrease in pH and increase in loss factor from 12 to 32 h (Fig. 7a). The pH and loss factor kept constant in last 4 h. The acidification tendency during storage matched well with other research (Gu and Hua, 2005). When stored at 5 °C, the acidification process and increase in loss factor were less important (Fig. 7b).

Milk is a complex mixture of water, lactose, fat, protein (mostly casein), minerals and vitamins distributed throughout colloidal and soluble phase (Mabrook and Petty, 2003a). The microorganisms present in the milk make the milk quality deteriorate over time, slowly at first and more rapidly subsequently. With the
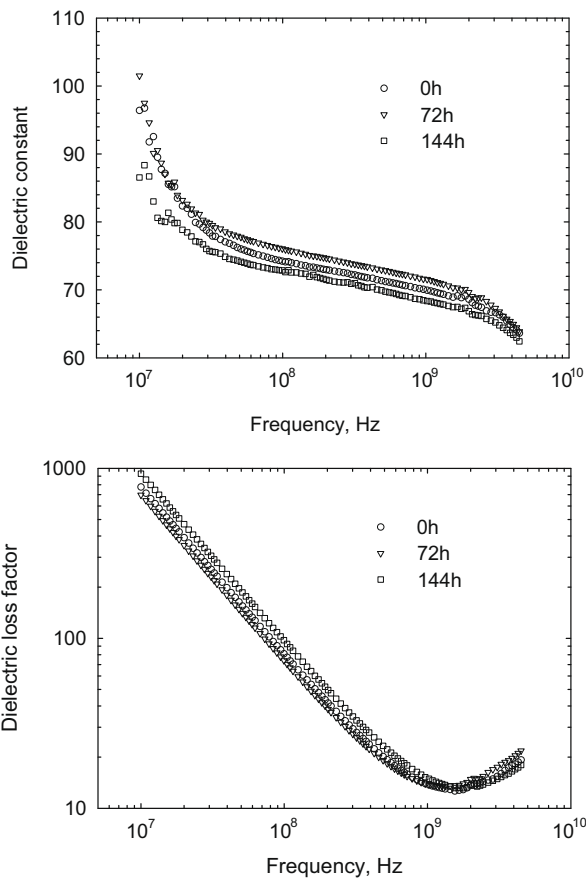
Fig. 6. Dielectric properties of raw milk from 10 to 4500 MHz at 22 °C at indicated times during 144 h storage at 5 °C.

Fig. 7. The change of loss factor (ε'') at 915 MHz and pH value of raw milk during storage at 22 (a) and 5 °C (b).

growth of microorganisms, the components in milk, such as lactose, protein and fat, are decomposed into various acids, especially lactic acid, and other inorganic materials with small molecules (Weng et al., 2006). This leads to a significant decrease in pH. During the breakdown of the milk compounds, the molecules become smaller, and the ionic concentration increases, which makes the loss factor increase over the storage time. It was also observed that floccules appeared in heated milk when milk pH was lower than 6.5.

The linear coefficient of determination between pH and loss factor for both storage conditions against frequency is shown in Fig. 8. The R^2 was higher than 0.89 from 10 to 1300 MHz. The highest, 0.983, appeared at 1100 MHz (Fig. 9). Their linear relationship can be described as

$$\varepsilon'' = a \cdot \text{pH} + b \tag{7}$$

where a is the slope of the regression line, and b is the intercept at $\varepsilon'' = 0$ in Fig. 9. If the loss factor is known, the pH can be calculated as:

$$\text{pH} = \frac{\varepsilon'' - b}{a} \tag{8}$$

Typical values of a, b, and R^2 at several selected frequencies are listed in Table 2. As it was described above, when pH was lower than 6.5, floccules began appearing in heated milk. So, the loss factor where the pH is equal to 6.5 can be used as a critical value in distinguishing milk freshness. For example, the milk could be regarded as deteriorated if the loss factor at 1100 MHz was higher than 13.8.
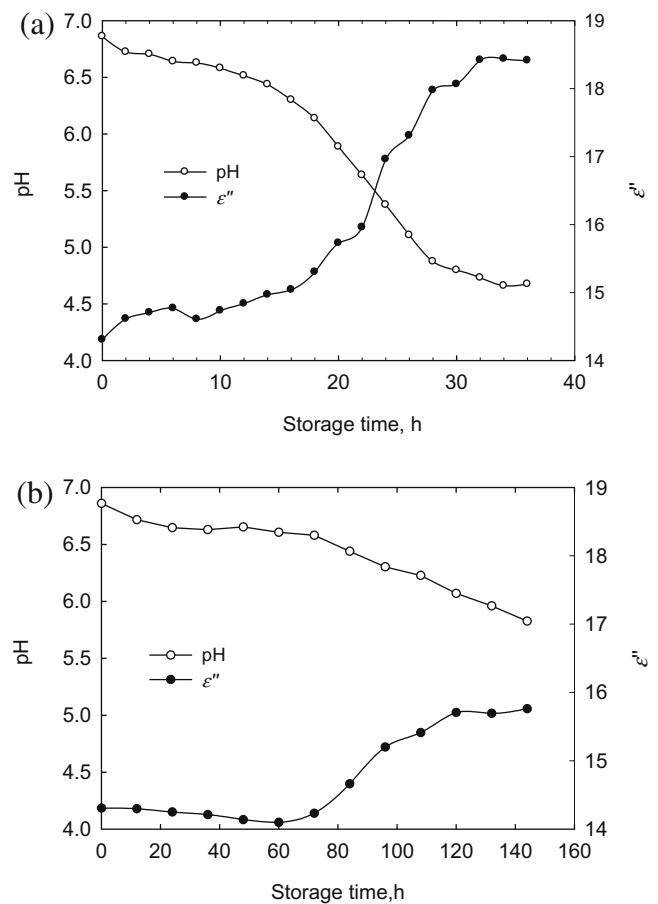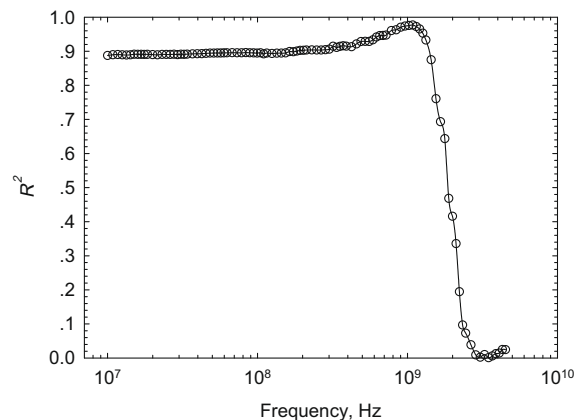

Fig. 8. Coefficients of determination (R^2) between the loss factor and pH values of raw milk during storage at 22 and 5 °C over the frequency range from 10 to 4500 MHz.

3.5. Penetration depth

The penetration depths in milk solution with 100% and 70% milk concentration and in raw milk at different storage periods of 0, 18 and 36 h over the frequency range from 10 to 4500 MHz at 22 °C are shown in Figs. 10 and 11, respectively. The penetration decreased with increasing frequency, and was higher in diluted milk than in raw milk (Fig. 10). It was the highest in fresh raw milk, and
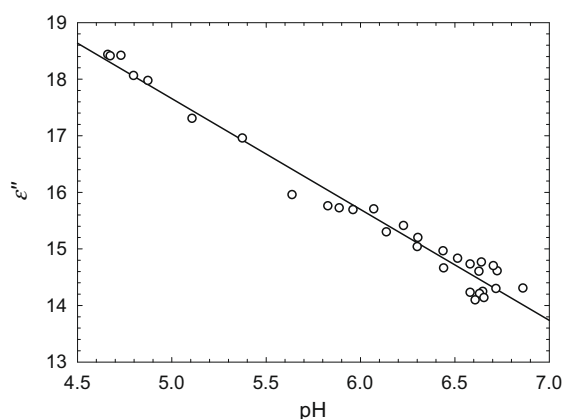
Fig. 9. Relationship between loss factor (ε'') at 1100 MHz and pH value at 22 °C for the stored raw milk at 22 and 5 °C with $R^2 = 0.983$.

Table 2
The coefficients in Eqs. (7) and (8) at four selected frequencies.

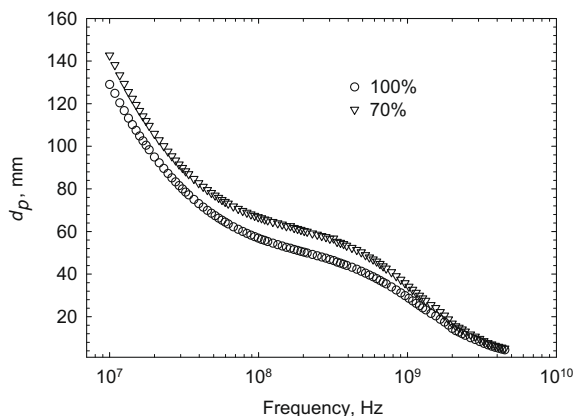| Coefficients	Frequency, MHz			
	27	41	915	1100
a	−69.761	−45.567	−1.965	−1.581
b	748.410	489.870	27.476	24.123
R^2	0.890	0.892	0.971	0.983

Fig. 10. Penetration depth (d_p) of electromagnetic energy in milk solutions of 70% and 100% milk concentrations at 22 °C.
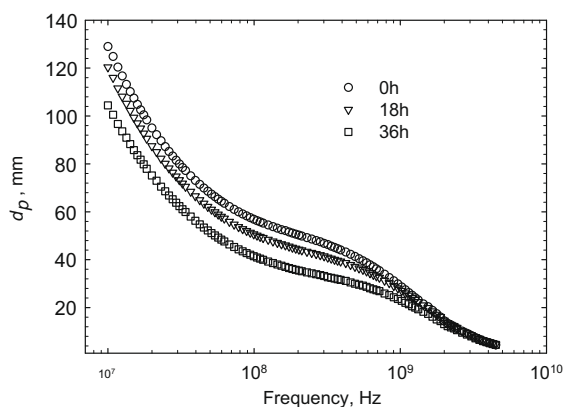

Fig. 11. Penetration depth (d_p) of electromagnetic energy in raw milk at indicated storage periods at 22 °C.

the lowest for 36 h storage at same frequency (Fig. 11). The penetration depth (80 mm) at 27 MHz in raw milk might develop practical RF pasteurization processes for large packages, resulting in continuous and uniform RF heating in large-scale samples. The penetration depth should be deep enough to provide sufficient information on sensing milk quality according to permittivity measurements.

4. Conclusions

The dielectric constant of milk decreased with the increasing frequency, and the loss factor had a minimum at about 1700 MHz over the frequency range from 10 to 4500 MHz at 22 °C. The raw milk had lowest dielectric constant when the frequency was higher than about 20 MHz, and had the highest loss factor over the detected frequency range when compared with diluted milk. There was a high linear coefficient of determination between the milk concentration and loss factor with the highest $R^2 = 0.995$ at 915 MHz. The dielectric loss factor of raw milk was linearly inversed to pH value during storage at 22 and 5 °C, while the coefficient of determination ($R^2 = 0.983$) reached the highest at 1100 MHz. The loss factor can be a good indicator in predicting milk concentration and freshness. The penetration depth increased with decreasing frequency, water content and storage time, which was large enough to detect dielectric properties changes in milk samples and provide large-scale RF pasteurization processes. The study provides some useful information for developing a fast and simple milk concentration and freshness sensor in food processing industry.

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