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Original Article

Determination of the concentration of alum additive in deep-fried dough sticks using dielectric spectroscopy

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ABSTRACT

The concentration of alum additive in deep-fried dough sticks (DFDSs) was investigated using a coaxial probe method based on dielectric properties in the 0.3–10-GHz frequency range. The dielectric spectra of aqueous solutions with different concentrations of alum, sodium bicarbonate, and mixtures thereof were used. The correspondence between dielectric loss and alum concentration was thereby revealed. A steady, uniform correspondence was successfully established by introducing $\omega \cdot \epsilon''(\omega)$, the sum of dielectric loss and conductor loss (i.e., total loss), according to the electrical conductivity of the alum-containing aqueous solutions. Specific, resonant-type dielectric dispersion arising from alum due to atomic polarization was identified around 1 GHz. This was used to discriminate the alum additive in the DFDS from other ingredients. A quantitative relationship between alum and sodium bicarbonate concentrations in the aqueous solutions and the differential dielectric loss $\Delta\epsilon''(\omega)$ at 0.425 GHz was also established with a regression coefficient over 0.99. With the intention of eliminating the effects of the chemical reactions with sodium bicarbonate and the physical processes involved in leavening and frying during preparation, the developed technique was successfully applied to detect the alum dosage in a commercial DFDS (0.9942 g/L). The detected value agreed well with that determined using graphite furnace atomic absorption spectrometry (0.9722 g/L). The relative error was 2.2%. The results show that the proposed dielectric differential dispersion and loss technique is a suitable and effective method for determining the alum content in DFDSs.

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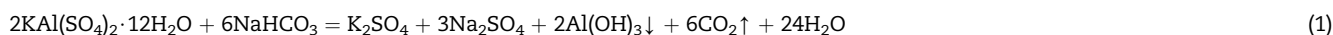
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1. Introduction

The deep-fried dough stick (DFDS), or Youtiao, is a traditional and inexpensive Chinese breakfast food, which is popular in China. The major ingredients of DFDSs are dough, baking soda (sodium bicarbonate), and alum (hydrated potassium aluminum sulfate). The preparation process of DFDSs involves fermentation and frying at high temperature. When alum is not used, the DFDS is small and hard. However, when using alum the DFDS is big, golden in color, crispy outside, and has better sensory characteristics. During the cooking process, alum and sodium bicarbonate undergo the following chemical reaction:



In this reaction, carbon dioxide gas is produced, while the $\text{Al}(\text{OH})_3$ is formed as a floc. The aluminum hydroxide floc mixes with the flour to form a membranoid substance, which wraps around the CO_2 . During the frying process, the carbon dioxide expands, making the DFDS swell. It is clear that there are chemical reactions involved. However, DFDS production also involves the following physical processes: floc aluminum hydroxide + flour \rightarrow membrane covering carbon dioxide gas \rightarrow expansion of the CO_2 when fried \rightarrow DFDS swelling.

Alum is approved as a food additive by the U.S. Food and Drug Administration. However, it is toxic to humans in large quantities. Aluminum overload (alum, of course, includes aluminum in its chemical composition) in humans can cause diseases such as kidney dysfunction, softening of the bones, neurological disorders [1–4]. The commonly used methods for detecting aluminum in samples are atomic absorption spectrophotometry [5,6] and inductively coupled plasma-related detection technologies [7–9]. Although these technologies have high sensitivity and good accuracy, the equipment required is expensive and complicated to operate. Therefore, a fast, simple, and sensitive method for the detection and quantification of aluminum was needed. Some technologies have been developed based on the physical properties of foodstuff, including acoustic, optical, magnetic, mechanical, thermal, and fluid properties. These techniques have been explored to detect food quality, but each technique has its application field [10]. The dielectric properties of food materials describe how these materials interact with the electromagnetic field [11]. The interest in the dielectric properties of agricultural materials and food products has primarily centered on predicting heating rates that describe the behavior of the food when subjected to the high-frequency fields used in dielectric heating applications (so-called *novel thermal treatments*) [12]. This technology is not limited by the food variety. It is also simple, rapid, nondestructive, and sensitive [10]. Based on dielectric difference spectroscopy, dielectric properties have been used to control and evaluate

food quality [13–20] and to detect food adulterated with other compounds [10,21,22].

The physical property to explain the effect of the electric field through a dielectric material is given by the Maxwell relations (Gauss law). Electric field is indicated as an epsilon (according to the notation suggested by the International Union of Pure and Applied Chemistry) and is defined as *permittivity*. Permittivity describes the displacement of the electric field, and therefore it is vectorially decomposed using a complex expression, where the real part describes the electric storage and is called *dielectric constant* (ϵ') and the imaginary part describes the *transformations* in other energies and is called *loss factor* (ϵ'') [23,24]. A relaxation spectrum, based on $\epsilon'(\omega)$, usually occurs at frequencies smaller than

10 GHz (the angular frequency is given by $\omega = 2\pi f$, where f is the frequency) in which the absorption $\epsilon''(\omega)$ is very small. The behavior here is that the spectrum increases with increasing ω . The behavior is mainly caused by the orientation and spatial polarization of the dielectric material. As frequency increases, a resonance spectrum is observed [$\epsilon'(\omega)$ increases, then decreases, and then increases again as ω increases]. This is accompanied by the appearance of an absorption peak in $\epsilon''(\omega)$ [at the same frequency as the *drop zone* in the $\epsilon'(\omega)$ spectrum] [24]. To investigate the dielectric properties of alum, and to develop a new detection method for alum additives in DFDS, both the dielectric dispersion spectrum $\epsilon'(\omega)$ and the absorption spectrum $\epsilon''(\omega)$ of alum were studied in this work.

Even with a constant amount of alum and sodium bicarbonate, the DFDSs produced are different if different proofing and frying methods were applied in the production process. If these DFDSs were subjected to dielectric spectroscopy directly, the results would be different and would mainly reflect the different production processes used. Therefore, to avoid such influences on the dielectric spectrum, it is necessary to first extract the alum and sodium bicarbonate from the DFDS. It is well-known that alum and sodium bicarbonate are readily soluble in water. However, they are difficult to dissolve after the frying process. In turn, extraction and preparation of an aqueous solution of alum and sodium bicarbonate from DFDSs is an effective method for determining the dielectric spectrum of alum and sodium bicarbonate additives. However, Ryyänen [25] stated that the most important factor in determining the dielectric property is still water. Thus, it is difficult to observe the essential characteristics of food using the dielectric properties of electrolytic solutions. Therefore, to obtain the characteristics of the dielectric spectra of alum and sodium bicarbonate and to develop methods for detecting the nonaqueous food characteristics of components using dielectric spectroscopy, this study made use of standard samples of alum and sodium bicarbonate in addition to the DFDS solutions.

2. Materials and methods

2.1. Materials and equipment

2.1.1. Materials

The refined starch (food grade) was purchased from Shanghai Kite Flour Co., Ltd. (Shanghai, China). Soybean oil (food grade) was purchased from Shanghai Kerry Food Industries Co., Ltd. (Shanghai, China). Alum [hydrated potassium aluminum sulfate or potassium alum, $KAl(SO_4)_2 \cdot 12H_2O$; analytical grade] was purchased from Shanghai Shenbo Chemical Co., Ltd. (Shanghai, China). Sodium bicarbonate (analytical grade) was purchased from Shanghai Avison Chemical Factory (Shanghai, China). Commercial DFDSs were purchased from the market in Lingang New City, Shanghai, China.

2.1.2. Equipment

Frying was carried out using a commercial frying pan (DeLonghi F885V; Yi Long Trading Co., Ltd., Shanghai, China). Dielectric spectroscopy was measured using a microwave network analyzer (N5230C PNA-L; Agilent Technologies Co., Ltd., Palo Alto, CA, USA). Aluminum concentration was determined using an atomic absorption spectrometer (ZEEnit 700; Analytik Jena Ltd., Jena, Thuringia, Germany). To implement real-time monitoring of temperature, temperature measurement and display were achieved using a thermocouple (DR030B Digital Recorder; Shanghai Da Hua instrument factory, Shanghai, China) and TDA series temperature display regulator (Ningbo Automation Instrument Factory, Ningbo, Zhejiang, China), respectively. Weighing and blending were carried out using an analytical balance (Sartorius BS224 S; Beijing Sartorius Instrument System Co. Ltd., Beijing, China) and magnetic stirrer hot plate (Guohua Magnetism Msier is 78-2; Changzhou Guohua Electric Appliance Co., Ltd., Changzhou, Guangdong, China), respectively.

2.2. Methods

2.2.1. DFDS manufacturing process

The dough was produced according to the general DFDS production method [26]. Deionized water was used together with flour, alum, and sodium bicarbonate in the ratio of 50:100:2:2 (by weight). A blank dough was also produced with no alum and sodium bicarbonate added. After the dough was made, it was wrapped in a plastic wrapper and left to stand for 3 hours at room temperature to ferment. After fermentation, the dough was cut into small equal quantities and then pulled to form a strip. These were put into the preheated frying pan and fried at 180°C for 1.5 minutes and then removed and allowed to cool at room temperature.

2.2.2. Dielectric spectroscopy

Standard solutions of alum, soda, and mixtures of the two were prepared with concentrations from 0.1 to 10 mg/mL in 150-mL beakers at 25°C using deionized water. Dielectric spectroscopy was performed using a coaxial probe (85070E-030; Agilent Technologies Co., Ltd.) and the network analyzer (PNA-L). The range of frequencies used in the test was from 0.3 to 10 GHz, and the number of measurement points was 1941.

The system was corrected for air, short circuit, and deionized water at 25°C. After calibration, the dielectric spectrum of 100 mL of deionized water was measured in a 150-mL beaker (the temperature of water was maintained at 25°C using a water bath and monitored using a thermocouple). The probe was placed into the deionized water at 25°C (the probe did not have any contact with the container) to test the dielectric properties and the test data were recorded. The dielectric spectra of the aforementioned standard solutions were also measured. The DFDSs were cut into 1-g samples and placed into 150-mL conical flasks. Deionized water (100 mL) was added and the solution was stirred for 24 hours at room temperature and covered with a foil seal to prevent evaporation of water. After filtering the mixtures, the solutions were added into a water bath at 25°C and the temperature was monitored using a thermocouple. Finally, the dielectric spectra of the solutions were measured.

2.2.3. Graphite furnace atomic absorption spectrometry

DFDS was chopped and placed in a nitrification tube. A pipette was then used to transfer 10 mL of nitrate (analytical grade) to the nitrification tube. Nitrification was carried out at 240°C in a high-temperature heating pipe. The smoke was observed until it no longer appeared brown and about 1 mL of liquid remained in the tube. Heating was then stopped.

The first configuration used included a 10 µg/L aluminum top standard solution. The instrument automatically diluted the sample to concentrations of 2, 4, 6, 8, and 10 µg/L. Having set the experimental operating conditions (the ashing temperature was 1200°C, atomization temperature was 2450°C, and the atomic preservation time was 3 seconds), the absorbance of the five standard solutions was determined. The mapping between the absorbance value and the concentration was determined, and a calibration curve was drawn. The standard calibration equation was determined by fitting the calibration curve. Then, the nitrification liquid obtained from the DFDS was analyzed according to the defined operating conditions to determine the corresponding absorbance, and the aluminum content was calculated numerically from the standard calibration equation. Preparation and measurement using standard sample solutions were carried out two times; preparation and measurement using DFDS samples were repeated 10 times. The results obtained were reproducible.

3. Results and discussion

3.1. Characteristics of the dielectric spectrum of alum

Compared with the behavior observed for deionized water (Fig. 1), it can be seen that the dispersion in the alum solution basically reflects the characteristics of the water. It is difficult to directly obtain information on the alum using the dielectric measurements. The dielectric absorption of the alum solution (ϵ'') shows significant absorption in the low-frequency range. In the higher-frequency range, ϵ'' is only slightly larger than that obtained with deionized water, as shown Fig. 1. At low frequencies in the range of 300 MHz to approximately 1–2 GHz, the loss factor is influenced by the ionic conduction mechanism. For frequencies between 1 and 2 GHz, the

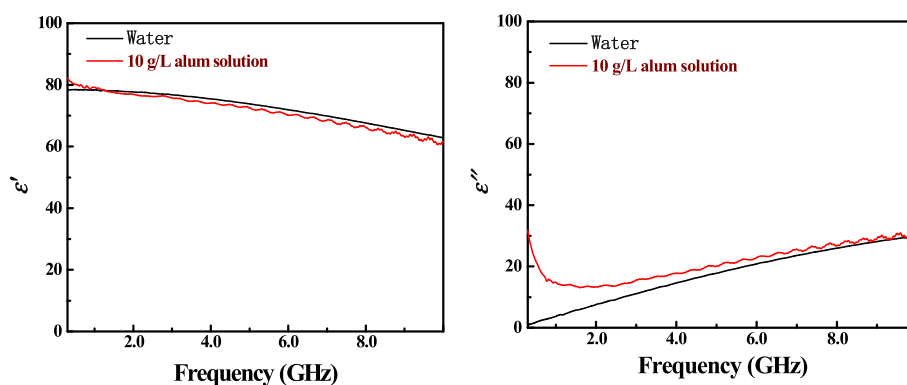


Fig. 1 – Dielectric spectra of aqueous solutions consisting of 10.0 g/L alum and deionized water.

dielectric mechanism shifts from ionic conduction to dipole polarization and for frequencies above 2 GHz, dipolar relaxation mechanism dominates the dielectric loss behavior [24,27]. However, with the aim of finding the rule of dielectric properties of alum solution and determining the alum concentration from the DFDS, the dielectric properties were also tested and analyzed at above 2 GHz.

However, the aforementioned results show that there is little difference between the dielectric dispersion characteristics of an aqueous solution of alum and deionized water. Therefore, the difference curves for dielectric dispersion ($\epsilon'_{\text{Alum solution}} - \epsilon'_{\text{water}}$) and dielectric absorption ($\epsilon''_{\text{Alum solution}} - \epsilon''_{\text{water}}$) were investigated. In the low-frequency region, the differential dielectric dispersion curve gradually decreases with increasing frequency. It presents a type of relaxation dispersion, which might be associated with molecular orientation polarization of the alum. Nevertheless, multiple dispersion fluctuations can be seen at frequencies near 1 GHz. In the dielectric absorption difference curve, absorption peaks appear coincident with the decreases in the dispersion. Specific details in a resonance spectrum usually originate from atomic and electronic polarization. The latter usually occur in the higher-frequency area. Thus, the features observed in the resonance spectrum arise from atomic polarization of the alum, and therefore, may be due to polarization of the Al, K, and S atoms in the alum. By contrast, in the higher-frequency area, the absorption peaks fail to show any correspondence with the dispersion, and therefore, fail to identify the source. Even so, the specific resonance spectrum is enough to reflect the presence of alum, and so can be used to identify the composition of the nonaqueous component.

To explore the influence of the concentration of alum on the dielectric properties of the solution, the different dielectric properties were investigated over a large concentration range. With a decrease in alum concentration, the dielectric dispersion difference curves become smoother, and the amplitude of the dielectric absorption difference curves is reduced at the same time, as shown in Fig. 2. The relaxation spectrum of alum (caused by orientation polarization) also decreased with a decrease in the alum concentration. This supports the rationality and validity of introducing the dielectric difference spectrum. Meanwhile, there was no significant frequency

shift or increase in specific resonant-type dielectric dispersion caused by atomic polarization near 1 GHz. The atomic polarization was not changed due to the different concentration of visible molecules. This further confirms that the characteristic frequencies are related to the alum.

It is worth noting that the dielectric absorption difference curve decreases monotonically with decreasing alum concentration in the low-frequency area. In other words, the intensity of the dielectric absorption difference curve is increased with increasing the concentration of the alum (at the same frequency). To some extent, this change in relaxation absorption reflects the concentration of alum. However, the absorption difference caused by the change in concentration is very different at different frequencies. Therefore, further research is required if we are to measure the alum concentration in the solution using this method.

For solutions of dielectric materials, any carrier migration that occurs in the solution also leads to leakage loss in addition to dielectric absorption loss. The sum of dielectric loss and leakage loss (i.e., the total loss) is given by $\omega \cdot \epsilon''$ (also referred to as the dielectric conductivity). It is not hard to deduce that the total loss should be expected to increase with

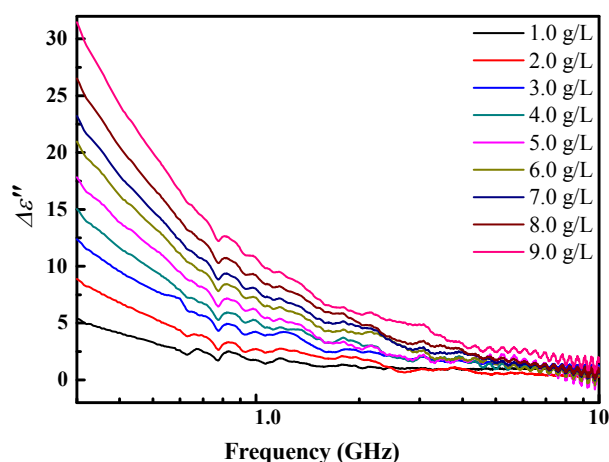


Fig. 2 – Differential dielectric spectra of different concentrations of alum solution.

an increase in alum concentration as the dielectric absorption increases with an increase in the concentration of alum. Thus, the total loss in alum solutions of different concentrations was investigated (i.e., we generated a difference curve for the total loss). As shown in Fig. 3, the total loss ($\omega \cdot \epsilon''$) is constant over a wide area, especially in the low-frequency band. The total loss also increases monotonically with alum concentration (N). Thus, it appears that the total loss difference curve is far more suitable for characterization of concentration.

Usually, to simplify the application of dielectric spectroscopy, a particular frequency is often chosen, and the dispersion or absorption strength is used to determine the concentration of a particular component. Regardless of the dielectric spectrum type (relaxation or resonant), both dielectric dispersion and absorption strength have obvious changes with frequency, especially near the maxima in the dielectric absorption. Choosing a dielectric property at a particular frequency to represent the concentration of a specific component in a dielectric material may bring about error caused by frequency shifts in the spectral line used. The stability of the total loss of alum with respect to frequency fundamentally avoids any errors caused by the use of dielectric dispersion or absorption in a particular frequency. However, considering the total loss and its frequency dependence near 1 GHz, which is specific to the dielectric spectrum of alum, it is seen that there is significant fluctuation in this region. Therefore, we should choose a low frequency to establish the corresponding relationship between dielectric properties and alum concentration when using the total loss dielectric curve.

3.2. Characteristics of the dielectric spectrum of sodium bicarbonate

The specific resonant-type features of the dielectric spectrum of sodium bicarbonate caused by atomic polarization appear in the high-frequency region. The features are different from those due to alum and so it is possible, to a certain extent, to avoid interference with the specific dielectric difference frequency spectrum of alum. As with the alum, decreasing the concentration of the sodium bicarbonate produces smoother

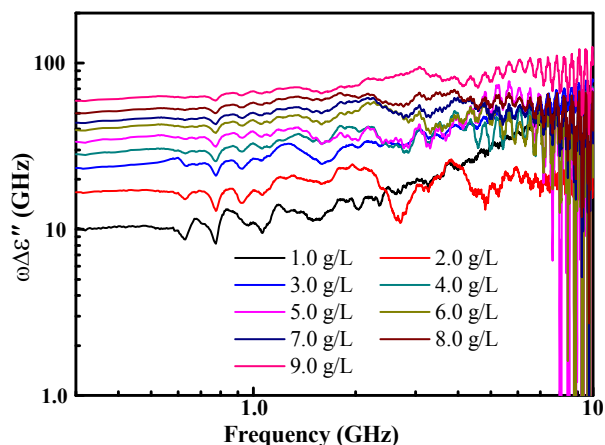


Fig. 3 – Total loss differential dielectric spectrum of alum solution.

changes in the dielectric differential dispersion curve. The amplitude of the dielectric absorption difference curve was reduced at the same time. This is quite striking in the low-frequency area. Thus, it is important to introduce the total loss.

As for alum, the total loss ($\omega \cdot \epsilon''$) is constant over a wide area, but especially so in the low-frequency region. In addition, the total loss increases monotonically with an increase in concentration of sodium bicarbonate. This is further proof of the rationality and validity of using the total loss difference spectrum to represent the concentration of a specific nonaqueous component. However, it is worth noting that it remains to be further investigated whether the use of the total loss difference spectrum is still valid or not when the sodium bicarbonate and alum are mixed, and after their reaction.

The dielectric differential spectrum of an aqueous solution of a mixture of sodium bicarbonate and alum is shown in Fig. 4. The sodium bicarbonate and alum were mixed to 2.000 g/L. In Fig. 4, the specific dielectric dispersion shows resonant-type behavior caused by atomic polarization in the mixed aqueous solution (blue dotted line), which is similar to that of alum (black dotted line). This characteristic dielectric difference spectrum resembling the resonant-type spectrum of alum itself may originate from the products formed during the reaction between the alum and sodium bicarbonate [such as $\text{Al}(\text{OH})_3$ and K_2SO_4] and may be caused by atomic polarization. Among the products formed, the quantity of Al and K is

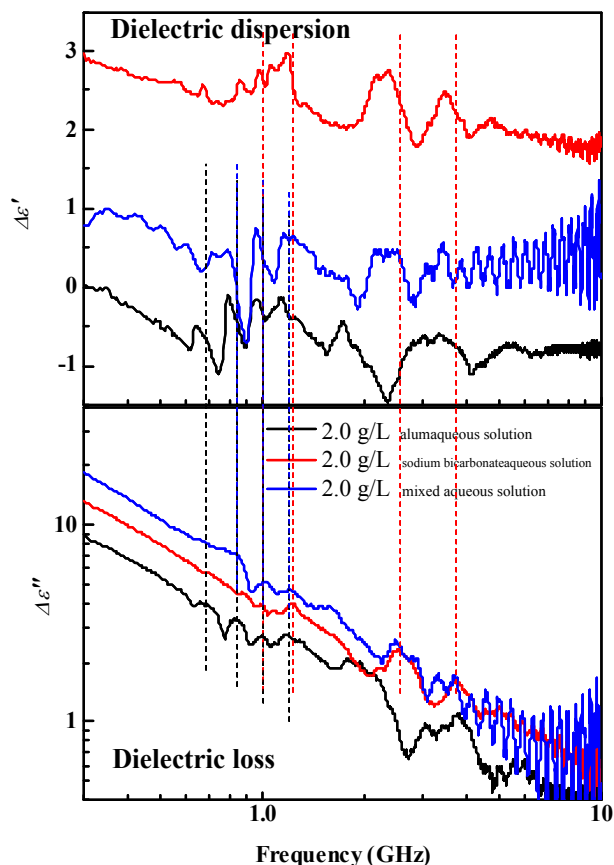


Fig. 4 – Differential dielectric spectra of single and mixed solutions of alum and sodium bicarbonate.

the same as in alum, and can be used to reflect the content of alum. Reaction products such as Na_2SO_4 do not appear to cause any new features in the dielectric resonance spectrum in the observed frequency area. The influence, if any, of the bicarbonate therefore only occurs in the relaxation-type dielectric spectrum in the relatively low-frequency area. Thus, it can be seen that the sodium bicarbonate had little influence on the specific dielectric spectrum of alum. The specific dispersion curve of the resonant type can also be used to determine the existence of the alum at 1 GHz.

The absorption amplitude of the dielectric absorption difference spectrum ($\Delta\epsilon''$) of the mixed solution is larger than that of either the alum or sodium bicarbonate alone (for the same concentration), especially in the low-frequency area. Thus, it is necessary to introduce the total loss ($\omega \cdot \epsilon''$) to calibrate the alum and sodium bicarbonate content in the mixed aqueous solution. Fig. 5 shows the total loss dielectric difference curves at different concentrations of the mixed solutions. The total loss in the low-frequency area of the spectra from the mixed solutions increases as the concentrations of alum and sodium bicarbonate increase. In addition, with an increase in the concentration of the sodium bicarbonate, it increases monotonically. Thus, it can be used to establish the corresponding relationship between the concentration of alum and total loss.

To simplify the application of the dielectric frequency spectrum, a specific frequency was selected to establish the relationship between the concentration of alum and total loss. An appropriate intermediate value to use is 0.425 GHz, as the total loss is constant in the low-frequency area (Fig. 5). The total loss and the concentration of alum (N) were found to have a linear relationship here. The dielectric differential absorption of the solute ($\Delta\epsilon''$) is zero when there is no solute in the solution ($N = 0$). The relationship between the concentration of alum and the differential absorption of the solute at 0.425 GHz has the following form:

$$N = 0.1514 \times \Delta\epsilon''_{0.425 \text{ GHz}}, R^2 = 0.99 \quad (2)$$

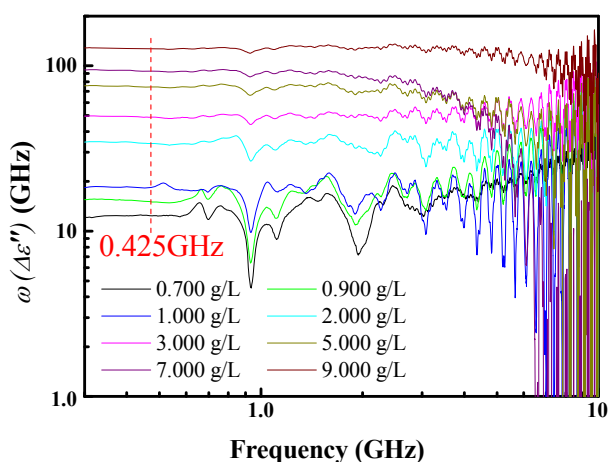


Fig. 5 – Total loss differential dielectric spectra of mixed solutions of alum and sodium bicarbonate.

where N is the concentration of alum (g/L), $\Delta\epsilon''$ is the differential absorption of the solute, and the regression coefficient is 0.99.

The relationship between the concentration of alum and the differential absorption of the solute can be further applied to the low-frequency area using the following expression:

$$N = 5.884 \times 10^{-11} \omega \cdot \Delta\epsilon'', R^2 = 0.98 \quad (3)$$

However, in the DFDS cooking process, whether or not the addition of flour and the high-temperature frying process influence this linear relationship (and even the specific resonance spectrum of alum) is yet to be addressed. Therefore, the dielectric properties of an aqueous solution of a DFDS need to be further studied.

3.3. Characteristics of the dielectric difference spectrum of alum in DFDSs

To explore the effect of the alum and sodium bicarbonate on the DFDS frying process, the DFDS supernatant was analyzed using the dielectric difference spectrum method. From observation of the frequency spectra, the dielectric difference spectra of the DFDS supernatant was found to be almost the same regardless of whether it was left to stand or stirred. These processes therefore have only little impact on the experimental results. Hence, the static postprocessing of the DFDS supernatant will be discussed in the following section.

Flour and cooking oil also have dielectric properties. To rule out the interference of these factors, some additive-free DFDS supernatant was used as the blank control group. The results show a resonant-type dispersion in the corresponding DFDS supernatant and mixed aqueous solution. This further proves that the specific dielectric spectrum of a standard solution can be used to determine the alum content in the DFDS supernatant.

To verify the applicability of the relationship between alum concentration and dielectric absorption difference obtained using standard samples, the total loss in the DFDS supernatant was compared with the mixed solution, as shown in Fig. 6A. The figure clearly shows that the total loss difference curve of the DFDS supernatant lies between the curves obtained using mixed solutions with concentrations from 0.900 to 1.000 g/L. This suggests that the concentration, N , of the alum was such that $0.900 \text{ g/L} < N < 1.000 \text{ g/L}$. According to the DFDS supernatant spectrum, at 0.425 GHz, the value of the dielectric absorption difference is $\Delta\epsilon''_{0.425 \text{ GHz}} = 6.3273$. Thus, using Eq. (2), the alum content in the DFDS was calculated to be $N = 0.9942 \text{ g/L}$. However, the minimum detectable concentration of alum was $0.0450 \mu\text{g/L}$.

3.4. Comparison and verification using graphite furnace atomic absorption

To verify the validity of the new detection method for alum additive, the graphite furnace atomic absorption method was used to determine the Al content of the DFDSs. The results show that the elemental Al content was $55.33 \mu\text{g/L}$ ($\pm 2.5\%$). This can be used to further determine the amount of alum in

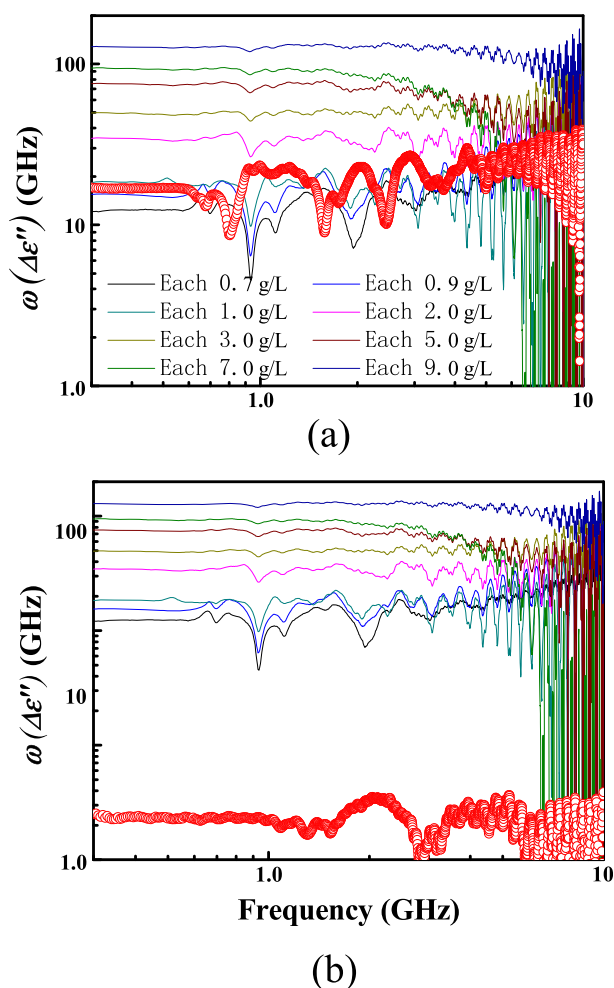


Fig. 6 – Total loss differential dielectric spectra of (A) mixed solutions and prepared deep-fried dough stick (DFDS) extracts and (B) commercial DFDS extracts.

the DFDS. The value derived is 0.9722 g/L, which compares well with the value obtained using the newly developed differential dielectric spectrum method (0.9942 g/L \pm 1.14%). The relative error between the two is 2.2%. In summary, the newly developed method can both qualitatively and quantitatively determine the amount of alum additive in the DFDSs.

3.5. Application of the dielectric differential spectrum method

The purpose of any new development technology is to use it in real applications. The new dielectric spectrum test method was developed to determine the alum in DFDSs in a simpler and more effective manner than other detection methods. Therefore, to test the practicability of the new method, commercial DFDSs were randomly purchased from the market in Lingang New City (Shanghai, China).

From the comparison of alum, sodium bicarbonate, and mixed aqueous solutions, we know that the dielectric dispersion difference curve of the commercial DFDSs has the

same trends as the specific resonant frequency spectrum of a mixed aqueous solution of alum and sodium bicarbonate. The results clearly show that alum has been added to the commercial DFDSs. To quantify the amount of alum involved, the total loss difference curves of the commercial DFDSs with different concentrations of mixed solution were compared, as shown in Fig. 6B. The amplitude of the total loss difference curve of the commercial DFDSs is lower than the different concentrations of the mixed solutions by one to two orders of magnitude and lower than the lowest standard curve (0.7000 g/L) by a factor of 50.

According to the differential absorption value, $\Delta\epsilon''_{0.425 \text{ GHz}} = 0.0873 (\pm 1.14\%)$, at a frequency of 0.425 GHz, the alum content in the commercial DFDS, as calculated using Eq. (2), is $N = 0.0132 \text{ g/L}$. This result is much smaller than the standard suggested for addition of alum to foodstuff, which has an upper limit of 0.158 g/L (in 1 kg food, Al cannot exceed 100 mg). This shows that the amount of alum in the commercial DFDSs is in accordance with the state-prescribed standards.

4. Conclusion

The characteristics of the alum were revealed after a detailed analysis. This study is based on the polarization properties of the ingredients involved. The frequency regions of the media associated with orientation and atomic polarization were selected and used to observe deionized water and aqueous alum solutions with different concentrations. Both the dielectric dispersion curve ϵ' and the dielectric absorption curve ϵ'' were investigated. A new method involving subtracting the dielectric dispersion and loss due to water from that of the food solution was proposed based on other studies involving food with high water content. The method was confirmed to be effective at detecting the nonaqueous ingredients. The new technology, based on the differential dielectric spectrum of the DFDSs, is simple in operation and effective in determining the added alum dose. Moreover, the instruments involved are cheap and readily available. The technology is based on the dispersion, dielectric loss, and leak loss caused by atomic polarization of the alum. In the limited time available for this study, we were able to rule out the occurrence of potentially negative effects on the alum dielectric characteristics due to several factors, including chemical reaction in the DFDS due to the main additive sodium bicarbonate and physical effects in the dough during the proofing and frying processes. It was not possible to include other potential factors. To further popularize the new technology, other applications of the differential dielectric spectrum method need to be developed aimed at other nonaqueous ingredients. The new component testing method also needs to be constantly improved and upgraded in future work.

Conflicts of interest

All contributing authors declare no conflicts of interest.

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REFERENCES

- [1] Polizzi S, Pira E, Ferrara M, Bugiani M, Papaleo A, Albera R, Palmi S. Neurotoxic effects of aluminium among foundry workers and Alzheimer's disease. *Neurotoxicology* 2002;23: 761–74.
- [2] Crisponi G, Nurchi VM, Bertolasi V, Remellib M, Faad G. Chelating agents for human diseases related to aluminium overload. *Coord Chem Rev* 2012;256:89–104.
- [3] Karmakar A, Zhang Q, Zhang Y. Neurotoxicity of nanoscale materials. *J Food Drug Anal* 2014;22:147–60.
- [4] Fu PP, Xia Q, Hwang HM, Ray PC, Yu H. Mechanisms of nanotoxicity: generation of reactive oxygen species. *J Food Drug Anal* 2014;22:64–75.
- [5] Nguyen KL, Lewis DM, Jolly M, Robinson J. Determination of soluble aluminium concentration in alkaline humic water using atomic absorption spectrophotometry. *Water Res* 2004;38:4039–44.
- [6] Parengam M, Judprasong K, Srianujata S, Jittinandana S, Laoharojanaphand S, Busamongko A. Study of nutrients and toxic minerals in rice and legumes by instrumental neutron activation analysis and graphite furnace atomic absorption spectrophotometry. *J Food Compos Anal* 2010;23:340–5.
- [7] Chen B, Zeng Y, Hu B. Study on speciation of aluminum in human serum using zwitterionic bile acid derivative dynamically coated C18 column HPLC separation with UV and on-line ICP-MS detection. *Talanta* 2010;81:180–6.
- [8] Bantan-Polak T, Mitrović B, Milačić R. The use of fast protein liquid chromatography with ICP-OES and ES-MS-MS detection for the determination of various forms of aluminium in the roots of Chinese cabbage. *Anal Chim Acta* 2005;540:83–9.
- [9] Mitrović B, Milacic R. Speciation of aluminium in forest soil extracts by size exclusion chromatography with UV and ICP-AES detection and cation exchange fast protein liquid chromatography with ETAAS detection. *Sci Total Environ* 2000;258:183–94.
- [10] Guo W, Liu Y, Zhu X, Wang S. Dielectric properties of honey adulterated with sucrose syrup. *J Food Eng* 2011;107:1–7.
- [11] Ahmed J, Ramaswamy HS, Raghavan VGS. Dielectric properties of butter in the MW frequency range as affected by salt and temperature. *J Food Eng* 2007;82: 351–8.
- [12] Sosa-Morales ME, Valerio-Junco L, López-Malo A, García HS. Dielectric properties of foods: reported data in the 21st century and their potential applications. *LWT Food Sci Technol* 2010;43:1169–79.
- [13] Liu S, Fukuoka M, Sakai N. A finite element model for simulating temperature distributions in rotating food during microwave heating. *J Food Eng* 2013;115:49–62.
- [14] Xue C, Fukuoka M, Sakai N. Prediction of the degree of starch gelatinization in wheat flour dough during microwave heating. *J Food Eng* 2010;97:40–5.
- [15] Peng J, Tang J, Jiao Y, Bohnet SG, Barrett DM. Dielectric properties of tomatoes assisting in the development of microwave pasteurization and sterilization processes. *LWT Food Sci Technol* 2013;54:367–76.
- [16] Castro-Giráldez M, Dols L, Toldrá F, Fito P. Development of a dielectric spectroscopy technique for the determination of key biochemical markers of meat quality. *Food Chem* 2011;127:228–33.
- [17] Castro-Giráldez M, Fito PJ, Fito P. Application of microwaves dielectric spectroscopy for controlling pork meat (*Longissimus dorsi*) salting process. *J Food Eng* 2010;97:484–90.
- [18] Castro-Giráldez M, Toldrá F, Fito P. Low frequency dielectric measurements to assess post-mortem ageing of pork meat. *LWT Food Sci Technol* 2011;44:1465–72.
- [19] Soltani M, Alimardani R, Omid M. Evaluating banana ripening status from measuring dielectric properties. *J Food Eng* 2011;105:625–31.
- [20] Ragni L, Cevoli C, Berardinelli A. A waveguide technique for non-destructive determination of egg quality parameters. *J Food Eng* 2010;100:343–8.
- [21] Lizhi H, Toyoda K, Ihara I. Discrimination of olive oil adulterated with vegetable oils using dielectric spectroscopy. *J Food Eng* 2010;96:167–71.
- [22] Guo W, Zhu X, Liu Y, Zhuang H. Sugar and water contents of honey with dielectric property sensing. *J Food Eng* 2010;97: 275–81.
- [23] Makul N, Rattanadecho P, Agrawal DK. Applications of microwave energy in cement and concrete—a review. *Renew Sustain Energy Rev* 2014;37:715–33.
- [24] Chandrasekaran S, Ramanathan S, Basak T. Microwave food processing. *Food Res Int* 2013;52:243–61.
- [25] Ryyänen S. The electromagnetic properties of food materials—a review of the basic principles. *J Food Eng* 1995;26:409–29.
- [26] China Mianshi Wang (CNMSW). DFDS production method. 2014. Available at: <http://www.cnmsw.net/news/2/5953.html>. [accessed: 22, October 2014].
- [27] McKeown M, Trabelsi S, Tollner EW, Nelson SO. Dielectric spectroscopy measurements for moisture prediction in Vidalia onions. *J Food Eng* 2012;111:505–10.