

# Estimating the Dielectric Constant of Cellulose Acetate Fiber Aggregation with Its Components Volume Fraction

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## ABSTRACT

For the sake of studying the relationship between dielectric constant ( $\epsilon$ ) of cellulose acetate fiber aggregation and volume fraction of its compositions (including cellulose acetate fibers, water and oiling agent), the dielectric spectrum of the aggregation was measured in two forms with a Broadband Dielectric Spectrum (BDS) test system- dry cellulose acetate fibers (dry samples) and oiled cellulose acetate fibers (oiled samples). The measurement data show that dielectric constant has a linear correlation with the volume fraction of cellulose acetate fibers and the oiling agent in the aggregation, and has a non-linear correlation with the volume fraction of water in the aggregation. A function between dielectric constant of the cellulose acetate fiber aggregation and volume fraction of the components was fitted with 1stOpt. The function is in close proximity to the measured data.

**Keywords:** cellulose acetate fiber aggregation; dielectric constant; Fitting Function; Volume Fraction

## INTRODUCTION

Cellulose acetate fibers are made from natural wood pulp. They are mainly used as filter materials for cigarette filters. An important production process for cellulose acetate fibers is oiling with a finishing solution. The oil content of cellulose acetate fibers is closely related to smoothness, anti-static properties, spinnability and so on. It is defined as the weight percent of oiling agent (non-fibrous material) based on the weight of dry fibers.

Several practical methods have been used to measure oil content, for instance, extraction, infrared spectrophotometry, atomic absorption, nuclear magnetic resonance spectroscopy and so on.

Extraction is one of the most widely used methods. It has high relative accuracy but is a time-consuming and environmentally hazardous method. Infrared spectrophotometry is environmentally and is reliable. Atomic absorption and nuclear magnetic resonance spectroscopy can be used during the spinning process in order to adjust the oiling process and control the oil content, but the test equipment is expensive.

Dielectric properties are an important characteristic of fibers. Researchers have made use of the textile materials dielectric properties for quality control purposes such as the measurement of water content [1], the improvement of unevenness in linear density [2], the control of static generation [3], the observation of impurities within fibrous [4,5] and so on. People have been devoting to new applications based upon dielectric character of textile materials for a long time. In this paper, dielectric constant is associated with the oil content of fibers.

Many dielectric mixture formulas were reviewed. Since Maxwell [6] defined electromagnetism mathematically, the question of predicting dielectric constant, or permittivity, of multicomponent mixtures has attracted much attention. Some approaches, such as the Maxwell Garnet formula [7], the Bruggeman formula [8], Taylor formula [9], Polder-van Santen formula [10] and so on, were proposed to predict permittivity. Hearle [11] performed extensive research on electrical properties of various fibers at frequencies ranging from 50 kHz to 200 kHz and tried to use a variety of air-fiber mixture models to estimate the value of capacitance, but found it difficult to explain those phenomena effectively. M. Peeters [12] successfully evaluated the dry mass and the moisture content of

wool independently by constructing an experimental device, but the result is useful only under restricted conditions such as in the case of low moisture regain. The equivalent dielectric properties of heterogeneous mixtures depend on the dielectric constant, volume fraction, grain size, shape and spatial distribution of the mixture composition [13]. Nelson, Kandala and Trabelsi Samir [14] investigated the relationship between dielectric constant and water content of materials and found that the dielectric constant increased with an increase in the water content. Kausik Bal and V. K. Kothari studied the dielectric constant of woven fabrics and compared the dielectric constant formulas of various air-fiber mixtures, concluding that the average values calculated by most of the equations were essentially equivalent [15]. They also characterized a three phase mixture consisting air, moisture and nylon 6 monofilaments and determined that the dielectric constant followed a quadratic curve with respect to volume fraction [16].

In this paper, two types of samples (including dry samples and oiled samples) were characterized using the BDS test system to determine the dielectric spectra. The relationship between dielectric constant and volume fraction of the mixture components was then fit to a function.

**MATERIALS**

The cellulose acetate fibers used in this study are cellulose acetate tows consisting of 300 cellulose acetate filaments with a linear density of 3 Denier. The fiber density is  $\rho_{fiber} = 1.36g/cm^3$ . Wet and oiled samples were prepared.

**PREPARATION OF DRY SAMPLES**

First, 21 oilless cellulose acetate tows with dry mass ranging from 0.2g to 0.9g were prepared by extraction. A typical sample is shown in *Figure 1*. The samples were then baked at  $105\pm 2^\circ C$  for an hour in an oven, packed in bags and dried under vacuum. Finally, separated into 3 groups (group  $i$ ,  $i=1, 2, 3$ ) of 7 samples (sample  $i-j$ ,  $i=1-3$ ,  $j=1-7$ ) per group.



FIGURE 1. Typical sample.

**PREPARATION OF FINISHING SOLUTION**

First, the oiling agent was prepared by blending an emulsifier consisting of span-20 (sorbitan monooleat) and tween-20 (poly-oxyethylene sorbitan monolaurate) in equal volumes, mineral oil and deionized water at the rate of 6:13:1 by volume. Its density is  $\rho_{oil} = 0.9g/ml$ . Seven types of finishing solutions consisting of the oiling agent and deionized water at different blend rates were then prepared. The concentrations of the oiling agent ( $C_k$ ) are shown in *Table I*, where  $k$  is the serial number of the finishing solutions.

TABLE me. Volume concentrations of oiling agent.

$k$	1	2	3	4	5	6	7
$C_k$ (%)	0	0.25	0.5	0.75	1	1.25	1.5

**PREPARATION OF OILED SAMPLES**

The oiled samples were prepared as follows. First, 21 dry samples were conditioned at standard atmospheric conditions for 24 h. Second, three groups of samples were soaked in seven finishing solutions for one minute with serial number  $k$  equal to  $j$  respectively. Finally, the finished samples were hung vertically for an hour to dry.

**MEASUREMENT AND RESULTS**

Measurements were conducted at a temperature of  $20^\circ C$  and RH of 65 percent. The readability of the electronic balance used in the mass measurements is 0.0001g, and its verification scale interval is 0.001g. The accuracy of relative impedance and capacity of the BDS test system is less than  $3 \times 10^{-5}$ , and the resolution is less than  $10^{-5}$ .

**MEASUREMENT OF DRY SAMPLES**

The measurements were conducted according to the following steps:

(1) An empty capacitor with two disposable electrodes and a Teflon gasket was constructed. The volume was  $V = 4.0212ml$ . The dielectric spectrum was measured with the BDS test system, and the measurement system was adjusted until the dielectric constant reached a value of 1. Therefore the influence of air could be ignored. The data was weighted and recorded as  $m_0$ .

(2) A sample capacitor was made using a dry sample and the empty capacitor. A sample in capacitor was placed as shown in Figure 2.

(3) The sample capacitor was weighed, and the weight recorded as  $m'_{i-j}$  ( $i=1,2,3$ ,  $j=1-7$ ), where  $i$  is the serial number of groups and  $j$  is the serial number of samples. The dielectric spectrum of the sample was tested at 58 frequencies ranging from 0.9 Hz to 20 MHz. The sample capacitor was weighed again and the weight was recorded as  $m''_{i-j}$ .

(4) Steps (2) and (3) were repeated on the remaining samples.



FIGURE 2(a). Sample capacitor.



FIGURE 2(b). The placing way of a sample in capacitor.

The mass of dry sample  $m_{i-j}$  was

$$m_{i-j} = \frac{m'_{i-j} + m''_{i-j}}{2} - m_0 \tag{1}$$

The volume of dry sample  $V_{i-j}$  was

$$V_{i-j} = \frac{m_{i-j}}{\rho_{fiber}} \tag{2}$$

The volume fraction of dry sample  $\phi_{i-j}$  was

$$\phi_{i-j} = \frac{V_{i-j}}{V} \times 100\% \tag{3}$$

**MEASUREMENT OF OILED SAMPLES**

The measurement method used for the oiled samples was slightly different with that used for dry samples. Each oiled sample was tested many times at different water contents.

(1) A sample capacitor was made using an oiled sample and the empty capacitor.

(2) The sample capacitor was weighed and the weight recorded as  $m'_{i-j-z}$  ( $z=1, 2, 3\dots$ ), where  $z$  is the serial number of the measurement of sample  $i-j$ . The dielectric spectrum of the sample capacitor was then measured. The sample capacitor

was weighed again and recorded as  $m''_{i-j-z}$ . The sample was removed from the capacitor and hanged for 5 minutes to release humidity. This step was repeated until  $m'_{i-j-z}$  was approximately equal to  $m'_{i-j-z+1}$ .

(3) Steps (1) and (2) were repeated until all the oiled samples were measured.

(4) The samples were baked at  $105\pm 2^\circ\text{C}$  for an hour, placed in sealed bags and stored in a vacuum dryer.

(5) A sample capacitor was made using a dry oiled sample and the empty capacitor.

(6) The sample capacitor was weighed and the weight recorded as  $m'_{i-j-0}$ . The dielectric spectrum of the sample capacitor was measured. The sample capacitor was weighed again and recorded as  $m''_{i-j-0}$ .

(7) Steps (5) and (6) were repeated until all the oiled samples were measured.

The mass of oiled sample  $m_{i-j-z}$  was

$$m_{i-j-z} = \frac{m'_{i-j-z} + m''_{i-j-z}}{2} - m_0 \quad (4)$$

The mass of dry oiled sample  $m_{i-j-0}$  was

$$m_{i-j-0} = \frac{m'_{i-j-0} + m''_{i-j-0}}{2} - m_0 \quad (5)$$

The mass of oiling agent on sample  $m_{oil-i-j}$  was

$$m_{oil-i-j} = m_{i-j-0} - m_{i-j} = \frac{m'_{i-j-0} + m''_{i-j-0} - m'_{i-j} - m''_{i-j}}{2} \quad (6)$$

The volume of oiling agent on sample  $V_{oil-i-j}$  was

$$V_{oil-i-j} = \frac{m_{oil-i-j}}{\rho_{oil}} \quad (7)$$

The volume fraction of oiling agent on sample  $\varphi_{oil-i-j}$  was

$$\varphi_{oil-i-j} = \frac{V_{oil-i-j}}{V} \times 100\% \quad (8)$$

The mass of water on oiled sample  $m_{w-i-j-z}$  was

$$m_{w-i-j-z} = m_{i-j-z} - m_{i-j-0} = \frac{m'_{i-j-z} + m''_{i-j-z} - m'_{i-j-0} - m''_{i-j-0}}{2} \quad (9)$$

The volume of water on oiled sample  $V_{w-i-j-z}$  was

$$V_{w-i-j-z} = \frac{m_{w-i-j-z}}{\rho_w} \quad (10)$$

Where  $\rho_w$  is water density.

The volume fraction of water on oiled samples

$\varphi_{w-i-j-z}$  was

$$\varphi_{w-i-j-z} = \frac{V_{w-i-j-z}}{V} \times 100\% \quad (11)$$

## RESULTS AND DISCUSSION

The results indicate that:

(1) In *Figure 3*, for dry samples,  $\varepsilon$  varies weakly with frequency, especially at high frequencies. At low frequencies, it increases slightly with a decrease of frequency. There is a dielectric relaxation phenomenon for materials- the dielectric constant increases at low frequencies. Lyons [17] pointed out that dielectric constant is not related to frequency when the water content of fibers is near zero. In *Figures 4a and 4b*, there is a significant linear correlation between the volume fraction of dry samples ( $\omega_{i-j}$ ) and  $\varepsilon$  over the measured range of frequencies. The dielectric constant of fibers is higher than that of air under the same conditions. When the volume fraction of fibers increases, the dielectric constant increased.

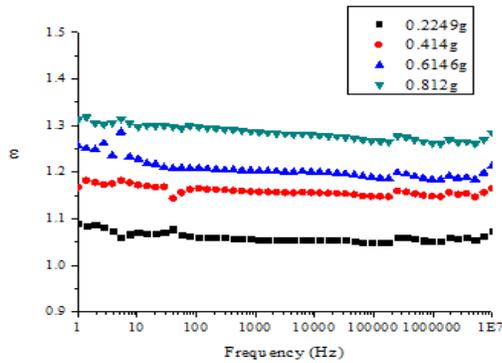


FIGURE 3 The dielectric spectrum of dry samples with different mass.

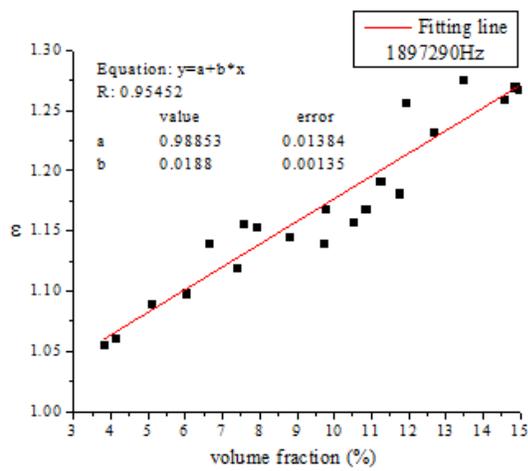


FIGURE 4(a). Relation between  $\epsilon$  and  $\phi_{i-j}$  at 1897290Hz.

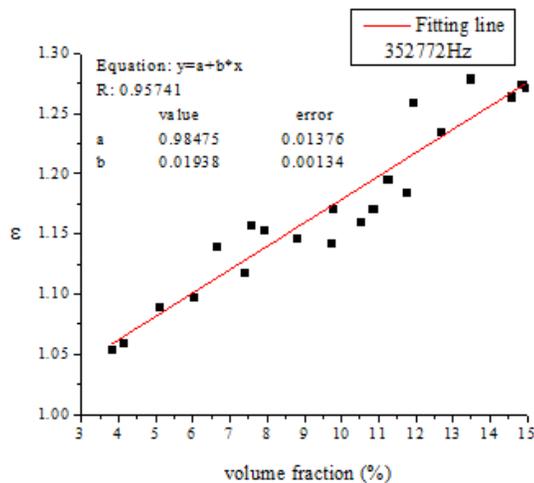


FIGURE 4(b). The relation between  $\epsilon$  and  $\phi_{i-j}$  at 352772Hz.

(2) Figure 5 shows that  $\epsilon$  of oil less samples increases with an increase water volume fraction and decreases with an increase in frequency. The dielectric constant of a material is closely related to volume fraction, and due to the dielectric relaxation phenomenon the dielectric constant at low frequency is higher than that at high frequency.

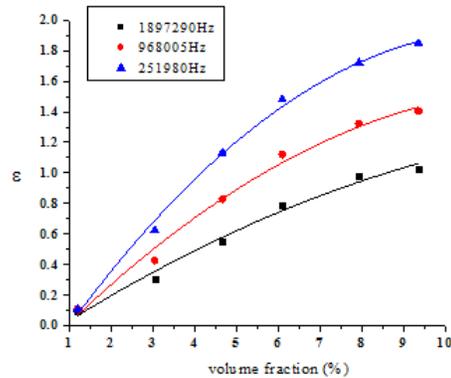


FIGURE 5. The  $\epsilon$  of a sample with different water volume fraction.

(3) Figure 6 shows the dielectric spectrum of four dry oiled samples with different masses. As the figure shows, the  $\epsilon$  increases with decreasing frequency at low frequencies, with little change at high frequencies. The main cause of this phenomenon is the dielectric relaxation phenomenon of the dry samples and moisture in the air. Figure 7 shows the linear correlation relation between  $\epsilon$  and volume fraction of the oiling agent in the fibers.

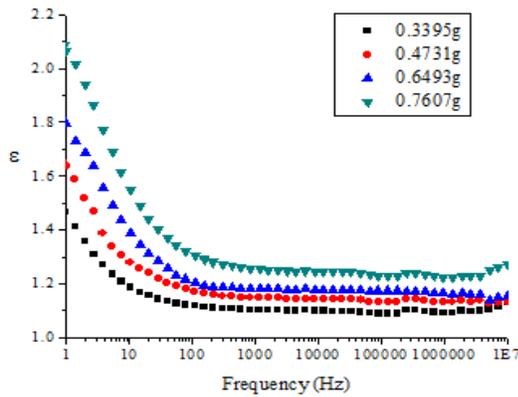


FIGURE 6. The dielectric spectrum of four oiled samples with different mass.

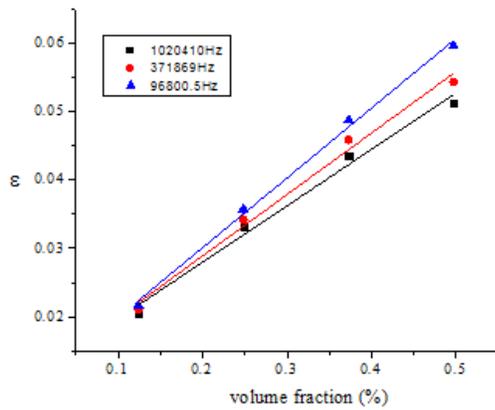


FIGURE 7. The  $\epsilon$  of oiling agent with different volume fraction.

(4) Figure 8 shows the comparison of the dielectric spectrum of a sample before and after it was oiled. At high frequencies the change in dielectric constant of due to the presence of the oiling agent on the fibers is not obvious because the volume fraction is too small. In this figure, it is noted that water makes more of a contribution to the dielectric constant than the oiling agent and the fibers due to the dielectric permittivity of water, which is many times higher than ordinary polymeric materials (in this case cellulose acetate).

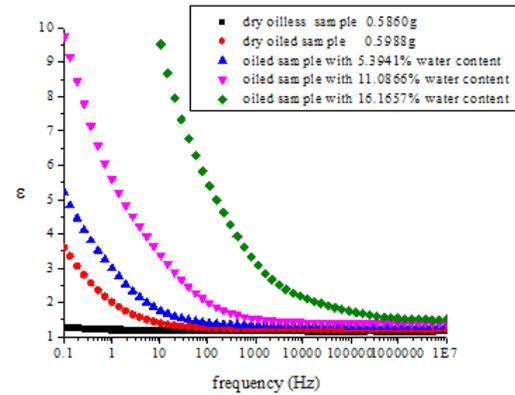


FIGURE 8. The comparison of the dielectric spectrum of a sample before and after it's oiled.

### FITTING THE FUNCTION TO THE DATA

Dielectric constant modeling normally adopts the method of physical modeling, which uses functions to analyze the dielectric relaxation process [18]. Common functions used are the Debye equation, the Cole-Cole circular arc equation, the Davidson-Cole asymmetric arc equation and the Havriliak-Negami equation and so on. However, there was not obvious relaxed peak according to the test data.

By analyzing the independent contribution to dielectric constant made by the components, a fitting function between the dielectric constant and the volume fraction of components of cellulose acetate fiber aggregation was developed.

It is defined that:

- (1)  $\epsilon$  is the total dielectric constant of the cellulose acetate fiber aggregation;
- (2)  $x$  stands for volume fraction of the cellulose acetate fibers, which contributes the dielectric constant  $\epsilon_x$ ;
- (3)  $y$  represents volume fraction of water in the aggregation and  $\epsilon_y$  is the dielectric constant it contributed;
- (4)  $z$  expresses volume fraction of the oiling agent of the aggregation and  $\epsilon_z$  is the dielectric constant contributed.

### **ESTABLISHMENT OF THE FUNCTION**

Many researchers studied the relationships between the total dielectric constant and the dielectric constants of the components, but it is difficult to develop an exact understanding and analysis of the multicomponent mixture dielectric constant. In the samples measured in this study, there is no chemical reaction between the compositions. For oiled samples, the volume of oil agent on the fibers is so small that the intermolecular interactions of the compositions have little influence on the total dielectric constant. Ignoring these interactions, a mathematical model was developed as follows:

(1) Assuming that every component makes a specific and independent contribution to the total dielectric constant, ignoring the influence of air, then total dielectric constant may be expressed as

$$\varepsilon = \varepsilon_x + \varepsilon_y + \varepsilon_z \quad (12)$$

(2) According to *Figure 4*, at high frequency, there is a significant linear correlation between dielectric constant and volume fraction of dry cellulose acetate fibers. That is

$$\varepsilon_x = ax + h_1 \quad (13)$$

In Eq. (13),  $a$   $h_1$  are undetermined coefficients,  $x \in (3.66\%, 16.46\%)$ .

(3) Kausik Bal and Kothari [16] pointed out that the total dielectric constant of a three phase mixture including moisture followed a quadratic curve with respect to the volume fraction of water present. According to *Figure 5*, at high frequencies, the curve is quadratic in shape. At high frequencies, according to *Figure 5*, the relational expression is assumed as follows.

$$\varepsilon_y = by + cy^2 + h_2 \quad (14)$$

$b$ ,  $c$  and  $h_2$  are undetermined coefficients,  $y \in (0, 8\%)$ .

(4) Compared to volume of water and that of the cellulose acetate fibers, the oiling agent volume is very small. At high frequencies the contribution to the dielectric constant made by the oiling agent is

not obvious. Combining *Figure 7* and *Figure 8*, the relationship between the volume fraction of oiling agent of the aggregation and the dielectric constant at high frequencies can be assumed as follows.

$$\varepsilon_z = dz + h_3 \quad (15)$$

$d$ ,  $h_3$  are undetermined coefficients and  $z \in (0, 0.75\%)$ .

Substituted Eq. (13), Eq. (14) and Eq. (15) into Eq. (12), therefore

$$\varepsilon = \varepsilon_x + \varepsilon_y + \varepsilon_z = ax + h_1 + by + cy^2 + h_2 + dz + h_3 \quad (16)$$

Let  $h = h_1 + h_2 + h_3$ , then the dielectric constant of the aggregation can be defined as

$$\varepsilon = ax + by + cy^2 + dz + h \quad (17)$$

where  $a$ ,  $b$ ,  $c$ ,  $d$ ,  $h$  are undetermined coefficients,  $a$  indicates a linear correlation between  $\varepsilon_x$  and  $x$  and  $c$  indicates that  $\varepsilon_y$  and  $y$  represents quadratic function relation,  $d$  indicates that there is a linear correlation between  $\varepsilon_z$  and  $z$  and  $h$  is the intercept.

### **FITTING THE FUNCTION**

The measured data show that the dielectric constants of the oiled samples have the same varying trend at high frequencies. Taking 179 groups of measured data (21 groups of dry oil less samples, 21 groups of dry oiled samples, 68 groups of oilless samples with different water content, with the remainder consisting of samples with different oil content and water content) at the frequency of 1897290Hz for example, the  $a$ ,  $b$ ,  $c$ ,  $d$ ,  $h$  were calculated with Eq. (17) and the values of  $x$ ,  $y$ ,  $z$  and  $\varepsilon$  with 1stOpt. The results are as follows.

$$\hat{a} = 0.0046015, \quad \hat{b} = 0.17005, \quad \hat{c} = -0.0017701, \\ \hat{d} = -0.11226, \quad \hat{h} = 1.10323, \quad R = 0.95676, \\ R^2 = 0.91540$$

Therefore, the multivariate nonlinear regression equation is

$$\varepsilon = 0.0046015x + 0.17005y - 0.0017701y^2 - 0.11226z + 1.10323 \quad (18)$$

$x \in (3.66\%, 16.46\%)$  ,  $y \in (0, 8\%)$  and  $z \in (0, 0.75\%)$ .

### VERIFICATION

Ten groups of independent variables and dependent variables were chosen randomly as original data. Then  $\varepsilon$  was calculated using Eq. (18) and compared with measured  $\varepsilon$  values. The comparison between original data and calculated  $\varepsilon$  is shown in Table II. As shown in Table II, relative errors are small, varying from 1.09404% to 5.36636%. The electronic balance verification scale interval is 0.001g, thus when weighing light samples, weighing errors are higher. For example No.3 in Table II, both the fiber and water levels are low, so the error is high, but acceptable. It can be inferred then that, ignoring the influence of air, Eq. (18) is of high reliability.

TABLE II. The comparison between the estimated and measured values of  $\varepsilon$ .

Number	$x$ (%)	$y$ (%)	$z$ (%)	Measured $\varepsilon$	Estimated $\varepsilon$	Relative error (%)
1	5.07787	0	0	1.09007	1.12660	3.35078
2	7.33978	0	0	1.11936	1.13700	1.57626
3	4.81913	0.79330	0	1.19506	1.25919	5.36636
4	11.8874	3.37088	0	1.65121	1.71104	3.62318
5	15.3662	5.42748	0	2.10516	2.04474	2.87003
6	6.11648	0.79330	0.11743	1.20654	1.25198	3.76597
7	6.90458	0.80324	0.23487	1.19794	1.24408	3.85201
8	12.8108	1.16259	0.13539	1.35947	1.34229	1.26407
9	12.2165	1.85268	0.15612	1.43519	1.45089	1.09404
10	8.51187	1.37024	0.19756	1.37788	1.34991	2.07199

### CONCLUSION

The cellulose acetate fiber samples were tested in dry and oiled states. In the case of the dry samples,  $\varepsilon$  increases with increasing volume fraction of cellulose acetate fibers but varies weakly with frequency, especially at high frequencies. To a certain extent,  $\varepsilon$  increases with increasing water volume fraction in the aggregation when the volume fraction of dry cellulose acetate fibers is constant. The  $\varepsilon$  values of dry oiled sample changes very little at high frequencies. There is a linear relation between the  $\varepsilon$  of the oiling agent of the aggregation and volume fraction. The data indicates that  $\varepsilon$  is related to volume fraction of fibers, water and oiling agent in the aggregation.

By analyzing the measured data, function relating dielectric constant and the variables was put forward. A function with 3 independent variables and a dependent variable was fitted with 1stOpt and the experimental data. The estimated  $\varepsilon$  based on Eq. (18) agrees well with the experiment data.

While concentration of the oiling agent is constant, the volume fraction of water can be calculated with that of oil agent. Then for a certain volume of fibers, the total dielectric constant is related clearly to its oil content. It may provide a method for the online test of oil content.

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