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## ЛАБОРАТОРНАЯ ТЕХНИКА

# CONCENTRATION MEASUREMENT WITH OPTICAL FIBERS FOR SMALL SAMPLE

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It is valuable to physically detect concentration of small quantity of liquid with suspended particles. In this paper, light scattering in different directions and optical lengths is simulated and analyzed based on Mie theory. Then, an equipment is designed with two receiving beams of optical fibers, one is for scattering and the other is for transmitting. Scattering angle can be adjustable in order to locate the most sensitive direction. The method of ratio of scattering light to transmitted light is used to reduce the influence of fluctuations of light source and the method of transmitting light is used to satisfy the large measure range. Under room temperature conditions, concentration of yeast extract is tested with 785 nm laser. The results indicate at 0 to 5%, there is linear relationship between transmitting light to transmitted light and concentration. While, for 5% to 10%, there is linear relationship between transmitting light and concentration. Furthermore, it's notable that maximum relative error on experiment is less than  $\pm 0.2\%$ .

### **INTRODUCTION**

Photoelectric detection, also known as optical density measurement, is based on the principle that the varying concentration leads to certain scattering and transmitting. In 1992, M.A. Hughes proposed an optical probe assembly to determine the concentration of bacteria in fluid media and ascertain the content of blood hemoglobin [1]. In 2006, a flexible turbidity sensing system that implements single and multiple infrared beam architectures was presented [2].

In recent years, there are more research on detection of suspended particles concentration in mixture samples, but for small samples is seldom given. This paper put forward a new equipment to physically measure the concentration of suspended particles in liquid by using optical fibers.

## THE THEORY OF SCATTERING AND TRANSMITTING

Suspended particles in a liquid will scatter and absorb the incident light, and the condition of scattering is different for every suspended particles. Light scattering and transmitting theory is the basis of concentration measurement.

## The Theory of Mie Scattering

Fig. 1 shows Mie scattering condition. A beam of light is emitted to a particle along with z axis, and it is scattered. In the scattering figure,  $\varphi$  is the angle between incident vibrating surface and the scattering

surface,  $\theta$  is scattering angle, and *r* is the length between particle and detector.

According to Mie scattering theory, when a spherical particle is radiated by a beam of parallel light as shown in Fig. 1, the scattered light intensity can be expressed in the following formulas [3]:

$$I_r = \frac{\lambda^2}{8\pi^2 r^2} [i_1 + i_2] I_0.$$
(1)



Fig. 1. Light scattering orthogonal reference frame.  $\phi$  is the angle between incident vibrating surface and the scattering surface,  $\theta$  is scattering angle.

Where  $I_r$  is the intensity of the scattered light in  $\theta$ direction and  $I_0$  is the intensity of the incident light to the particle,  $\lambda$  represents the wavelength of light and rrepresents the distance between particle and receiving optical fiber.  $i_1$  and  $i_2$  are the scattering light intensities, which are in two orthogonal directions of incident polarization, respectively and related to three parameters: m,  $\alpha$ , and  $\theta$ , where m is the refractive index of the particle;  $\theta$  is the direction of scattered light;  $\alpha$  is defined by  $\alpha = \pi D/\lambda$ , which indicates the size parameter, D is the diameter of the spherical particle.

The two light intensity functions  $i_1$  and  $i_2$  depend on the scattering amplitude functions  $S_1$  and  $S_2$ , they are stated as follows:

$$i_{1}(\theta) = |S_{1}(\theta)|^{2};$$
  

$$i_{2}(\theta) = |S_{2}(\theta)|^{2}.$$
(2)

In the above function,  $S_1$  and  $S_2$  are the scattering amplitude functions of the light corresponding to  $i_1$ and  $i_2$ , respectively. The functions  $S_1$  and  $S_2$  are expressed as follows:

$$S_{1} = \sum_{l=1}^{\infty} \frac{2l+1}{l(l+1)} [a_{l}\pi_{l}(\cos\theta) + b_{l}\tau_{l}(\cos\theta)];$$

$$S_{2} = \sum_{l=1}^{\infty} \frac{2l+1}{l(l+1)} [a_{l}\tau_{l}(\cos\theta) + b_{l}\pi_{l}(\cos\theta)].$$
(3)

The complex-valued Mie coefficients  $a_l$  and  $b_l$  depend on the size parameter of particle  $\alpha$  and on the refractive index of particle *m*, in which *m* is complex. The angle eigenfunction  $\pi_l$  and  $\tau_l$  depend on the scattering angle only.  $a_l$  and  $b_l$  can be expressed as follows:

$$a_{l} = \frac{\psi_{l}(\alpha)\psi_{l}'(m\alpha) - m\psi_{l}'(\alpha)\psi_{l}(m\alpha)}{\xi_{l}(\alpha)\psi_{l}'(m\alpha) - m\xi_{l}'(\alpha)\psi_{l}(m\alpha)};$$

$$b_{l} = \frac{m\psi_{l}(\alpha)\psi_{l}'(m\alpha) - \psi_{l}'(\alpha)\psi_{l}(m\alpha)}{m\xi_{l}(\alpha)\psi_{l}'(m\alpha) - \xi_{l}'(\alpha)\psi_{l}(m\alpha)}.$$
(4)

In equations (4),  $\psi_l(z)$  and  $\xi_l(z)$  are expressed in

terms of spherical Bessel functions.  $\psi'_l$  and  $\xi'_l$  are the first derivative of the  $\psi_l$  and  $\xi_l$  to their independent variable. In order to simplify the expression, *z*, which represents the refractive index of particle *m* or the product of *m* and the size parameter  $\alpha$ , is introduced in the following formulas, which depend on the equations (4).

$$\psi_{l}(z) = (z\pi/2)^{1/2} J_{l+1/2}(z);$$

$$\xi_{l}(z) = (z\pi/2)^{1/2} [J_{l+1/2}(z) - iY_{l+1/2}(z)].$$
(5)

Where  $J_{l+1/2}(z)$  and  $Y_{l+1/2}(z)$  denote the Bessel functions of the first and the second kind, respectively.

By derivation,  $\psi'_l$  and  $\xi'_l$  are gained. They are expressed in terms of recursive expressions  $\psi_l$  and  $\xi_l$  in order to use Bessel function within Matlab and simplify the calculation to reduce the calculation time. They are

$$\psi'_{l}(z) = \psi_{l-1}(z) - \frac{l}{z} \psi_{l}(z);$$
  

$$\xi'_{l}(z) = \xi_{l-1}(z) - \frac{l}{z} \xi_{l}(z).$$
(6)

In equation (3),  $\pi_l$  and  $\tau_l$  is only decided by the scattering angle, and they are independent on the refractive index and size parameter of the particle. They are defined in the following formulas:

$$\pi_{l}(\cos\theta) = \frac{P_{l}^{(1)}(\cos\theta)}{\sin\theta};$$

$$\tau_{l}(\cos\theta) = \frac{d}{d\theta}P_{l}^{(1)}(\cos\theta).$$
(7)

Where  $P_l$  is the associated Legendre function, and  $P_l^{(1)}$  is the associated Legendre function of the first class.

In order to get  $S_1$  and  $S_2$ , the initial conditions of  $\psi_l$ ,  $\xi_l$ ,  $\pi_l$  and  $\tau_l$  should be known. Substitute l = 1 into the equations (5) and (7), the initial conditions of them can be gained, as follows:

$$\psi_{l}(z) = \frac{1}{z}(\sin z - \cos z);$$
  

$$\xi_{l}(z) = \frac{1}{z}(\sin z + i \cos z) - (\cos z - i \sin z);$$
 (8)  

$$\pi_{l}(z) = 1; \quad \tau_{l}(z) = \cos \theta.$$

Based on the initial conditions derived from formula (8), Mie coefficients  $a_l$  and  $b_l$  are calculated from equations (5) and (6), and the angle eigenfunction  $\pi_l$ and  $\tau_l$  may be calculated, too. And then, the vibratory amplitude functions of the scattered light  $S_1$  and  $S_1$  are calculated from the result of equation (4) and equation (7). So the relationship between the light intensity and the scattering angle are described accurately.

Now according to the first equation of (5) and the corresponding initial condition, the authors designed a program to calculate the value of  $\psi_l$ , by using the Bessel function of the first kind within Matlab directly, and the calculation of  $\xi_l$  are carried out in the same way by using the Bessel functions of the first and the second kind. Because the recursive formula between  $\psi_l$  and  $\psi'_l$  has been obtained in the previous part of the



Fig. 2. The distribution of Scattering intensity when  $D = 0.1 \,\mu\text{m}$  (a), 1  $\mu\text{m}$  (b), 10  $\mu\text{m}$  (c).  $\lambda = 0.785 \,\mu\text{m}$ , m = 1.5 - 0.5i.

paper, the calculation of  $\psi'_l$  may be achieved. Also, the calculation of  $\xi'_l$  are completed in the same way. The program substitutes the values of *m* and  $\alpha$  into the functions,  $\psi_l$ ,  $\psi'_l$ ,  $\xi_l$ , and  $\xi'_l$  are calculated, and so the Mie scattering coefficients  $a_l$  and  $b_l$  are calculated. The rest parameters  $\pi_l$  and  $\tau_l$  are calculated with use of the Legendre function of the first kind within Matlab.

Ratio of scattering and incident intensity,  $10^{-3}$ 



**Fig. 3.** Relationship between receiving light intensity and optical length for three diameters of particles: 20  $\mu$ m (*I*), 10  $\mu$ m (*2*), 1  $\mu$ m (*3*).

Defining concentration of a sample of liquid as N, there is the following expression based on equation (1):

$$I_r = N \frac{\lambda^2}{8\pi^2 r^2} [i_1 + i_2] I_0.$$
(9)

Intensity of scattering light  $I_r$  is proportional to the concentration of suspended particles N as wavelength of incident light  $\lambda$ , intensity of incident light  $I_0$ , distance of detection r, and  $i_1$ ,  $i_2$  are certain.

In a sample of liquid under test, based on scattering theory the optical multi-scattering may happen when concentration of suspended substance is high to a limit. When the multi-scattering happens, the relationship between scattering light  $I_r$  and concentration of suspended particles N becomes nonlinear. So the detection method with scattering light is not fit for high concentration.

#### The Theory of Lambert-Beer Transmitting

Based on Lambert-Beer transmitting theory, suppose a testing region which has cross-section area of A, length of l with unit volume containing the number of N microparticles of the same shape, size and facing-light area all a, the number of microparticles in the thin layer with the thickness of dl is *NAdl*. When light with intensity of I come through the thin layer, the equation is given as follows by definition of extinction efficiency Q:

$$-AdI = IQNAdl. \tag{10}$$



Fig. 4. The constitution of measure equipment.

Suppose the intensity of incident light is  $I_0$ , extinction efficiency Q = ka then from integral the equation (10) there is

$$I = I_0 \exp[-Nkal]. \tag{11}$$

Following equation (11), the intensity of transmitting light has the reverses ratio of the concentration when incident light intensity  $I_0$ , size parameters a,



Fig. 5. The assembly drawing of sample cell structure.

proportionality coefficient K and transmitting optical path l are certain [4].

## The Method of Scattering Light-to-Transmitting Light Ratio

S/T (Scattering light-to-transmitted light ratio) method is used to measure the concentration by the ratio between scattering intensity and transmitted intensity. It was known, the concentration can also be obtained using the scattering light and transmitted light alone. But the fluctuation of light source will bring indispensable error to the measurement result. Undoubtedly, S/T method would eliminate the influence of the fluctuation of light source, and improve the stability.

But S/T method is only used with low concentration because scattering light method will lose with high concentration. So, S/T method is for low concentration, and transmitting light method is for high concentration, thus high precision and large measure range becomes possible.

## THE SIMULATION

The next work is to find some rules based on the numerical simulations with MATLAB.

The Mie theory declares that  $i_1$  and  $i_2$  are two important factors in the studying of light scattering. This paper simulates the trend of  $i_1$  and  $i_2$  along with the increasing of scattering angle which ranges from 0° to 180° shown as Fig. 2. In the simulation, the wavelength of incident light is defined to be 785 nm, the re-



Fig. 6. The principle of signal detection amplifier.

fractive index is 1.5-0.5i, and the size of particle diameter ranges from 0.1 to 10  $\mu$ m.

It is shown that the scattering light intensity behaves an obvious trend along with the increase of the size of particles in the following figures. Fig. 2a shows scattering light distribution tending to be symmetrical, where the diameter of particle is smaller than the wavelength of incident light. It is clearly indicated that particles cause light to be scattered at both forward and backward direction in  $0^{\circ}$  to  $180^{\circ}$  range when the diameter is much smaller than the wavelength of incident light. From the comparison of the three figures, the value of forward scattering light intensify is more and more high with the increase of diameter, yet the optical back scattering is weakened. When the diameter increases to a certain limit, the scattering intensity mainly concentrated in the forward. The phenomenon is basically consistent with the theory. As the intensity of scattering light is different in different angles, it is necessary for measure scattering angle adjustable in order to find the optimal type of detection.

The length between suspended substance and receiving fiber will affect the luminous flux received. Figure 3 shows the relationship between receiving light intensity and optical length for three diameters of particles. From Fig. 3, it indicates that the light intensity quickly becomes low when the optical length near to a certain limit, so the optical length should not be too long. The simulations provide the basis for the design of equipment.

## THE CONSTITUTION OF MEASUREMENT EQUIPMENT

The concentration measurement equipment for small sample with optical fibers is composed of regulated power supply, fiber laser, reception fiber, photocell, signal detection amplifier, data acquisition card and PC. It is as shown in Fig. 4. In the equipment, ST785D10G fiber laser of Situo is used as light source. Because the output connector size of fiber laser is small, it's suitable for the structural design of a small sample cell. Reception fiber is a plastic fiber which core diameter is thick so that it's easy to receive more scattering and transmitting signals. The photocell which peak wavelength is near to the laser is used as a optical-electrical converter in the equipment. The high-speed data acquisition card USB2010 of Altai is exerted to collect data to input PC and LabVIEW in PC is utilized to process the data.

#### The Structure of Sample Cell

The sample cell with length of 8 mm, width of 5 mm, depth of 5 mm is designed, based on the reasons that: the size the sample cell should be small enough as the simulation results; and the detection optical path has to be large enough to satisfy precision requirement. Besides, the surface of sample cell is blackened to prevent influence of stray light reflected by the cell wall. As it has been analyzed, intensity distribution of scattering light is determined by the ratio of particle size and incident light wavelength. Angle of scattering light is adjustable to find the angle at which the linearity is the best, it's precision is 2 with the principle of slide calipers. Fig. 5 is the assembly drawing of the cell.

#### The signal detection amplifier

Due to the scattering and transmitting signals are so weak, especially the scattering signal which is several micrometer level, it's necessary for I/V conversion and amplifier-filter. In the equipment, a twostage operational amplifier which the first is I/V conversion and second is amplifier is used. Fig. 6 shows the principle of circuit. The voltage of output is:

$$V_{out} = R_f I_{in} (R_5 / R_4), \tag{12}$$

 $R_f I_{in}$  is the voltage of I/V conversion,  $R_5/R_4$  is the amplification factor of second stage.



Fig. 7. The scattering signals of different scattering angles vary with the suspended fluid concentration.



**Fig. 8.** The S/T (1) and the transmitting signal (2) for  $90^{\circ}$  vary with the suspended fluid concentration.

## TEST AND DATA ANALYSIS

The yeast extract is utilized as suspended particulates Under the room temperature, after the suspending fluid of different concentrations are prepared, the experiments are carried for scattering angles from  $60^{\circ}$ to  $120^{\circ}$  respectively.

Fig. 7 shows that the scattering signals of different scattering angles vary with the suspending fluid concentration. As seen from the figure, the curve linearity is best at 90°. This result is consistent with the theory of Mie scattering. The scattering light at 90° is less affected by the suspended fluid color changing and surrounding environment changing and so on. Therefore, the 90° scattering angle is identified as the test direction of the scattering light.

Fig. 8a shows the S/T of 90° vary with the suspended fluid concentration, vertical axis indicates  $\ln[I_s/(I_s + I_t)]$ . As seen from the figure, 0 to 5%, the curve is nearly linear; while more than 5% the curve is bending. So we can define the measure range of S/T as 0-5%.

Based on Lambert-Beer transmitting theory, we can obtain the formula of concentration N following equation (11):

$$N = -\frac{1}{kal} \ln(I_t/I_0). \tag{13}$$

Where  $I_t$  is the transmitting signal, 1/kal is constant, N has a inversely-proportional relationship with  $\ln(I_t/I_0)$  and a proportional relationship with  $\ln(I_0/I_t)$ . We use  $\ln(I_0/I_t)$  to deal with data. Fig. 8b shows the condition of transmitting signal of 90° vary with the suspended fluid concentration. As seen from the figure, 0 to 2%, there is a little bending in the curve, while more than 2% the curve is nearly linear. So the measurement range of S/T method is determined as 5–10%.



**Fig. 9.** (a) The linear that S/T and concentration for 0-5%; (b) The linear that transmitting light and concentration for 5-10%.

Fig. 9a is the calibration curve of the equipment that S/T and concentration for 0-5% with slope k = 0.6681, intercept coefficient b = -3.9590, fitting related coefficients is 0.9984; while Fig. 9b is the calibration linear that transmitting light and concentration for 5-10% with slope k = 0.2612, intercept coefficient b = -0.5614, fitting related coefficients is 0.9967.

## CONCLUSION

Based on the theory of light scattering and transmitting, for the concentration measurement of small sample, this paper presents a detect equipment with fiber that combines S/T method and transmitting method. Under the room temperature, a number of tests are carried and experimental findings are analyzed, the result indicates that 0-5%, the curve is nearly linear by using the S/T method; 5-10%, the curve is nearly linear by using the transmitting light method. Besides, the experiment is repeatable and the maximum relative error is less than  $\pm 0.2\%$ . The equipment enables rapid measurement of small sample and has a good application prospect in the field of MEMS.

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