

# Design and implementation of measurement system for milk fat content based on optical fiber sensor

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The aim of this paper is to design an optical fiber sensing system for determination of fat content in milk. The measurement system is composed of light source, Y-type optical fiber, photodetector, thermoelectric cooler (TEC), preamplifier, second-level amplifier, A/D converter, microprocessor, data storage module, data display and key module. According to Mie light scattering theory, absorbency is adopted as the optical parameter representing the milk fat content; Homogenization is realized by TEC to ensure that the scattering coefficient keeps invariant; Based on the standard model of the system established beforehand, the processor can calculate the fat content in milk. Through calibration and prediction, we can choose the best testing temperature. Results of this study indicate the feasibility and real-time performance of using this measurement system for milk fat analysis.

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*Keywords:* Fat content, Y-type optical fiber, EDTA, Data modeling

## 1. Introduction

Milk is a popular nutritional food and is of particular interest due to importance in the dairy products market. Fat and protein are two nutrients of milk, and their concentrations are routinely monitored during the commercial production and in the final product. The fat in milk belongs to natural fat which is easy for the body to absorb because to rich short and medium chain fatty acids [1]. It is also a carrier of fat-soluble vitamins [2]. So a fast and accuracy quantificational method for the detection of components in milk is needed [3].

Currently, the most widely used methods for detecting the fat content in milk mainly include chemical analysis method, middle-infrared spectroscopy method, near-infrared spectroscopy method and ultrasonic method. There are mainly four chemical analysis methods: Rose-Gottlieb method, Geber method, Babcock method and Tesa method [4-5]. Though these methods achieve higher detection accuracy, they are time and labor consuming, high cost and off-line by nature [6]. At present, near-infrared spectroscopy method [7-8], middle-infrared spectroscopy method [9-10] and ultrasonic method [11] have become the mainstream directions for fat content determination relying on high speed, simple operation and nondestructive measurement. In addition, some other measurements are also used to measure the fat content in milk [12-29]. In [13], Gaustavsson et al. indicated that it was possible to design a thermal conductivity probe, capable of recording the fat content in milk with a sensitivity of better than 0.1%, within a total measurement time of 1 s. In [18], Mabrook et al. confirmed that the milk

conductance was predominantly determined by the salt fraction, while the presence of fat resulted in a decrease in the milk conductance with increasing fat content. In [20], Nunes et al. studied the complex permittivity of milk at room temperature. The variation of six parameters with fat content suggested that they might be useful to roughly determine the milk fat content.

Milk is a concentrated dispersion with eighty-five to ninety percent of water and ten to fifteen percent of dry matter. Among them, lactose and inorganic salt can dissolve in milk, fat can form a stable emulsion in milk and protein can form a suspending liquid. According to Mie theory, only the big globules of the fat and protein can cause obvious light scattering. The light scattering of the other particles can be ignored due to their small grain size and low concentration [30]-[32]. Therefore, the scattering light can absolutely determine the content of the fat and protein in the milk.

The intensity of scattering light is closely related to the number, size distribution of particles, and consequently, the respective component content [33-34]. The scattering coefficient is also correlated with the size of the particles tested. The standard model of the measurement system depends on the intensity of scattering light and the scattering coefficient of the sample. However, the grain size of milk fat globule will vary in a large range with the breeds and feeding season of the cows [35]. Therefore, milk samples must be homogenized before testing to ensure the unique standard model [36-37]. Homogenization refers not to uniform mixing, but means that the particle size distribution of the milk samples should be identical through increasing temperature and



the wavelength of 1060 nm was selected as the light source, and the matched avalanche diode was selected as the photodetector.

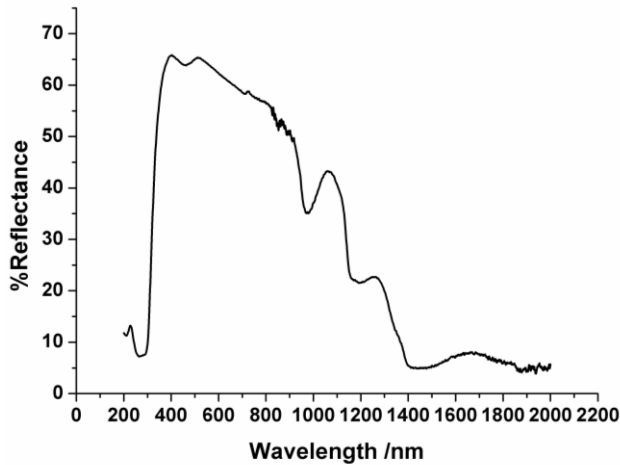


Fig. 2. Reflectance spectra of milk.

The particle size of fat ranges from 0.1 $\mu$ m to 10 $\mu$ m with a mean of 4 $\mu$ m for 100% homogenised milk, protein particle size is typically an order of magnitude smaller [39]. According to the scattering theory, when the size of the scattering particle is less than one fifth of the light wavelength, this type of scattering belongs to Rayleigh scattering; When the size of the scattering particle is larger than three tenths of the light wavelength, this type of scattering belongs to Mie-scattering [34]. The intensity of Mie-scattering is much more stronger than that of Rayleigh scattering. Considering of the characteristics of the milk and the light source, the scattering of fat particle belongs to Mie-scattering basically and the scattering of protein particle (casein in the main) belongs to Rayleigh scattering. According to the analysis above, the scattering spectra mainly includes the information of fat content. So the fat content is suitable for measuring in the mode of scattering [40]-[41].

### 2.3.2 Y-type optical fiber

A schematic of the structure of the Y-type optical fiber is shown in Fig. 3. Quartz optical-fiber bundle with a diameter of 2.1mm was used in the input terminal of the fiber. Each fiber has a core diameter of 0.1mm and a numerical aperture of 0.22. Glass optical-fiber bundle with a diameter of 2.1mm was used in the output terminal. Each fiber has a core diameter of 0.05mm and a numerical aperture of 0.37. In order to enhance the coupling efficiency, a self-focusing optical fiber with a length of 20mm was respectively used on the input port and the output port of the fiber. The fiber probe had a composite structure of above mentioned fibers. In order to decrease the spot radius and collect more scattering light, the

self-focusing optical fiber was also used on the port of fiber probe. A shielding ring with a shape of trumpet could be fixed on the probe of the Y-type optical fiber for reducing the influence of the stray light. The inner wall of the shielding ring should be coated with black pigment to absorb the stray light.

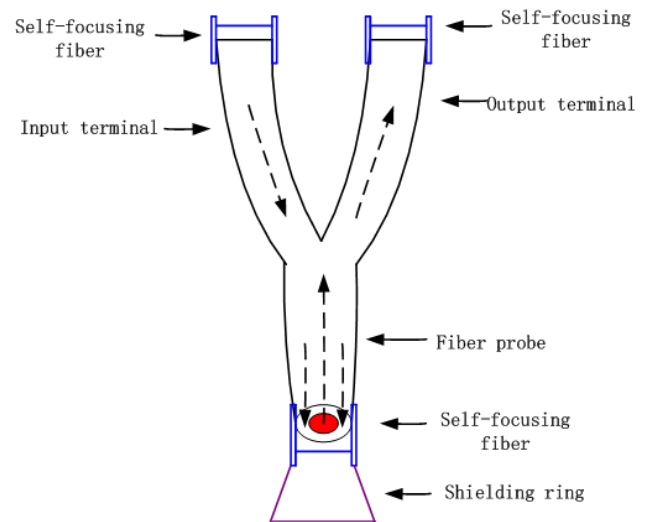


Fig. 3. Schematic of the Y-type optical fiber.

### 2.3.3 Preamplifier and second-level amplifier

In this paper, amplification of the faint signal is realized by a high gain CMOS operational amplifier (ICL 7650). Fig. 4 (a) gives the scheme of the preamplifier based on ICL7650.  $I_S$  is the tested current output by the photodetector;  $R_2$  together with  $R_3$  and  $R_4$  constitute the T-network to increase the stability of high gaining and reducing the noise. Subsequently, the output voltage  $V_O$  can be expressed as follows:

$$V_o = -I_S \left( R_2 + R_4 + \frac{R_2 R_4}{R_3} \right) \quad (1)$$

In order to diminish the drift of the operational amplifier and fluctuation of the light source, a reference signal is adopted in the system. The amplification circuit used in the reference circuit is the same as that used in the measurement circuit shown in Fig. 4 (a). Compared to the measurement branch, the reference branch doesn't receive the optical signal reflected by the sample. The second-level amplification circuit is used to amplify the difference between the measurement signal and reference signal. ICL7650 is also adopted as the amplifier of the second-level amplification circuit. Fig. 4(b) gives the scheme of second-level amplifier. The second-level amplifier adopts differential amplification and the output voltage  $V_{out}$  can be expressed as follows:

$$V_{out} = \left(1 + \frac{R_3}{R_1}\right) \left(\frac{R_4}{R_2 + R_4}\right) V_1 - \frac{R_3}{R_1} V_2 \quad (2)$$

where  $V_1$  is the output voltage of the preamplifier of the measurement circuit,  $V_2$  is the output voltage of the preamplifier of the reference circuit. Make sure that the values of  $R_1$  and  $R_2$  are identical, and the values of  $R_3$  and  $R_4$  are identical. Consequently, the expression of the output voltage  $V_{out}$  is shown as follows:

$$V_{out} = \frac{R_3}{R_1} (V_1 - V_2) \quad (3)$$

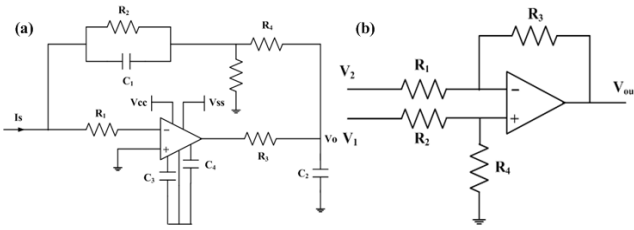


Fig. 4. Two-stage amplifier of weak photocurrent (a) the preamplification circuit; (b) the second-level amplification circuit.

### 2.3.4 TEC temperature control module

Thermoelectric cooler TEC-12704 whose working voltage is 12V and maximum working current is 2A was used here. TEC-12704 is driven by the chip LMD18200 which is a H-Bridge designed for motion control application. The LMD18200 whose working voltage is up to 55 V and maximum working current is 3 A can output bipolar current so that the TEC-12704 can be changed in the mode of heating or refrigerating optionally. DS18B20 was adopted as the temperature sensor of the system. The microprocessor can control TEC-12704 to refrigerate or heat the sample according to the difference between the real-time temperature and the required temperature. So the sample temperature can be fixed at required temperature quickly and accurately. Fig. 5 shows a schematic diagram of the TEC temperature control module.

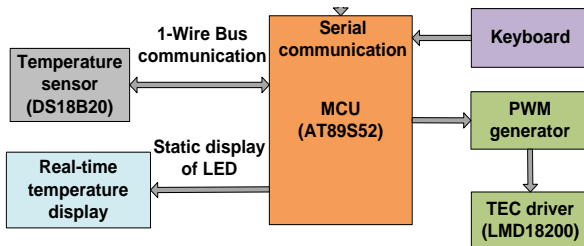


Fig. 5. Schematic diagram of the TEC temperature control module.

## 2.4 Modeling and measurement principle

### 2.4.1 System modeling

Diffuse reflectivity ( $R$ ) which represents the ratio of diffuse reflection beam intensity to incident intensity can be expressed as:

$$R = 1 + \frac{K}{S} - \left[ \left(\frac{K}{S}\right)^2 + 2\left(\frac{K}{S}\right) \right]^{1/2} \quad (4)$$

where  $K$  is absorptivity and  $S$  is scattering coefficient. In practical measurement,  $R$  is not easy to obtain. It is usually replaced by relative diffuse reflectivity which takes a nonabsorbing material in near-infrared region ( $\text{BaSO}_4$ ) as reference and comparison. Relative diffuse reflectivity of the tested sample whose thickness is infinite can be expressed as:

$$R_{\infty} = \frac{R_{\text{sample}}}{R_{\text{ref}}} \approx 1 + \frac{K}{S} - \left[ \left(\frac{K}{S}\right)^2 + 2\left(\frac{K}{S}\right) \right]^{1/2} \quad (5)$$

where  $R_{\text{sample}}$  is the diffuse reflectivity of milk sample and

$R_{\text{ref}}$  is the diffuse reflectivity of reference material. Using

diffuse reflection plate built by barium sulfate as reference

material,  $R_{\infty}$  can be expressed as:

$$R_{\infty} = \frac{V_{\text{sample}}}{V_{\text{ref}}} \quad (6)$$

where  $V_{\text{sample}}$  and  $V_{\text{ref}}$  are the voltages (output by the second-level amplifier) representing the diffuse reflection beam intensity of the milk sample and the reference material, respectively.

Absorbency in diffuse reflection spectroscopy can be expressed as:

$$A = -\lg(R_{\infty}) = -\lg\left(\frac{V_{\text{sample}}}{V_{\text{ref}}}\right) = -\lg\left\{1 + \frac{K}{S} - \left[ \left(\frac{K}{S}\right)^2 + 2\left(\frac{K}{S}\right) \right]^{1/2}\right\} \quad (7)$$

It can be seen from the equation that there is a logarithmic function relationship between  $A$  and  $K/S$ .

The logarithmic function relationship can be replaced by linear relationship in a certain value range of  $K/S$ , namely:

$$A = a_0 + b_0 \frac{K}{S} \tag{8}$$

Results of tissue optics indicated that the relationship between absorptivity ( $K$ ) and the fat content is linear around 1060 nm [42]. In the thin pure fat solutions, the size distribution of fat particle is homogeneous so the scattering coefficient can keep invariant [37]. Therefore, equation (8) can be modified into the following equation:

$$A = a_1 + b_1 c_p \tag{9}$$

Equation(9) can also be written as:

$$c_p = aA + b \tag{10}$$

where  $A$  is the absorbency of the milk sample which can be obtained through equation (7),  $C_p$  is fat percentage content of the thin pure fat solutions,  $a$  and  $b$  are both the equation coefficients. This equation is the modeling principle of the system.

### 2.4.2 Measurement principle

In the thin pure fat solutions, the mean particle sizes of the fat and protein are 4000nm and 120nm, respectively. The other particles dissolve in the milk so the light scattering of these particles can be ignored. Because the contents of the two substances are tested using only one optical parameter (Absorbency), the measurement should be done separately in two steps. Firstly, fresh milk samples were diluted with EDTA solution which can dissolve the protein into small globules. After this operation, the milk contained only one kind of big globules, the fat. Secondly, the fat content could be determined using light scattering method.

The protein in 1.5ml milk can be completely dissolved with 25ml saturated solution of EDTA [25]. Let  $C$  represent the fat percentage content of the milk sample.  $C$  is given as following:

$$C = \frac{m_{fat}}{m_{milk}} \tag{11}$$

where  $m_{fat}$  is the mass of the fat contained in the milk and  $m_{milk}$  is the mass of the milk sample. Let  $C_p$  represent the percentage content of the thin pure fat solution.  $C_p$  is given as following:

$$C_p = \frac{m_{fat}}{m_{milk} + m_{EDTA}} \tag{12}$$

where  $m_{EDTA}$  is the mass of the EDTA saturated solution. According to equation (11) and (12), we can obtain:

$$C = C_p \left(1 + \frac{m_{EDTA}}{m_{milk}}\right) \tag{13}$$

The main purpose of calibration is to establish the standard model of the system. The steps of calibration can be described as follows:

(1) Select 40 different samples with volume of 1.5ml respectively whose fat content has been known and measure the mass of these samples  $m_{milk}$ . Measure out 25ml EDTA saturated solution, weigh it ( $m_{EDTA}$ ) and make 40 copies of this reagent. Transform the fat content of the milk sample  $C$  into the fat content of the thin pure fat solution  $C_p$  using equation (13).

(2) Add 25ml EDTA saturated solution into each milk sample prepared beforehand. Measure the scattering light intensity of each sample output by the second-level amplifier ( $V_{samp}$ ). Meanwhile, measure the light intensity of the barium sulfate plate output by the second-level amplifier ( $V_{ref}$ ). Then, absorbency ( $A$ ) can be calculated by equation (7).

(3) A linear equation can be obtained by data fitting ( $A, C_p$ ) using equation (10). This equation can be used as the standard model and loaded into the processor. According to the standard model, the processor can give the fat content of the milk sample.

The measuring steps can be described as follows:

(1) Measure out 1.5ml milk sample and weigh it ( $m_{milk}$ ); Measure out 25ml EDTA saturated solution and weigh it ( $m_{EDTA}$ ); Add EDTA into the milk sample.

(2) Measure the scattering light intensity of the thin pure fat solution output by the second-level amplification module ( $V_{samp}$ ); Using equation (7), absorbency ( $A$ ) can be calculated; According to standard model established in calibration,  $C_p$  can be calculated.

(3) The fat content of the milk sample ( $C$ ) can be obtained by equation (13).

In order to ensure the stability of the measurement system, we can use the method of multi-times measurement. The A/D converter will obtain 16 data at one time. Removing the largest three ones and the least three ones, we can get an average value among the other ten data. Repeating this process ten times, we can get ten average values. Then a final value, which is output by the A/D converter, can be obtained based on the ten average values. Fig. 6 shows the data processing flow of this system. In Fig. 5, M=16 means the A/D converter will obtain 16 data at one time and N=10 means the process should be repeated ten times.

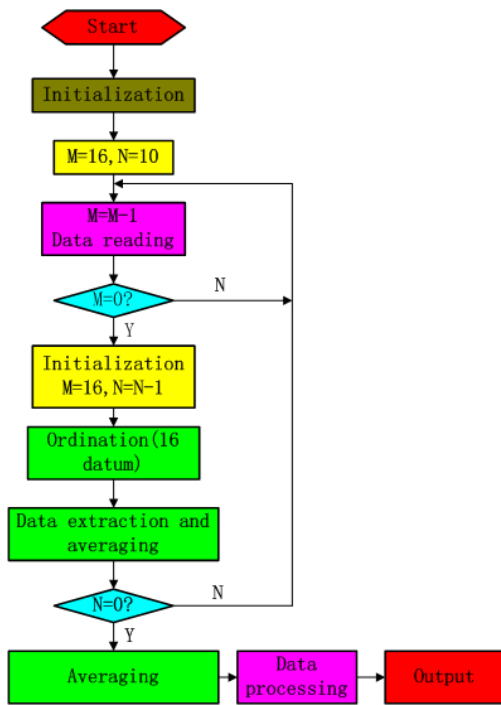


Fig. 6. Data processing flow of the system.

### 3. Results and discussions

#### 3.1 Calibration

According to the principle of system modeling in section 2.4.1, the scattering coefficient of the fat must remain constant so the milk samples must be homogenized before testing. In this paper, the high-temperature type was adopted. We selected 40 fresh milk samples with fat content ranging from 1.2% to 5.3% (Table 2 shows the transformation from  $C$  into  $C_p$ ). Absorbency of these samples were measured at five different temperatures, namely 25°C, 30°C, 35°C, 40°C and 45°C. Then, five one-dimensional linear equations could be obtained by data fitting ( $A, C_p$ ). According to the evaluation parameters of the linear equations, we can select the optimum homogenization temperature.

Table 2. Concentration transformation of milk samples.

$c$ (%)	$c_p$ (%)	$c$ (%)	$c_p$ (%)	$c$ (%)	$c_p$ (%)	$c$ (%)	$c_p$ (%)
5.3	0.308	4.3	0.249	3.3	0.191	2.3	0.133
5.2	0.302	4.2	0.244	3.2	0.185	2.2	0.127
5.1	0.296	4.1	0.238	3.1	0.18	2.1	0.121
5	0.29	4	0.232	3	0.174	2	0.116
4.9	0.285	3.9	0.226	2.9	0.168	1.9	0.11
4.8	0.279	3.8	0.22	2.8	0.162	1.8	0.105
4.7	0.273	3.7	0.214	2.7	0.156	1.7	0.099
4.6	0.267	3.6	0.209	2.6	0.15	1.6	0.093
4.5	0.261	3.5	0.203	2.5	0.145	1.5	0.087
4.4	0.255	3.4	0.197	2.4	0.139	1.4	0.081

Scatter diagrams of absorbency for 40 samples at five temperatures are shown in Fig. 7. The horizontal axis represents the diffuse reflectance absorbency, and the vertical axis represents the fat content of the thin pure fat solution. The fitting standard equations are shown as follows:

Model Equation 1 (25°C):

$$y_1 = -0.527x_1 + 0.331 \quad (14)$$

Model Equation 2 (30°C):

$$y_2 = -0.616x_2 + 0.34 \quad (15)$$

Model Equation 3 (35°C):

$$y_3 = -0.614x_3 + 0.346 \quad (16)$$

Model Equation 4 (40°C):

$$y_4 = -0.621x_4 + 0.381 \quad (17)$$

Model Equation 5 (45 °C):

$$y_5 = -0.519x_5 + 0.337 \tag{18}$$

where  $x_1, x_2, x_3, x_4$  and  $x_5$  are the diffuse reflectance absorbency at above five temperatures,

respectively;  $y_1, y_2, y_3, y_4$  and  $y_5$  are the prediction values of the fat content for the thin pure fat solution. The evaluation parameters of the five model equations are shown in Table 3.

Table 3. Evaluation parameters of model equations.

Model	R	R <sup>2</sup>	SEC	F
1	0.98	0.96	0.0137	917.67
2	0.984	0.969	0.0122	1181.543
3	0.991	0.983	0.009	2169.266
4	0.997	0.994	0.005	6715.212
5	0.987	0.974	0.0114	1412.927

Standard error of calibration (SEC) is a key factor to evaluate the model equation. The smaller the value SEC is, the better the model equation is. Equation 4 possesses the minimum value of SEC. The correlation coefficient R and determination coefficient R<sup>2</sup> of the five equations are all near to 1 and the Equation 4 possesses the minimum values of R and R<sup>2</sup>. The significance test demonstrated a significant correlation between the absorbency and the fat content of the thin pure fat solution in all of the five equations at 5% level of significance.

It can be seen from these analyses that equation 4

gives the best modeling effect. There are reasons as follows: when the temperature is low, fat globules are easy to bond together, thus causing uneven distribution. If the samples are not homogenized at higher temperature, the model equation will be partially inaccurate; when the temperature is high, the membrane of the fat globule will be destroyed, thereby affecting the measurement accuracy. Therefore, the error caused by the temperature difference can not be ignored. Considering the evaluation parameters of model equations, the optimum temperature adopted in the test is 40 °C.

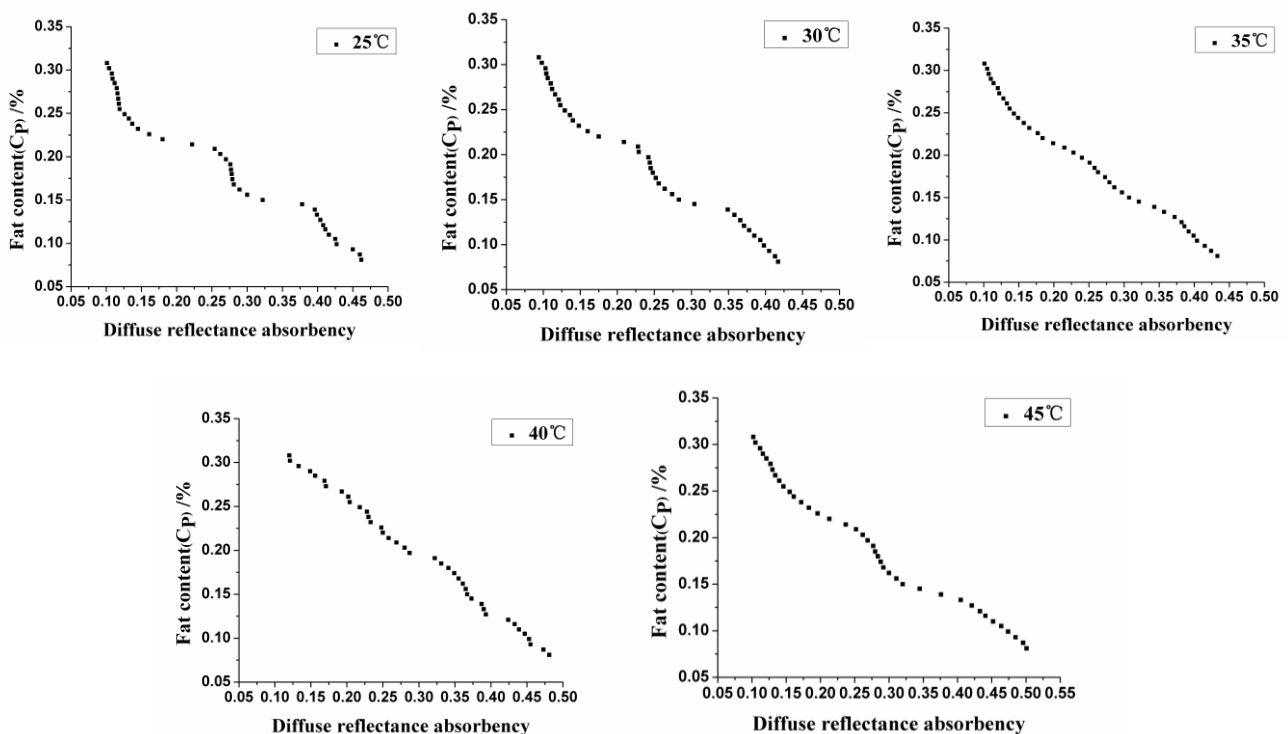


Fig. 7. Scatter diagrams of absorbency for 40 samples at five temperatures.

### 3.2 Predication of fat content

In order to verify the measurement accuracy of the system, the fat concentration predication were carried on here. We prepared ten milk samples with volume of 1.5ml respectively, added 25ml EDTA saturated solution into each milk sample, calculated the absorbency of each

sample at five temperatures using equation (7) and then adopted the model equations 1-5 to predict the fat content of the thin pure fat solution ( $c_p$ ). Finally, we could obtain the fat content predictive values of the milk samples ( $c$ ) by equation (13). The data of the test samples are shown in Table 4.

Table 4. Experimental design of milk sample composition.

$c$ (%)	Mass of the milk sample-1.5ml (g)	Mass of EDTA-25ml (g)	$c_p$ (%)
3.85	1.5403	25.005	0.223
3.45	1.5392	25.005	0.2
3.25	1.5387	25.005	0.188
2.95	1.5382	25.005	0.171
2.65	1.5375	25.005	0.153
2.35	1.5367	25.005	0.136
2.15	1.5364	25.005	0.124
1.95	1.536	25.005	0.113
1.65	1.5354	25.005	0.096
1.35	1.5348	25.005	0.078

Prediction evaluation parameters of the five model equations are shown in Table 5 and the prediction results are shown in Table 6. The linear dependence of fat content obtained by the light scattering method with that obtained by the reference method is shown in Fig. 8. It can be seen from Table 5 that model equation 4 possesses the minimum value of standard error of prediction (SEP) and the maximum value of prediction correlation coefficient ( $r_p$ ). Fig. 8 and Table 6 demonstrate that model equation 1 and model equation 5 have large prediction errors. The reasons are as follows: When the temperature is low, fat globules are easy to float upward because to uneven distribution, thus causing measuring deviation; When

temperature is high, the deviation is mainly resulted from the destruction of fat globule and water evaporation.

From all the data shown in Fig. 8, it is clear that equation 4 possesses the minimum measuring deviation. Prediction data of the equation 4 are shown in Table 7. The results show that every difference value is smaller than 0.14%, and the root mean squared difference is 0.081%. The optimum testing temperature selected in the prediction experiment is in coincidence with that selected in the calibration experiment. Therefore, we can use the equation 4 to realize fat content measurement, that is to say, the measurement must be implemented at constant temperature of 40°C.

Table 5. Prediction evaluation parameters of model equations.

Model	1	2	3	4	5
$r_p$	0.952	0.979	0.989	0.996	0.977
SEP	0.014	0.0095	0.0068	0.0044	0.01



Table 6. Prediction results of the five model equations.

Fat content $c$ (%)	Prediction value (%)				
	Model 1	Model 2	Model 3	Model 4	Model 5
3.85	4.13	4.05	4.05	3.9	3.96
3.45	3.89	3.32	3.52	3.53	3.42
3.25	3.21	3.27	3.27	3.11	3.32
2.95	3.17	3.17	3.05	2.83	3.21
2.65	2.92	2.88	2.74	2.65	2.98
2.35	2.09	2.13	2.22	2.41	22.26
2.15	2.03	2.24	1.97	2.07	2
1.95	1.95	2.02	1.85	1.85	1.8
1.65	1.78	1.63	1.65	1.7	1.51
1.35	1.59	1.29	1.26	1.38	1.24

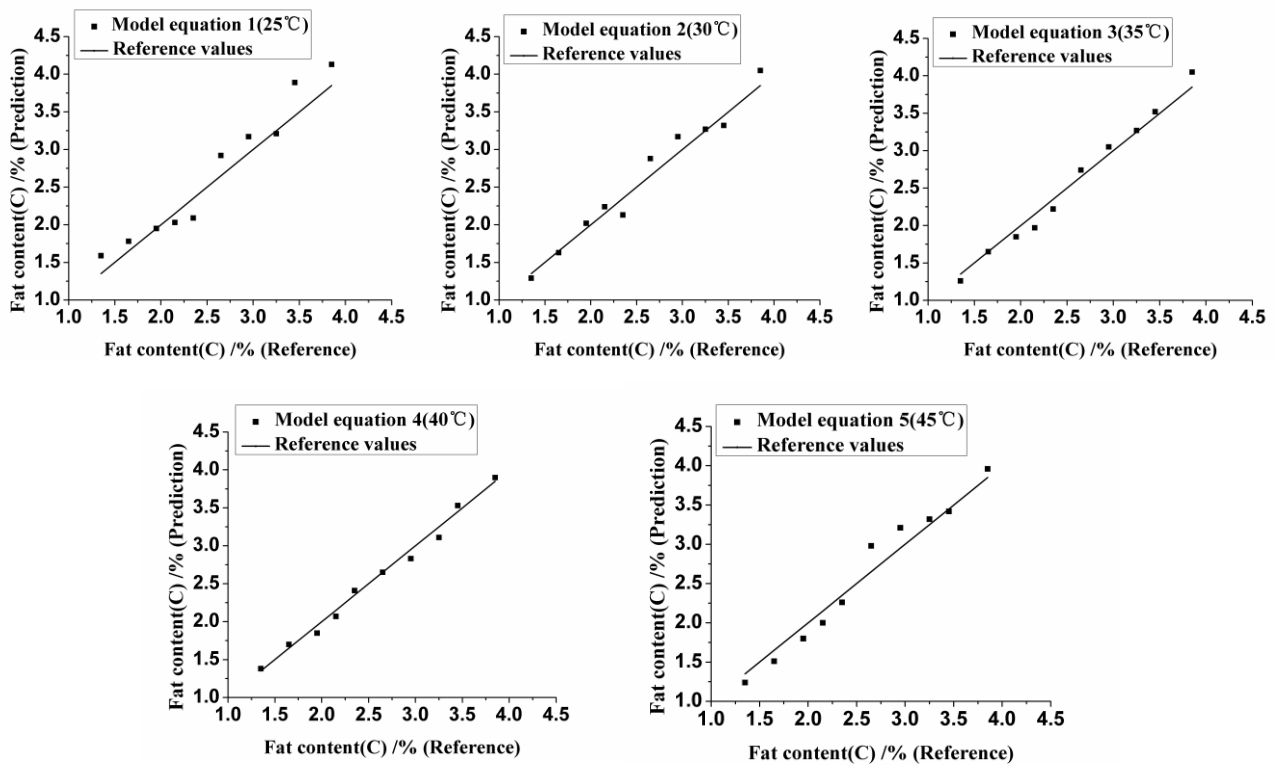


Fig. 8. Scatter diagrams of fat content prediction for five model equations.

Table 7. Measurement results of the model equation 4.

$c$ (%)	$c_p$ (%)	Absorbency	Prediction of $c_p$ (%)	Prediction of $c$ (%)	Error(%)
3.85	0.223	0.249	0.226	3.9	0.05
3.45	0.2	0.284	0.205	3.53	0.08
3.25	0.188	0.323	0.181	3.11	0.14
2.95	0.171	0.349	0.164	2.83	0.12
2.65	0.153	0.366	0.154	2.65	0
2.35	0.136	0.389	0.139	2.41	0.06
2.15	0.124	0.421	0.12	2.07	0.08
1.95	0.113	0.437	0.11	1.85	0.1
1.65	0.096	0.455	0.098	1.7	0.05
1.35	0.078	0.485	0.08	1.38	0.03

#### 4. Conclusions

A new-style measurement system for fat content in milk based on Y-type optical fiber is explained in this paper. EDTA saturated solution is added into the milk to dissolve the protein in milk before testing. Constant temperature control for homogenization is realized through TEC to ensure that the scattering coefficient remains constant.

According to the measurement results at different temperatures, this system achieves the best effectiveness at the temperature of 40°C: the maximum error is 0.14% and the root mean squared difference is 0.081%.

With the advantages of miniaturization, strong adaptability and high accuracy, the system can realize the measurement of milk fat content indoor or outdoor, and it can also be used to control other equipment to filter the inferior products. The system designed here can also be applied to measure the content of major compound in the other kinds of latex.

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