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Moisture content online measurement in the sludge by ultrasonic reflection method

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Abstract

Moisture content is an important parameter of solid waste degradation in landfill. The traditional gravimetric method used for determining the moisture content of sludge is very time-consuming and cannot achieve online measurement of sludge moisture content. This paper proposes an ultrasonic reflection method to measure the moisture content of sludge online, the sludge only needs to be dried once, and then the online measurement can be realized. The specific process is as follows: by analyzing the ultrasonic characteristic parameter under different moisture content, the quantitative relationship between them can be obtained, and then the sludge moisture content can be deduced by the ultrasonic characteristic parameter. In this paper, the relationship between the ultrasonic characteristic parameter of the three sludge and the moisture content was analyzed, and the quantitative relationship was determined. The sludge moisture content calculated by this method is very close to the actual value. This method provides a new research idea for the online measurement of sludge moisture content.

Keywords: Moisture content; Sludge; Ultrasonic reflection

1. Introduction

Sludge is the product of sewage treatment, and the moisture content is generally very high. The percentage of the weight of the moisture content in the sludge to the total weight is the sludge moisture content ^{1,2}. Usually, when the moisture content is above 85%, the sludge is in a fluid state; when it is 65% to 85%, it is in a plastic state; when it is lower than 60%, it is in a solid state. The raw sludge will undergo a series of harmless treatment after production, making the moisture content of the sludge gradually reduced to meet the sewage sludge discharge standards of municipal sewage treatment plants ³. Decreasing the sludge moisture content, however, consumes a large amount of flocculant and increases the cost of the treatment plant. Thus, the real-time determination of the sludge moisture content is very important to save as much money as possible while meeting emissions standards.

The methods that can be used to determine the moisture in wood chips, biomass, seeds and other materials are diverse, but few can be applied to sludge ⁴⁻⁶. Determination of the moisture content by drying the sludge is nowadays an accepted method ^{7, 8}. This method is well established and does not demand sophisticated laboratory equipment, but it is time-consuming and cannot truly meet the operational requirements of sewage treatment plants. It is vital to come up with a method that can measure the moisture content of the sludge in real time.

The ultrasonic methods are attracting more and more attention because of its feature of strong penetration, wide frequency range and on-line non-contact measurement⁹. Ultrasonic methods can mainly be divided into reflection methods and transmission methods according to the different mechanisms and probe arrangements, and have been widely used in nondestructive testing, multiphase flow rate and flow pattern measurement, particle

concentration and size distribution in industry¹⁰⁻¹². Compared with transmission methods, ultrasonic reflection method has the advantages of no effect on the flow field, easy to operate, and more suitable for industrial sites^{13, 14}. Therefore, ultrasonic reflection method is chosen to measure the moisture content of the sludge in real time in this paper.

A novel method for measuring the moisture content of sludge based on ultrasonic reflection is proposed in this paper, with plexiglass as the material of the measuring container. The basis of the method is that ultrasound will be reflected at the interface when it passes through two media with different acoustic resistances¹⁵. That is, when the ultrasound enters the sludge through the wall of the plexiglass container, reflection will occur on the wall of the plexiglass container due to the different acoustic resistances of the plexiglass and the sludge. As the moisture content of the sludge changes, so does the intensity of the reflection. Three types of sludge (pharmaceutical sludge, municipal sludge and tannery sludge) were studied in this paper to analyze the relationship between reflection intensity and moisture content. The results showed that there was a linear relationship between them for the three sludge types, thus confirming the feasibility of the method. However, the sludge in solid state cannot completely fit the wall of the plexiglass container, and the measurement results will be affected in the presence of air. As a result, the measuring range of the method is limited to fluid and plastic sludge, and cannot be applied to sludge in solid state. Although the range of moisture content is limited, this method provides a new research direction for the online measurement of sludge moisture content and has high industrial value.

2. Methodology

2.1 The principle of measurement

Sound is the propagation of vibrations through a medium, and unlike electromagnetic waves, the propagation of sound waves requires an elastic medium. Depending on the frequency, sound waves can be divided into infrasound (frequency less than 20 Hz), audible sound (frequency between 20 Hz and 20 kHz) and ultrasound (frequency greater than 20 kHz). Ultrasound is a kind of high-frequency sound wave, which has the advantages of good directivity, strong penetration ability, and focused power. The unique advantages of ultrasonic waves make it widely used in many fields such as chemical, mechanical and medical industry.

When ultrasonic waves propagate through a medium, they interact with the medium and a variety of physical and chemical reactions occur. Reflection occurs at the boundary, when the ultrasonic wave enters another medium with different acoustic resistances from one medium¹⁶. Acoustic impedance refers to the damping and confrontation of energy propagation by the medium or sound transmission structure, which can be divided into two parts, acoustic resistance as an important part, is related to friction and is expressed in acoustic systems as the effect of a pore barrier on sound. Acoustic resistance is the product of density and sound velocity, as shown in Equation (1). The reflection coefficient is obtained by the acoustic resistance of the two, as shown in Equation (2)¹⁷,

$$Z = \rho c \quad (1)$$

$$R = (Z_2 - Z_1) / (Z_2 + Z_1) \quad (2)$$

where subscripts 1 and 2 refer to the original propagating medium and the medium that reflects the sound waves, respectively.

The measurement method in this paper is based on ultrasonic reflection. The larger the reflection coefficient R , the larger the received echo signal, the higher the signal-to-noise ratio,

and the more accurate the result ¹⁸.

2.2 Materials

Three different kinds of sludge were selected for the experiment, namely municipal sludge, tannery sludge and pharmaceutical sludge. First, the density of the sludge was determined by dividing the weight by volume. The weight of an empty container with a volume of 50 mL is measured as m_1 , then the container is filled with sludge, and its weight is measured again as m_2 . The change in weight reflects the weight of the 50 mL sludge, then the density of the sludge can be calculated.

$$\rho = \frac{m_2 - m_1}{50 \text{ mL}} \quad (3)$$

The sludge is then placed into a cylindrical plexiglass container, which has the radius of 5 cm, the height of 2 cm and the thickness of 1 mm. The transceiver is placed on the sludge and is used to transmit and receive ultrasonic signals. The transducer, signal generator that control transmission and reception, and data acquisition card are the same as those in Section 3. The emitted ultrasonic waves will encounter the upper side wall of the container after passing through the sludge, and will be reflected by the lower side wall. By comparing the time point of transmitting the signal t_1 and receiving the signal t_2 , the time of ultrasonic propagation in the sludge can be obtained. It should be noted that the distance that ultrasound passes through during this time is 4 cm, because the ultrasound propagates to the wall and is reflected back by the wall. The sound velocity in the sludge can then be calculated by dividing distance by time (as shown in Fig.1).

$$c = \frac{4 \text{ cm}}{t_2 - t_1} \quad (4)$$

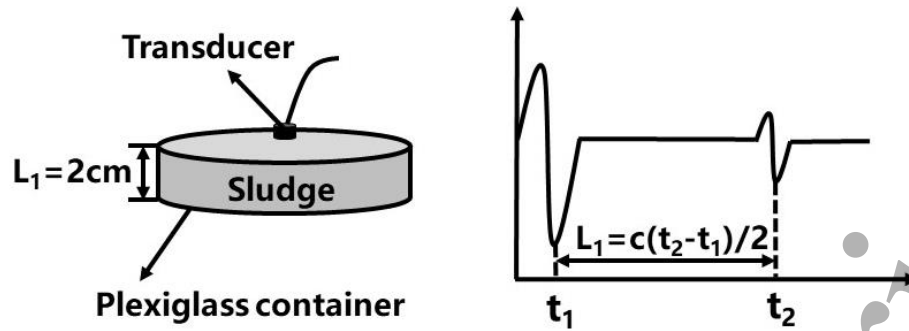


Fig. 1. Schematic of sound velocity measurement in sludge

Finally, the density and sound velocity of the three types of sludge obtained are shown in Table 1.

Table. 1 The density and sound velocity of the three types of sludge.

Materials	Density (kg/m ³)	Sound velocity (m/s)	Acoustic resistance
			(10 ³ kg/(m ² .s))
Municipal sludge (Sludge 1#)	1841	1573.96	2897.66
Tannery sludge (Sludge 2#)	1824	1573.94	2870.87
Pharmaceutical sludge (Sludge 3#)	1860	1573.56	2926.82

The dried sludge was ground through a 200-mesh screen and elemental analysis was performed (Table 2) to characterize three types of sludge more accurately. XRF analysis of ash was then carried out by Axios wavelength dispersive X-ray fluorescence spectrometer, and the results are shown in Table 3. The analysis shows that there is little difference in the elements of the three types, and the elements are mainly calcium, iron, chlorine and carbon, among which

the calcium is the most abundant element of the tannery sludge, while the iron occupies the highest content in the municipal and pharmaceutical sludge¹⁹.

Table. 2 Elemental analysis of three kinds of sludge.

Elemental analysis /%	C	H	N	S	O
Tannery sludge	21.88	3.23	2.67	2.21	17.77
Municipal sludge	27.51	4.29	6.89	0.74	13.96
Pharmaceutical sludge	22.32	3.47	3.04	0.58	19.39

Table. 3 XRF analysis of three kinds of sludge.

XRF/%	Ca	Fe	Cl	Na	Si	Al	P
Tannery sludge	36.09	33.75	1.26	2.84	6.56	3.95	2.12
Municipal sludge	14.54	38.89	0.0116	0.399	19.91	7.09	7.48
Pharmaceutical sludge	18.1	33.05	0.12	1.79	2.03	31.58	8.25
XRF/%	Mg	Cr	Ti	K	Mn	Sr	Zn
Tannery sludge	1.6	1.49	1.05	0.573	0.244	0.126	0.139
Municipal sludge	3.21	0.0638	1.77	3.54	0.383	0.301	0.562
Pharmaceutical sludge	0.6	0.072	0.093	0.145	0.189	0.062	0.854

Then, a thermos-gravimetric analyzer (NETZSCH TG209 F1 Libra) was employed to conduct a thermal gravimetric analysis of the sludge samples after drying, with the temperature range of 50-900°C and the rise rate of 20°C/min^{20, 21}. FIG. 2 shows the thermos-gravimetric (TG) and derivative thermos-gravimetric (DTG) curves of the three types of sludge.

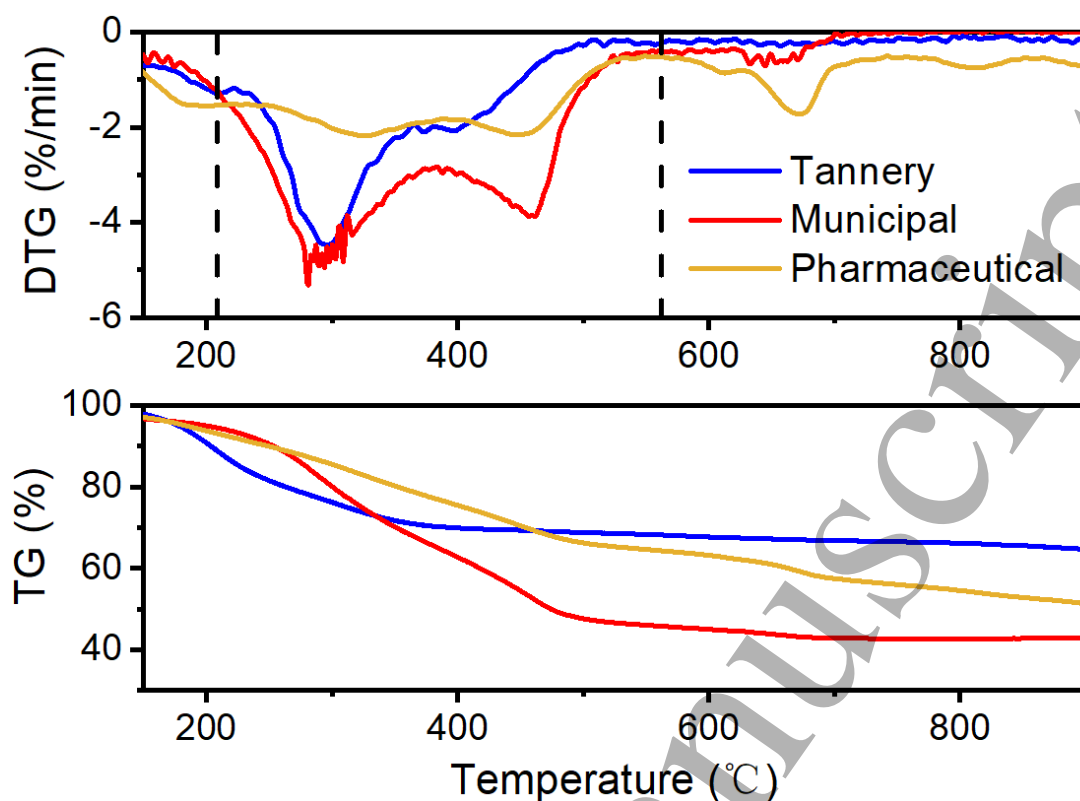


Fig. 2. DTG and TG curves of sludge at the heating rate of 20°C/min

As shown in the differential heat curve, the pyrolysis process of sludge can be divided into three stages. The first stage is the dehydration process of the sample, the water in the sludge is heated to evaporate, because the sample has been dried before, so the evaporation of this process is mainly bound water, and the weight loss rate is not high. When the temperature rises to 208°C, the second stage is entered, and the organic matter in the sludge is decomposed by heat into light oil, bio-char and small molecular biogas, and the weight loss rate is the highest in this stage. Tannery sludge has an obvious weight loss peak (about 300 °C), which can be understood as having an important organic component decomposed at this temperature, while the other two have two weight loss peaks, which means that more kinds of organic components decompose. The weight loss rate of pharmaceutical sludge is obviously small, indicating that the content of organic components is low. The third stage (when the temperature is higher than 560°C) is mainly the secondary decomposition of solid residue, it can be seen that only

pharmaceutical sludge has a weight loss peak, indicating that there is a high thermal stability of polymer organic matter at high temperature decomposition^{22, 23}.

2.3 Selection of measuring container

In this paper, the ultrasonic waves travel through the walls of the container into the sludge. While considering two media that affect ultrasound reflection, the measuring container is generally regarded as medium 1, and the measured sludge is considered as medium 2, so the acoustic resistance of the container and the sludge affect the reflection coefficient directly. The larger the reflection coefficient R , the larger the received echo signal, the higher the signal-to-noise ratio, and the more accurate the result. Thus, the selection of the measurement container is very important to make the coefficient of reflection R get bigger, and the measurement results more accurate. Since the acoustic resistance of sludge Z_2 cannot be changed, it can be concluded that the smaller the acoustic resistance of container Z_1 , the bigger the reflection coefficient R . Therefore, materials with smaller acoustic resistance are more appropriate for measuring container in this paper.

After the investigation of the sound velocity, density, hardness and other properties of various materials, plexiglass was finally selected as the measuring container with density of 1180 kg/m³ and sound velocity of 2730 m/s. In addition, the plexiglass has a high transparency and helps to make sure the sludge fits closely to the vessel wall during the measurement process. The size of the plexiglass container is 10cm×10cm×15cm, and the wall thickness is 5 mm.

3. Experiments

3.1 Preparation before the experiment

Divide the sludge into 8 parts (marked 1 to 8), then measure the weight of 8 drying trays and

record them as x_n ($n=1-8$), which are used for holding the sludge in the oven. Drying the 8 parts of sludge in an oven, one part of the sludge was taken out every 3 hours. Note that the first part of the sludge ($n=1$) is used as a control group, and does not need to be dried. After 21 hours, 8 parts of the sludge were all taken out separately.

Each sludge that is taken out goes through the following process: first, the sludge was moved to the plexiglass container for measurement, note that at this step, it is essential to make sure that the walls of the sludge to be measured and the plexiglass container fit tightly; Next, the ultrasonic reflection system is used to measure the intensity of reflections, which occur at the interface between plexiglass and sludge. The corresponding reflection intensity is denoted as y_n ; Then the sludge was reloaded into the drying tray and its total weight was measured as z_n ; Finally, the sludge is returned to the oven to dry to constant weight w_n (the weight of the drying tray is included)^{24, 25}.

3.2 Measurement of the reflection intensity

The process of measuring the reflection intensity y_n is completed by the ultrasonic reflection system, which consists of a transceiver integrated transducer, a plexiglass container, a pulse signal generator, a signal acquisition card and a computer.

The pulse signal generator (DPR 300 of JSR Company) can be used to generate high voltage electrical excitation pulses with amplitudes ranging from 100 V to 475 V with repetition rates changed from 100 Hz to 5 kHz. The ultrasonic is reflected back after passing through the wall of the container with a thickness of 5 mm, and the time of such a cycle is

$$T = \frac{5 \text{ mm} \times 2}{2730 \text{ m/s}} = 3.66 \mu\text{s}$$

A repetition rate of 100 Hz (100 cycles of pulse signals emitted per second) can fully meet

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the time precision, and ensure that all useful signals are not lost. Besides, the minimum pulse repetition rate 100 Hz can help to reduce the burden of sampling and subsequent signal analysis. Finally, pulses with an amplitude of 225 V and a repetition rate of 100 Hz were selected in the experiment.

The transceiver (Guangdong Shantou Ultrasonic Electronics Co., Ltd.) is mainly composed of shell, protective film, piezoelectric wafer, sound absorbing material and so on, ultrasonic waves are transmitted or received by piezoelectric wafers. The transducer used in the experiment has a central frequency of 2.5 MHz, and the wafer size of piezoelectric ceramics is 6 mm. The data acquisition card (PCIe 8912 from Beijing ART Technology Co., Ltd.) has a high-speed and reliable sampling rate of 250 MS/s and an analog bandwidth up to 100 MHz, and can fully meet the experimental requirement. It is triggered by the high voltage pulse generator, which makes the signal transmission and reception realize synchronization.

The pulse signal from the high voltage signal generator controls the transceiver integrated transducer, which clinging to the outer wall of the container to emit a certain intensity of ultrasound. Ultrasound will pass through the walls of the container and be reflected when it encounters the sludge in the plexiglass container. At the same time, the pulse signal generator will also send out a trigger signal, prompting the signal acquisition card to start signal acquisition. The reflected signal y_n is received by the integrated transceiver, and collected by the signal acquisition card to the software for subsequent analysis and processing. In this way, the process of measuring the intensity of the reflection is completed. The detailed experimental setup is shown in Fig. 3.

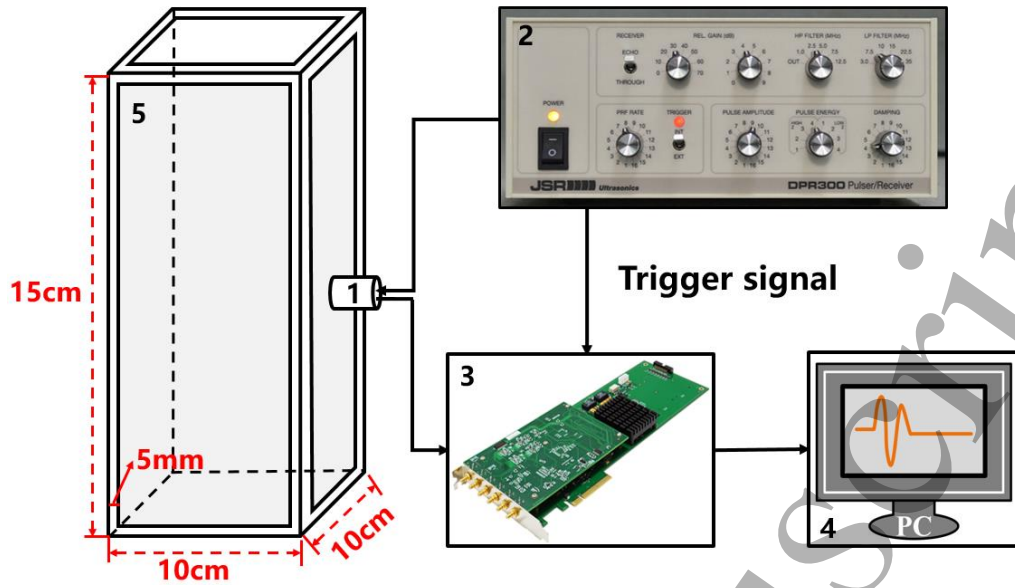


Fig. 3. The detailed experimental setup (1- transceiver integrated transducer, 2- pulse signal generator, 3-signal acquisition card, 4-computer, 5-plexiglass container).

4. Results

As can be seen from Fig. 2, the bound water and macromolecular organic in the sludge start to decompose when the temperature is higher than 200°C. Therefore, the decline of sludge weight with the increase of temperature is thought to be caused by the evaporation of free water. The results can be analyzed accordingly.

The moisture content m_n can be easily obtained by the following equation:

$$m_n = \frac{z_n - w_n}{z_n - x_n}$$

where x_n is weight of drying trays, z_n is total weight of drying trays and sludge, w_n is constant weight after drying, $z_n - w_n$ is the weight of the water that was dried, $z_n - x_n$ is the total weight of the sludge.

Taking sludge 1# as an example, the calculation of the moisture content of the sludge after different drying times is shown in Table 4. It can be seen that the initial moisture content of the

sludge is 84.25%, and after drying for 21 hours, the moisture content of the sludge is gradually reduced to 66.44%. As the sludge continues to be dried, it cannot fit closely to the wall of the measuring container due to its solid appearance, resulting in inaccurate measurement results. Therefore, for the 1# sludge, the relationship between moisture content and reflectance intensity was analyzed in the range of moisture content from 66.44% to 84.25%.

The reflection intensity of the sludge 1#, 2# and 3# under different moisture content are shown in Fig. 4, Fig. 5 and Fig. 6, respectively. The first segment of the signal in each figure can be thought of as a transmit signal, which is only related to the control of the signal generator and does not change with the difference in the measurement environment. The red dash line in the picture shows the reflected signal of ultrasound at the interface between the plexiglass container and the sludge. It can be seen that with the decrease of moisture content, the intensity of the reflected signal increases gradually. This can be explained by the fact that the acoustic resistance of the sludge varies with the moisture content. The density of water is 1000 kg/m^3 , the sound velocity is 1500 m/s , so the acoustic resistance is about $1500 \times 10^3\text{ kg/(m}^2\cdot\text{s)}$, that is, the acoustic resistance of water is much smaller than that of sludge. As the moisture content decreases, the percentage of water in the sludge decreases, the percentage of the sludge increases, and thus the acoustic resistance increases. It can be seen from equation (2) that in the case of the plexiglass container unchanged (Z_1 is the fixed value), the reflection coefficient R increases as the acoustic resistance of the sludge increases (Z_2 becomes larger), which leads to the reflection intensity gets larger.

Table 4: The moisture content of the sludge after different drying times.

n	Weight of drying trays x_n (g)	Total weight of drying trays and sludge z_n (g)	Constant weight after drying w_n (g)	Weight of water in the sludge $z_n - w_n$ (g)	Weight of the sludge $z_n - x_n$ (g)	Moisture content $(z_n - w_n) / (z_n - x_n)$
1 (0h)	9.55	94.83	22.98	71.85	85.28	0.842519
2 (3h)	9.22	80.09	21.43	58.66	70.87	0.827713
3 (6h)	9.56	79.26	23.73	55.53	69.70	0.7967000
4 (9h)	9.38	84.54	27.66	56.88	75.16	0.756786
5 (12h)	9.60	87.00	30.48	56.52	77.40	0.730233
6 (15h)	9.57	92.87	33.05	59.82	83.30	0.718127
7 (18h)	9.53	79.84	31.40	48.44	70.31	0.688949
8 (21h)	9.60	62.04	27.20	34.84	52.44	0.664378

Taking the moisture content and the peak-to-peak value of the reflection intensity as the horizontal and vertical coordinates respectively. Quantitative relationship between reflection intensity and the moisture content of three sludge is shown in Fig. 7. There is a good linear relationship between the reflection intensity and moisture content for the three types of sludge, which proves the feasibility of the method in the measurement of moisture content.

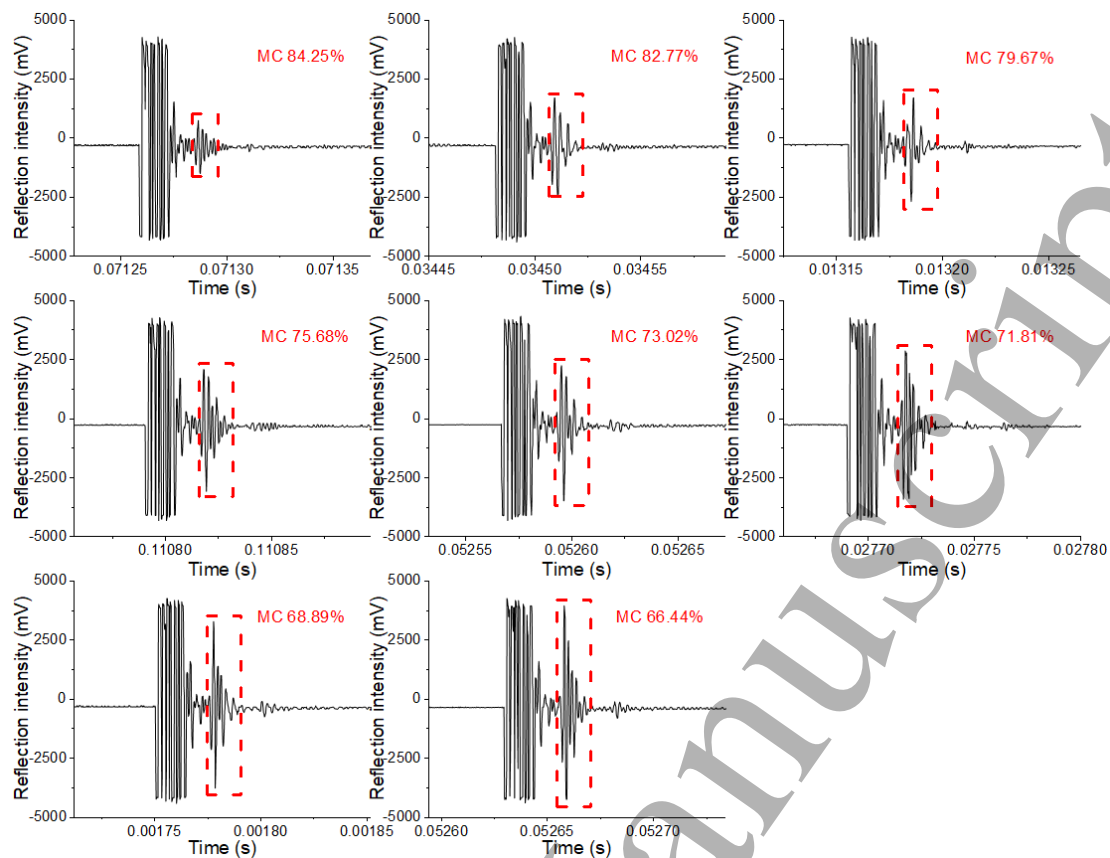


Fig. 4. The reflection intensity of the 1# sludge under different moisture content.

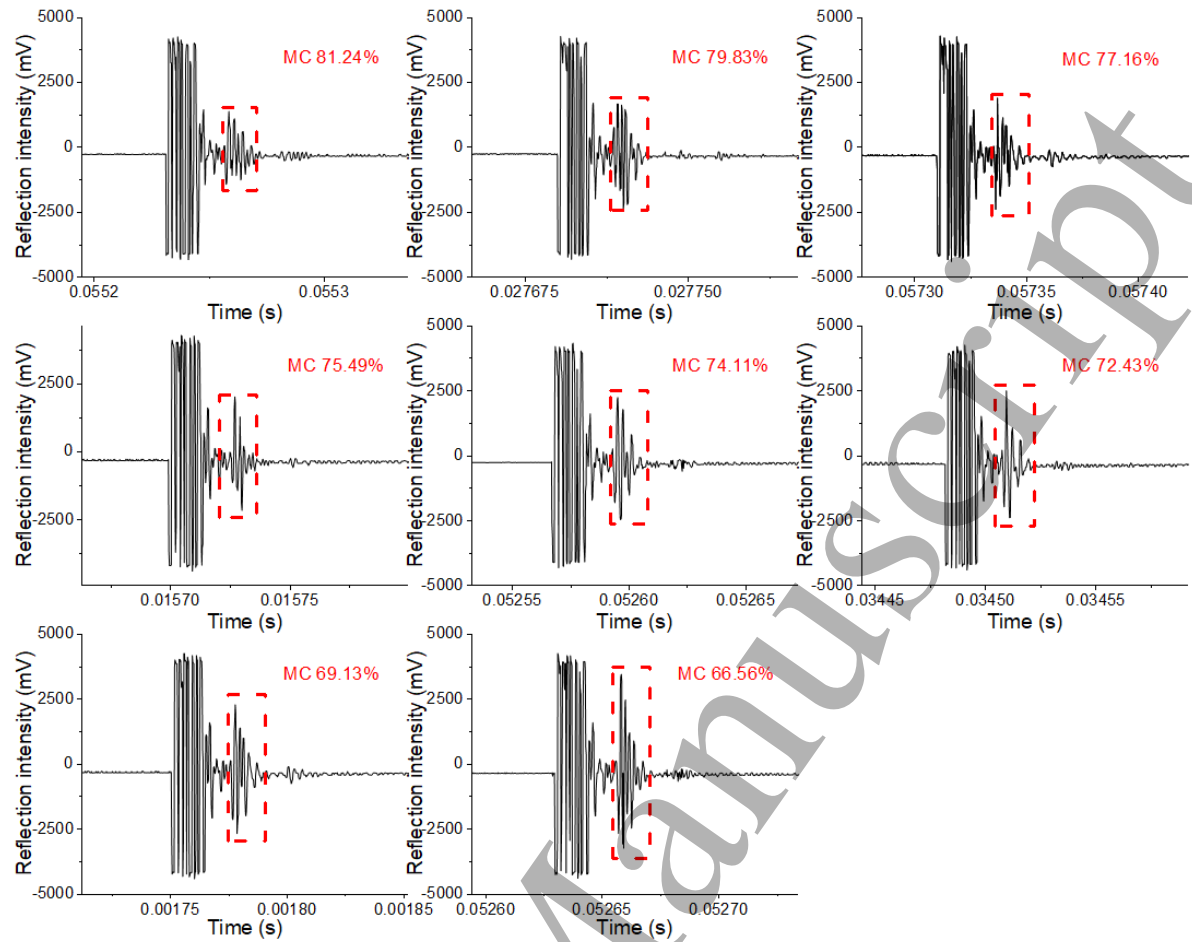


Fig. 5. The reflection intensity of the 2# sludge under different moisture content.

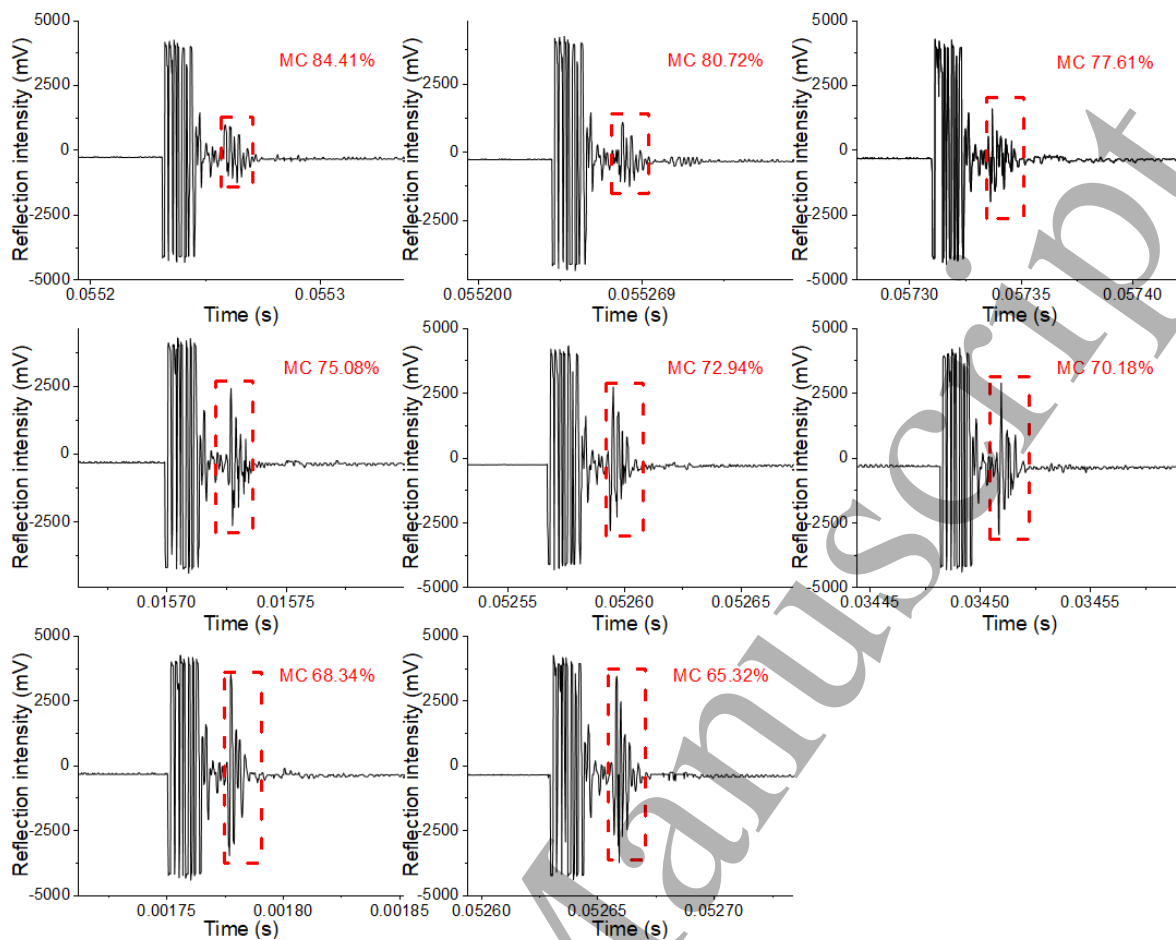


Fig. 6. The reflection intensity of the 3# sludge under different moisture content.

In practice, this method can be used to carry out instant on-line moisture content measurement. First, the moisture content of sludge under different drying time can be calculated directly after drying the sludge, and the reflectance intensity can be obtained with the ultrasonic reflection system, then the linear relationship between the reflection intensity and moisture content is determined. Thus, the moisture content to be measured can be reversed by this linear relationship after measuring the reflectance intensity. The specific analysis process is shown in Fig. 8.

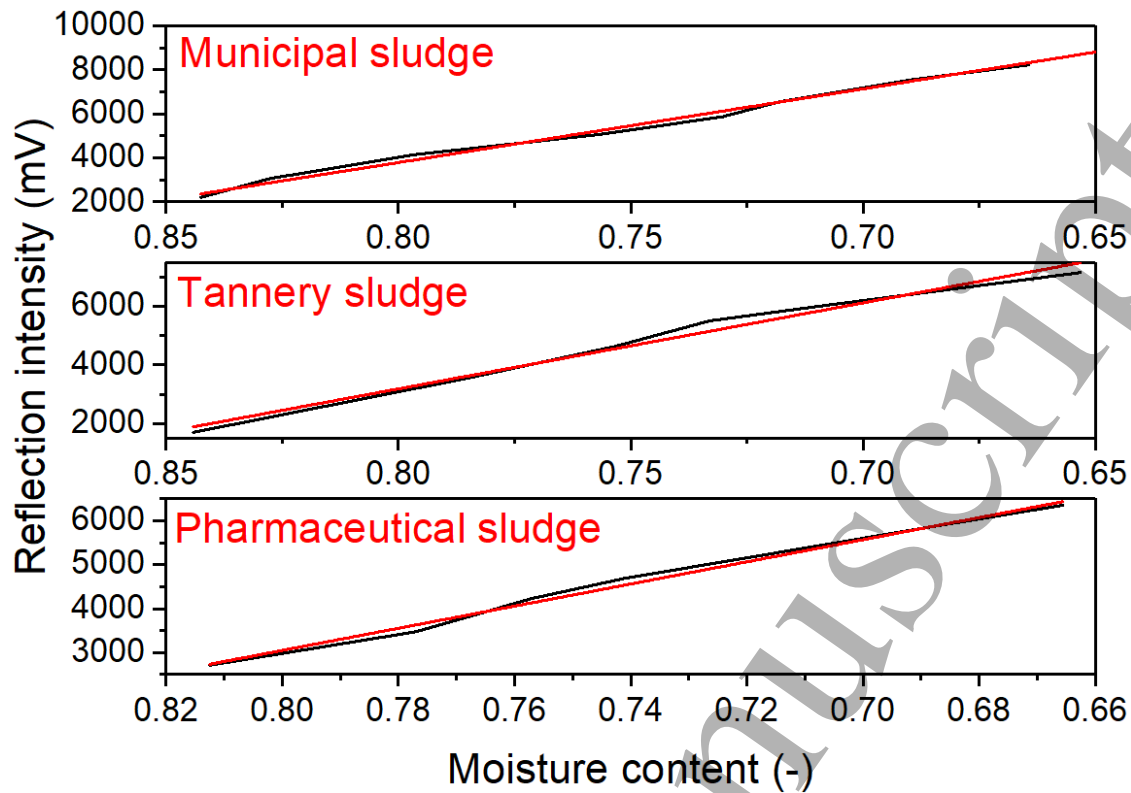


Fig. 7. Quantitative relationship between reflection intensity and the moisture content of three sludge.

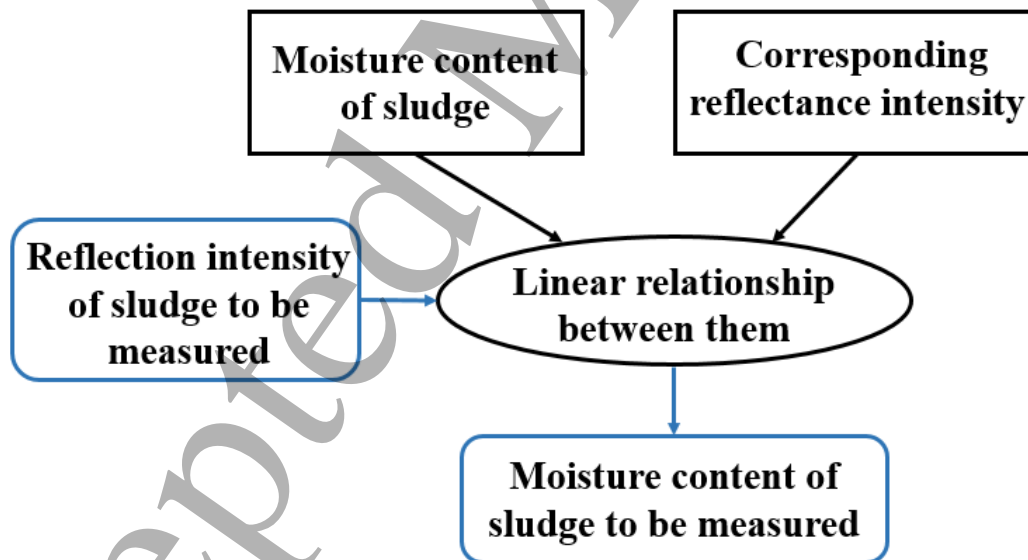


Fig. 8. The specific analysis process in this work

It should be noted that the sludge with the moisture content reduced to about 60% has begun to agglomerate (similar to solid), and its scattering intensity cannot be accurately measured, so the moisture content that can be measured by this method has a certain range.

4. Conclusion

A new ultrasonic method for measuring the moisture content of sludge is presented in this paper. Plexiglass was selected as the material of the measuring container to obtain better experimental results after investigating a variety of materials. The principle of this method is that reflection occurs when ultrasound passes through the plexiglass container and enters the sludge in the container, and the intensity of reflection is related to the magnitude of the acoustic resistance of the two media. Therefore, the reflection intensity can be used to reflect the acoustic properties of the sludge.

Three types of sludge (municipal sludge, pharmaceutical sludge and tannery sludge) were studied in the paper. By analyzing the relationship between the reflection intensity of three different sludge with the change of moisture content, we found that with the decrease of the moisture content, the acoustic resistance of the sludge gradually grows, and the reflection coefficient shows an upward trend. Specifically, there was a good linear relationship between them.

This result provides a new research direction for the online measurement of moisture content. In the industrial site, if it is necessary to obtain the moisture content of sludge in time, the relationship between the reflectance intensity and the sludge moisture content can be determined by this method first, and then the moisture content can be inverted after obtaining the reflective intensity of the sludge to be measured. Compared with the traditional gravimetric method, the method saves a lot of time and electricity, thus has certain industrial value. However, it should be noted that due to the poor fluidity of solid sludge, which cannot fit closely with the measurement container, this method can only be applied to the measurement

of non-solid sludge (moisture content greater than 60%). Subsequent studies will focus on the use of ultrasound in measuring the moisture content of sludge with the moisture content less than 60%.

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Declaration of interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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