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Identification of Moisture Components of Slurry after Pressurized Electro-osmotic Dewatering by Thermogravimetric Analysis

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ABSTRACT

Increasing quantities of slurry from industries and urban construction is one of the societal problems due to the high transportation costs and more restrictive environmental regulations. The slurry contains a large quantity of water; therefore, slurry dewatering is a prerequisite for subsequent utilization. However, the existing mechanical dewatering (MD) technique using only pressure has some limitations to achieve satisfactory water content. Pressure and electric dewatering in simultaneous operation, usually called pressurized electro-osmotic dewatering (PEOD), could serve as a promising method to enhance dewatering efficiency. During the dewatering process, geotechnical properties vary with the moisture content of different components such as free water and absorptive water, consequently influencing the engineering properties of the slurry in the applied environment. However, few studies have investigated the identification of moisture components of slurry after PEOD. Therefore, a method with clear physical significance to identify them is important. This study investigated the identification of different components of soil water by means of thermogravimetric analysis (TGA). The findings demonstrate that PEOD under 20V has better dewatering efficiency with a final moisture content of 32.6%. From the TGA results, we concluded that the quantity of free water remaining in the slurry is as follows: PEOD under 20V < PEOD under 10V < MD. The absorbed water is the most dominant after PEOD under 20V. Overall, the method and results in this study provide a new perspective on determining the slurry moisture component as well as understanding the mechanism of pressure-electricity interactions in the dewatering process.

Keywords: Slurry; Mechanical dewatering; Pressurized electro-osmotic dewatering; Thermogravimetric; Free water; Absorbed water

1 INTRODUCTION

China's urbanization construction has made remarkable achievements in the past ten years. At the same time, construction waste has also increased as a by-product of the renewal and maintenance of urban infrastructure and the construction and transformation of industrial and civil buildings. From the perspective of social economics, there is a clear correlation between the urbanization rate and economic development. At present, China's urbanization rate is still a certain gap compared with the 80% level of developed countries. The annual output of construction waste in China is higher than the output of domestic waste. Many cities are facing or will soon face the problem of "construction waste encirclement" (Hua et al., 2022). How to carry out reasonable, harmless sustainable disposal of construction waste has become an urgent problem to be solved (Osmani & Villoria-Sáez, 2019; H. Wang et al., 2022). According to 2021 statistical data, construction slurry or slurry waste is one of the largest construction wastes in terms of output, making it a key target for resource utilization. The construction slurry refers to the residual slurry generated from pile foundation, foundation pit enclosure, slurry shield, pipe network excavation, and other construction of various buildings (structures). Due to its high water content and dewatering difficulty, there are relatively few researches and applications on reutilization. The landfill is still the main disposal method of construction slurry, which causes a large amount of resource waste due to its unsustainability and also produces security risks.

Slurry dewatering represents the general process of removing moisture from slurry and generally is accomplished by mechanical methods. However, the existing MD method has some limitations in achieving satisfactory water content (Li et al., 2021; Zhang et al., 2020). Electro-osmosis was first applied to civil engineering in the middle 1930s (Casagrande, 1949). Several studies have shown that electro-osmosis could remove water from soft soils and improve shear strength efficiently (Hu et al., 2019; Rao et al., 2022). PEOD is an emerging technology for deep dewatering of slurry. Mahmoud et al. (2011) indicated that PEOD could remove an extra 10% - 24% water compared with MD. Several studies have shown that PEOD may be a solution to dewater with lower energy expenditure compared to thermal drying and with considerable savings of time compared to MD (Rao et al., 2021; Zhang et al., 2020). Previous research on PEOD focused on moisture content (Mc), energy consumption (Ec), electrical parameters, temperature, joule heat, and pH (Mahmoud et al., 2016; Zhang et al., 2020).

Different soil water types, such as free water and absorbed water, have distinct effects on the mechanical, physicochemical and engineering properties of soils, particularly for unsaturated clayey soils. The geotechnical properties of slurry change as the moisture content changes during the dewatering process; therefore, identifying and quantifying the soil's water components from total water content is required. However, few studies have been conducted to investigate the impact of PEOD on variation of different slurry moisture components. TGA was used to investigate the identification and quantification of different components of soil water including free water, capillary water, and bound water (Yan-long et al., 2015). TGA has been widely used in soil science research in recent years, and some researchers have used it to identify, classify, and quantify various types of soil water (Cheng & Heidari, 2017; H. K. Wang et al., 2020). Y. J. Wang et al. (2011) used the TGA technique to determine the fraction of soil water that is bound to soil colloids. Yan-long et al. (2015) applied an integrated approach incorporating isothermal adsorption and thermogravimetric analyses to determine the content of different types of bound water on the surfaces of four loess soil samples. However, the preceding studies only looked at slurry dewatering and did not look into the effect of PEOD on slurry moisture components. The proposed methods investigate the variation of slurry moisture components on construction sites during PEOD since moisture component changes affect the engineering properties of the slurry in the applied environment.

In this paper, dewatering protocols are performed on a self-designed innovative PEOD apparatus. To investigate the efficiency of the coupling mechanism of electric field and mechanical pressure, the MD at various pressures (1, 1.5, 2, and 2.5MPa) and PEOD at various voltages (10, 20V) are investigated. A simulation of slurry sample was made using a combination of kaolin and bentonite soil. After the dewatering experiment, soil samples are collected from the MD (end and middle area) and PEOD (middle, cathode, and anode area) to take into account the effect of different moisture content. The variation of moisture content after PEOD influences the engineering properties of the slurry, therefore TGA technology is used to identify the slurry moisture components. Finally, MD and PEOD under various conditions are compared to determine the best dewatering efficiency, as well as TGA results to identify the soil moisture components.

2 MATERIALS AND METHODS

This section first introduces the sample preparation and test procedure in detail and then the principles of PEOD and TGA experiments are described.

2.1 Slurry samples

A high-purity kaolin powder (K299133, Shanghai Aladdin Biochemical Technology Co., Ltd) and bentonite (Wuxi Ding Long Minerals CO., Ltd) were used for the experiment. The kaolin and bentonite soils were mixed with different percentages (80% and 20% respectively) with an initial water content (water from natural source) to simulate the slurry soil samples (slurry from construction site) for the experiments. The physical properties were obtained according to the Chinese Standard Specifications of Soil Testing (SL 234–1999). The particle size distribution of the sample was measured by the Eyetech laser particle size analyzer (Ambivalue, Netherlands). The physical properties and particle size of the sample are summarized in Table 1.

Table 1. Physical properties and particle size of kaolin and bentonite

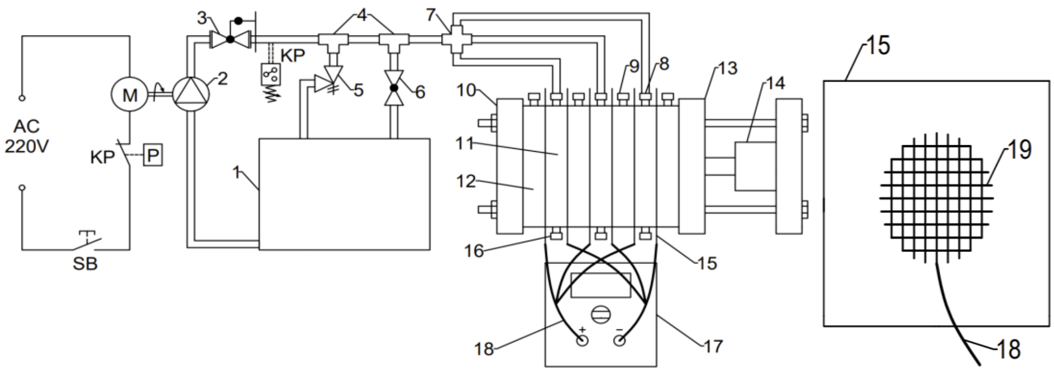
Samples	Particles size (D50)	Liquid limit	Plastic limit
Kaolin	8.2μm	64.9%	40.3%
Bentonite	12.3μm	321.3%	34.3%

Experiments were carried out using a weight of 300 g of soil (240g and 60g of kaolin and bentonite respectively). The initial moisture content was set to 120% based on the liquid limit equilibrium of kaolin and bentonite, as well as the proportion of each soil, and the solution (soil and water) was mixed with a mixer until it was homogeneous and the water/solid ratio was 1.2. The thickened slurry was placed in the filter-press cell. Filter cell sides were covered by a filter cloth of 1-3μm aperture.

2.2 Experimental apparatus

2.2.1 Self-designed pressurized electroosmosis apparatus

A deep slurry dewatering method using pressurized electro-osmotic includes a cylindrical laboratory filter-press cell (cake cross-section: 298.45cm^2; volume: 353.43cm^3), an automatic press water pump, a filter cloth made from polypropylene, a DC power supply of 20V and 5A, and a copper as electrodes were used in this study as shown in Figure 1.



Push and switch Button (SB); Pressure relay (KP); Motor (M); Water supply tank (1); Water pump (2); Pressure regulating valve (3); three-way pipe (4); Spring safety valve (5); Pressure relief valve (6); four-way pipe (7); Water inlet (8); Feed inlet (9); Thrust plate (10); Diaphragm filter plate (11); Filter frame (12); Piston hold-down plate (13); Hydraulic cylinder (14); Filter cloth (15); Water outlet (16); DC power box (17); Conductor (18); Copper conductive metal mesh (19).

Figure 1. *A self-designed pressurized electroosmosis device*

For achieving the pressurized electroosmosis test, a lab-scale set-up was specially designed for making the electroosmosis cell able to withstand mechanical loading and so, additional pressure application. The pressurized system consists of a laboratory filter press which includes a hydraulic cylinder, a diaphragm filter plate, a pressure relay, and a filter cloth. The hydraulic cylinder is connected to the filter press and the pressure is controlled by a pressure gauge. Through a hydraulic cylinder, the pressure is applied to the filter press plate to maintain them fixed and tighten during the dewatering operation.

The electro-osmosis system consists of filter cloth made of polypropylene material with 1-3microns of filtering accuracy, electrodes (copper), and a DC power supply unit with a maximum output power of 20V and 5A that is connected to three electrodes: copper as the anode in the middle and the cathode electrode at both ends. Small holes, 0.25 mm in diameter, were drilled in the electrodes to allow the drainage of the electro-osmotic flow. During the electro-osmosis process, slurry is retained by the filter cloth and only water can pass through leading to the dehydration phenomenon.

2.2.2 TGA apparatus

The identification of moisture components of slurry during PEOD is determined by TGA. TGA experiments were performed using TGA2 equipment (Mettler-Toledo, Switzerland) with an accuracy of 0.1μg. The samples after PEOD were taken out and put in hermetic containers to prevent water loss.

The furnace of TGA was heated from 30°C up to 300°C (to remove all soil water without destroying minerals) at rates of 9°C/min under a nitrogen atmosphere at a flow rate of 50 mL/min.

The basic principle of TGA is that as a sample is heated, its mass changes. This change can be used to determine the composition of a material or its thermal stability, up to 1000°C. Usually, a sample loses weight as it is heated up due to decomposition, reduction, or evaporation. A sample could also gain weight due to oxidation or absorption. While in use, the TGA machine tracks the change in weight of the sample via a microgram balance. Temperature is monitored via a thermocouple. The TGA can also track changes in weight as a function of time. Data can be graphed as weight percent or time vs temperature (°C).

2.3 Experimental methods

Firstly, slurry was dewatered for different pressure (1; 1.5; 2 and 2.5MPa) under a constant time of 2h until reached no more than 1g of removal water between an interval of 1 min. Secondly, the electroosmosis is introduced under constant pressure (2MPa) and different voltage (10; 20V) for 40min until reached to no more than 1g of removal water between an interval of 1min. The pressure, time, and voltage values are selected according to the filter press equipment as well as for some safety reasons. They are used for experimental issues. For PEOD tests, the electric field was applied after the MD. During electroosmosis pressure and electricity are applied simultaneously to improve dewatering efficiency. Finally, once the experiment is finished, the slurry cake is taken out from the filter plate.

The results were used to draw the moisture content graph under different dewatering conditions. The moisture content (Mc) of slurry was calculated as follows:

$$Mc = \frac{m_0 - m}{m} \times 100\% \quad (1)$$

where m_0 and m are the mass of initial and dried sludge, respectively.

TGA is a thermal analysis technique in which the mass of a substance is observed as a function of temperature or time as the sample is subjected to controlled temperature and atmospheric environments (Escalante et al., 2022). Experiments are carried out inside a thermogravimetric (TG) analyzer, with the sample placed on a precision balance inside a furnace. During the experiment, the furnace is in charge of heating and cooling the sample. When soils are heated, the main thermal events include the release of water, decomposition of organic matter, and dehydroxylation of the soil minerals (Yan-long et al., 2015). To conduct the TGA analysis for identification of slurry moisture components, a sample of soil is taken after the experiment from the MD (end and middle area) and PEOD (middle, cathode, and anode area).

3 RESULTS AND DISCUSSION

3.1 Effect of MD under different pressure

To evaluate the pressure effect, dewatering experiments under different pressure (1; 1.5; 2; 2.5MPa) were conducted at a fixed time of 2h. Because high pressure squeezes more water from sludge pores, it resulted in a better dewatering effect. When a pressure of 1; 1.5; 2 and 2.5MPa was applied, the moisture content of slurry reached a minimum of 90.8%; 85.6%; 82.5 and 80.3% respectively. Due to the limited anti-pressure ability of the filter press, we did not further raise the pressure.

The moisture content of the slurry cake gradually decreased from 90.87% to 80.36% with increasing pressure from 1 to 2.5MPa. This result was also consistent with the results obtained by our researchers (Zhang et al., 2020), higher pressure resulted in a better dewatering effect. The corresponding amount of removal water was 29.7g, 40.7g, 46.2g, and 50g for 1, 1.5, 2, and 2.5MPa, respectively (see Figure 2).

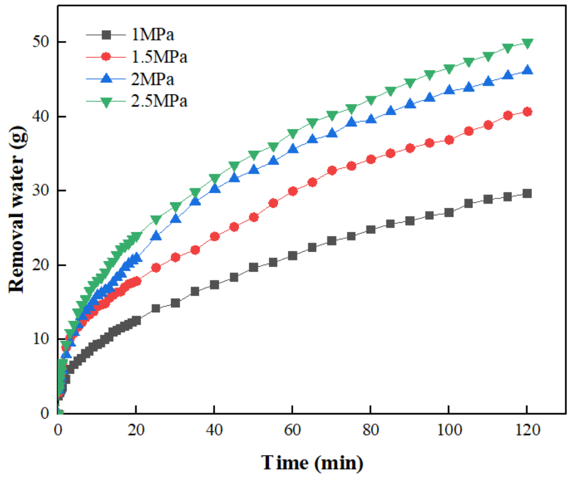


Figure 2. *Quantity of removal water with dewatering time under different pressure*

However, the effect of mechanical pressure on the moisture content may be inconspicuous as the mechanical pressure increase to a greater certain degree, which may be attributed to the higher pressure operated may block the pores between the sludge particles, leading to the weakening compressibility and lower permeability of water (Zhang et al., 2020).

3.2 Effect of PEOD under different voltage

Dewatering performance is highly dependent on voltage, and thus their effects on PEOD mode were evaluated. Figure 3 illustrates the evolution law of removal of water under different voltages (10; 20V).

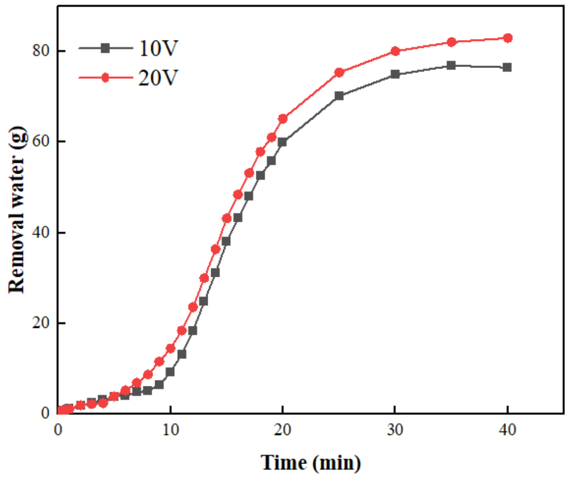


Figure 3. *Quantity of removal water with dewatering time under different pressure*

The moisture content of sludge reduced from 48.9% to 32.7% with the increasing voltage from 10 to 20V, respectively. The corresponding amount of removal water was 76.5g and 83g for 10V and 20V, respectively (see Figure 3a). This result indicated that higher voltage means more water removal and then keeps constants. As reported by earlier studies (Mahmoud et al., 2016; Tao et al., 2020) the voltage significantly affected the final drying degree of slurry cake through dewatering kinetics. Tuan et al. (2008) also pointed out that there was a positive correlation between voltage and the dewatering effect.

3.3 Thermogravimetric analysis of slurry sample under MD and PEOD

In this work, the organic matter contents of slurry sample can be ignored because the soil sample that had been used is a combination of high-purity kaolin and bentonite without organic matter. The slurry samples are collected from different sides after each experiment because the cake moisture content varies by side. After the MD the samples are collected from the end and middle side and the anode, cathode, and middle side after the PEOD experiment (see Figure 4).

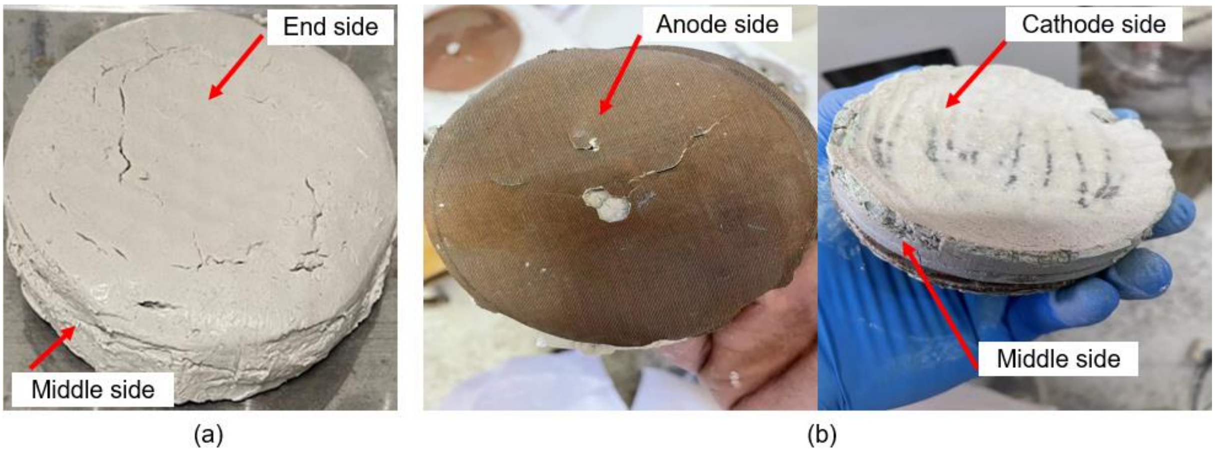


Figure 4. *Sample collection for TGA experiment:* (a) After MD; (b) After PEOD

During MD, the TGA curves of different parts of soil show that sample mass decreases with the decrease in pressure (see Figure 5). For the end area (Figure 5a), the curve can be divided into three stages. The first stage is from room temperature 30°C to 135°C. With the increase in temperature, sample mass rapidly declined. Once the temperature reached 135°C, the sample mass rate declined; the weight loss rate is 25.85% at 135°C. The second stage is from 135°C to 170°C. After 135°C, the TGA curve had an initial smooth change before becoming stable. This change indicates that it entered a new stage of water dehydration (H. K. Wang et al., 2020). At 170°C, the weight loss rate was 26.54%. The third stage begins at 170°C, in this stage, sample mass decreased very slowly (almost stable), indicating that temperatures higher than 170°C still caused some water desorption. At 300°C, the weight loss rate was 26.85%.

For the middle area (Figure 5b), the curve was similar to the end area curve and can be divided into three stages as well. The first stage is from room temperature 30°C to 125°C. With the increase in temperature, sample mass rapidly declined. Once the temperature reached 125°C, the sample mass rate declined; the weight loss rate is 50.21% at 125°C. The second stage is from 125°C to 135°C. After 125°C, the TGA curve had an initial smooth change before becoming stable. This change indicates that it entered a new stage of water dehydration. At 135°C, the weight loss rate was 50.57%. The third stage begins at 135°C, in this stage, sample mass decreased very slowly (almost stable), indicating that temperatures higher than 135°C still caused some water desorption. At 300°C, the weight loss rate was 50.91%. The results indicate that slurry sample recorded the highest rate of weight loss in the middle area and the rate of weight loss is located at the beginning of the experiment.

The TGA analysis results for the PEOD is shown in Figure 5. As can be seen, sample mass decrease with the decrease in voltage. For the PEOD experiment under 10V (Figure 5c), the TGA curve can be divided into three parts. The first part was a temperature region from 30°C to 120°C. With the increase in temperature, sample mass rapidly declined. Once the temperature reached 120°C, the sample mass rate declined; the weight loss rate is 28.58% at 120°C. The second part, the range of 120°C to 135°C, showed a total weight loss rate of 29.64% at 135°C. This change indicates that it entered a new stage of water dehydration. The third part was a high-temperature region (i.e.,135°C to 300°C), which showed a weight loss of approximately 30.37%. During the third part, sample mass decreased very slowly (almost stable).

Similar to the PEOD experiment under 10V, the PEOD experiment under 20V (Figure 5d) can also be divided into three parts. The first part was a temperature region from 30°C to 125°C. With the increase in temperature, sample mass rapidly declined. Once the temperature reached 125°C, the sample mass rate declined; the weight loss rate is 22.53% at 125°C. The second part, the range of 125°C to 155°C, showed a total weight loss rate of 23.19% at 155°C. This change indicates that it entered a new stage of water dehydration. The third part was a high-temperature region (i.e.,155°C to 300°C), which showed a weight loss of approximately 23.80%. During the third part, sample mass decreased very slowly (almost stable).

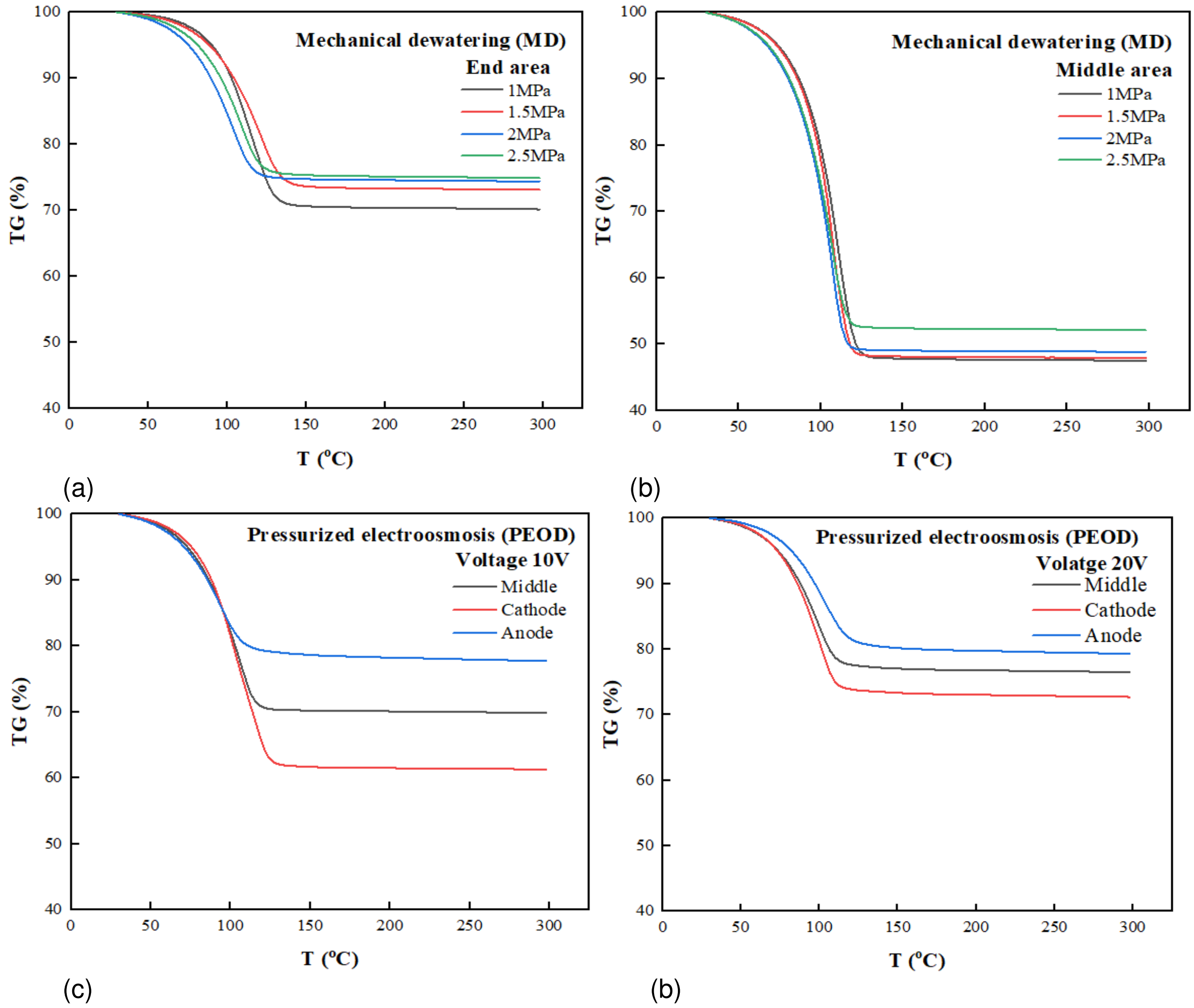


Figure 5. *Thermogravimetric analysis of slurry sample under MD and PEOD: (a) End area, (b) Middle area, (c) Voltage 10V and (d) Voltage 20V.*

The results indicate that slurry sample recorded the highest rate of weight loss under 10V of voltage; the highest rate of weight loss for slurry cake is located in the cathode area and the rate of weight loss is located at the beginning of the experiment.

3.4 Differential thermogravimetric curves of slurry sample under MD and PEOD

The differential thermogravimetric (DTG) curves can be obtained by differentiating the TGA curves. The first derivative of the weight loss percentage against temperature or time yields a DTG curve. The DTG curves can provide both quantitative and qualitative information. The qualitative analysis involves identifying material or differentiating between reactions. The quantitative analysis identifies the peak height and its corresponding temperature for the maximum weight loss sections of a curve (Escalante et al., 2022).

To evaluate the moisture components of slurry sample after MD and PEOD experiment, derivative weight changes of slurry sample were conducted under different voltages (10 and 20V) for different sides of slurry sample. The DTG analysis results are shown in Figure 6.

Results from DTG curve revealed a peak that varied according to the experimental condition. However, this study observed that DTG curves peaking corresponds to the high loss rate of the sample mass. Firstly, for DTG curve under MD at the end area (Figure 6a), the DTG temperature peaks were 115°C, 118°C, 101°C, and 106°C for 1, 1.5, 2, and 2.5MPa respectively. Secondly, for the curve under MD at the middle area (Figure 6b), the DTG temperatures peaks were 109°C, 106°C, 104°C, and 104°C for 1, 1.5, 2, and 2.5MPa respectively. Thirdly, for DTG curve for PEOD under 10V of voltage (Figure 6c), the DTG temperature peaks were 101°C, 106°C, and 92°C for the middle, cathode, and anode sides respectively. Lastly, for DTG curve for PEOD under 20V of voltage (Figure 6d), the DTG temperature peaks were 95°C, 95°C, and 101°C for the middle, cathode, and anode sides respectively. We concluded that the peak form DTG corresponded to the dehydration of slurry sample.

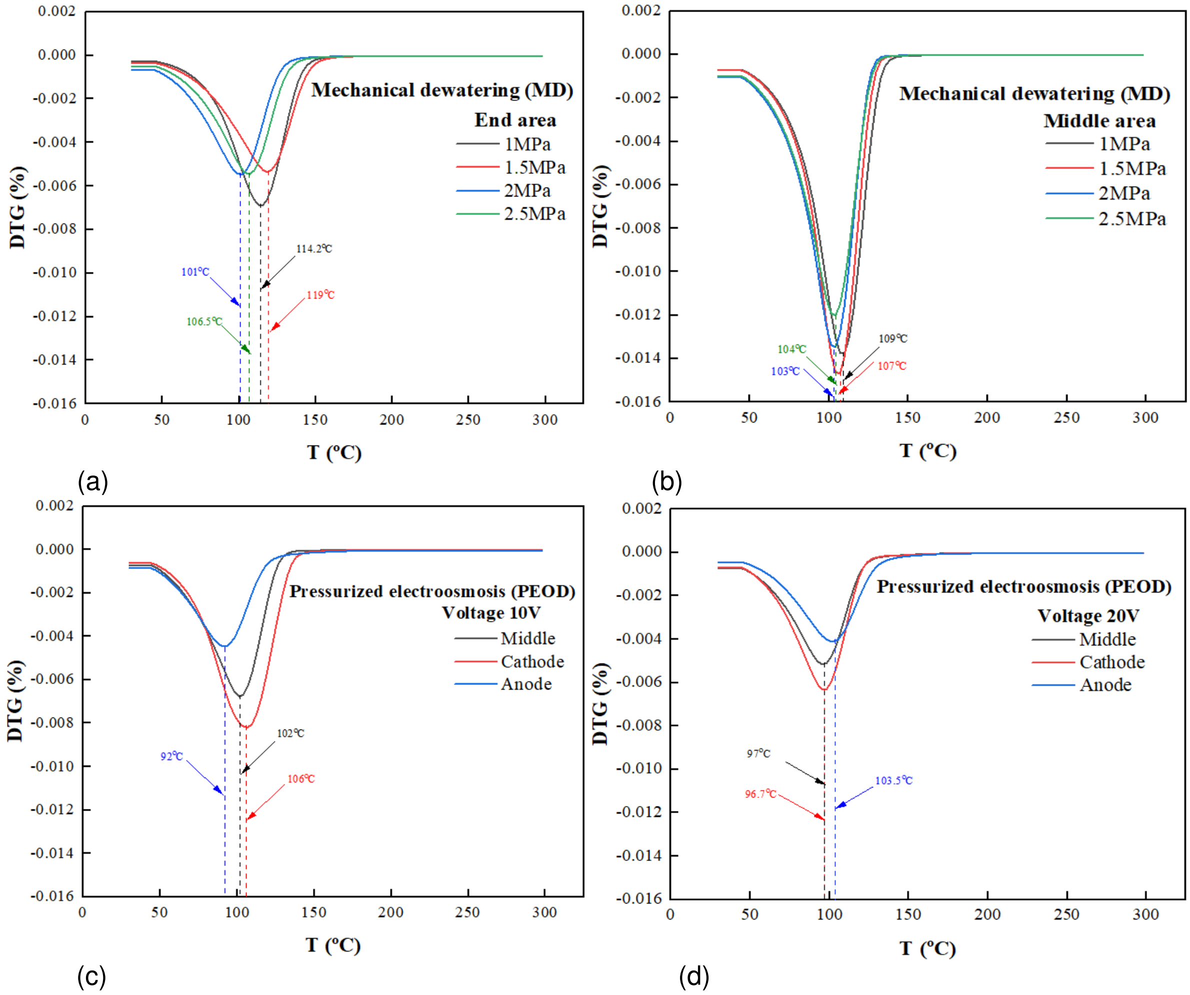


Figure 6. *Differential Thermogravimetric curves of slurry sample under MD and PEOD: (a) End area, (b) Middle area, (c) Voltage 10V and (d) Voltage 20V*

For DTG curve under MD (Figures 6a and b), an initial fast decrease (high derivative weight change) of mass is observed during heating the sample to 115°C. The weight of the sample decreased and the derivative weight change increased slightly when heated to 160°C. The weight loss became stable from 160°C to 300°C, and the derivative weight changed from asymptotic to zero. When the sample reached 300°C, the differential thermogravimetric curves were close to zero. This observation indicated that the dehydration of slurry sample finished at 300°C. For the DTG curve under PEOD, as can be noticed, the moisture content for the PEOD under 10V is higher than that for the PEOD under 20V.

3.5 Discussion about the variation of the soil moisture component during dewatering process

In this study, the variation of soil moisture components is fundamentally observed from TGA results. A DTG curve generated by plotting the weight change over time vs temperature can better show the curve's change. When there is no weight loss, DTG is equal to zero. A maximum slope on the TG curve is represented by the derivative curve's peak. A shift in slope occurs on the TG curve when DTG is minimum but not zero, which is known as an inflection. The desorption temperature of free water is low, and the desorption temperature of adsorbed water is high (Vyazovkin et al., 2011). Then, in the process of increasing free water, the peak value of DTG tends to be near the low-temperature area, and in the process of decreasing free water, DTG tends to be near the high-temperature area.

For DTG curve under MD (Figures 6a and b), as can be noticed, the moisture content in the middle area is higher than that in the end area. The curve peaking position is inclined to the left (low-temperature area) with the increase in pressure. We concluded that the quantity of free water after the MD is the most dominant.

From the DTG curve for PEOD under 10V (Figure 6c), it can be seen that the water at the anode side < water at the middle side < water at the cathode side. Therefore the smaller quantity of water in the soil, most the DTG curve position is oriented to the left (low-temperature area) indicating that the quantity of free water after the PEOD under 10V is the most dominant. The DTG curve for PEOD under 20V (Figure 6d) shows that the smaller quantity of water in soil, most the DTG curve position is oriented to the right (high-temperature area) indicating an opposite phenomenon. We concluded that after PEOD under 20V, the absorbed water is the most dominant.

In the temperature range higher than 160°C, all tested mass changes are smaller than 0.1%. Combined with the desorption temperature of soil water in the literature (Yan-long et al., 2015), it can be concluded that all free and absorbed water in the soil is removed.

4 CONCLUSIONS

In this study, the pressurized electro-osmotic dewatering was shown to significantly enhance the dewaterability of slurry, and the identification of different moisture components of soil water including free water and absorbed water from thermogravimetric analysis (TGA) data were investigated.

Some findings are summarized as follows: (1) PEOD under 20V has better dewatering efficiency with 32.6% moisture content; (2) The quantity of free water remaining in the slurry is as follows: PEOD under 20V < PEOD under 10V < MD; (3) For the MD and PEOD under 10V, the quantity of free water after the MD and PEOD under 10V is the most dominant; (4) For the PEOD under 20V, the quantity of absorbed water is the most dominant.

It is recommended to further innovate the process of large-scale dewatering of construction slurry in future research and propose measures to improve the dehydration performance during the process. In addition, it is suggested to quantify in terms of percentage the different types of moisture components after pressurized electroosmotic dewatering.

5 ACKNOWLEDGEMENTS

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