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The thermodynamic properties variation of cemented clay after treatment at high temperatures



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HIGHLIGHTS

• The thermodynamic properties of cemented clay were explored at 25-900 °C.

• The CIE-L* a* b* system be used to analyze the color of the sample surface.

• Variation of thermal conductivity are evaluated by the wave velocity and mass loss.

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ABSTRACT

Cemented clay materials are often used in a different engineering projects but concrete exhibits different thermal properties at different temperatures. Therefore, it is important to have a thorough understanding of the thermodynamic properties of cemented clay when it is used in a particular structure or exposed to a particular temperature. At high temperatures, a series of changes occur in the internal structure of cemented clay, which include changes to the alkalinity level, mass, density, wave velocity, thermal conductivity and compressive strength. In this study, the cemented clay samples are exposed to temperatures from 25 °C to 900 °C in a heating furnace. Alkalinity was judged by observing the change in the surface color of the samples treated with phenolphthalein. At the same time, using the CIE-L* a* b* system to further analyze the color of the sample surface, to explore the physical and chemical reactions of the sample during the temperature increase process. It is found that the quality, wave velocity, thermal conductivity and compressive strength of cemented clay are all decreased with the increase of temperature, especially 200–600 °C. In addition, the mass loss rate and wave velocity are used to evaluate the thermal conductivity of the cemented clay samples. And the compressive strength is evaluated by the change of density.

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1. Introduction

Cemented clay materials are often used in a different engineering projects, such as buildings, bridges and tunnels. Research work on the performance of cemented clay exposed to high temperatures has been abundant [1–14]. Studies also show that concrete exhibits different thermal properties at different temperatures [15–16]. Therefore, it is important to have a thorough understanding of the thermal properties of cemented clay when it is used in a particular structure or exposed to a particular temperature [17]. The thermal properties of cemented clay can be examined through thermal conductivity, specific heat, thermal diffusivity, thermal expansion and mass loss [18]. For instance, Shin et al. examined

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https://doi.org/10.1016/j.conbuildmat.2018.06.137 0950-0618/© 2018 Elsevier Ltd. All rights reserved. the thermal properties of cemented clay used in a South Korean nuclear power station under high temperatures with the transient heat wire method [19]. Yan et al. examined the thermal conductivity of clay with C40 high performance cement at high temperatures, and the thermal conductivity of clay at a constant temperature by using the guarded hot plate method, respectively [20]. Khaliq and Kodur tested the thermal conductivity of selfcompacting concrete under high temperatures, and found that its thermal conductivity decreases with increases in temperature [21].

At high temperatures, a series of changes occur in the internal structure of cemented clay [1,22]. Therefore, the thermal conductivity of cemented clay under different engineering conditions needs to be examined to assess the damage in the cemented clay structure [23–25]. At the same time, a number of academics or research institutes have provided equations for the thermal conductivity of cemented clay with temperature [26,27]. These







empirical formulas are very helpful for studying the specific thermal conductivity of cemented clay. However, there are few studies that examine the changes in the alkalinity of cemented clay at high temperatures. When changes in the alkaline level of cemented clay with temperature are determined, cemented clay can then play a better role in different practical applications. Moreover, research work on the various relationships among the thermodynamic parameters of cemented clay are also lacking.

In this paper, the changes in the alkalinity level of cemented clay at different temperatures are shown by using the phenolph-thalein reagent and $CIE-L^* a^* b^*$ system. At the same time, the mass loss rate and wave velocity are used to evaluate the thermal conductivity of the cemented clay samples. And the compressive strength is evaluated by the change of density.

2. Experimental tests

In the experiment, cemented clay samples were made in a temperature controlled environment at 20 °C–22 °C. A 70.7 mm × 70.7 mm × 70.7 mm mold was used to prepare the cemented clay. Table 1 shows the mix proportion of the cemented clay. The cement is ordinary Portland cement with a strength of 42.5 MPa. Fine sand was added as the fine aggregate, and the particle size of the fine sand was 0 mm–2.5 mm. The mixing water was tap water. In accordance with the mix proportion, we added water to the stated ingredients. Then the mix was placed into the mold which had been coated with Vaseline for ease of removal. The chemical composition of the materials are provided in Table 2.

High temperature testing was carried out with a ash volatile analyzer (CTM-100) (QIULONGYIQI and China). The CTM-100 is composed of a controller and muffle furnace. The samples were heated in the muffle furnace. The test samples were placed into the furnace, and heated to the designated temperature (25 °C, 50 °C, 100 °C, 200 °C, 400 °C, 600 °C, 800 °C, and 900 °C respectively) at a rate of 10 °C/min. Each sample was kept at its designated temperature for about 90 min. When the furnace temperature naturally declined to 200 °C, the door of the muffle furnace was opened. When the furnace temperature was less than 100 °C, the samples were removed and cooled down to room temperature. The mass and thermal conductivity of these samples were tested before and after being heated. The mass was measured by using an electronic balance, with a precision of 0.01 g. The thermal conductivity testing of the cemented clay samples was carried out by using a DRE-2C thermal conductivity tester. The transient plane heat source (TPS) technique was used with the DRE-2C thermal conductivity tester and the Hot Disk as the thermal conductivity probe. In particular, the surface of the samples was polished prior to testing with the DRE-2C thermal conductivity tester. The wave

Table 1		
Mix proportions	of cemented	clay (kg/m ³).

Portland cement	fine sand	clay	water
9.6	36	2.4	12

Table 2

Chemical composition of cemented clay.

Chemical composition	Content (%)
Calcium oxide (CaO)	54.6
Silicon dioxide (SiO ₂)	31.7
Aluminum oxide (Al ₂ O ₃)	7.3
Ferric oxide (Fe ₂ O ₃)	1.7
Phosphorus pentoxide (P ₂ O ₅)	0.21
Sodium oxide (Na ₂ O)	0.58
Potassium oxide (K ₂ O)	0.62

velocity of the samples was measured by using an RSM-SY5 nonmetal ultrasonic detector. The samples were tested by using a phenolphthalein reagent after the heat treatment, and it was observed the surface of the samples changes color with different temperatures. The compressive strength of the samples is tested by the WES-D1000 electrohydraulic servo test machine. And the loading rate is 0.2 kN/s.

3. Results

3.1. Surface color characteristics

The cemented clay samples contain calcium hydroxide (Ca $(OH)_2$) and calcium bicarbonate ($Ca(HCO_3)_2$) which have high levels of alkaline, and turn red when they come into contact with phenolphthalein. The samples were exposed to different temperatures, and their surface color showed changes at different temperatures. Fig. 1 shows a photo of all the samples after heat treatment. The samples were uniformly sprayed with a 1% phenolphthalein ethanol solution. After ten minutes, changes in the surface color of the samples were observed (see Fig. 2). In order to better describe the color change characteristics of the samples after treatment with phenolphthalein, we tested the L* a* b* value of the surface color by using a TES-135A color meter. In the CIE- L* a* b* system, L* represents bright differences, a* represents red and green differences, and b* represents the difference between yellow and blue. The results of the ICE- L* a* b* tested are shown in Fig. 3. As we can see from Fig. 3(a), the surface of the specimen is dark before 400 °C. At 400–900 °C. the surface of the specimen brightened obviously. The greater the value of the a*, the more the color tends to be red, as shown in Fig. 3(b). At the same time, the more red the color of the surface, the stronger the alkalinity of the samples. The larger the b^{*} value, the more yellow the surface of the samples is, as shown in Fig. 3(c). From room temperature to 400 °C, a series of chemical reactions took place within the cemented clay.



Fig. 1. All samples after heat treatment.

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