Response of thermal conductivity of loess after high temperature in northern Shaanxi burning rock area, China

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Research Article

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Abstract

The thermal conductivity of loess plays an important role in ecological restoration design and the calculation of roadbed and slope stability. This study uses 60 loess samples from Shaanxi Province of China as raw material. The loess samples were heated to high temperature in an unclosed muffle furnace and then cooled to room temperature. Subsequently, the heat conduction coefficient (λ), specific heat capacity (c) and thermal diffusion coefficient (α) of the sample were measured. Between 23–900 °C, with temperature increasing, λ decreased from 0.90 to 0.46 W/m·K, c decreased from 1.60 to 1.23 J/kg·K, α decreased from 0.59 to 0.38 m²/s. The test results show that, after being exposed to high temperature, the thermal conductivity of loess tends to weaken. This has a very important relationship with the pore characteristics of loess.

1. Introduction

Northern Shaanxi is located in the loess plateau and is rich in coal resources. But the ecological environment is fragile. The thermal conductivity of loess not only plays an important role in ecological restoration design, but also is an important parameter in the calculation of roadbed and slope stability (Santi et al., 2013; Zihms et al., 2013). Coal spontaneous combustion exists in many mining areas in northern Shaanxi (Fig. 1). The high temperature it produces changes the thermal conductivity of overlying loess to a great extent (Biro et al., 2019).

Some experimental results show that the loess becomes strongest when calcined at 600 °C, followed by 500 °C and 700 °C, while the strength at 800 °C decreases sharply (Ng et al., 2018; Li et al., 2018). However, in engineering applications, how to accurately calculate its strength, thermal conductivity is an indispensable parameter.

Thermal conductivity is an important parameter reflecting the change of structure, density, pore, humidity and other properties of loess, and it will change regularly with the increase of temperature (Sun et al., 2016a; Tang et al., 2017). After heat conduction parameters changed, how to choose the thermal conductivity value scientifically and reasonably plays an important role in the accuracy of engineering calculation and numerical simulation, such as maintenance and reconstruction of loess road and slope after high temperature.

The spontaneous combustion of shallow coal seams in northern Shaanxi can produce a high temperature of up to 1000 °C, and the thermal conductivity of overlying loess will change to a certain extent (Zhang et al., 2018). Although many scholars have studied the thermal characteristics of loess, research on its thermal conductivity after being subjected to high temperatures is lacking. Temperature is one of the most important factors affecting the thermal conductivity of loess (Oikonomopoulos et al., 2015). Guo et al. (2012) measured the thermal conductivity of loess samples in a temperature range of 17–22 °C. The results show that the thermal conductivity of loess
material increases slightly with increases in temperature and fluctuates greatly at lower temperatures (Guo et al., 2012).

In this paper, the effect of temperature on thermal conductivity of loess is measured and analyzed, and the main reasons for the change of thermal conductivity are discussed by measuring mass loss rate, chromaticity and microscopic observation.

2. Experimental Tests

2.1 Sample preparation and experimental procedure

The sampling site was in Northern Shaanxi Province, China. The sampling horizon was Quaternary loess. The mineral composition of loess is mainly quartz (50%), feldspar (20%), clay minerals (20%) and a small amount of carbonate minerals (10%) (Liu, 2002). As can be seen from figure 2, the grain size of loess samples is relatively uniform. The content of silty soil in loess is high.

First, loess was uniformly broken. 80g loess was compacted into samples of the same size. A small cylinder with a diameter (± error) of 50 ± 1 mm and height of 20 ± 1 mm was fabricated at room temperature. The compaction density is 2.0g/cm³.

In this experiment, ten different temperatures with 60 samples were used: 23 °C, 100 °C, 200 °C, 300 °C, 400 °C, 500 °C, 600 °C, 700 °C, 800 °C, and 900 °C. The heating condition was a non-closed environment in a furnace and the cooling mode was natural cooling to room temperature.

Samples were weighed before heating. After the furnace reached the target temperature, the temperature was held constant for 1 h to ensure that the samples were uniformly heated. After natural cooling to room temperature, samples were weighed again and then sealed with plastic wrap. Finally, parameters such as chromaticity and thermal conductivity were determined.

2.2 Experimental instruments

Weights were measured using a electronic balance with an accuracy of 0.01 g (JE102-JE2002, Hangzhou Hengyi Instrument Technology Co. Ltd.). The samples were uniformly broken by a multifunctional sample preparation grinder (DY-800Y, Xinyuan Coal Quality Analysis Instrument Co. Ltd., Hebi City, Henan Province). The equipment for making samples (KC-505T) was independent research and development. Produced by Cixi Kecheng Machinery Co. Ltd.

Samples were heated in a furnace (SDTGA200, Hunan Sundy Science and Technology Co. Ltd.) with a maximum temperature rating of 1300 °C.

The color of the samples was measured by a precision colorimeter (NR10QC, 3NH Technology Co. Ltd). The parameter Δa* > 0 indicates red chromaticity and the higher the Δa* value, the more obvious the red;
The parameter $\Delta b^* > 0$ indicates yellow chromaticity and the higher the $\Delta b^*$ value, the more obvious the yellow.

The thermal conductivity of the sample was tested by a thermal parameter analyzer (TPS 2500S Hot Disk Company, Sweden). A laser beam was used to irradiate the sample, infrared detector was used to measure the temperature rise on the back of the sample, calculate the thermal diffusivity of the sample (Cai et al., 2011). Its internal resistance value was $R = 6.936806 \, \Omega$, applicable temperature range $-10–1000 \, K$, and thermal conductivity test range $0.005–500 \, W/(m\cdot k)$. The measurement accuracies of thermal conductivity, specific heat capacity, and thermal diffusivity were ±3%, ±7%, and ±5%, respectively.

Uses the HCT-3 differential thermal balance (Beijing Hengjiu Scientific Instrument Factory, China) to test the thermogravimetric.

The instrument used for microscopic observation is STEMI-508 binocular stereoscopic microscope (Carl Zeiss Microscopy, GmbH, Germany) and JSM-7610F field emission scanning electron microscope (Japan Electronic Dwarf Club).

The test instrument and basic process are shown in Figure 3.

3. Results

3.1 Heat conduction performance

The relationship between thermal conductivity and temperature in loess after high-temperature treatment is shown in Figure 4. heat conduction coefficient ($\lambda$), specific heat capacity ($c$), and the thermal diffusion coefficient ($\alpha$) had negative exponential relationships with temperature ($T$) in the range 23–900 °C.

As can be seen from Figure 4a, from 23 to 200 °C, the heat conduction coefficient $\lambda$ of loess samples decreased by 37.8% from 0.90 to 0.56 W/m·K, with a high rate of decline. The $\lambda$ decreased by 17.9% from 0.56 to 0.46 W/m·K, with a low rate of decrease from 200 to 900 °C. There was a negative exponential relationship between $\lambda$ and $T$ in the loess samples, as per Eq. 1:

$$\lambda = 0.300 \exp \left( -\frac{T}{24.946} \right) + 0.427 \exp \left( -\frac{T}{509.447} \right) + 0.373$$

$$R^2 = 0.871$$

As can be seen from Figure 4b, at 23–300 °C, the specific heat capacity $c$ decreased by 19.4%, from 1.60 to 1.29 J/kg·K. After that, as the temperature increased to 900 °C, the $c$ only decreased by about 4.7% from 1.29 to 1.23 J/kg·K. There was a negative exponential relationship between the $c$ and $T$ of loess samples, as per Eq. 2:

$$c = 0.296 \exp \left( -\frac{T}{40.428} \right) + 0.370 \exp \left( -\frac{T}{1056.893} \right) + 1.068$$

$$R^2 = 0.912$$
The variations in the thermal diffusion coefficient $\alpha$ and heat conduction coefficient of loess specimens after heating were very similar (Tarnawski et al., 2018). As can be seen from Figure 4c, when heated to 200 °C, the $\alpha$ decreased by 25.4% from 0.59 to 0.44 m$^2$/s. Then, at 200–900 °C, the $\alpha$ fluctuated greatly, decreased by 13.6% from 0.44 to 0.38. Overall, there was a close negative exponential relationship between the $\alpha$ and $T$ of loess samples, as per Eq. 3:

$$\alpha = 0.106 \exp\left(-\frac{T}{494.711}\right) + 0.181 \exp\left(-\frac{T}{495.008}\right) + 0.297$$

$$R^2 = 0.755$$

### 3.2 Change in mass

After weighing the mass of samples before and after the experiment, it was found that the mass of each sample decreased by varying degrees after high-temperature exposure, with the mass-loss rate ($M$) increasing with temperature (Eq. 4). As shown in Figure 5, during Stage I (temperature increase from 23 °C to 200 °C), the mass-loss rate increased rapidly from 0% to 0.0359%. In Stage II (200–600 °C), the mass-loss rate increased from 0.0359% to 0.0621% and the mass-loss rate was slightly reduced. In Stage III (600–900 °C), the mass-loss rate was relatively stable and in the range 0.0609–0.0668%.

$$M = \frac{m_0 - m_t}{m_0} \times 100\%$$

where, $M$ is the mass-loss rate, $m_0$ and $m_t$ represent the sample mass at room temperature and after heating, respectively.

### 3.3 Changes in apparent color

Color is a more observable feature. Changes in the surface color of the loess samples treated at different temperatures were obvious (Sanmartín et al., 2011). They changed gradually with increases in treatment temperature (Karaman et al., 2006; Fig. 6). At 300 °C, the color of the loess samples began to show the characteristics of a dark system. After that, with increases in temperature, the degree of redness in the samples became more obvious.

As can be seen from Figure 7a, the red chromaticity $\Delta a^*$ was stable at -0.33–0.008 at temperatures of 23–200 °C (Stage I; slope $k = 0.232$). At a temperature of 300 °C, the value of $\Delta a^*$ increased rapidly from near zero to 2.97. Between 300 and 700 °C (Stage II), $\Delta a^*$ increased from 2.97 to 6.79 (slope $k = 0.955$). At 700–900 °C (Stage III), $\Delta a^*$ increased from 6.79 to 11.35 (slope $k = 2.280$).

From Figure 7b, the yellow chromaticity $\Delta b^*$ value increased from -0.82 to 0.16 in the first stage (23–300 °C; slope $k = 0.354$). In Stage II (300–700 °C), $\Delta b^*$ increased from 0.16 to 5.6 (slope $k = 1.36$). In Stage III (700–900 °C), $\Delta b^*$ increased from 5.6 to 13.47 (slope $k = 3.935$).
In general, the variations in $\Delta a^*$ with temperature and $\Delta b^*$ with temperature were similar; both increased with temperature, with the change being very obvious at 300 °C.

4. Discussion

Thermogravimetric (TG) and differential thermal analysis (DTA) tests were carried out at room temperature to analyze the causes of mass loss and chromaticity changes of samples at high temperatures (Fig. 8). Variations in loess with temperature were observed to occur within three main stages: 23–200 °C, rapid decline of TG curve and unstable rise of DTA curve; 200–650 °C, steady decline of TG curve. DTA has changed from a steady rise to a steady decline. And 650–900 °C, the TG curve gradually stabilized and the DTA curve continued to decline steadily. These are described in more detail below.

(1) At temperatures of 23–200 °C, there is an obvious endothermic valley in the DTA curve (the mass loss is relatively obvious). The main change was the evaporation of free water, adsorbed water, and partially bound water from the samples (Li et al., 2015; Rau et al., 2014). The rate of water loss was rapid in this range, causing the highest mass-loss rate. However, changes in chromaticity were not obvious (Evrard et al., 2019; Cheng et al., 2019).

(2) At temperatures of 200–650 °C, from the DTA curve, it can be seen that the endothermic valley mainly appears in this stage too, which is in the state of mass loss. The main change was due to the precipitation of mineral structure water and crystal water. In this temperature range, the samples were completely dehydrated (Wang et al., 2000; Sun et al., 2016b). In addition, CaCO$_3$ decomposition, emitting CO$_2$ (Eq. 5). There was also a small amount of mass loss (Hajpál, 2002; Milheiro et al., 2005) and the mass-loss rate was low. At the same time, due to the higher temperature, the Fe$^{2+}$ in the cement can be separated more easily. The Fe$^{2+}$ gradually oxidized to Fe$^{3+}$ and the color of the soil gradually tended towards red (Hu et al., 2013; Murad and Wagner, 1998; Manhães et al., 2002).

\[
\text{CaCO}_3(s) \xrightarrow{\Delta} \text{CaO}(s) + \text{CO}_2(g) \tag{5}
\]

(3) At temperatures of 650–900 °C, it can be seen from the DTA curve that there is an exothermic peak in this temperature range, and the mass loss stops. The oxidation of iron became more thorough, with the intensity of the red color indicative of the degree of iron oxidation (Manhaes et al., 2002; Milheiro et al., 2005).

From the above analysis, we can see that before 650 °C, it is mainly the process of dehydration and the combustion of organic matter, as well as the decomposition of CaCO$_3$, so that their original space is filled with air. The thermal conductivity of air is much lower than that of liquid and solid matter. Therefore, the thermal conductivity of the sample decreases rapidly at this stage (Paul et al., 2002; Fig. 9a).
At 650–900 °C, the main change of the sample is that the iron ions are gradually oxidized with the increase of temperature. The chromaticity changes obviously and the mass loss tends to be stable (Wang et al., 2021a). Therefore, we also carried out microscopic observation on the samples with binocular stereoscopic microscope and scanning electron microscope (Fig. 10). As can be seen from Figure 10a, there is no obvious crack in the sample at 500 °C. At 600 °C, obvious cracks appeared, while at 900 °C, the cracks increased and multiple cracks appeared. From Figure 10b, there are fewer pores in the sample at 500 °C. The porosity in the sample tends to increase at 600 °C. At 900 °C, the pore size in the sample increases, and the number of pores in the sample increases.

When heating up, the water in the sample evaporates, and the shrinkage of the surface is greater than that of the interior, which leads to cracks in the interior of the sample. In the cooling process, due to the fast cooling rate of the sample surface, the thermal stress caused by the difference between internal and external temperature also promotes the formation of cracks (Wang et al. 2003; Monteiro and Vieira 2004). The thermal expansion coefficient of different minerals is different, which leads to the deformation and evolution of adjacent minerals into fractures (Ahmad et al. 2008; Hummel, 2010; Wang et al., 2021b). Therefore, the increase of cracks and pores can reduce the thermal conductivity (Hideo and Hideyuki, 2004; Fig. 9b).

Figure 11 shows that in the range where there was a high mass-loss rate (23–300 °C), the thermal conductivity decreased rapidly, while in the ranges with lower mass-loss rates (300–500 °C and 600–900 °C) (Hiraiwa and Kasubuchi, 2000), the thermal conductivity of the loess samples decreased relatively slowly. This is mainly because the thermal conductivity of materials with lower moisture content is also smaller (O"Donnell et al., 2009; Duan, 2016). Overall, the mass-loss rate $M$ of loess samples had strong linear relationships with thermal conductivity $\lambda$, thermal diffusion coefficient $\alpha$, and specific heat capacity $c$. The models are as follows (Eq. 6; Eq. 7; Eq. 8):

$$\lambda = 0.875 - 6.352M \ (R^2 = 0.935) \quad (6)$$

$$\alpha = 0.605 - 3.803M \ (R^2 = 0.816) \quad (7)$$

$$c = 1.543 - 4.481M \ (R^2 = 0.913) \quad (8)$$

### 5 Conclusions

1) When the heating temperature is less than 200 °C, the heat conduction coefficient $\lambda$ and thermal diffusion coefficient $\alpha$ decrease greatly with the increase of temperature. The values of $\lambda$ and $\alpha$ are basically in a stable state at 200–500 °C. At 600 °C, there is another sharp decrease, and then it is basically stable to 900 °C. Before 300 °C, specific heat capacity $c$ continued to decrease greatly with the increase of temperature. As the temperature continues to rise to 500 °C, $c$ increases slightly, and then decreases slowly until the temperature reaches 900 °C.
2) In the range of 23–200 °C, the mass-loss rate ($M$) of loess was relatively large, while changes in chromaticity were not obvious. At temperatures of about 200–650 °C, the $M$ was relatively stable, whereas the redness and yellowness of the samples began to increase obviously. At temperatures in the range 650–900 °C, the change in $M$ was not obvious, whereas redness and yellowness were further enhanced.

3) When the mass-loss rate of loess was large, the thermal conductivity decreased rapidly. In the low mass-loss rate temperature range, the thermal conductivity decreased slowly. Overall, the mass-loss rate of loess had strong linear relationships with thermal conductivity $\lambda$, thermal diffusion coefficient $\alpha$, and specific heat capacity $c$.

**Declarations**

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**Declarations Conflict of interest**

On behalf of all authors, the corresponding author states that there is no conflict of interest.

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Figures

Figure 1
Coal spontaneous combustion in a coal mine in northern Shaanxi

Figure 2

Soil particle size distribution map of the sample
Figure 3

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Figure 4

Variations of thermal conductivity with temperature, where $\lambda$ is the heat conduction coefficient, $c$ is the specific heat capacity, and $\alpha$ is the thermal diffusion coefficient.
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Changes in the color of samples at different temperatures

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Figure 9

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