

Experimental study on the effect of calcination on the volcanic ash activity of diatomite

Liguang Xiao & Bo Pang

School of Materials Science and Engineering, Jilin Jianzhu university, Changchun 130118, China

E-mail: xlg627@163.com, 1004625664@qq.com

Abstract: The volcanic ash activity of diatomite was studied under the conditions of aerobic calcination and vacuum calcination by the combined water rate method, it was characterized by XRD, BET and SEM. The results showed that the volcanic ash activity of diatomite under vacuum conditions was higher than that of aerobic calcination, 600°C vacuum calcination 2h, the combined water rate of diatomite-Ca(OH)₂-H₂O system was increased from 6.24% to 71.43%, the volcanic ash activity reached the maximum value, the specific surface

1 Introduction

Volcanic ash activity, refers to the ability of natural or artificial volcanic ash materials to form hydrated products by chemical reaction of water and calcium hydroxide at room temperature [1]. Diatomite is a non-metallic mineral formed by the deposition of unicellular algae, the main component of diatomite is amorphous SiO₂ [3, 4], which has a certain volcanic ash activity and as an active admixture for the preparation of high performance concrete. The main mechanism of diatomite for concrete is the formation of hydrated calcium silicate gel (C-S-H) by reactive SiO₂ and Ca(OH)₂ produced by cement hydration reaction, which improves the filling density of concrete and improves the morphology of hydration products [5]. In this study, the volcanic ash activity of diatomite was studied by the combined water rate method [2], and the volcanic ash activity of calcined diatomite under conditions of aerobic and anaerobic was compared.

2 Experimental process

2.1 Experimental materials

Diatomaceous soil: from Changbai County, Jilin Province; Lime: quicklime.

2.2 Experimental Instruments

HY-1600°C high temperature energy-saving electric furnace, TM3030 scanning electron microscope (SEM), VC-Sorb 2800TP specific surface and pore size analyzer, X-Ray Diffractometer System 554800 X-ray diffractometer, KSXL-1216 vacuum calciner, DM-L frequency conversion planetary Type ball mill.



2.3 Experiment Method

2.3.1 Calcination. The diatomite after grinding processing was placed in a muffle furnace respectively at different calcination temperatures (500°C, 600°C, 700°C, 800°C, 900°C) calcined for 2h. The diatomite after grinding processing was placed in a vacuum muffle furnace respectively at different calcination temperatures (500°C, 600°C, 700°C, 800°C, 900°C) vacuum calcined for 2h.

2.3.2 Volcanic ash activity test. 50g calcined diatomite and 50g lime were taken, water-cement ratio was 9:1, diatomite-Ca(OH)₂-H₂O system was compound, it was conserved at 80°C for 3d. Two samples 1g (accurate to 0.0001g) of hydrated powder dried at 60°C were weighed, and placed in a burned constant weight crucible. Then the two samples was heated to 975°C burning to constant weight in a muffle furnace. After removal, samples were cooled to constant temperature in a desiccator and weighed. The combined water rate of the hydration product was calculated by:

$$W = (C_1 - C_2) / C_2 - W_x / (1 - W_x)$$

In formula: W is the combined water rate of the hydrated product; C₁ is the mass of the hydrated sample after drying at 60 °C; C₂ is the mass of the hydration sample after burning at 975 °C; W_x is loss on ignition of unhydrated sample.

2.3.3 Characterization method. All samples were heated at 105°C for 1h before the test. The surface of the samples was analyzed by X-ray diffraction. The surface morphology was analyzed by TM3030 scanning electron microscope. The specific surface area was measured by VC-Sorb 2800TP specific surface and pore size analyzer.

3 Experimental results and analysis

3.1 Loss on ignition analysis

Figure 1 showed the Loss on ignition of diatomite at different calcination temperature, from Figure 1 showed that the loss on ignition of diatomite was 13.5%, the high loss on ignition of diatomite, high organic content, With the increase of calcination temperature, the loss of diatomite was gradually reduced, As diatomite was produced with a variety of organic matter composition of animal and plant residues, Under conditions of high temperature organic matter of diatomite was decomposed, organic matter became into small molecule and volatilization that loss on ignition was reduced. Under conditions of different calcination temperatures the loss on ignition of diatomite under anaerobic calcination was lower than aerobic calcination, it indicated that the anaerobic calcination organic matter and crystal water was removed efficiently, Under the condition of anaerobic calcination, the organic matter was became C and attached to the diatom shell. The calcination process would not produce a lot of CO₂ and avoided environmental pollution.

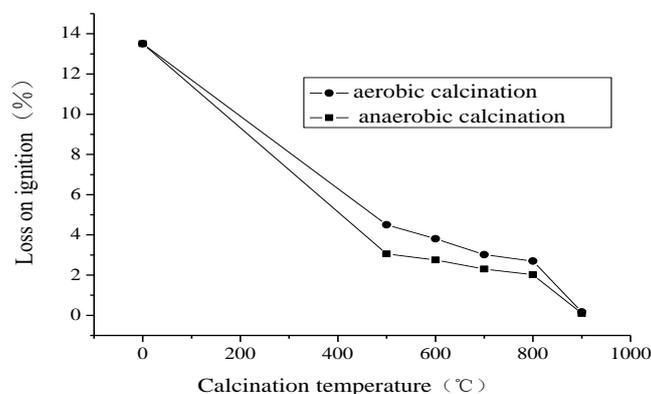


Fig 1. Diatomite loss at different calcination temperatures

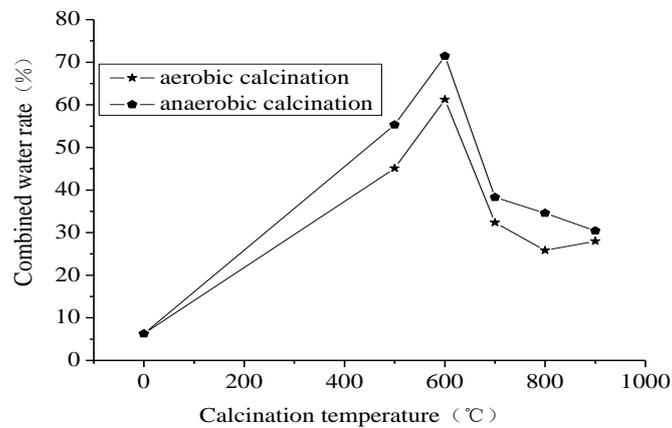


Fig 2. Bound water ratio of diatomite- $\text{Ca}(\text{OH})_2\text{-H}_2\text{O}$ at different calcination temperatures

3.2 Combined with water rate analysis

Fig 2 showed the combined water ratio of diatomite- $\text{Ca}(\text{OH})_2\text{-H}_2\text{O}$ system after 2h calcination at different temperatures, As shown in Fig 2, the calcination temperature of the diatomite was increasing, combined with water rate increased to a certain value and then decreased, volcanic ash activity reach to the maximum at 600°C and then decreased. And the activity of volcanic ash under anaerobic calcination conditions was higher than that under aerobic calcination conditions, Due to the SiO_2 of diatomite reacted with $\text{Ca}(\text{OH})_2$, formed the hydrated calcium silicate (C-S-H) gel, The diatomite- $\text{Ca}(\text{OH})_2\text{-H}_2\text{O}$ was measured , the higher the rate of binding water, the more C-S-H gels were produced, the stronger volcanic ash activity. With the temperature increased more than 600°C, the specific surface area decreased, the surface of diatomite a large number of OH^- was removed under high temperature, so the number of bridge oxygen increased gradually, resulted in the number of active SiO_2 reduced gradually. In addition, the pore structure of diatomite was improved greatly under anaerobic condition, and the organic matter of diatomite became C under oxygen-free calcination and adhered to the surface of the diatomite, The surface of diatomite was modified to improve the reaction of diatomite with $\text{Ca}(\text{OH})_2$, so the activity of volcanic ash under anaerobic calcination was higher than that under aerobic calcination.

3.3 XRD analysis

Fig. 3 showed the XRD patterns of raw diatomite and calcination diatomite, The original soil contained quartz and feldspar band was more obviously, after calcination the feldspar and quartz peak was reduced significantly, The dispersion peaks of amorphous SiO_2 in the vicinity of $2\theta = 22^\circ$ were still significant, indicated that the calcination removed the impurities on the surface of the diatom shell, purified the main component of diatomite SiO_2 .

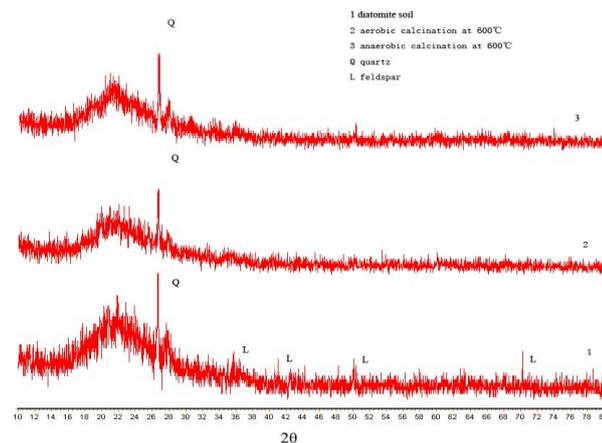


Fig 3. XRD patterns of native and calcined diatomite

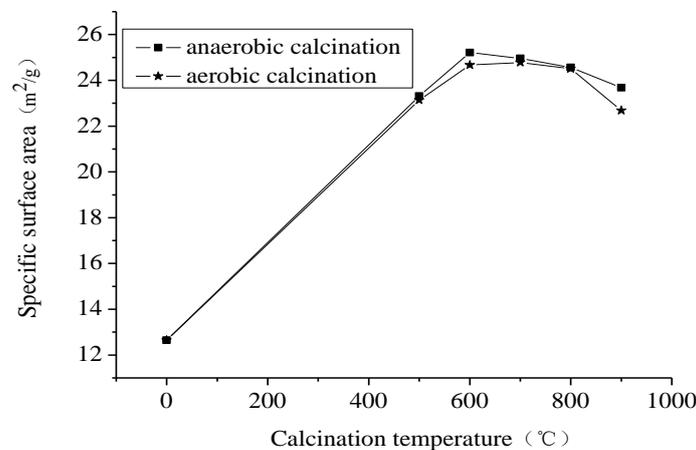


Fig 4. Specific surface area of diatomite at different calcination temperatures

3.4 BET analysis

Figure 4 showed the specific surface area of the diatomite at different calcination temperatures under conditions of aerobic calcination and anaerobic calcination. It can be seen from Figure 4 that specific surface area of original soil was $12.65\text{m}^2/\text{g}$. The specific surface area of diatomite increased firstly and then decreased with the increase of calcination temperature, and reached the maximum at 600°C . Respectively, the specific surface area of anaerobic calcination and aerobic calcination were $25.21\text{m}^2/\text{g}$ and $24.67\text{m}^2/\text{g}$. The specific surface area decreased from 600°C , which was in accordance with the results of the combined water rate analysis. The main reason was that the impurities were removed in the microporous of diatomite and the specific surface area of diatomite increased with increasing temperature. When the temperature reached to 600°C , the microporous of the diatomite melt and the average pore size increased. Therefore, the specific surface area of diatomite decreased gradually. The specific surface area under the condition of anaerobic was larger slightly than aerobic condition. So the effect of aerobic or anaerobic calcination on the specific surface area of diatomite was not significant.

3.5 SEM analysis

Figure 5, Figure 6, Figure 7 were the SEM of diatomite soil, 600°C anaerobic calcination and 600°C aerobic calcination. It can be seen from the figures: there were more impurities on the surface of diatomite shell before calcination, after calcination, the similar plant detritus were reduced obviously, and the pore structure on the shell became clear, most of the diatom shells were retained intact. The internal pore structure of diatomite was dredged highly through removing impurities under high temperature calcination, and was consistent with the results of XRD.

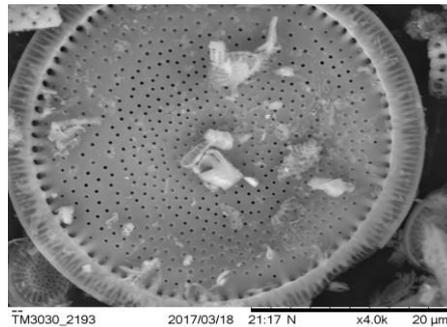


Fig 5. Diatomite soil

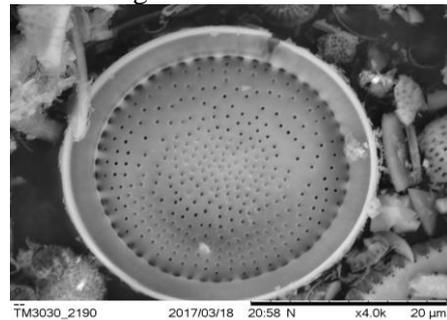


Fig 6. Anaerobic calcination 600°C

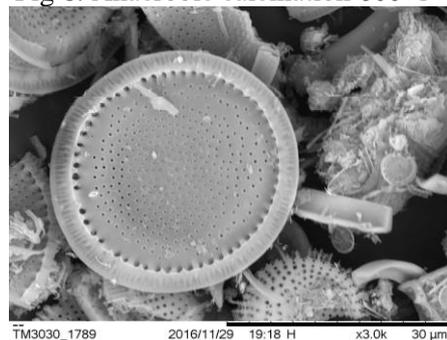


Fig 7. Anaerobic calcination 600°C

4 Conclusions

In this experiment, the volcanic ash activity of diatomite calcination in anaerobic condition was higher than aerobic condition calcination, the activity of volcanic ash was the highest at 600°C for 2h; The improvement of volcanic ash activity of diatomite was mainly due to the improvement of pore structure and surface structure under high temperature, calcination diatomite under anaerobic conditions, reduced CO₂ emissions, organic matter was formed C by anoxic calcination, and C was attached to the surface of the diatomite, The surface of diatomite played a modified role, which promoted the reaction with Ca(OH)₂ that cement hydration product, the anaerobic calcination diatomite as a pozzolanic active admixture was worth further study.

Acknowledgments

This work was financially supported by the National Key Technologies Research and Development Program of China (No. 2016YFC0701002).

References

- [1] Lian & Zhang. (2001). A rapid method for assessing the activity of volcanic ash. *J. Journal of Building Materials*, (3): 119-141.

- [2] Mao, Huang, Luo, Liu & Zhang. (2016). Study on the Test Method of Volcanic Ash Activity of Volcanic Gray Materials. *J. Nonmetallic ore*, 39(3):4-6.
- [3] Rahhal V & Talero R. (2004) Influence of two different fly ashes on the hydration of portland cements. *J. Journal of thermal analysis and calorimetry*, 78(1): 191-205.
- [4] Xiao & Pang. (2017). Research and Development of Diatomite in Indoor Functional Building Materials. *J. Journal of Jilin Architectural University*, 34(1):37-40.
- [5] Zhang & Huang.(2011). Evaluation method of volcanic ash activity and its influencing factors. *J. Materials Herald*, 25(15):104-106.