The Investigation about Gel Properties of New Composite Materials with Meta-kaolin

Jiaqian Ren* and Meng Li

Department of urban and rural planning, School of architecture, Southeast University, Nanjing 210096, PR China

*Corresponding author

Abstract. As one of the most promising building materials, the composite materials of metakaolin have a wide range of applications and excellent performance. Owing to the lack of research on the early-high-strength characteristics of MK, this article tries to make some meaningful attempts. In terms of the characteristics of hydration calcium silicate with gelation performance, this paper studied the preparation of metakaolin, the type of hydrothermal synthesis products, and the corresponding hydration dehydration phase and its rehydration characteristics. Through the exploration and research of the metakaolin hydrothermal synthetic process, it has prepared a new type of early-high-strength concrete regulator and opened up a new way for the effective application of kaolin. The preparation process, structure, and properties of MK-based concrete performance regulators were systematically investigated by XRD and SEM analysis. The macroscopic properties of the MK-based concrete performance regulator were discussed through coagulation time, hydration heat, and compressive strength performance.

1. Introduction

Kaolin is a natural mineral that is rich in our reserves and has a long used widely in light industry, chemical, building materials, petroleum, medicine, health, and other fields because of its physical and chemical properties, involving in the specific surface area, cation exchange capacity and ion absorption [1-5]. Besides, owing to a friendly environmental feature of low CO2 emission, the earliest research of kaolin polymer was found by Joseph Davidovits in 1972 [6]. Cement concrete of kaolin component pavement has good construction performance and excellent durability, and the road structure is strong, anti-skid performance and wear resistance, having a long service life, so ordinary cement with metakaolin becomes an interesting subject [7-8]. On account of the good properties of the composites of metakaolin polymer and cement, lots of investigators spent much attention and time to consider composite materials [9-13]. Metakaolin polymer materials include three-dimensional amorphous aluminosilicate binders [14], which can synthesize under normal temperature state through kaolin [15-16]. As the base of aluminosilicate, metakaolin is the most extensive source [17].

Except for the mechanical properties and durability of MK composite material, another reason for using metakaolin to replace cement is that environmental problems because of the high energy consumption of cement compared to low CO₂ emission of metakaolin. As the composite materials, including cement and metakaolin, they can be as the supplementary cementitious materials (SCMs). The regulator based on metakaolin demands needs to have some specific and strict features, such as good curing behavior, strength and durability development, less time conservation, etc. To well
mechanical properties and elevated durability, the metakaolin-based polymer produced by regulator and cement requires specific conditions including curing time, conservation temperature. For example, successful polymer hydration needs 50 and 80°C to suitable value. Many silicate materials (such as Portland cement, slag, volcanic ash material), as the composition of calcium material, can be combined with water at room temperature or hydrothermal reaction to produce hydration products. Hydration products are mainly hydration Calcium silicate, hydrated calcium aluminate, and hydrated calcium sulphoaluminate. Many scholars have studied and discussed the thermodynamics, kinetics and hydration product composition and structure of hydration reaction, and the thermal stability (dehydration temperature) of hydration products. In recent years, kaolin cement-based materials show people a fascinating development prospects with a wide range of applications and excellent performance, but this research work is still in the initial stage, having an urgent need to improve people's understanding, strengthen investment and its formation mechanism, preparation technology, modification technology, and application development research. Kaolin cement-based materials research and development have a profound impact on non-metallic mineral resources development and utilization.

2. Experimental

2.1. Experimental Scheme and Technical Route
The experimental scheme is based on the preparation of reasonable raw materials, kaolin from the preparation of meta-kaolin, and then hydrothermal synthesis for intermediates, and finally through a reasonable calcination system to obtain dehydration phase. Based on the research system of hydrated calcium silicate (CSH) dehydrated phase cementitious material, a new type of cementitious material containing the CSH dehydration phase was prepared.

2.2. Dehydration Phase Calcination System
After drying and grinding, kaolin was calcined by the muffle furnace, then the kaolin was obtained by heating for set time. The purpose of hydrothermal synthesis is to allow meta-kaolin and CaO for full hydration reaction under hydrothermal conditions. Hydrothermal synthesis of the hydrate was calcined to obtain a dehydrated phase of Silicate or Aluminate. The instrument used for the muffle furnace, control muffle furnace heating rate of 10°C /min, reach a predetermined temperature rise after 60-80 minutes, and then remove the sample quenching.

2.3. Test Methods
X-Ray Diffraction (XRD): The powder diffraction pattern was determined by the D / max-RB X-ray diffractometer of Japan RIGAKU. Scanning electron microscopy (SEM): The JSM-5610LV scanning electron microscope produced by JEOL (Japan Electronics) Co., Ltd.

3. Results and Discussions

3.1. Characteristics of Calcined Kaolin
The active component is kaolinite (2Al2O3 • 4SiO2 • 4H2O) in kaolin, and it is dehydrated with appropriate temperature (650 – 800°C) to form metakaolin.
In Fig.1 and Fig.2 $\theta$ is a typical kaolin diffraction peak between 35° and 40°. It can be seen from the figure that the main characteristic peaks of kaolinite are kaolinite, illite, and quartz ($\alpha$-SiO$_2$), and it also shows that kaolin raw material contains a certain amount of quartz, which will adversely affect the activity of meta-kaolin. Fig.2 shows that the strongest characteristic peaks of kaolin ($d = 0.715$, $d = 0.373$) disappeared after calcination at 750°C for 2h, and the X-ray diffraction peak was diffused as a typical amorphous structure diffraction peak. This shows that kaolin becomes the amorphous crystalline after calcination. The ionic bond in kaolin breaks, which reduces the number of crystals, then kaolin is converted to meta-kaolin. Both XRD patterns could see obvious quartz (SiO$_2$) characteristic peaks.
It can be observed that the grain shape of kaolin is flaky before and after calcination from Fig. 3. Before calcination, the particles are dispersed (figure 3-a,c), and the crystal structure is neatly arranged. After calcination, the particles are reduced, the sticking particles increase, the gap decreases, and the crystal structure is destroyed with the disorderly arrangement (figure 3-b,d).

3.2. Regulator Composition and Formation Process
The composition of hydrated calcium silicate and hydrated calcium aluminate was studied in detail, and its thermal stability (dehydration temperature) was analyzed. The experiment was carried out by XRD, SEM, IR, and TG. The mechanism of the preparation of the dehydrated phase was studied to establish the research system of aluminosilicate dehydrated phase cementitious material.

It can be seen that the regulator contains many components, and the diffraction peaks overlap each other from Fig. 4. There are mainly C12A7, C11A7CaCl2, CaSO4, CaO, β-C2S, β-CS, and SiO2. There is no phenomenon of strength contraction and the age of each sample exceeds the standard sample strength. The cement strength, which incorporates 5% and 10% of the regulator, is similar between the early 1d and 3d Conservation and the compressive strength of the standard cement is greatly improved.
It can be observed that the structure of the regulator is similar to the CSH gel from Fig.5. The particle fineness is small, the pores are loose, and the pore spacing is much smaller than that of the hydrothermal synthesis product. The ball is wrapped by the Mesh filaments to form the reunion phenomenon, and these phenomena can explain the specific surface area of the regulator is very large.

4. Conclusion
In this paper, the preparation process of a kaolin-based concrete performance regulator and its structure and performance mechanism are studied systematically. The conclusions of the study are as follows:
(1) After kaolin raw material calcined at 750°C, crystal structure damaged, crystal structure transfer to the amorphous structure, and form active meta-kaolin. After the hydrothermal synthesis of kaolinite as the main component, CSH gel, hydrated calcium aluminate C4AH13, C3AH6, hydrated garnet C3ASH4 were also produced.
(2) The regulator includes C12A7, C11A7CaCl2, CaSO4, CaO, β-C2S, β-CS, and SiO2.

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