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Effects of Calcined Nano Attapulgite Clay on the Mechanical and Durability Properties of Cement Mortar

Yihong Xu[1,2*]
[1]Liaoning provincial college communications, Shenyang, Liaoning, China
[2]Liaoning Province Bridge Safety Engineering Professional Technology Innovation Center, Shenyang, Liaoning, China
*Corresponding Author.

Abstract:

In order to study the effect of the calcined nano attapulgite clay on Mechanical and Durability the properties of cement mortar, using the box-type resistance furnace to high temperature calcination, the dispersion, mechanical and durability properties of calcined nano attapulgite clay additives was investigated. At the same time, the micro structure of the cement mortar of adding the attapulgite clay was revealed. The results indicate ultrasonic dispersion can improve the dispersion of calcined nano-attapulgite clay in water. When the content is less than 2%, it can basically guarantee that the calcined nano-attapulgite clay will not settle in water for a short time. Compared with the normal cement mortar, mixed with the calcined nano attapulgite clay can reduces the fluidity of cement mortar, improve the mechanics and durability of the cement mortar. When the cement mortar of adding the 0.01 attapulgite clay, in 56 days of flexural strength than ordinary cement mortar increased by 6.43%, the compressive strength increased by 12.97%, the chloride ion diffusion coefficient of 28 days reduced by 19.84%. Microscopic test results indicated that the appropriate amount of attapulgite clay can promote hydration process of the cement, improve the microstructure of cement mortar, reduce internal cracks and pores. The microscopic structure was more compact.

Keywords: *calcination, attapulgite clay, cement mortar, mechanical properties, diffusion coefficient, microstructure*

I. INTRODUCTION

With low price, simple construction and broad source, cement mortar and concrete are extensively used in civil engineering structures across the world [1]. Nevertheless, the cold environment, erosion of sulfate, seawater and deicing salt in the north lead to premature material deterioration, with the geotechnical structure life far shorter than designed service life, incurring huge economic losses [2,3]. At present, durability of concrete structures is enhanced mainly through doping of nano-scale mineral admixtures. Introduced into cement-based materials by taking advantage of the unique interface, quantum and size of nano-materials, it increases the mechanical properties of concrete and allows more compact microstructure, thereby lowering the permeability of chloride ions and strengthening durability of the structure itself. In spite of this, nanomaterials are difficult to prepare, highly priced, and involve uneven standards, so little progress is made in its engineering applications [4-6]. Accordingly, it is of positive significance to find mineral admixtures with low cost and distinct feature advantages to increase the durability of cement-based materials.

Nano-attapulgite clay contains hydrous magnesium-rich aluminum silicate of a fibrous structure. With chemical formula Mg5Si8O20 (OH) 2(OH2) 4·4H2O, it has unique colloidal properties, strong adsorption capacity. Resistant to salt and alkali and high temperature, the material has small shrinkage, easy dispersion and good suspension stability [7]. Moreover, with abundant reserves and low price, it can also accelerate hydration reaction and strengthen the cohesion and bonding force of cement slurry, which is thus widely used in fine chemicals, new building materials, environmental protection, pesticides, etc.[8-13]. Xue Lili et al. [14] modified the polymer mortar using four kinds of attapulgite, finding that the mechanical properties and impermeability of the polymer waterproof mortar were optimum when 2% nano-attapulgite was doped. Shen Wenzhong et al. [15] studied the effect of modified attapulgite clay on the strength and working performance of fresh mortar. It was found that modified attapulgite clay reduced the mortar stratification and improved working performance, while mortar compressive and flexural strength were decreased. Liu Jing [16] studied the effect of attapulgite soil and air-entraining agent on the thixotropic properties of fresh concrete, finding that double mixing of attapulgite and air-entraining agent could increase the static yield stress of fresh concrete and enhance the concrete ability to maintain slope. Li Wei et al. [17] studied the effect of nano-attapulgite clay dosage on the shrinkage rate, compressive shear bond strength and softening coefficient of granular thermal mortar, and recommended the optimal dosage through experiments. It was found that the mechanical properties of mortar gradually decreased with the addition of attapulgite clay. Under 8% dosage of attapulgite, the thermal mortar strength was nearly 20% lower than that of ordinary mortar. Qu Meiyan et al. [18] studied the effect of attapulgite clay on the performance of cement-stabilized macadam, finding that doping of attapulgite clay can improve the unconfined compressive strength of cement-stabilized macadam and reduce the dry shrinkage coefficient. Lv Dan [19] mixed nano-attapulgite with mineral admixtures to study the effect of different dosages of mineral admixtures on concrete durability. It was found that addition of mineral admixture reduced the strength of nano-attapulgite concrete in the early stage, but with strength increased later. Meanwhile, the carbonation property of specimens was decreased.

Despite certain progress made in the research of nano-attapulgite clay mortar material, nano-attapulgite clay is easy to agglomerate in water, forming secondary particles. It is urgent to solve the problem of how to improve the dispersibility of nano-attapulgite clay. Its mechanics, durability and microscopic properties still require further study. This paper investigates the dispersibility of nano-attapulgite clay in water under different dosages and its effects on the mechanics, permeability and microstructure of cement mortar.

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II. EXPERIMENT

2.1 Raw Materials

The nano-attapulgite is clay produced by Changzhou Dingbang Mineral Products Technology Co., Ltd., and the cement is ordinary Portland cement of grade PO·42.5R produced by Dalian Xiaoyetian Cement Plant. **TABLE I** displays the main components of nanoclay and cement. Standard sand was "China ISO Standard Sand" (No.: GSB08-1337) produced by Hebei Yuke Instrument Equipment Co., Ltd.; distilled water was used for chloride ion diffusion test, and tap water was used as mixing water.

TABLE I. Chemical composition of cement and nano-attapulgite clay

MATERIAL	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	K_2O	Na_2O	Lol
CEMENT	59.3	21.91	6.27	3.78	1.64	2.41	--	--	4.96
NANO-ATTAPULGITE CLAY	1.8-2.5	50.4-61.3	9.4-9.5	4.0-5.0	9.3-10.5	--	0.2-0.6	0.5-1	11.94-13.46

2.2 Specimen Preparation

In the experiment, nano-attapulgite clay was calcined at 650 ℃ in a box-type resistance furnace. The dosage of nano-attapulgite clay accounted for 0%, 1%, 3% and 5% of the cement mass. The specimens were numbered ATB-0, ATB-1, ATB -3, ATB-5, specifically shown in TABLE II. The specimen has a dimension of 160mm×40mm×40mm, and 4 groups were prepared, with 3 specimens in each group, which were used to determine the compressive and flexural strength of nano-attapulgite cement mortar specimens of different ages. For cylindrical test mode with a dimension of Φ100mm×50mm, three groups were prepared, with 3 specimens in each group, which were used to determine the chloride ion diffusion coefficient of the nano-attapulgite cement mortar specimens of different ages. When preparing the cement mortar, firstly pour the attapulgite clay into the cement mortar mixer after 30min ultrasonic treatment, add cement, and mix it for 2min according to the relevant regulations in the current standard: "Test Regulations for Highway Engineering Cement and Cement Concrete" (JTGE30-2005). Then, add 20% remaining water, quickly stir for 4 min. The prepared specimens were cured in a standard curing box until the test age for testing.

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TABLE II. Specimen number and mix ratio

SPECIMENT NUMBER	WATER/ML	STANDARD SAND/G	NANO-ATTAPULGITE CLAY/G	CEMENT/G
ATB-0	225	1350	--	450
ATB-1			4.5	445.5
ATB-3			12.5	437.5
ATB-5			22.5	427.5

2.3 Experimental Method

The nano-attapulgite clay suspension was ultrasonically dispersed by an ultrasonic disperser at an ultrasonic power of 3000KHz. The suspension was dispersed for 30min and then evaluated for ultrasonic dispersion.

The specimen was cured to the test age, and the fluidity of the cement mortar specimen was measured according to the relevant provisions in "Determination of the fluidity of cement mortar" (GB/T 2419-2005). The measurement was carried out with a ruler along the two directions perpendicular to each other at the mortar bottom. The average value of the measured results was taken as the fluidity of the cement mortar, with accuracy up to 1mm.

The flexural and compressive strengths of cement mortar were measured in accordance with the "Test Regulations for Highway Engineering Cement and Cement Concrete" (JTGE30-2005).

The chloride ion diffusion coefficient of cement mortar was measured by HMK-10 concrete multifunctional chloride ion durability tester, and the chloride ion diffusion coefficient of cement mortar specimens was tested for different ages.

Scanning electron microscope was used to test the microstructure of the 28-day-old specimens. Before the test, the test specimens were dried, and the samples were selected, placed and fixed on a metal base, then sprayed with gold and used for SEM measurement.

III. RESULTS AND DISCUSSION

3.1 Effect of Calcination on the Microstructure of Nano-attapulgite Clay

The scanning electron microscope images of the attapulgite clay before and after high-temperature

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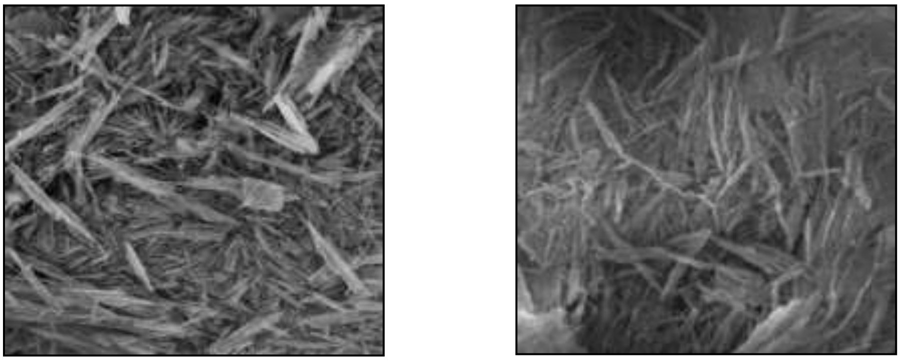
calcination are displayed in Fig 1. According to the microstructure diagram, the fibrous rod like crystal has a length of about 0.5-1 μm and a diameter of about tens of nanometers, presenting a disordered fibrous structure. After calcination, the crystal structure of nano-attapulgite clay does not collapse, but still maintain a complete crystal structure. Crystal structure has significantly decreased aggregation after calcination, with rod bundle volume increased and crystal structure loosened, which facilitates the dispersion in cement-based materials.



(a) before calcination (b) after calcination

Fig1: SEM image of nano-attapulgite clay

The XRD patterns of the nano-attapulgite clay before and after calcination are illustrated in Fig 2. The figure shows that the diffraction peaks of the nano-attapulgite clay disappear after calcination, with abundant SiO2 and alkali metal salts generated, and the crystalline properties are unstable, with pozzolanic activity increased.

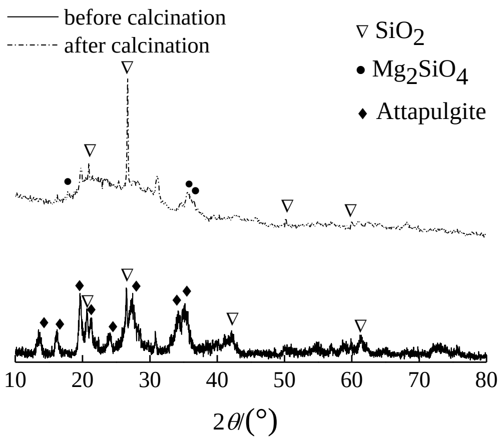


Fig 2: XRD pattern of nano-attapulgite clay

3.2 Dispersibility of ATB Dosage in Water

To assess the dispersibility of calcined nano-attapulgite clay in water, in the experiment, the water-to-binder ratio was 0.5, the ATB dosage was 1%, 3% and 5% of the cementitious material mass, and ATB suspension was subject to ultrasonic dispersion. By comparison with the unsonicated suspension, variation of sedimentation volume over time was investigated under different ATB dosages. Fig 3 shows that, with the increase of nano-attapulgite dosage, the sedimentation volume gradually increases. Under

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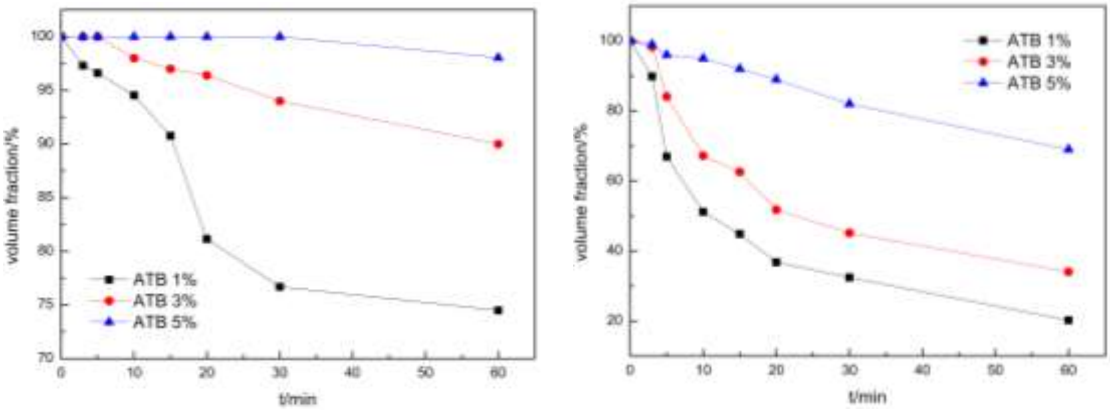
nano-attapulgite dosages of 1%, 3% and 5%, the sedimentation volume after 60 min standing decreased by 72.83%, 34.07% and 29.62% compared with that before ultrasonic dispersion. Hence, ultrasonic dispersion can improve the dispersibility of nano-attapulgite clay in water. When the dosage is within 2%, it can be basically guaranteed that nano-attapulgite clay does not settle in water in a short time, but with the increase of nano-attapulgite dosage, the settlement gradually increases



(a) before ultrasonic dispersion (b) after ultrasonic dispersion

Fig 3: variation of sedimentation volume over time under different ATB dosages

3.3 Effect of Nano-attapulgite Clay Dosage on Cement Mortar Fluidity

Fig 4 shows the effect of nano-attapulgite clay dosage on cement mortar fluidity. The figure shows that the cement mortar fluidity gradually decreases with the increase of nano-attapulgite clay dosage. Under 0% dosage of nano-attapulgite clay, the cement mortar fluidity is 199mm. Under 3% dosage of attapulgite, the cement mortar fluidity drops to 145mm. Under 5% dosage of attapulgite, the cement mortar fluidity drops to 134mm, which is 32.66% lower than that of the benchmark specimen, resulting in poor construction performance of cement mortar. For its reason, the water in the cement mortar is adsorbed by the nano-attapulgite clay, resulting in decreased free water in the slurry, which increases the friction between the attapulgite clay and the cement particles, makes the mortar thicker and lowers the mortar mobility. Hence, the dosage of nano-attapulgite clay should be strictly controlled in the construction process to prevent agglomeration of disorganized fibrous rod crystal structures from damaging its working performance.

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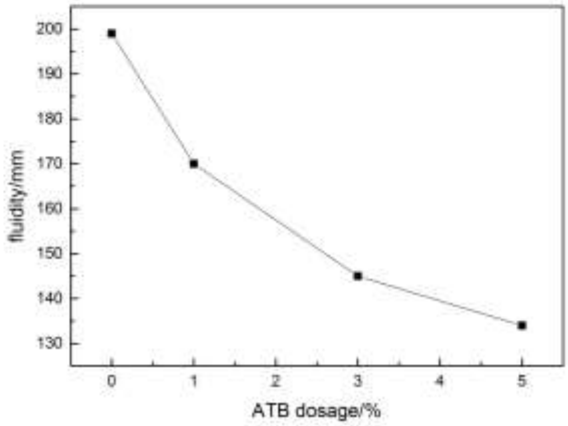

Fig 4: effect of ATB dosage on cement mortar fluidity

3.4 Effect of Nano-attapulgite Clay Content on Mechanical Properties of Cement Mortar

Based on the existing research results [14-17], it is found that after direct doping of uncalcined nano-attapulgite clay, a higher water-water-cement ratio is required under the strong adsorbability of nano-attapulgite clay to maintain its working performance, thus resulting in generally reduced mechanical properties of cement-based materials. High-temperature calcination increases activity of nano-attapulgite clay, leading to greatly enhanced mechanical properties.

Fig5 displays the effect of calcined nano-attapulgite clay dosage on the flexural strength of cement mortar. It suggests that nano-attapulgite clay can increase the flexural strength of cement mortar. Under 1%, 3% dosage of nano-attapulgite clay, cement mortar specimen at the age of 3d and 7d has a flexural strength not much different from that of the benchmark specimen. Under 5% dosage of nano-attapulgite clay, cement mortar has a flexural strength 2.87% and 5% higher than that of ordinary cement mortar, respectively. Under curing age 28d, cement mortar doped with 3%, 5% nano-attapulgite clay has a flexural strength 1.63% and 5.47% higher than that of ordinary cement mortar, respectively. At curing age 56d, cement mortar doped with 1%, 3% nano-attapulgite clay has a flexural strength 6.34% and 7.25% higher than that of ordinary cement mortar, respectively. Hence, in the early age, the flexural strength of cement mortar doped with nano-attapulgite clay increases slowly as the curing age prolongs, and when the curing age exceeds 28d, the flexural strength of cement mortar increases rapidly. At curing age 56 d, the nano-attapulgite clay creates a pozzolanic effect in the later stage, which hastens the secondary hydration and increases the flexural strength of the cement mortar.

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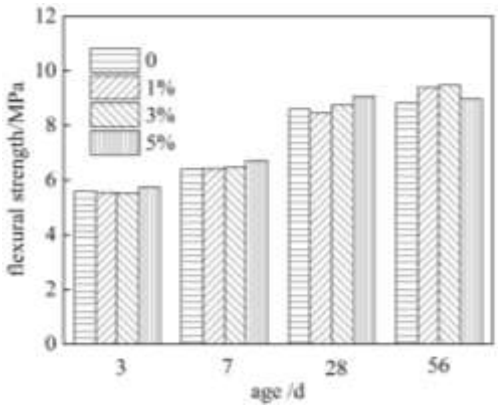

Fig 5: effect of ATB dosage on flexural strength of cement mortar

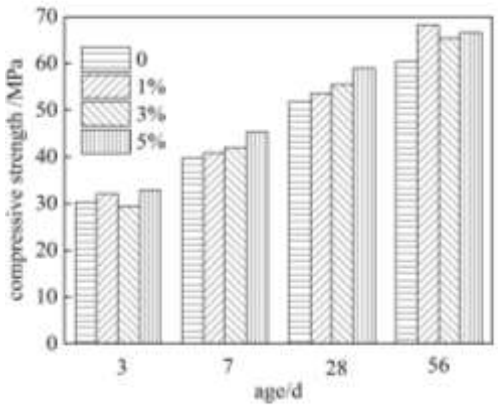

Fig 6: effect of ATB dosage on compressive of cement mortar

Fig 6 illustrates the effect of different dosages of nano-attapulgite clay on the compressive strength of cement mortar. The figure indicates that an appropriate amount of nano-attapulgite clay can increase the compressive strength of cement mortar specimens. In the early stage of curing, nano-attapulgite clay has a compressive strength not much different from that of ordinary cement mortar. At curing age 7d and 28d, the compressive strength of cement mortar increases with the additional nano-attapulgite doping. At curing age 28d, the cement mortar doped with 1%, 3% and 5% nano-attapulgite has a compressive strength 3.44%, 7.29% and 14.02% higher than that of ordinary cement mortar, respectively. At curing age 56, cement mortar doped with 1%, 3% and 5% nano-attapulgite has a compressive strength 12.97%, 8.27% and 10.29% higher than that of ordinary cement mortar, respectively. This is mainly because the increased pozzolanic activity of the nano-attapulgite clay hastens the hydration of the cement, leading to greater compressive strength.

From the above analysis, it can be seen that an appropriate amount of calcined nano-attapulgite clay can enhance the mechanical properties of cement-based materials, which accords with the research results of Kawashima et al. [20, 21].

3.5 Effect of Nano-attapulgite Clay Dosage on Chloride ion Diffusion Coefficient of Cement Mortar

Fig 7 demonstrates the effect of nano-attapulgite clay dosage on the cement mortar microstructure. The figure indicates that different dosages of nano-attapulgite clay can reduce the chloride ion diffusion coefficient of cement mortar. In particular, at an early age, under 5% dosage of attapulgite, cement mortar has the greatest resistance against chloride ion penetration. At 3d age, dosage of 1%, 3% and 5% nano-attapulgite reduces chloride ion diffusion coefficient by 25.95%, 29.58% and 33.97% compared to ordinary specimens, respectively. At 7d age, dosage of 1%, 3% and 5% nano-attapulgite reduces chloride ion diffusion coefficient by 34.21%, 31.56% and 36.34% respectively compared with ordinary specimens. At 28d age, dosage of 1%, 3% and 5% nano-attapulgite reduces chloride ion diffusion coefficient by 20.63%, 26.90% and 33.25% compared with the ordinary specimen, respectively. This is mainly due to the pozzolanic and filling effects of nano-attapulgite clay. As a nano-material, it has tiny particle diameter,

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making it easily fill the pores between the cement particles during the cement slurry hydration, thus resulting in greater microstructure compactness. On the other hand, cement mortar produces a large amount of C-S-H gel during the hydration, which perfects the cement mortar microstructure and enhances the cement mortar resistance against chloride ion erosion.

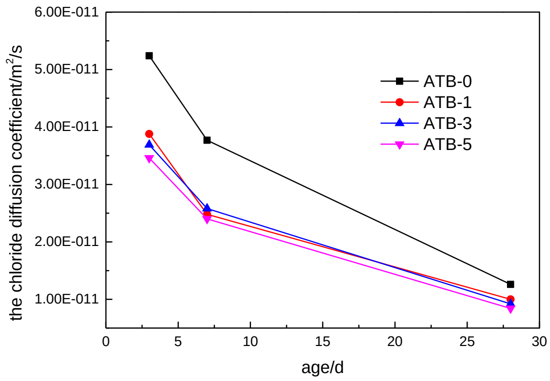

Fig 7: effect of ATB dosage on chloride ion diffusion coefficient of cement morta

3.6 Effect of Nano-attapulgite Clay Dosage on Cement Mortar Microstructure

To study the effect of different dosages of nanoclay on cement mortar microstructure, the specimens with a curing age of 28d were taken out, and small cubes of 1cm×1cm×1cm were selected within the range of 10-15mm from the bottom. The surface was sprayed with gold and then subject to SEM analysis. The microscopic morphology of the hardened crystals of cement mortar doped with different amounts of nano-attapulgite clay is shown in Fig 8. Fig 8(a) reveals that the microstructure of the cement mortar without calcined attapulgite has obvious pores, and the crystal products are not tightly bonded. CH crystal and needle-stick C-S-H gel are overlapped with each other, displaying mesh interwoven skeleton structure and relatively loose interior of the microstructure. After doping of nano-attapulgite clay, cement mortar has more compact microstructure and more hydration products. Where, cement mortar doped with 1% nano attapulgite has evenly distributed hydration particles, fewer pores, relatively dense microstructure, displaying block superposition and dense layered bonding. For cement mortar doped with 3% nano attapulgite, it can be clearly seen from the microstructure that CH crystals and rod-like crystals are interspersed in the C-S-H gel, making them intertwined, with cracks and pores reduced, resulting in dense microstructure. For cement mortar doped with 5% nano attapulgite, it can be clearly seen from the microstructure that a small amount of rod-like crystals are interspersed in the C-S-H gel, displaying mesh superposition in the interior structure. There are obvious cracks and pore structure in each layer, resulting in poor bonding of the cementitious structure, so the mechanical properties of cement mortar are reduced. The analysis suggests that, an appropriate amount of nano-attapulgite clay can accelerate cement hydration and perfect the cement mortar microstructure.

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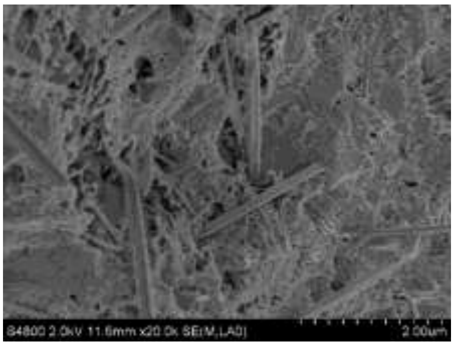
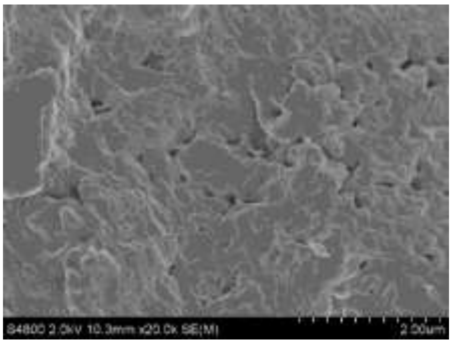
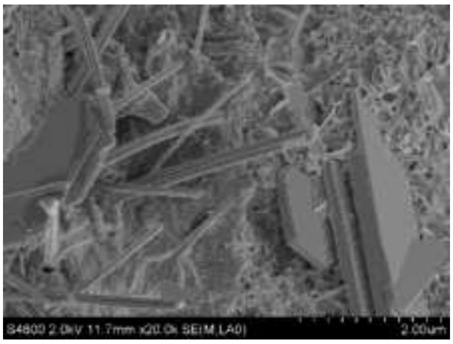


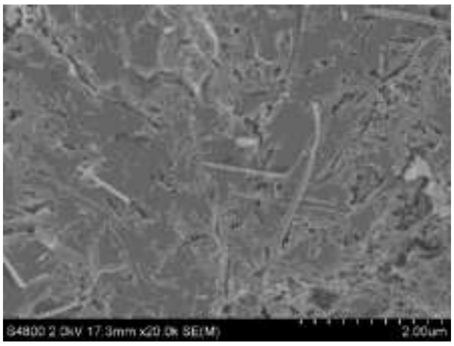
(a) ATB:0%



(b) ATB: 1%



(c) ATB: 3%



(d) ATB: 5%

Fig 8: SEM morphology of ATB cement mortar at 28d

IV. CONCLUSION

(1) Through high-temperature calcination of nano-attapulgite clay, the volume of rod bundles increases and the crystal structure becomes loose, which facilitates dispersion in cement-based materials, but increased dosage of nano-attapulgite clay content increases the cement mortar fluidity and downgrades the working performance.

(2) Ultrasonic dispersion can increase the dispersibility of nano-attapulgite clay in water. With the increase of dosage, the dispersibility deteriorates. When the dosage is within 2%, it can be basically guaranteed that the calcined nano-attapulgite clay will not settle in water within a short period of time.

(3) Doping appropriate amount of calcined nano-attapulgite clay can improve the mechanical properties of cement mortar. Compared with specimens before doping of nano-attapulgite clay, at 56d age, the specimens with 1% doping of nano-attapulgite clay has a compressive strength increased by 12.97% and a flexural strength increased by 6.43%. When the dosage exceeds 5%, the mechanical properties are decreased.

(4) Appropriate amount of nano-attapulgite clay can reduce the chloride ion diffusion coefficient of cement mortar. In particular, in the early age, under 5% dosage of nano attapulgite, the cement mortar has

greatest resistance to chloride ion penetration.

(5) Microscopic studies suggest that an appropriate amount of nano-attapulgite clay can accelerate the cement hydration and perfect the cement mortar microstructure. Cement mortar doped with 5% calcined nano-attapulgite clay has worse microstructure.

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