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Study on Compressive Strength of Cement Mortar Containing Rice Husk Ash and Silica Fume under Freeze-Thaw Cycles

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Abstract - *To investigate the influence of rice husk ash (RHA) on the compressive strength and freeze-thaw resistance of cement mortar, the WAW-1000 universal testing machine was conducted to analyze the compressive mechanical properties of cement mortar specimens under varying RHA replacement ratios and freeze-thaw cycles. Scanning Electron Microscope (SEM) was further used to analyze the changes in the microstructure of cement mortar. The results demonstrated that the compressive strength of cement mortar increased with the increased of RHA replacement ratios. After curing for 28 days, the compressive strength of specimens with 5% RHA replacement for silica fume increased by 70.4% compared to the control group. With an increasing number of freeze-thaw cycles, the rate of compressive strength reduction in cement mortar containing rice husk ash and silica fume was significantly slower than the control group. SEM microstructural analysis revealed that RHA effectively mitigates damage induced by freeze-thaw cycles, thereby improving the durability of cement mortar.*

Key Words: Rice husk ash; Cement mortar; Freeze-thaw cycles; Freeze-thaw resistance; Compressive strength

1. INTRODUCTION

The cold regions are widely distributed in the northern of China [1]. With the shift of China's social economy and infrastructure construction towards the western region, the development of cold region engineering has progressed rapidly. However, buildings in cold regions are inevitably affected by freeze-thaw cycles [2], which can cause significant harm to the projects. According to relevant data statistics [3], the cement industry has a large energy consumption, leading to the greenhouse gas emissions account for 8% to 10% of the global total. At the same time, natural resources are becoming scarcer, and the use of industrial waste in cement concrete is becoming more common [4]. For example, the use of slag and fly ash in concrete can not only reduce resource waste and the pollution caused by waste accumulation, but also promote the development of new low-carbon and environmentally friendly building materials, generating excellent economic and environmental benefits [5-6].

China, the world's largest rice-growing country, produces about 40 million tons of rice husks every year [7]. However, due to high hardness and difficult decomposition of rice husks, serious environmental pollution has arisen. Researchers from around the world have actively discussed and conducted experimental studies on the utilization of rice husk ash. Countries, such as the United States, the former Soviet Union, Japan, and India, began to study its properties at an early stage. China has carried out more systematic research since the 1980s. Through research, it has been found that the chemical composition of rice husks calcined by high temperature is similar to silica fume, containing a large amount of highly active silica. Incorporating it into cement-based composites can significantly improve the mechanical properties [8], workability [9], and durability [10].

Therefore, this study uses the rice husk ash calcined at a controlled temperature of 600°C to replaces part of the silica fume, investigating its influence on the compressive mechanical properties and frost resistance of cement mortar. The results will provide a reference for the widespread application of rice husk ash in concrete.

1.1 Raw Materials

In this study, the rice husk is calcined into ash at a controlled temperature of 600°C. The grade of cement is ordinary Portland cement of 42.5. The fineness modulus of the sand is 1.69, and the silica content of the silica fume is 96%. The mixing and curing water used are distilled water.

1.2 Specimen Preparation

Firstly, Put the rice husks into the hydrochloric acid solution, stir thoroughly, and soak for 1 hour. Then, repeatedly rinse the acid-soaked rice husks with distilled water until the rinsing solution is in a neutral state. Next, put the rinsed rice husks into an oven for drying. Subsequently, put the dried rice husks into a muffle furnace and calcine them at a stability temperature of 600°C for 3 hours. Finally, put the calcined rice husks into an ice-water mixture at 0°C for rapid cooling. After cooling, grind them into a fine powder and sieve it through a 180-mesh sieve.

The mix proportion in this study is based on the research findings from existing references [11-13]. The materials mix proportions (kg/m^3) are as follows: cement: sand: water = 330:1210:160.

In the experiment, the cement mortar mixture is mechanically stirred and then fabricated the specimens with 6 mix proportions. The detail progress is as follow.

Prepare specimens with rice husk ash replacement ratios for silica fume of 0%, 10%, 30%, 50%, 70%, and 90%, and mark them with G1, G2, G3, G4, G5, and G6 respectively. According to the mix proportion and the requirements in the Test Method for Mechanical Properties of Ordinary Concrete (GB50081) to prepare and cure the specimens.

Table -1: Cement mortar ratio

Groups	water	Sand	cement	Rice husk ash	Silica fume
G1	0.038	0.423	0.104	0	0.011
G2	0.038	0.423	0.104	0.001	0.010
G3	0.038	0.423	0.104	0.003	0.008
G4	0.038	0.423	0.104	0.005	0.006
G5	0.038	0.423	0.104	0.008	0.004
G6	0.038	0.423	0.104	0.010	0.001

The 70.7mm×70.7mm×70.7mm cubic mold is used to make the specimens of cement mortar. Pour the raw materials for 6 groups with different mix ratios, 3 specimens in each group. Then, remove the specimens and soak them in water at (20 ± 2)°C for curing for 4 days.

1.3 Experimental methods and instruments

The freeze-thaw cycle is carried out for cement mortar specimens in accordance with the standard GB/T 50082-2009[14] Standard for Test methods for Long-term Performance and Durability of ordinary concrete. The freezing temperature is -20±2℃, the thawing temperature is 20±2℃. The freezing and thawing time are both 4 hours, and one cycle lasts 8 hours. The number of freeze-thaw cycles in this study is 20, divided into three groups: 0, 10, and 20 cycles. The specimen without freeze-thaw cycles was used as the control group.

The instrument type used for freeze-thaw cycles is STDW-40D high and low temperature test chamber, the temperature is set -20℃, the specimen is put in and the freezing time is set to 4 hours. At the end of 4 hours, the specimen was taken out and put into room temperature (20±2℃) for 4 hours. When the thawing time is over, one freeze-thaw cycle is completed. After reaching 0, 10 and 20 cycles, the basic physical of the specimen were measured, and carried out static uniaxial compression test to analyze the mechanical properties.

Static uniaxial compression test equipment is WAW-1000 universal testing machine, the test adopts the displacement loading method, the loading rate is 0.01m/s, the maximum loading force is 1000kN.

Before the static uniaxial compression experiment, the zero point of the force measuring should be corrected and adjusted. The cross-section of specimen was measured with a vernier caliper. Then, put the specimen in the center of the platen. During the experiment, apply the load slowly and evenly. Record the peak load when the specimen is compressed, and continue recording the load until the cement test block is compressed to the point of failure. After the experiment, the uniaxial compressive stress-strain curve of cement specimen is obtained according to the experimental data.

2. EXPERIMENTAL DATA ANALYSIS

The stress-strain curves of cement mortar specimens under different freeze-thaw cycles exhibit unimodal and typical characteristics of plastic materials. In the compaction stage, the stress of the cement mortar specimen increases gradually with the increase of strain. When entering the elastic deformation stage, the stress-strain curve is basically linear. With the increase of strain, the slope of stress-strain curve increases, and the deformation of the specimen is also intensified, and the specimen enters the plastic stage. After reaching the peak stress, it finally enters the failure stage. It can be seen from Fig. 1,2 and 3 that with the increase of freeze-thaw cycles, the peak stress of rice husk ash specimens with different contents as they reach failure decreases gradually. The peak stress is at a minimum when freeze-thaw cycles reach 20. With the increase of rice husk ash replacement ratio, the peak strain of specimens with different freeze-thaw cycles increases first and then decreases.

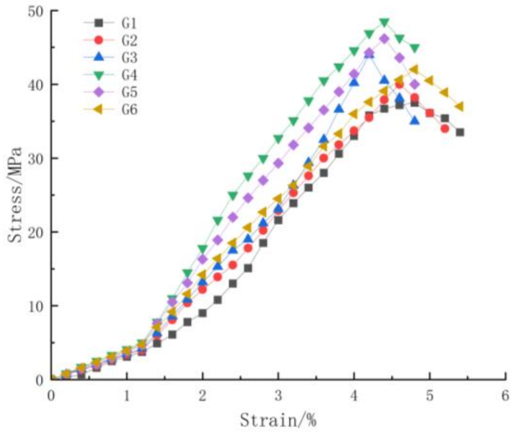


Fig -1: The 0th freeze-thaw cycle

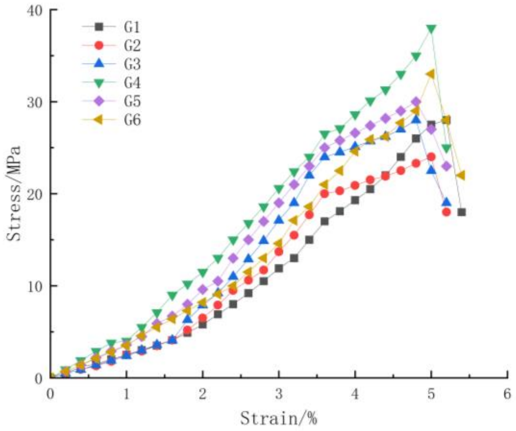


Fig -2: The 10th freeze-thaw cycle

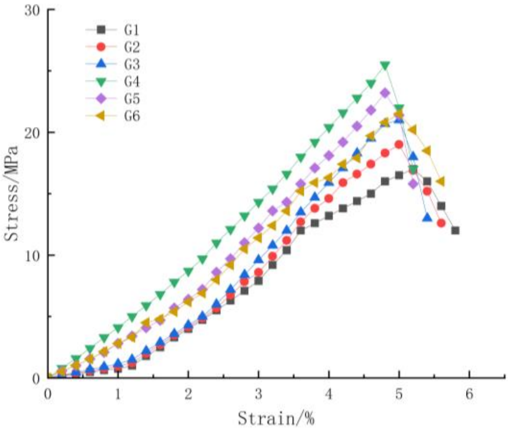


Fig -3: The 20th freeze-thaw cycle

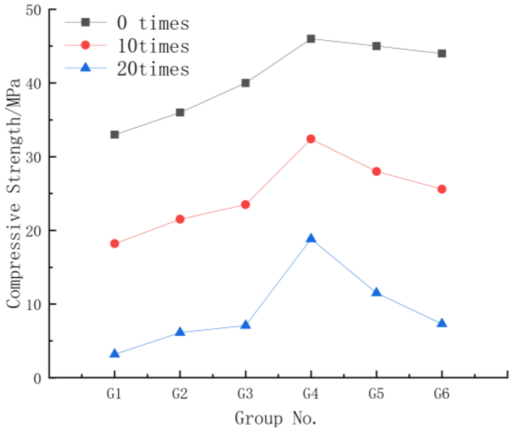


Fig -4: Compressive strength cement mortar after different freeze-thaw cycles

Figure 4 shows the compressive strength test results of specimens containing different contents rice husk ash and silica fume after curing 28d. The specimens contained 30% and 50% of rice husk ash replacing silica fume is higher compressive strength than the 100% silica fume. Therefore, when the proportion of rice husk ash replacing silica fume is less than 50%, the compressive strength of cement mortar can be improved. The compressive strength of the cement mortar specimens with 70% and 90% rice husk ash replacing silica fume is higher than the 100% silica fume. Moreover, under equivalent dosage conditions, the specimens incorporating only SF demonstrated lower compressive strength than those with combined incorporation of RHA and SF. This is because when the particle size and the mass fraction of SiO_2 are not much different, the existence form of SiO_2 will cause the difference in hydration reaction, leading to varying effects on compressive strength [15]. In addition to amorphous SiO_2, rice husk ash also contains a certain amount of crystalline phase SiO_2, which cannot promote hydration [16]. With the increase of freeze-thaw cycles, the compressive strength of rice husk ash and cement mortar specimens with different dosage decreases gradually. This is because the volume of water inside the specimen increases when it freezes, causing stress and deformation. During the thawing process, the water penetrates the interior, leading to further damage, which reduces the compressive strength of the specimen. After 20 freeze-thaw cycles, the compressive strength decreased significantly. Due to significant internal damage to the mortar, obvious cracks appeared on the surface, and granular material began to fall off. As a result, the strength of the mortar decreased significantly at this stage.

3. FAILURE MODE ANALYSIS

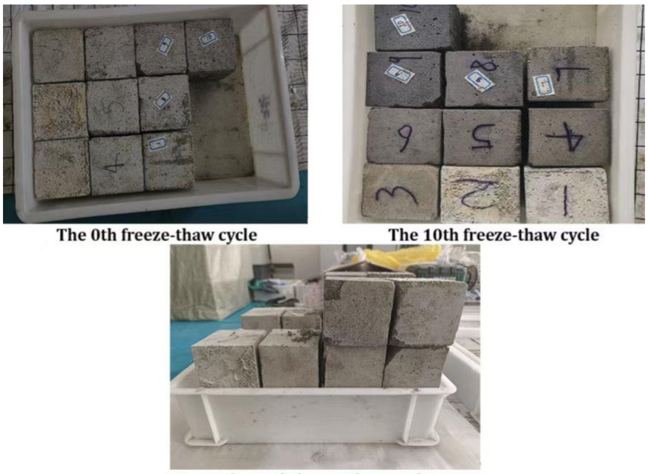

The 0th freeze-thaw cycle
The 10th freeze-thaw cycle
The 20th freeze-thaw cycle

Fig -5: Apparent characteristics after freeze-thaw cycles

It can be seen from Fig -5 that with the increase of freeze-thaw cycles of cement mortar, the surface erosion holes increase and the surrounding corners are passivated. This phenomenon shows that with the same dosage, the surface damage of the specimen contained different rice husk ash and silica fume will be more serious with the increase of freeze-thaw frequency.

The failure patterns of specimens under different freeze-thaw cycles can be seen from Figure 6. Figure 6 shows the shear failure occurs gradually with the increase of the number of freeze-thaw cycles of the specimen. The more freeze-thaw cycles of the specimen, the more macroscopic cracks will be generated during failure. The water inside the mortar gradually penetrated into the entire specimen. Because of thermal expansion and contraction, the water inside the specimen continuously transforms from liquid state to solid state, resulting in more and more cracks and pores inside the specimen. At this time, the mortar specimens have started to become brittle and exhibit peeling after loading, leading to a reduction in the compressive strength of the specimen.

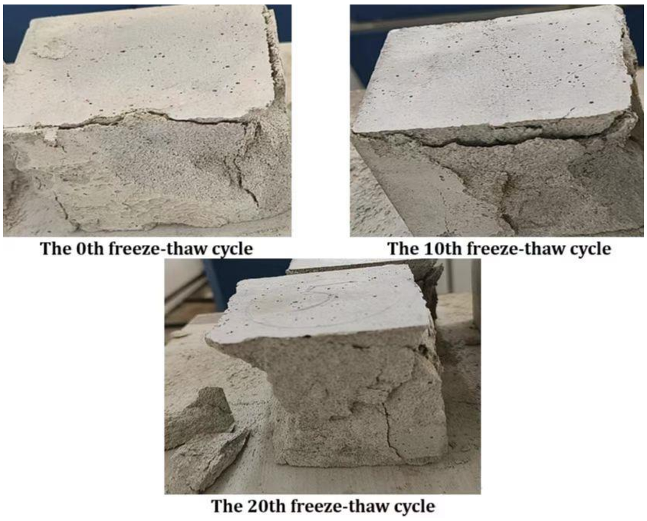

The 0th freeze-thaw cycle
The 10th freeze-thaw cycle
The 20th freeze-thaw cycle

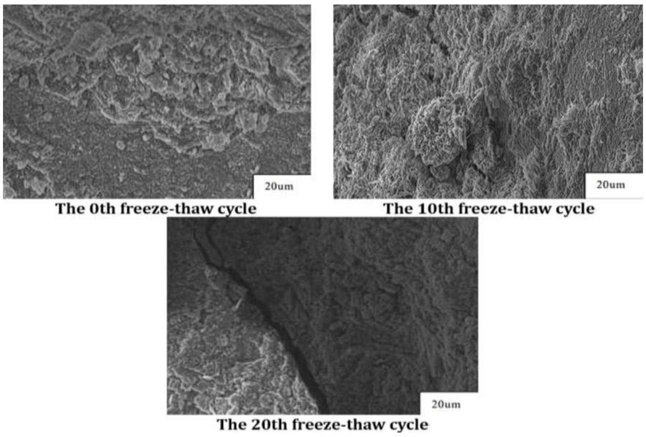
Fig -6: Failure mode of G4 specimen after freeze-thaw cycles

4. MICROSTRUCTURAL ANALYSIS

In order to investigate the microstructure changes of the specimen containing the different rice husk ash and silica fume content under different freeze-thaw cycles, the SEM test was carried out to analyze the internal microstructure of the specimen.

Select proper and small fragments from the broken specimen and soak them with ethanol. After soaking, put the small fragments into the drying oven at 60℃ and lasting for 24 hours. After drying, the fragments were conducted to the diffraction gold spraying and then placed in a Flex SEM 1000 electron microscope scanner for SEM testing.

As shown in Figure 7, during the initial stages of freeze-thaw cycles, the G4 group specimens exhibited minimal damage to the binder caused by frost heave forces [17], owing to their denser internal structure, larger contact area, and increased binder thickness.



The 0th freeze-thaw cycle
The 10th freeze-thaw cycle
The 20th freeze-thaw cycle

Fig -7: Microstructure images of G4 specimen after freeze-thaw cycles

With the increase of freeze-thaw cycle, the expansion pressure from freeze-thaw cycles gradually damages the intergranular cement. Under the periodic expansion pressure, the internal crack initiate, expand, and eventually cause fracture. The internal water is also in the process of "liquid - solid - liquid" transformation brings the two mainly damages on the specimen. On the one hand, when the water freezes into ice, the volume of the specimen increases and further expand the internal cracks ang pores. On the other hand, when the ice is transformed into water, it continuously penetrates into the specimen and erodes the cracks and pores. Otherwise, under freeze-thaw conditions, the cement is dissolved by water and migrates with water. As a result, the connection between the particles of the specimen with large water content is completely destroyed, and some particles become loose. Macroscopically, the internal pores of the specimen gradually expanded and the surface particles fell off.

5. CONCLUSION

In this study, rice husk ash, cement, silica fume, sand, and water are used as raw materials to prepare cement mortar specimens containing different amounts of rice husk ash. The rice husk ash is mixed into the cement mortar to replace part of the silica fume, which not only reduces the amount of silica fume required but also decreases the cost of the cement mortar. This helps to prepare energy-saving, environmentally friendly building materials and achieve the goals of energy conservation and environmental protection in green building construction. The properties of rice husk ash cement mortar were studied experimentally, with the dosage of rice husk ash used as a variable in the compressive strength tests. Additionally, compressive tests were conducted on cement mortar specimens subjected to different freeze-thaw cycles, followed by macro-structural and micro-structural comparative analysis.

As the proportion of rice husk ash replacing silica fume increases, the damage to the specimens initially decreases, then increases, causing the structure to become loose and resulting in a decrease in compressive strength.

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