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Effect of straw carbonization on properties of cement-based straw fiber composites

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Abstract. The raw materials of straw fibers were treated by different methods of nitrogen filling and carbonization at different temperatures. The cementitious materials consisted of ordinary Portland cement with strength grade 52.5 and sulphoaluminate cement with a ratio of 3:7. The surface morphology and material analysis of the fibers were carried out by means of SEM and EDS. The SiO₂ content in the surface layer of carbonized straw fibers was 86.69% higher than that of common straw fibers, and the interfacial bonding of the composites was more compact because of the hydration reaction. The initial setting time of composites with carbonized straw fibers is shorter than that of ordinary straw fibers, the flexural strength is slightly reduced by 6.3%, and the compressive strength is significantly increased by 21.93%.

1. Introduction

Straw fiber is a kind of green and renewable biomass material with wide sources, large aspect ratio and high specific strength. It can well restrain the early cracking of concrete. Straw fiber cells are composed of cellulose and lignin, etc. When straw fibers are mixed into alkaline concrete mixtures, they are eroded and hydrolyzed to glucose, mannose and other monosaccharides in alkaline environment. The monosaccharide molecules and calcium ions in concrete mixtures form calcium gluconate coated on the surface of cement particles^[1], which hinders the contact between cement and water. It hinders the hydration process and retards the development and improvement of strength. Biricik^[2] et al. determined that wheat straw was incinerated at low temperature, and the content of SiO₂ in ash was as high as 73%. Therefore, in this paper, the surface of straw fiber is moderately carbonized to reduce its surface organic matter content, improve the interface of straw fiber cement-based composites, and reduce the retarding effect of straw fiber on cement.

2. experiment

2.1 Experimental Raw Materials

Cement: "Polar Bear" brand sulphoaluminate cement, ordinary Portland cement, strength grade is 52.5; water reducer: water reducing effect of 25% polycarboxylic acid series superplasticizer; diatomite: Jilin Lin jiang area third-grade original soil, grinding for 20 minutes and then calcined at 600 °C for 2 hours. Straw fiber: ordinary annual reed straw in Jilin Province, which is dried, dusted and crushed into straw length of 5 mm to 25 mm for reserve; water: common laboratory tap water; sand: standard quartz sand.



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Table 1. Chemical composition of cement (%)

Main components	CaO	Al ₂ O ₃	SO ₃	SiO ₂	Fe ₂ O ₃	Other
SAC	42.36	36.34	8.47	5.69	1.37	5.77
P. O	52.03	5.79	5.21	25.71	3.89	7.37

2.2 Experimental Instruments

Electronic Analysis Balance, WEW-300B Microcomputer Screen Display Hydraulic Universal Testing Machine, SBY-60 Standard Constant Temperature and Humidity Maintenance Box, NJ-160A Concrete Mortar Mixer, Oven, JR-KZ300DS Flexural and Compressive Strength Integration Machine, KSXL-1216 High Temperature Vacuum Furnace with Nitrogen Filling Protection, Desktop TM3030 Electron Scanning Microscope, Electron Scanning Microscope-Energy Spectrum Analyzer

2.3 Specimen preparation and testing

The prepared straw fibers are evenly laid in crucibles and placed in nitrogen-filled high-temperature vacuum furnace. Five different temperatures are set up, namely 175°C, 200°C, 225°C, 250°C, 275°C, respectively. Sulphoaluminate cement and ordinary Portland cement are mixed in proportion of 3:7 to form a new cementitious material, which is mixed with straw fibers with different carbonization time, and the amount of straw fibers accounts for 2% of the total cementitious material. The water-cement ratio was 0.5 and the cement-sand ratio was 1:3, marked as C₁, C₂, C₃, C₄ and C₅ respectively. The blank group was labeled with C₀* without any fibers, and the C₀ labeled with ordinary straw fibers. After mixing evenly, the mortar is put into the test mould of 40mm×40mm×160mm. After 24 hours, the demoulding was put into the standard maintenance box for 28 days, and the measurement was taken out.

Cement strength test refers to GB/T 17671-1999 "Cement mortar strength test method (ISO method)" for testing. The morphology of the samples was observed by thermal field emission scanning electron microscopy (TFESEM), and the apparent composition of the samples was analyzed by energy dispersive spectrometer (EDS).

3. Results and discussion

3.1 Micromorphology of Carbonated Straw Fiber

When straw fibers are added into cement matrix composites, the dry shrinkage stress produced by hardening cementitious materials can be "absorbed" by fibers, which can reduce the internal stress produced by the materials themselves. Meanwhile, the random distribution of fibers can effectively "passivate" the stress concentration at the tip of cracks, thus effectively improving the flexural strength of the composites^[3]. However, in alkaline environment, biomass straw fibers will precipitate a large number of polysaccharides dispersed around the fibers, delaying C₂S hydration and hindering ettringite formation; more seriously, it will hinder the growth of Ca (OH)₂ crystals, resulting in the increase of setting time of cement paste and the formation of "retarding" phenomenon. The interfacial strength between fibers and cementitious materials is poor and the strength of concrete is reduced^[4]. The carbonization of straw surface is to degrade the polysaccharides on the surface of fibers by increasing the temperature appropriately in the vacuum nitrogen protection environment, so as to reduce the precipitation during mixing. In Fig. 1, a ~ e is the suitable time for carbonization, and the carbonization temperature is 175 ~ 275°C in turn. With the increase of temperature, the structure of straw fiber is gradually destroyed to varying degrees. When the temperature rises to 275 °C, the structure of straw fiber is destroyed most seriously by high temperature. The crack resistance and toughness of concrete are obviously reduced.

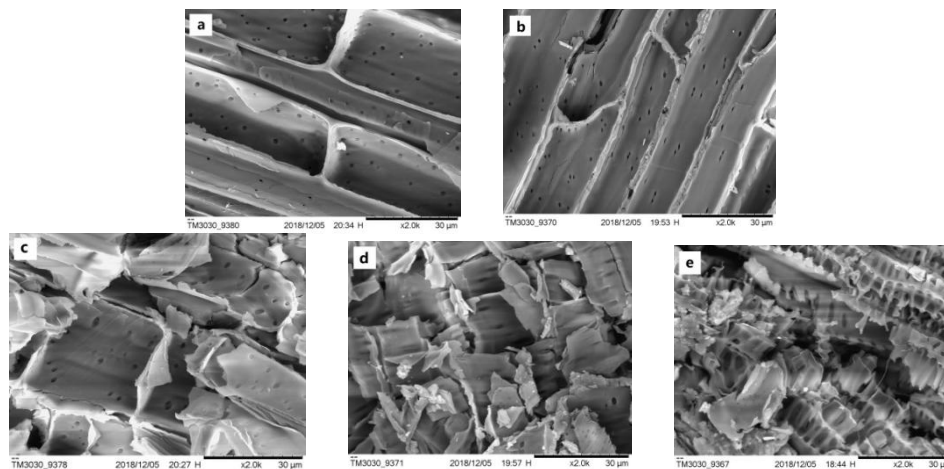


Fig1. (a) SEM image of straw fibers carbonized at 175°C;(b) SEM image of straw fibers carbonized at 200°C;(c) SEM image of straw fibers carbonized at 225°C;(d) SEM image of straw fibers carbonized at 250°C;(e) SEM image of straw fibers carbonized at 275°C;

3.2 Test results and analysis of composite materials

A new composite cementitious material was prepared by mixing sulphoaluminate cement and Portland cement. The effect of straw fibers at different carbonization temperatures on the strength of the composite material was studied by controlling the amount of straw fibers.

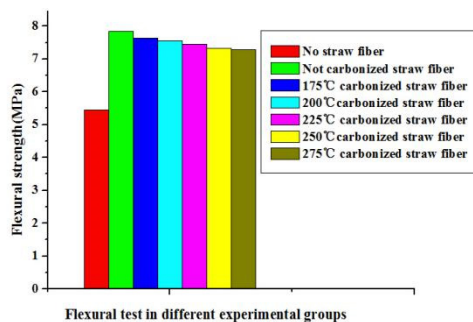


Fig2. Effect of different carbonization temperatures on flexural strength of straw fibers

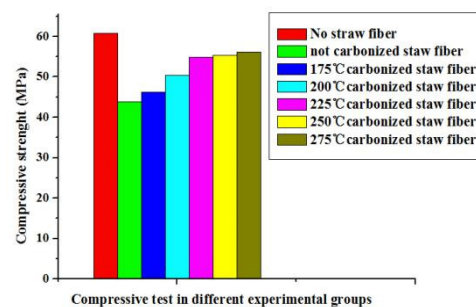


Fig3. Effects of different carbonization temperatures on compressive strength of straw fibers

From Fig. 2, it can be seen that the composites without straw fibers have the lowest flexural strength, the ordinary straw fibers have a significant increase of 30.4%, and the lowest flexural strength with carbonized fibers has a slight decrease, but the low flexural strength of the composites with most carbonized straw fibers is still 25.24% higher than that of the composites without fibers. With the increase of carbonization temperature of straw fibers, the degree of carbonization is deepening. Excessive carbonization temperature destroys the complete macromolecular structure of the fibers, thereby reducing the tensile strength of the straw fibers. Scanning electron microscopy (SEM) of carbonized straw fibers in Fig. 1 shows that the surface of straw is covered with a substance similar to "straw ash". With the increase of carbonization temperature, the ash layer of straw becomes thicker and thicker, and the volume of fiber which can provide tensile strength becomes smaller and smaller. When the composite material is damaged by external force, the ability to absorb transverse tensile stress decreases, so the flexural strength decreases. From Fig.3, it can be seen that the compressive strength of the composites without straw fiber is the highest, and the compactness of the composites decreases when the fibers are added into the mixtures, and the compressive strength of the composites decreases. With the increase of carbonization temperature, the compressive strength

increases obviously. The maximum compressive strength of carbon straw fiber is 21.93% higher than that of common straw fiber.

3.3 Interface analysis of composite materials

In order to further explore the reasons for the increase of compressive strength of carbonized straw fibers in cementitious materials, EDS was used to analyze the surface micro-area of common straw and carbonized straw respectively. Fig. 4 and Fig. 6 are the interface bonding diagrams of two different kinds of straw fibers with hydration products. The comparison of the two diagrams shows that the interface layer of the carbonized straw fibers is stronger, and the gap between the carbonized straw fibers and hydration products is smaller, and the combination of the two is more compact. Fig. 5 and Fig. 7 are EDS analysis maps of common straw fibers and carbonized straw fibers respectively. The comparison shows that the content of Si increases after carbonization of straw, and the main source of Si is SiO_2 . The mass percentage of Si element in the surface layer of straw fibers before carbonization was 15.73%, and the content of SiO_2 was 33.65% after analysis. After carbonization, the mass percentage of Si element in the surface layer of straw fiber is 29.36%, and the content of SiO_2 is 62.87%. Compared with the straw fibers before carbonization, the surface SiO_2 content increased by 86.69%. The main component of "straw ash" is a substance with high activity of free silicon, in addition to a small amount of sodium salt, potassium salt, and other inorganic salts. Highly active free silicon itself does not have or has less adhesive [5], but under alkaline conditions active SiO_2 is stimulated to react with free calcium hydroxide. Calcium silicate hydrate and other viscous gel substances are formed at the interface between straw fibers and cementitious materials, which makes the interface transition layer between the two more compact. At the same time, after carbonization, the inorganic content of the fiber surface increases, and the amount of polysaccharides released during mixing decreases. The initial setting time of cement can be obviously shortened by comparing the two. Therefore, the addition of carbonized straw fiber significantly reduces the initial setting time of sludge, and significantly improves the compressive strength of the composite.

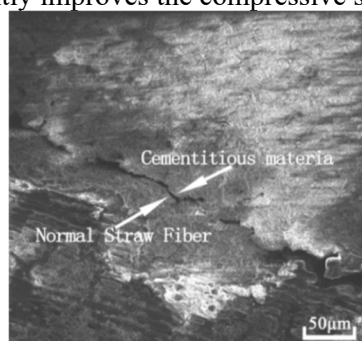


Figure4. Interface between common straw fibers and cement

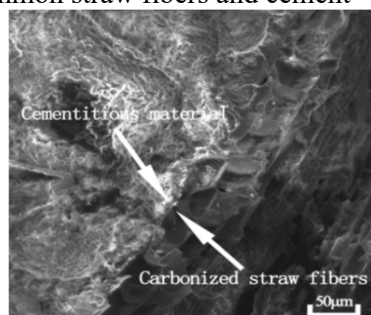


Figure6. Interface of Carbonated Straw Fiber in Contact with Cement

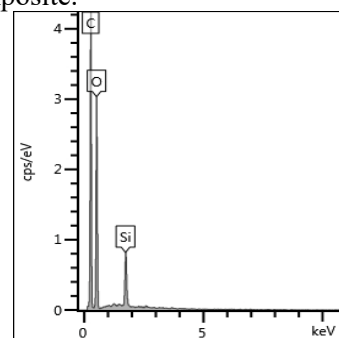


Figure5. EDS Atlas of common straw fiber surface

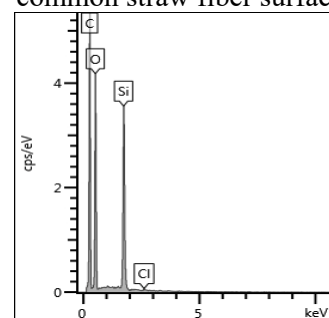


Figure7. EDS Atlas of carbonized straw fiber surface

4. Conclusion

After high temperature carbonization, straw fibers reduce the organic matter content in the surface of the fibers, effectively inhibit the precipitation of sugar, shorten the initial setting time of cement, and improve the retardation of straw fibers in cement mixtures. Under the microscope, the combination between carbonized straw fibers and hydration products was more compact.

The flexural strength of the composite increased by 30.4% and the compressive strength decreased by 27.84% with the addition of common straw fiber. Compared with the blank group, the flexural strength decreased slightly by 6.3% and the compressive strength increased by 21.93%.

The content of SiO₂ on the surface of untreated straw fibers is 33.65%, and that of carbonized straw fibers is 62.87%. Compared with that before carbonization, the content of SiO₂ on the surface of straw fibers is increased by 86.69%. The new SiO₂ participates in the hydration reaction of cement and improves the interfacial strength.

Acknowledgments

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