



Research paper

Effect of different amount of CNFs on the properties of concrete and cementitious materials

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Abstract: With the rapid development of the construction industry and higher requirements on the properties of materials, extensive studies have been made to improve the property of the concrete and cementitious materials. This paper mainly studies the mechanical property, anti-chlorine ion diffusion, anti-chlorine ion diffusion, anti-freezing performance, hydration process, microstructure and rheological property of the concrete and cementitious materials after adding cellulose nanofibers. Results showed that the compressive strength of C40 concrete with 0.15% cellulose nanofibers added was 75.72 MPa at 56 days of age, 23.11% higher than that of the control group. It was also higher than that of concrete with 0.20% cellulose nanofibers admixture added. When the content of cellulose nanofibers was 0.15%, the flexural strength reached the maximum value of 6.55 MPa, improving by 24% compared with the control group. Under the circumstances of 150 freeze-thaw cycles, the mass loss rate of C50 concrete with 0.15% CNFs admixture registered at 0.41%, reducing by 0.81% compared with the control group. However, when the cellulose nanofibers increased to 0.20%, the mass loss rate of the concrete reached 0.48%, indicating that adding an appropriate amount of cellulose nanofibers could improve the performance of the concrete. The study provides a strong scientific basis for modifying concrete and cementitious materials.

Keywords: CNFs, concrete, cementitious materials, mechanical properties, chloride resistance ion diffusion properties, hydration heat, rheological properties

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1. Introduction

Concrete is a building material that used frequently in construction. The performance of the concrete bears a directly impact on the quality and service life of the project. In recent years, Cellulose Nanofibers (CNFs), as a new type of concrete additives and admixture of cementitious materials, have attracted wide attention [1–3], as they can improve the mechanical property, crack resistance and durability of cementitious materials. In response to the need of concrete modification, Sun et al. proposed to add different amounts of CNFs to the concrete. Results showed that a moderate amount of CNFs admixture could enhance the flexural strength of the material without compromising its workability [4]. Kamasamudram et al. studied how different amount of CNFs added to the concrete affected the compressive, flexural and splitting properties of the material. They found that the compressive strength of the concrete with CNFs could improve by 15.7% and splitting strength, by 19.2% [5]. Based on the results of similar tests, this study added different amount of CNFs to the concrete in order to find out the effect of the admixture on the properties of the concrete and cementitious material. The paper has three parts. The first part introduced the materials and methods used for the test. In the second part, different CNFs was added to measure the properties of the modified concrete, including mechanical property, chloride diffusion resistance, frost resistance, heat of hydration and rheological property. The third part drew a conclusion on experimental results and put forward directions for future improvement.

2. Test methods and materials

2.1. Test materials

The concrete applied in the test is composed of cement, fly ash, stone, sand and water reducing agent. To be exact, there was P-O52.5 grade high strength silicate cement produced by Shanshui Group, class II fly ash, limestone with a particle size of 5–20 mm, ordinary river sand supplied by a mixing station, and polycarboxylic acid water reducing agent produced by Shandong Hongteng Biotechnology Company with a solid content of 50%. The sand was washed and dried with clean water as there was mud. The water reducing agent was blended according to the percentage of the mass of cement. The CNFs used was a white gel-like substance produced by Huaxiang Biological Co. Ltd, whose carboxylated substitution degree was 0.28 [6, 7].

2.2. Test methods

2.2.1. Pre-dispersion of cellulose nanofibers

To leverage the modification effect of CNFs, it should be pre-dispersed. Firstly, gel-like CNFs and water were poured into a wall-breaker according to the ratio of 1:2 to obtain the initial CNFs suspension. Then the suspension was poured into a 20-degree-celsius magnetic stirrer and stirred for 15 minutes. During the mixing process, the stirrer was covered with

a cling film to prevent CNFs from splashing out. The suspension was stewed for 10 minutes before being placed in an ultrasonic disperser to be stewed for 20 minutes twice. Finally, the configured suspension of CNFs was mixed with the water according to a moderate proportion and the admixture was stirred by a glass rod [8].

2.2.2. Mixing ratios with different amount of CNFs

To analyze how different amount of CNFs affects the properties of the concrete, several proportions were designed for the study. There were two grades of concrete: C40 and C50, which had different proportion of compositions. For C40, the fly ash was 135 kg, cement 312 kg, sand 805 kg, stones 958 kg, water reducer 0.6%, and water 157.5 kg. For C50, the fly ash was 140 kg, cement 320 kg, sand 716 kg, stone 1074 kg, water reducer 0.65%, and water 151.6 kg. C50 contained more fly ash, cement, sand, stone, and water reducing agent compared with C40. The CNFs were added as a proportion of the mass of cementitious materials, namely 0%, 0.05%, 0.1%, 0.15% and 0.20%. Table 1 shows the proportions.

Table 1. Concrete mix design

Grade	Group number	Fly ash kg	Cement kg	Sand kg	Stones kg	Water reducing agent %	Water kg	CNFs per cent
C40	C0	135	315	805	985	0.6	157.5	0
	C1	135	315	805	985	0.6	157.5	0.05
	C2	135	315	805	985	0.6	157.5	0.10
	C3	135	315	805	985	0.6	157.5	0.15
	C4	135	315	805	985	0.6	157.5	0.20
C50	C0	140	320	716	1074	0.65	151.6	0
	C1	140	320	716	1074	0.65	151.6	0.05
	C2	140	320	716	1074	0.65	151.6	0.10
	C3	140	320	716	1074	0.65	151.6	0.15
	C4	140	320	716	1074	0.65	151.6	0.20

2.2.3. Mechanical property

Non-standard specimens with $100 \times 100 \times 100$ mm were used for the test of the compressive and flexural strength. A SANTS300t pressure testing machine was used, with loading speed setting at 0.6 MPa/s and stress control setting as the control mode. Each group had three specimens. The measured value of three specimens was averaged to be the final result, which is accurate to 0.1 MPa. The test was conducted on the 7th, 28th, and 56th day of age. Additional discount strength testing was also performed on the 28th day [9, 10].

2.2.4. Resistance to diffusion of chloride ions

The resistance of the concrete to chloride diffusion was tested using Rapid Chloride Migration (RCM). Each group was set with three cylindrical specimens whose diameter $d = 100$ mm and height $h = 100$ mm. The specimens were placed in a curing room for 28 days. Then they were cut to a size of 50 ± 2 mm along the centre and applied to RCM test. The specimens were saturated with water for 24 hours using an intelligent vacuum saturator that contained $\text{Ca}(\text{OH})_2$ solution. At the same time, the surface of the specimen was sprayed with a colour-developing indicator by the RCM tester. After the surface became slightly dried, 0.1 mol/L AgNO_3 solution was sprayed. When there was white silver nitrate precipitate clearly visible on the surface of the specimen, the depth of intrusion was measured with a suitable straightedge at intervals of 10 mm from the centre to each side for seven times. The measurement was accurate to 0.1 mm [11–13].

2.2.5. Frost resistance

To assess how the frost resistance of the concrete was, a quick-freeze method was applied. Cyclic freeze-thaw tests were designed using a quick-freeze tester. Each set of tests was consisted of three $100 \times 100 \times 400$ mm specimens and the temperature at the centre was set between -16°C to -5°C . The test was conducted for 200 times. The mass loss rate (MLR) and relative dynamic elastic modulus (DEM) of the specimens were measured every 50 cycles. The average MLR and relative DEM were taken as the arithmetic mean of the three specimens. After the specimens reached the age of conservation, the moisture was wiped off from the surface, followed by a checked. The initial mass was weighted before the dynamic elastic modulus could be determined. The specimens were soaked into the fast-freezing test machine. When the freezing and thawing cycle reached a certain number, the water surface should be 25–50 mm above the top surface, the relative DEM was decreased to 60%, or the MLR reached 5% when the test was over [14].

2.2.6. Heat of hydration

Gelling materials was used to make the slurry. Firstly, the mixed CNFs suspension and gelling materials were added simultaneously to the mixing pot according to certain proportions and the pot ran at low speed for 30 seconds. Then the material was scraped off the side of the mixing pot and was stirrer at low speed for 30 seconds. This was followed by high-speed rotation for 60 seconds. The flow state of the admixture was observed during the rotation. Finally, the hydration heat test was applied under the temperature of $20 \pm 2^\circ$, and the test should be completed within 40 minutes [15].

2.2.7. Microstructure

Scanning Electron Microscope (SEM) was used to measure the concrete specimens. The specimens were first cut into pieces of suitable size, and then the pieces were cut into thin slices of 1–2 mm in thickness by using a low-speed cutter. The thin slices were then sprayed with gold. The 2000 \times and 5000 \times microscopes were used to observe how CNFs affected the microstructure of the concrete.

2.2.8. Rheological property

A rotational rheometer was used to test the rheological property of the cement paste. The rheological test was divided into three phases: pre-shear phase, stationary phase and formal test phase. A total of 140 groups of data were collected for the test, including 70 groups collected when the shear rate increased from 0 to 100 s^{-1} and 70 collected when the shear rate decreased from 100 s^{-1} to 0. The data were obtained from the downward curve. The 70 sets of data were analysed under the Bingham model. The strain in the Bingham model was mainly controlled by the excess of applied stress over the ultimate stress, which is calculated as in Eq. (2.1).

$$(2.1) \quad \tau = \tau_0 + \mu\gamma$$

In Eq. (2.1), τ refers to the shear stress, τ_0 refers to the yield stress, μ refers to the plastic viscosity, and γ refers to the shear rate.

3. The effect of adding different amount of CNFs on the property of the concrete and cementitious materials

3.1. Effect of adding different amount of CNFs on the compressive property of the concrete

The study first verified the effect of different amount of CNFs on the mechanical property of C40 and C50 concrete. The effect of CNFs admixture on the compressive strength of the concrete is showed in Fig. 1. The compressive strength increases first and then decrease with increasing content of CNFs. For C40 concrete, the effect at 7th day of age is not significant. It is mainly because the CNFs admixture has caused a delay in the early hydration of cementitious materials. The compressive strength of the concrete specimens with 0.05% CNFs added increases to 24.90% at 28th day of age. When the content of CNFs is 0.15%, the compressive strength of the concrete can reach as high as 65.15 MPa. But when the content increases to 0.20%, the compressive strength shows a decreasing trend. When the CNFs takes up 0.15% of the concrete specimen, its compressive strength at 56th day of age can reach 75.72 MPa, which is 23.11% higher than the control group and higher than the concrete with 0.20% CNF. The main reason is that CNFs are not uniformly dispersed and agglomerate, thus resulting in poor modification effect. Meanwhile, As CNFs within the specimen aggregate, there are cracks in the matrix, which in turn reduces the compressive strength of the concrete. Adding different amount of CNFs to C50 concrete has similar effect on the compressive strength. When the content of CNFs is 0.15%, the compressive strength of the specimens at the 7th day of age is 56.87 MPa. The compressive strength of the modified concrete can reach 71.56 MPa at 28th day of age, which is 33.60% higher than that of the control group. When the content of CNFs is 0.2%, the compressive strength of the concrete at 56th day of age is 16.19% lower than that with 0.15% CNFs, and the

decrease margin of C50 concrete is more significant than that of C40. This is mainly because C50 concrete includes relatively fewer cementitious materials, and adding CNFs will reduce the fluidity of the cementitious materials, thus reducing the compressive strength.

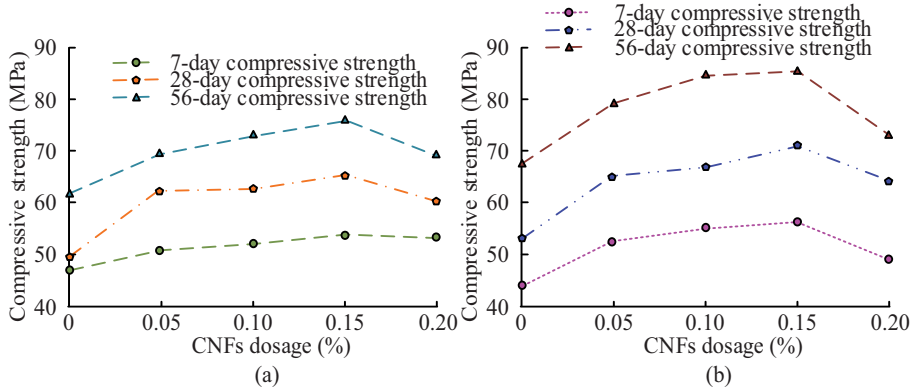


Fig. 1. Effect of CNFs on the compressive strength of C40 and C50 modified concrete: a) Change curve of compressive strength of C40 concrete, b) Change curve of compressive strength of C50 concrete

3.2. Effect of adding different amount of CNFs on the folding strength of the concrete

The effect of CNFs admixture on the flexural strength of the modified concrete is showed in Fig. 2. In Fig. 2, adding a moderate amount of CNFs to the concrete can significantly enhance the flexural strength. For C40 concrete, the flexural strength increases to 6.14 MPa when the CNFs admixture is 0.05%, or 16% higher compared with the concrete without CNFs admixture. The flexural strength continues to increase along with the increase of CNFs admixture and starts to decrease after reaching the maximum value. When the CNFs admixture is 0.15%, the flexural strength reaches the maximum of 6.55 MPa, improving by about 24% compared with the control group. For C50 concrete, the flexural strength

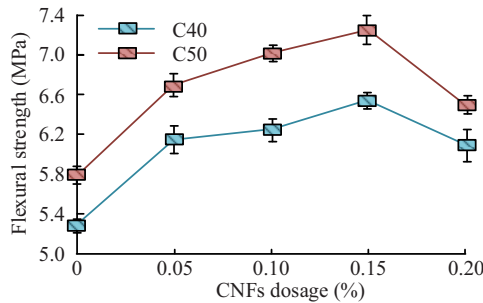


Fig. 2. Effect of CNFs admixture on the flexural strength of modified concrete

reaches the maximum value at 7.28 MPa when the CNFs admixture is 0.15%, improving by about 25% compared with the control group. This indicates that the CNFs admixture can improve the flexural strength. However, if too many CNFs are added, agglomeration may occur, which will undermine the modification effect.

3.3. Effect of adding different amount of CNFs on the anti-chlorine ion diffusion property of the concrete

RCM test was used to determine the diffusion depth of chloride ions and the unsteady state chloride diffusion coefficient of the concrete. By different amount of CNFs to the concrete, the resistance of the concrete to chloride diffusion was verified. The trend of chloride ion diffusion depth of the modified concrete with different amount of CNFs is shown in Fig. 3. The chloride ion diffusion depth of the concrete decreases and then increases with the increase of the CNFs admixture, and the diffusion shows an upward trend. For C40 concrete, when the CNFs is 0.15%, the diffusion depth of chloride ions is 12.54 cm, 4.25 cm lower than that of the control group. For C50 concrete, the trend of the diffusion depth is similar to that of C40. The diffusion depth of chloride ions is 10.05 cm, 4.11 cm lower than the control group. However, when the content of CNFs increase to 0.20%, the diffusion depth of chloride ions actually increases to 12.01 cm. This is mainly because of the agglomeration of CNFs when their content is high, so that the contact area between CNFs and the concrete slurry has been narrowed. At the same time, there are pores between the agglomerated CNFs and the concrete slurry, which enlarges the pores and cracks in the internal structure and widens the chloride ion diffusion depth in the concrete matrix. It indicates that adding CNFs has a positive effect on the anti-chlorine ion diffusion of the concrete, but the content of CNFs should not be too high, otherwise it will undermine the anti-chlorine ion diffusion. In addition, the chloride ion diffusion of C50 is shallower than that of C40. This suggests that C50 has better chloride ion erosion resistance after being added with CNFs.

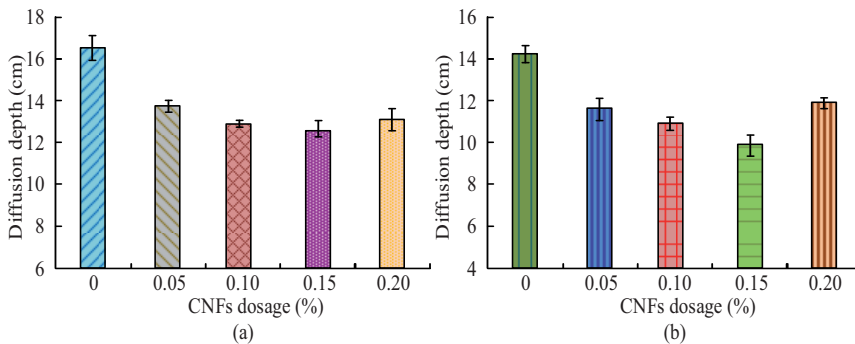


Fig. 3. The changing trend of the depth of chloride ion diffusion: a) Depth of chloride ion diffusion in C40 concrete, b) Depth of chloride ion diffusion in C50 concrete

Figure 4 shows the trend of unsteady state chloride diffusion coefficient of the concrete added with different amount of CNFs. As CNFs takes a higher proportion, the unsteady chloride diffusion coefficient of the modified concrete presents a downward trend followed by an upward one. When the content of CNFs is 0.15%, the unsteady chloride diffusion coefficient is the lowest. For C40 concrete, when CNFs takes up 0.15%, the unsteady chloride diffusion coefficient is reduced by 36.59% compared with that of the control group to $5.52 \times 10^{-12} \text{ m}^2/\text{s}$. For C50 concrete, the unsteady chloride diffusion coefficient is only $4.51 \times 10^{-12} \text{ m}^2/\text{s}$, reducing by 43.01% compared with that of the control group. However, when the content of CNFs increases to 0.20%, the unsteady state chloride diffusion coefficient will increase along with it, because high proportion of CNFs would result in agglomeration, thus reducing the modification effect. This indicates that a moderate amount of CNFs can improve the anti-chlorine ion diffusion of the concrete. In addition, CNFs have short-circuit diffusion effect, which can promote hydration and further improve the anti-chlorine ion diffusion property of the concrete.

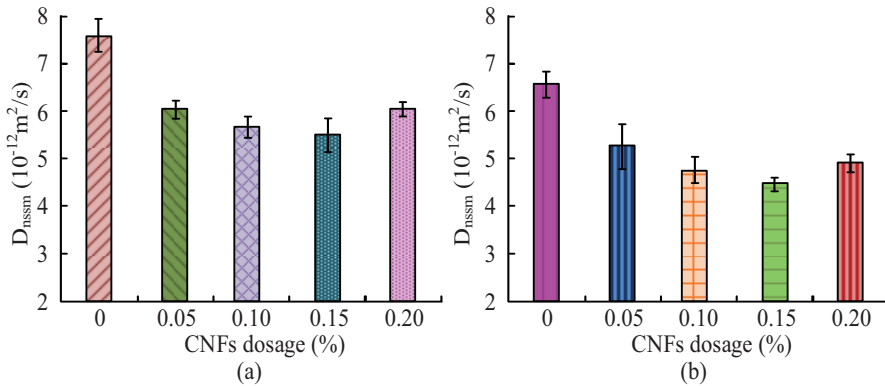


Fig. 4. Change trend of non-steady state chloride ion diffusion coefficient: a) C40 non steady state chloride ion diffusion coefficient, b) C50 non steady state chloride ion diffusion coefficient

3.4. Effect of adding different amount of CNFs on the frost resistance of the concrete

A study was designed to analyse the effect on the frost resistance of the concrete after adding different amount of CNFs. The performance is mainly verified by the mass loss rate, relative DEM and the appearance state of the concrete specimens. The mass loss rates of C40 and C50 concrete under different freeze-thaw cycles are shown in Fig. 5.

From Fig. 5, it can be seen that as there are more freeze-thaw cycles, the MLR of the concrete increases as well. Meanwhile, under the same freeze-thaw cycle, the MLR of the concrete shows a downward trend first and then increases as CNFs takes up a higher proportion. This is because adding CNFs can change the nature of the concrete surface and reduce the flow of water in the pores. It can also fill the pores inside the concrete, which

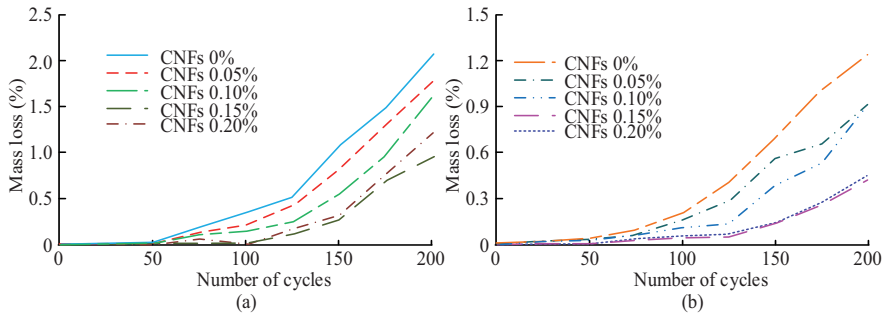


Fig. 5. Mass loss rate under different freeze-thaw cycles: a) C40 quality loss rate, b) C50 quality loss rate

in turn improves the frost resistance. The lowest MLR is observed for both types of the concrete when the CNFs admixture is 0.15%. For C40 concrete, the MLR does not change significantly until the freeze-thaw cycle is 100. When the freeze-thaw cycle is 150, the MLR increases rapidly. When the number of freeze-thaw cycle is 200, the mass loss rate of the concrete that contains 0.15% CNFs is 0.95%, 1.13% lower than that of the control group. For C50 concrete, the change of MLR is similar to that of C40. At 200 freeze-thaw cycle, the MLR of the concrete that contains 0.15% CNFs is 0.41%, which is reduced by 0.81% compared with the control group. However, when the CNFs admixture increases to 0.20%, the MLR rebounds to 0.48%. It is mainly due to the incomplete hydration of the cement, caused by the reaction between CNFs functional groups and the water. In addition, too many CNFs added to the concrete may lead to agglomeration, which reduces the bonding rate between the cement paste and the aggregate, thus reducing the frost resistance of the concrete.

This paper also studies the relative dynamic elastic modulus at different amount of CNFs admixture to accurately measure the internal damage and assess the soil frost resistance. The changing curves of relative DEM of C40 and C50 concrete with different amount of CNFs admixture are displayed in Fig. 6. From Fig. 6, it is known that the relative DEM of the concrete decreases with the increase of the cycle. At the same time, the relative DEM

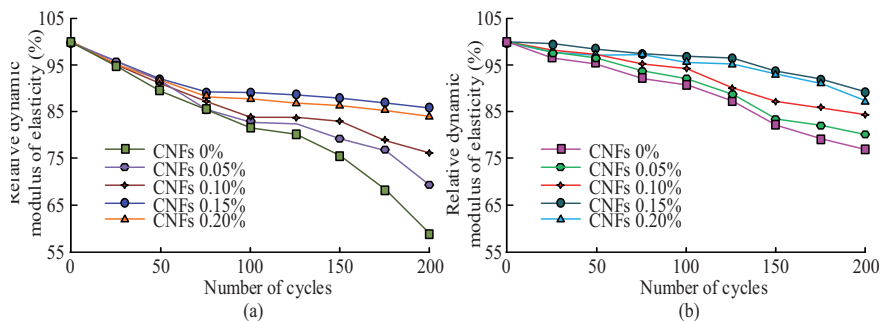


Fig. 6. Change curve of relative dynamic elastic modulus under different amount of CNFs: a) Relative dynamic elastic modulus of C40 concrete, b) Relative dynamic elastic modulus of C50 concrete

can increase by adding an appropriate amount of CNFs. Under the same cycle, the relative DEM shows an increasing and then decreasing trend as an increasing amount of CNFs is added to the concrete. For C40 concrete, the relative DEM of the concrete that contains 0.15% CNFs is significantly better than the concrete with CNFs at other levels of content. When the cycle is 100, the relative DEM is as high as 89.42%, improving by 9.67% compared with the control group. However, when the content of CNFs increases to 0.20%, the frost resistance is lower. For C50 concrete that contains 0.15% CNF, the relative DEM is better than the concrete with other CNFs proportions. The relative kinetic elastic modulus at 100 cycle is as high as 97.53%, improving by 2.13% compared with that of the concrete with 0.20% CNFs admixture. This is because too many CNFs would result in agglomeration. The CNFs cannot be sufficiently bonded with aggregates, so that the frost resistance would be undermined. Therefore, it is necessary to control the appropriate amount of CNFs in the concrete to obtain the best performance.

3.5. Effect of adding different amount of CNFs on the hydration heat of the concrete

The effect of adding different amount of CNFs on the heat of hydration of cementitious materials is also studied. The trend of the exothermic rate of hydration of cementitious materials within 120 hours at different CNFs admixtures is displayed in Fig. 7.

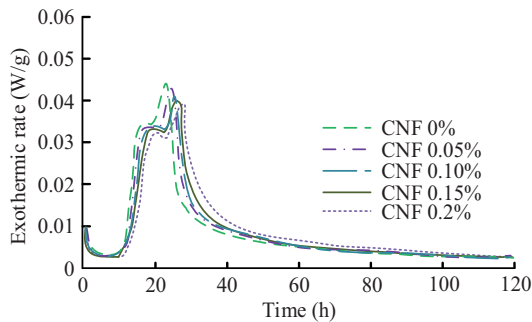


Fig. 7. Changing trend of hydration heat release rate within 120 hours

From Fig. 7, the exothermic rate of hydration of cementitious materials decreases gradually with the increase of CNFs content. At the same time, it can be seen that the higher the content of CNFs is, the slower the maximum exothermic rate appears. In the control group, the maximum exothermic rate of hydration occurs at about 21 hours, with the maximum exothermic rate being 0.045 W/g. When the content of CNFs is 0.15%, the maximum exothermic rate of hydration is delayed for about 5 hours to 26 hours, with the maximum exothermic rate at 0.041 W/g. When the content of CNFs reaches 0.20%, the maximum exothermic rate of hydration comes the latest, at 28 hours, with the maximum exothermic rate of hydration being 0.040 W/g. This indicates that the exothermic rate of hydration of cementitious materials can be reduced and delayed if an appropriate amount of CNFs is added to the concrete.

Figure 8 shows the curves of cumulative hydration heat release of gelling materials with different content of CNFs for 120 hours. In the first 30 hours, the cumulative exothermic heat of hydration of the cementitious material decreases as the content of CNFs increases. This is because CNFs will hinder the reaction between particles and the water by adsorbing on the surface of the material. At the same time, the special functional groups within the CNFs have a complexation reaction with the gelling material, which can delay the hydration reaction. However, after 30 hours, as the increase of CNFs content, the cumulative hydration exotherm shows an increasing trend. When the content of CNFs reaches 0.20%, there is a significant increase in the cumulative heat release compared with the specimens without CNFs. This indicates that adding CNFs can promote the hydration reaction of cement.

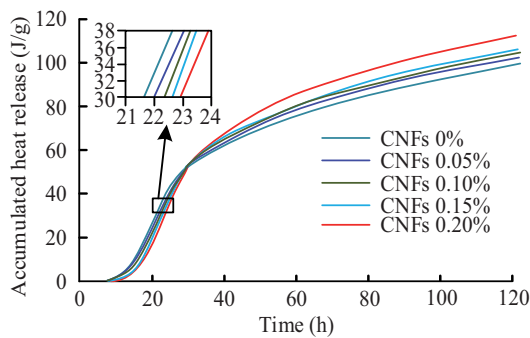


Fig. 8. 120 hours of cumulative heat release

3.6. Effect of adding different amount of CNFs on the property of the concrete from a microscopic point of view

This paper studies the effect of adding different amount of CNFs on the property of the concrete from a microscopic point of view. The SEM scanning electron microscope are used to scan the specimens. The scanned images under 2000 \times magnification are shown in Fig. 9. From Fig. 9, it can be seen that the holes and cracks in the concrete matrix decrease with the increase of the proportion of CNFs, and their densities gradually increase. It indicates that adding CNFs can effectively fill the holes in the concrete to enhance the compactness. The improvement of microstructure may contribute to the mechanical property and crack resistance.

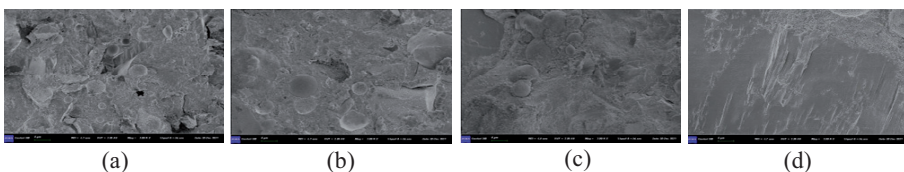


Fig. 9. Scanned images under 2000 \times SEM electron microscope: a) CNFs 0%, b) CNFs 0.05%, CNFs 0.15%, CNFs 0.20%

3.7. Effect of different amount of CNFs on the rheological property of the concrete

This paper studies the effect of adding amount of CNFs on the rheological property of cementitious materials, and the changing curve of shear stress and shear rate of the cement paste, as is shown in Fig. 10. The shear stress of the cement paste increases along with the shear rate. It also increases with the increase of proportion of CNFs, and the rheological property of the cement paste decreases. When the shear rate is 60 s^{-1} , the shear stress of the cement paste with 0.2% CNFs is as high as 200 Pa, exceeding the control group by 110 Pa. The main reason is that the functional groups within the CNFs have enhanced the ability of combining with water. CNFs can adsorb more water, which results in the reduction of free water in the cement paste. Since water is a key factor for the flow of the cement paste, reduced free water leads to a decrease in the rheological property of the cement paste, which is manifested as an increase in the shear stress. This phenomenon suggests that adding CNFs to the concrete can adjust the rheological property of the cement paste, thus affecting its workability and crack resistance.

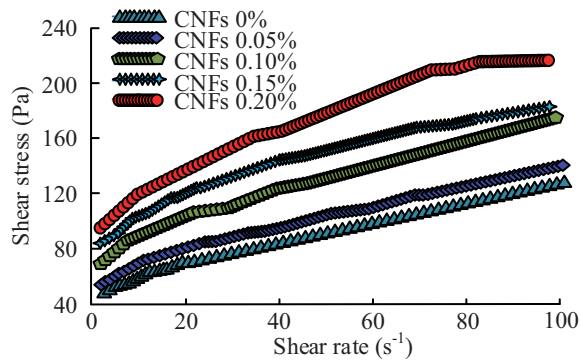


Fig. 10. Changing curve of the shear stress and shear rate

4. Conclusions

CNFs, as a new type of nanomaterials with excellent physical and chemical properties, are expected to have wide application in concrete modification. The study added different amount of CNFs to the concrete and analysed the effect on the properties of the concrete and cementitious materials. For C50 concrete, when the content of CNFs is 0.15%, the diffusion depth of chloride ions in the concrete is reduced by 37.51% compared with the control group. When the content of CNFs increases to 0.20%, the chloride ion diffusion depth of the concrete increases as well. For C40 concrete, the unsteady state chloride diffusion coefficient is reduced by 36.59% to $5.52 \times 10^{-12} \text{ m}^2/\text{s}$ compared with the control group when the CNFs is 0.15%. For C50, under the same content of CNFs, the unsteady state chloride diffusion coefficient is reduced by 43.01% to only $4.51 \times 10^{-12} \text{ m}^2/\text{s}$ compared with the control group. However, when the content of CNFs increases to 0.20%, the unsteady state chloride diffusion

coefficient also increases. At 0.15% CNFs, the appearance of the maximum exothermic rate of hydration is delayed by about 5 hours to 26 hours compared with the control group, with the maximum exothermic rate being 0.041 W/g. At 0.20% CNFs, the maximum exothermic rate of hydration comes the latest, at about 28 hours, with the maximum exothermic rate of hydration being 0.040 W/g. The maximum exothermic rate of hydration at 60 s is about 0.040 W/g. When the shear rate is 60 s^{-1} , the shear stress of the cement paste with 0.2% CNFs is as high as 200 Pa, 110 Pa higher than that of the control group. It indicates that adding different amount of CNFs can enhance the properties of the concrete and cementitious materials. However, the existing CNFs dispersion method relies on the environment and equipment, so the environment for the test needs to be further improved.

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Received: 2023-11-01, Revised: 2023-12-28