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Effect Mechanisms of Toner and Nano-SiO_2 on Early Strength of Cement Grouting Materials for Repair of Reinforced Concrete

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Abstract: The reinforced concrete invariably involves some diseases (e.g., crack, void, etc.) due to the complex service conditions. These diseases are usually repaired to extend the service life of reinforced concrete by using cement grouting materials. In order to meet a certain color need of reinforced concrete, toner is mixed into the cement grouting materials. However, the toner has a negative effect on the early strength of cement grouting materials. Unfortunately, the mechanism of the negative effects of toner is still unclear, and no effective and targeted measures have been put forward. Hence, the main work of this paper reveals the mechanisms of the toner and nano-SiO_2 (N-S) in the hydration process and the strength generation of the cement grouting materials in the case of different curing ages and nano-SiO_2 contents via the scanning electron microscopy test (SEM), X-ray diffraction test (XRD), differential scanning calorimetry test (DSC), and Fourier transform infrared spectroscopy test (FTRI). Results show that: (a) the toner hinders the generation of AFt and CH crystals (especially for 1-day and 3-day), which delays the hydration process and weakens the early performance of cement grouting materials; (b) the N-S promotes the hydration process and the formation of C-S-H gels, so as to effectively increasing the early strength and reducing (but not eliminate) the adverse effect of toner on cement grouting materials; (c) With the increase of every 1% N-S, the flexural strength of 1-day, 3-day, and 7-day average increased by 11.3%, 2.9%, and 0.9%, respectively, and the compressive strength of 1-day, 3-day, and 7-day average increased by 0.8%, 0.3%, and 0.1%.

Keywords: cement grouting materials; effect mechanisms; early strength; reinforced concrete; nano-SiO_2; toner

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1. Introduction

Reinforced concrete is one of the most important materials in civil structures. Owing to complex service conditions, it invariably involves some diseases (e.g., crack, void, etc.) in the reinforced concrete [1–3]. In this case, cement grouting materials are used to repair the diseases to extend the service life of reinforced concrete [4,5]. In recent years, different cement grouting materials have been developed to address various engineering properties (e.g., high fluidization, early strength, and low shrinkage) in previous studies and obtain satisfactory engineering applications. However, owing to some specific engineering conditions, the reinforced concrete [6–8] must meet a certain color need. Hence, the toner is tried to mix with cement grouting materials to modify the structure's appearance. Unfortunately, the toner inevitably influences the physical and mechanical properties of the cement grouting materials.

Khedaywi et al. [9] analyzed the effect of waste toner on the performance of cement-based materials and found that ductility and penetration decrease with the waste toner content increase. Parvan et al. [10] revealed the effect of carbon black on cement-based materials, which slowed down the hydration and hardening of cement-based materials. Zhang et al. [11] studied the feasibility of the application of waste carbon in cement-based materials. They found that 10% of waste carbon instead of cement-based materials has the least effect on the performance of cement mortar, but it still increases the initial setting and final setting time. When the carbon content increases, the mechanical properties of cement materials will be significantly weakened. Obviously, the previous studies indicated that the toner could significantly reduce the fluidization and strength of the cement grouting materials, which is against the engineering application of the cement grouting materials [12–14]. Unfortunately, the previous studies neither provide modified techniques nor reveal the mechanisms of the toner on the cement grouting materials. The purpose of this study is to address the above problems.

In recent years, nano-SiO_2 (N-S) has been widely adopted to modify cement-based materials owing to it can establish a new network to chemically bond with hydration products to generate C-S-H gel [15]. Ltifi et al. [16] revealed the effect of three different contents (3%, 5%, and 10%) of the N-S on the early performance and hydration mechanism of cement grouting materials with the constant water-cement ratio. Emamian et al. [17] established the formula to predict the mechanical resistance of cement materials with N-S by using the F-T cycle based on experiments. Liu et al. [18] revealed the effect of the N-S on the macroscopic properties of cement mortar by means of hydration heat analysis and XRD. The above research showed that it effectively shortened the setting time of cement and promoted the hydration process. Hosseini et al. [19] studied the effect of two kinds of N-S hydrosols with different surface areas on the early performance of cement mortars with low-activity blast furnace slag (BFS). They found that BFS and N-S can jointly improve the mechanical properties of mortar. Garcia et al. [20] analyzed the coordinated effect of N-S and metakaolin on improving the service life of cement-based mortar in aggressive environments at an early age. Gerlinde et al. [21] assessed the effect of SAPs and nano-silica on the hydration progress and the hardened properties. Liu et al. [22] investigated the influence of N-S on recycled concrete and found that N-S accelerated the setting speed of cement mortar and refined the pore size of recycled concrete. Chithra et al. [23] studied the influence of N-S on high-performance concrete with 40% fine aggregate of copper slag and revealed that colloidal nano-silica is a filling material for improving microstructure and promoting the strength characteristics of materials. Wang et al. [24] explored the influence of N-silica on the volume of cement paste and found that its addition accelerated the volume change of cement paste and concrete. According to the above research, it is a good choice to improve the cement grouting material modified with toner and N-S (in the following text, 'cement grouting material modified with toner and N-S' is abbreviated to 'CRTS').

Hence, the effects of the toner and N-S on macroscopic properties (compressive and flexural strength, fluidity, and dry shrinkage rate) of the CRTS were studied. The microstructure and hydration products were observed to reveal the effect mechanisms of the toner and N-S on the early strength of the CRTS via SEM tests, XRD tests, DSC tests, and FTIR tests. This study both reveals the mechanisms of the toner on the cement grouting materials and provides modified techniques to reduce the negative impact of toner, which improves the engineering application of cement grouting materials.

2. Materials and Methods

2.1. Materials

2.1.1. Raw Materials

The cement grouting materials prepared and applied in this paper include four kinds of raw materials, such as cement of the Shanlv P. O. 42.5R, water-reducing agent (WA), accelerating agent (AA), and expansion agent of the UEA (EA). The basic technical properties of the four types of raw materials are presented in our studies [25].

2.1.2. Additive

The toner is adopted to change the appearance of cement grouting materials, which basic performance is shown in Table 1. The toner used in this paper is an additive used for coloring cement grouting materials (pigment), and its composition is mainly carbon black. However, previous studies have proved that the addition of toner has a negative impact on the cement grouting materials. Hence, the nano-SiO_2 (N-S) is added to satisfy the high performance of the cement grouting materials with toner. The basic properties of the N-S are shown in Table 2. The macro morphology of the above materials is shown in Figure 1.

Table 1. Basic technical properties of toner.

Particle Size (nm)	Specific Surface Area (m^2/g)	Tinting Strength (%)	PH Value	Appearance
12	425	125	1.7	Black powder

Table 2. Basic technical properties of N-S.

Particle Size (nm)	Specific Surface Area (m^2/g)	Bulk Density (g/cm^3)	Purity (%)	Appearance
15	600	0.21	99.8	White grainy

(**a**) (**b**)

Figure 1. Additive. (**a**) Toner; (**b**) nano-SiO_2.

2.2. Methods

According to the Chinese standards [26], the engineering performance of CRTS is determined.

The SEM, XRD, DSC, and FTIR samples of the cement grouting materials can be prepared using the methods presented in Figure 2. Samples (4 cm × 4 cm × 16 cm) were prepared for cement grouting materials after curing age (1-day, 3-day, and 7-day). Then, the samples were further prepared into sheets (2 cm × 2 cm × 1 cm) according to the requirement. The sheet samples were soaked in absolute ethanol for 7 days and then dried for one day at 40 °C. In order to better carry out a microscopic test, the sample was finally processed into cubic blocks (1 cm^3). SEM samples: the cubic blocks were dried for two days at 40 °C. The other three microscopic tests also have the following process. The above cubes were ground into powder and then dried for two days at 40 °C. The XRD, DSC, and FTIR samples were prepared by passing through 80, 150, and 80 square sieves, respectively. The corresponding equipment can be found in Figure 3.

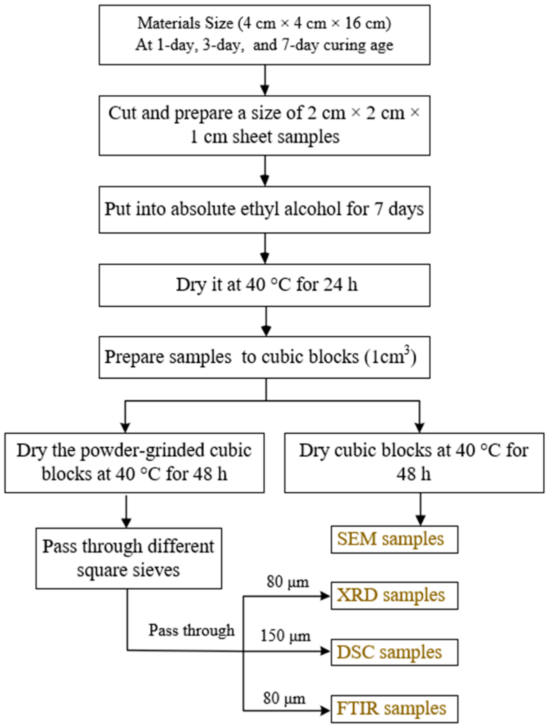
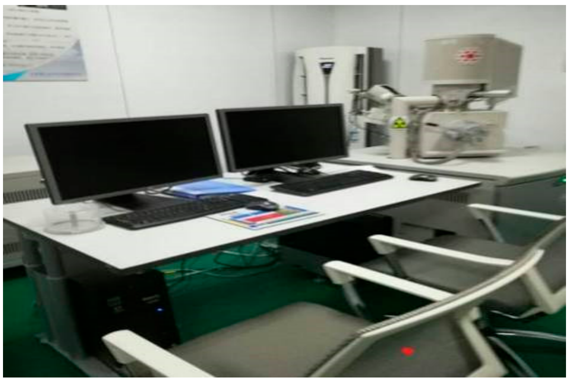
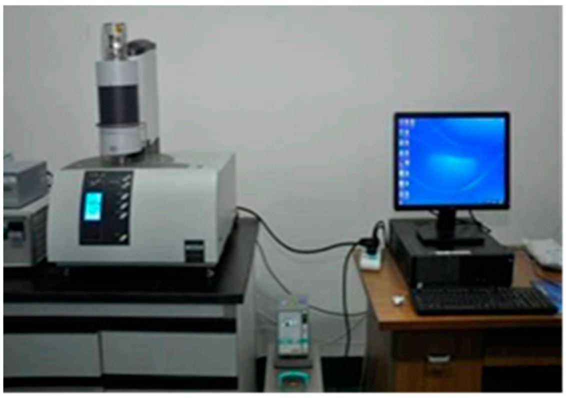


Figure 2. Sample preparation process.

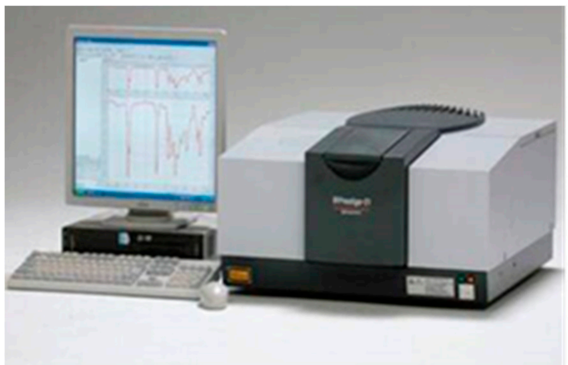


(**a**) SEM.

(**b**) XRD.



(**c**) DSC.



(**d**) FTIR.

Figure 3. Corresponding equipment.

The basic information of the corresponding equipment can be found as follows:

- **SEM** (Version: FEI Quanta 250, Anton Paar GmbH, Graz, Austria)

- Observe the microstructure of hydration products.
- **XRD** (Version: AXS, Bruker Corporation, Billerica, MA, USA)
- Investigate the types of hydration products Condition: 10°/min scanning speed, 10–65° scanning angle.
- **DSC** (Version: SDT 650, TA Instruments, New Castle, DE, USA)
- Analyze the content of hydration products by mass and heat change. Condition: 0–700 °C, 15 °C/min heating rate, nitrogen atmosphere.
- **FTIR** (Version: Nicolet 5700, Thermo Fisher Scientific—CN, Shanghai, China)
- Investigate functional group characteristics. Condition: 400–4000 cm^{-1} spectral.

3. Engineering Properties of the CRTS

According to our studies [27], the formula of the CRTS can be determined as follows: water–cement ratio = 0.56, WA = 1.2%, AA = 2.5%, EA = 5%, and toner = 4%.

As shown in Table 3, the compressive and flexural resistance of the cement grouting materials with toner is lower than without toner, while the fluidity is slightly higher. It proves that the addition of toner weakens the engineering properties of cement grouting materials. The proportion of flexural strength of CRTS to that of conventional cement grouting is 67% (1-day), 88% (3-day), and 90% (7-day), respectively. The proportion of compressive strength of CRTS to that of conventional cement grouting is 80% (1-day), 87% (3-day), and 88% (7-day), respectively. This shows that the adverse effect of toner on the strength formation of cement grouting materials is mainly reflected in the early stage (1-day). It speculates that the toner may limit the formation of C-S-H gels. The effect of toner will be further researched by microstructure studies. However, the early mechanical properties are very important for the application of cement grouting materials. Hence, the N-S was tried to optimize the performance of cement grouting materials with toner.

Table 3. The performance of cement grouting materials with toner.

Toner	Fluidity (s)	Flexural Strength (MPa)			Compressive Strength (MPa)			Shrinkage Rate (%)	
		1-Day	3-Day	7-Day	1-Day	3-Day	7-Day	7-Day	28-Day
Yes	11.41	2.31	7.16	9.61	8.76	22.63	30.28	0.018	0.113
No	10.38	3.43	8.18	10.66	10.91	26.02	34.38	0.019	0.108

The engineering properties of the CRTS with different N-S (nano-SiO_2) contents are presented in Table 4. With the increase of 1% N-S, the flexural strength of 1-day, 3-day, and 7-day average increased by 11.3%, 2.9%, and 0.9%, and the compressive strength of 1-day, 3-day, and 7-day average increased by 0.8%, 0.3%, and 0.1%, respectively. Undoubtedly, it indicates that the N-S can effectively decrease the disadvantageous effect of toner and improve the early intensity of the CRTS.

Table 4. The performance of cement grouting materials with different nano-SiO_2 contents.

Nona-SiO_2 Content (%)	Fluidity(s)	Flexural Strength (MPa)			Compressive Strength (MPa)			Shrinkage Rate (%)	
		1-Day	3-Day	7-Day	1-Day	3-Day	7-Day	7-Day	28-Day
0	11.41	2.31	7.16	9.61	8.76	22.63	30.28	0.018	0.113
1	11.89	2.81	7.47	9.76	9.91	23.62	31.85	0.024	0.105
2	12.34	3.05	7.69	9.92	10.79	24.69	31.56	0.017	0.112
3	12.92	3.16	7.81	9.86	11.21	25.26	31.22	0.022	0.110
Standard	9–13	-	-	≥2	-	-	10–30	-	<0.5

4. Microstructure Analysis

The hydration product has a decisive influence on the strength of materials. Hence, this section discussed the influences of toner, curing age (1-day, 3-day, and 7-day), and N-S contents (0%, 1%, 2%, and 3%) on the hydration products and microstructure of different cement grouting materials.

4.1. Microstructures of the Cement Grouting Materials with Pure Toner

The microstructures of the cement grouting materials, with or without toner at different curing ages, are presented in Figure 4.

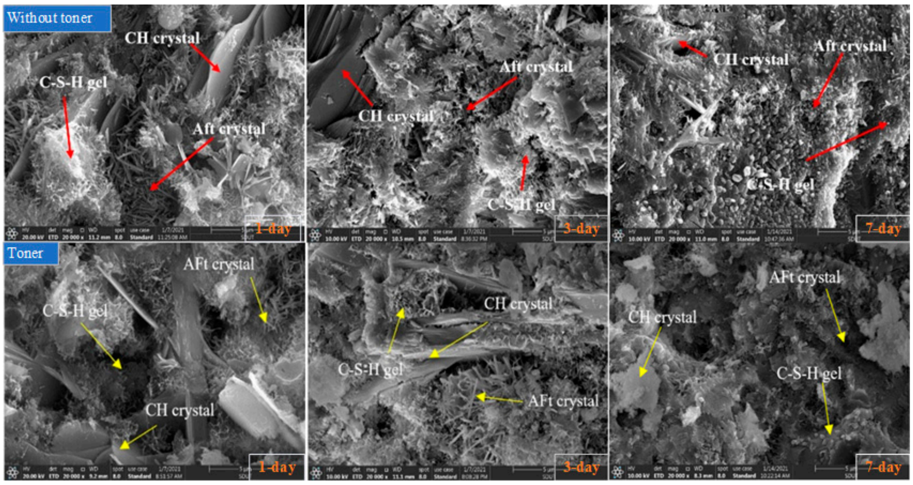


Figure 4. The microstructures of different cement grouting materials.

At the standard curing conditions, C-S-H gels and AFt in cement grouting materials without toner have basically formed. The C-S-H gels cross-clings to other hydration products to form a compact three-dimensional spatial structure, which provides early strength for the cement grouting materials. Compared without toner, the microstructure of the cement grouting materials with toner is loose and not dense. Meanwhile, its voids and cracks are more obvious, and the hydration product distribution is more dispersed due to the lack of contact.

With the increase in curing age, C-S-H gels in cement grouting materials without toner continue to increase, its wrapping effect on CH crystals is more obvious, and AFt is almost invisible. The compactness of the microstructure is strengthened, which indicates that hydration has been further enhanced. The hydration process of cement grouting materials without toner is faster and more sufficient by comparing the microstructure of cement grouting materials with and without toner. Moreover, the changes and development of cement grouting materials with toner hydration products are relatively slow. Therefore, it can be speculated that the mechanism of the influence of toner on the strength formation of the CRTS is to delay the transformation of CH crystals to C-H-S gels and destroy the existing CH crystal structure.

4.2. CRTS

The microstructures of the CRTS with different N-S contents at the curing age of 1-day, 3-day, and 7-day are shown in Figure 5.

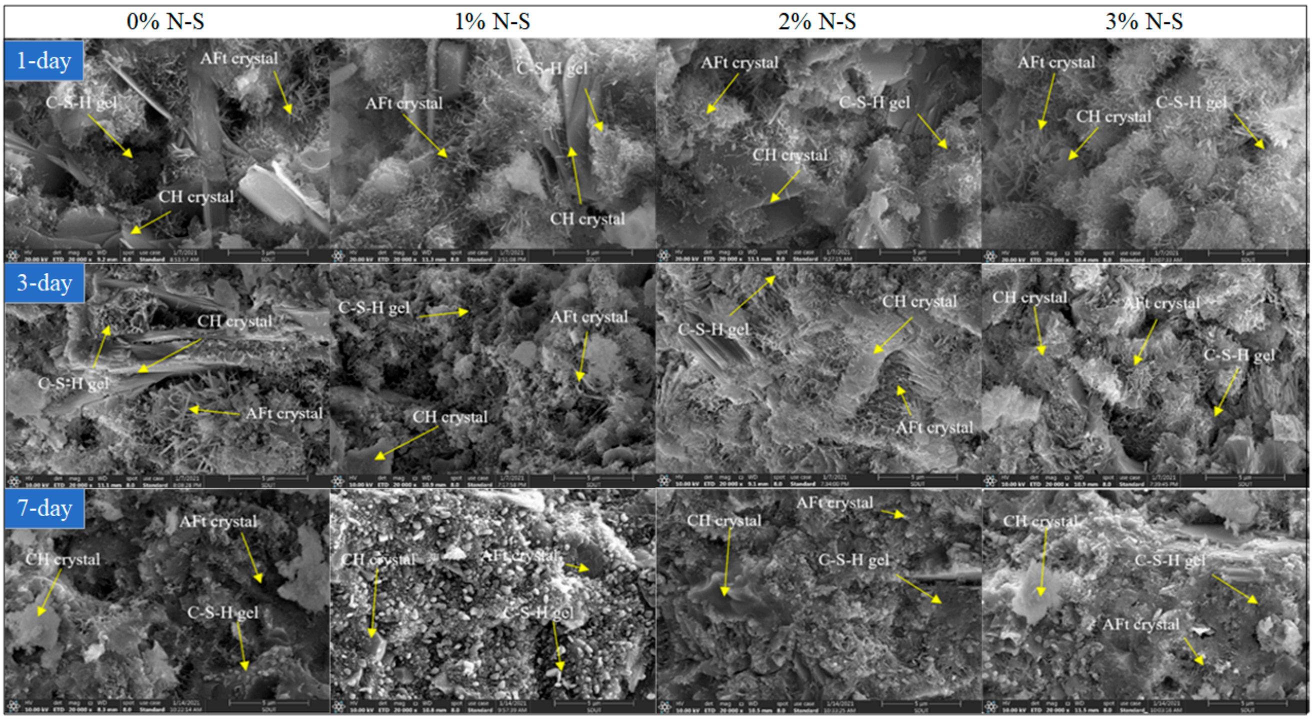


Figure 5. Microstructures of the cement grouting materials with the CRTS. (×20,000).

The CH crystals, C-S-H gels, and AFt can be observed in each CRTS. The microstructure of the CRTS without the N-S is shown that the CH crystals are mostly exposed on the surface, and AFt and C-S-H gels are dispersed mutually. This is because its hydration degree is not sufficient, which results in weak adhesion action of AFt and C-S-H gels.

However, their morphological distribution and interaction relationship varied with the change in the N-S contents. As the increase in N-S, the size of CH crystals gradually decreases, and its shape tends to be plate-like from lamellar, while C-S-H gels gradually increase. These results indicated that the increase in N-S contents can accelerate CH crystals consumption and C-S-H gels formation. The C-S-H gels and AFt gradually connected to each other and formed an interlaced skeleton structure. This explains why the compressive and flexural strength of the CRTS increases with the increase in N-S contents.

Furthermore, the void gradually decreases with the increase in N-S contents. The addition of N-S contents promotes the transformation of CH crystals and the formation of C-S-H gels, in particular, limiting the size distribution and density of voids. After curing for 3 days, the form of hydration products in the CRTS has basically formed and tended to be stable, and the increase in N-S has no obvious difference. It shows that the N-S mainly promotes the formation and development of early hydration products, and its effect gradually decreases with the growth of curing age.

5. Hydration Products of the CRTS

5.1. X-ray Diffraction Analysis

As shown in Figure 6, the XRD results of the CRTS and without toner were observed at different curing ages and with different N-S contents.

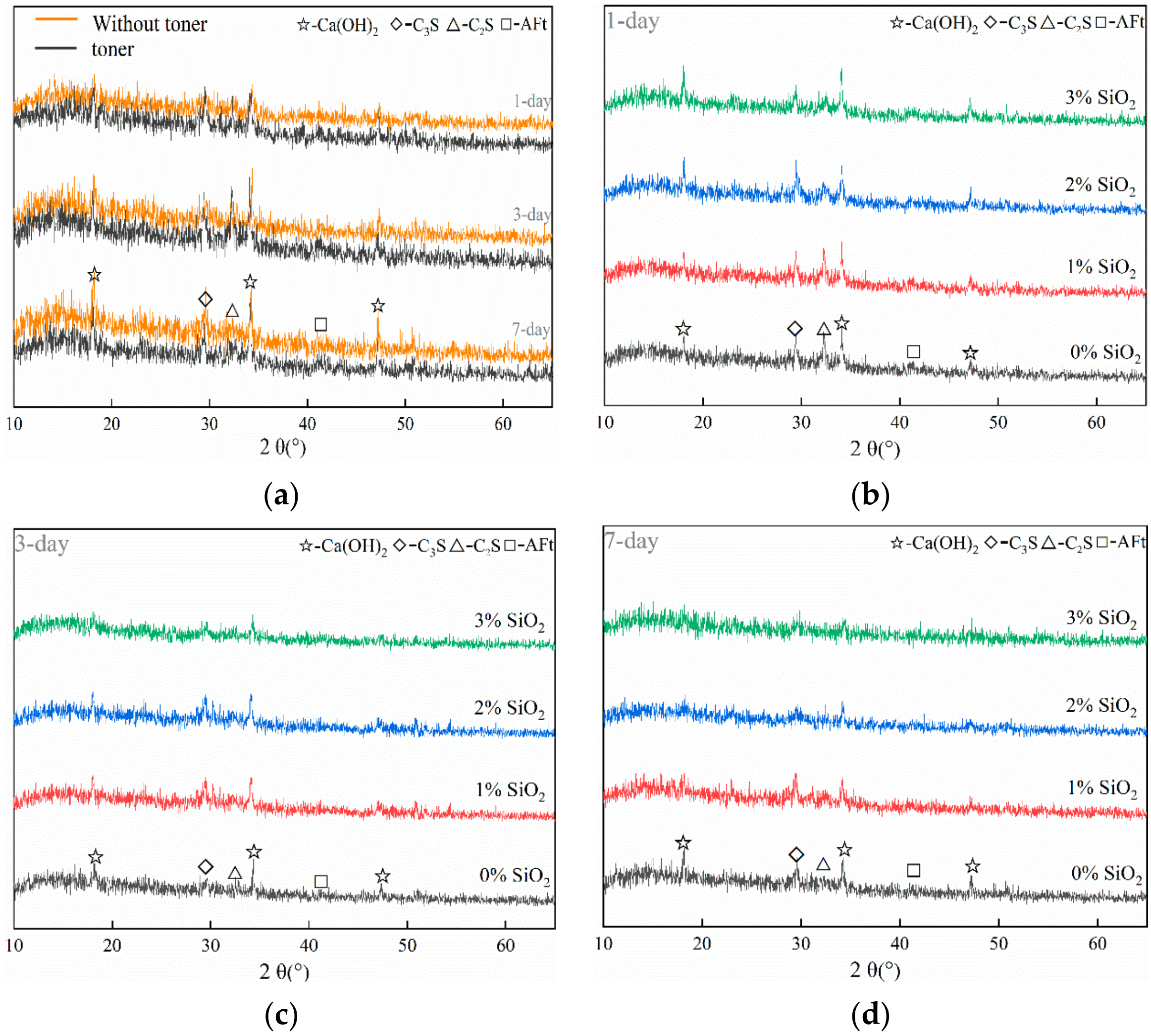


Figure 6. FTIR spectrums. (**a**) Cement grouting materials without the N-S, (**b**) CRTS at 1-day, (**c**) CRTS at 3-day, and (**d**) CRTS at 7-day.

According to Figure 6a, it can be found that the CH crystal diffraction peaks of the cement grouting materials with toner are low at the 1-day curing age, and its intensity of diffraction peak at the 3-day curing age is similar to that of the cement grouting materials without toner (1-day). This is due to that the addition of toner hinders the early hydration process and weakens the early performance of the materials. Moreover, compared with the cement grouting materials without toner, the CH crystals diffraction peaks at the 3-day and 7-day curing age are higher. It shows that the addition of toner delays the conversion from CH crystals to C-H-S gels, leading to the accumulation of CH crystals and higher diffraction peak intensity of CH crystals in the XRD. The macroscopic performance shows that the compressive and flexural strength of the cement grouting material with toner is relatively low.

In Figure 6b–d, it can be observed that the diffraction peak of CH crystals and AFt of the cement grouting materials with toner and nano-SiO_2 (CRTS) increases gradually as the increase in the N-S contents at the 1-day curing age, while the intensity of the characteristic peak of C_2S (dicalcium silicate) and C_3S (tricalcium silicate) decreases continuously. This is because the addition of the N-S contents accelerated the consumption of clinker in the CRTS and promoted the rapid formation of AFt and CH crystals. Moreover, with the increase in N-S, the attenuation degree of the diffraction peak intensity of C_3S is evidently higher than that of C_2S in the early curing period. It indicated that N-S mainly promotes the early hydration process of the CRTS by changing the balance of the C_3S hydration reaction and accelerating the conversion from C_3S to hydration products. During the curing age of 3-day and 7-day, the diffraction peak of hydration products gradually flattens, and the change due to the effect of the N-S content also decreases.

Hence, the above results indicate that the addition of the N-S contents reduces the hindrance effect of toner on the hydration process in the CRTS while mainly promoting the early hydration process of the CRTS. It benefits the rapid formation of CH crystals, thereby promoting the formation and stabilization of C-S-H gels.

5.2. Differential Scanning Calorimetry

At different curing ages, the mass loss (TG curve) and heat flow curve (DSC curve) of the CRTS with different N-S contents are shown in Figure 7. There are two obvious stages (S_I and S_{II}) of mass loss deriving from the thermal decomposition in the curves revealed. The mass loss at the S_I stage (150~200 °C) is the decomposition of AFt and C-S-H gels. Meanwhile, the S_{II} massless stage (400~450 °C) is formed by the decomposition and dehydration of CH crystals.

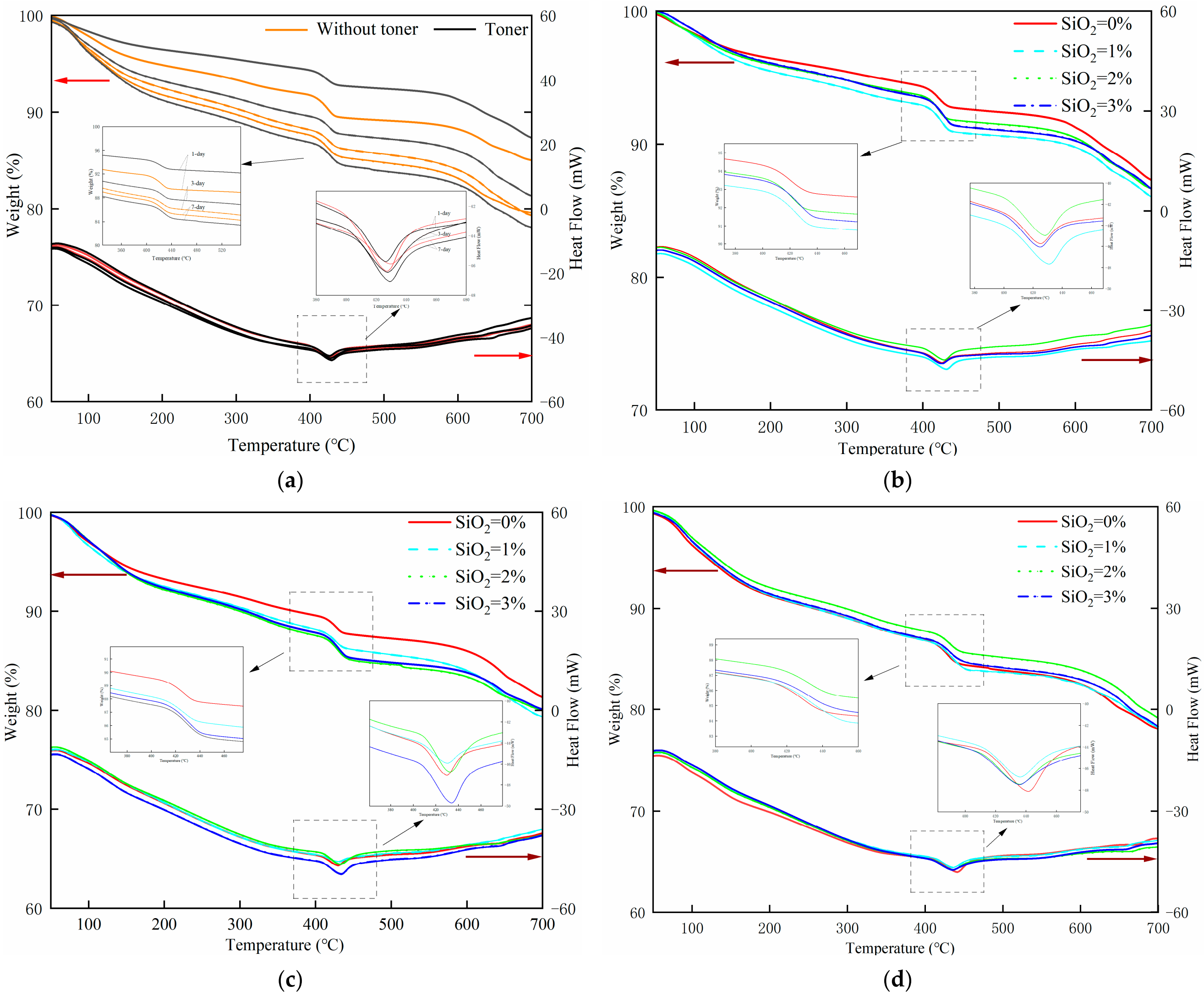


Figure 7. TG-DSC curves of the samples with the different curing ages: (**a**) cement grouting materials without the N-S, (**b**) CRTS at 1-day, (**c**) CRTS at 3-day, and (**d**) CRTS at 7-day.

As shown in Table 5, the mass loss and enthalpy change of the CRTS have two obvious stages with different curing days and N-S contents. The N0 represents cement grouting materials without toner, and the N-S.

Table 5. The mass loss of the CRTS with different stages.

SiO_2(%)	S_I (150~200 °C)			S_{II} (400~450 °C)			Total Mass Loss (0~700 °C)		
	1-Day	3-Day	7-Day	1-Day	3-Day	7-Day	1-Day	3-Day	7-Day
0	2.9	6.0	8.2	2.9	5.2	5.0	88.4	82.6	78.4
1	3	6.4	9.5	2.6	3.4	5.2	86.5	80.0	78.1
2	3.7	7.1	7.4	2.4	3.5	4.2	85.7	79.2	77.8
3	4.3	7.2	8.1	2.7	3.4	3.7	85.5	78.5	76.9
N0	4.6	7.1	8.1	2.5	4.8	3.4	85.2	77.5	75.7

As shown in Table 5, higher mass loss and enthalpy change of the two obvious stages ($\mathbf{S}_I$ and $\mathbf{S}_{II}$) are achieved for the N-S addition when compared to the CRTS without the N-S. This indicates the addition of the N-S promotes the hydration process of the CRTS. Moreover, at the curing age of 1-day and 3-day, with every 1% increase in the content of N-S, the mass loss of the $\mathbf{S}_I$-stage and $\mathbf{S}_{II}$-stage increases, respectively, on average, 0.25%, 0.70%, 0.35%, 0.10%, 0.60%, and 0.40%. However, the increase rate of mass loss decreases when the N-S content is 2~3%. This is because the agglomeration effect of excess N-S particles leads to inadequate hydration of the CRTS and shows concurrently that 2% N-S can obtain the best hydration gain for the CRTS.

A similar trend in mass loss was observed for the I-stage and II-stage at the 1-day curing age. However, the mass loss in $\mathbf{S}_I$-stage increased higher than that in $\mathbf{S}_{II}$-stage with the increase in curing days. It can be speculated that the disadvantageous effect of toner on the hydration process in the CRTS is overcome by the addition of the N-S. Meanwhile, the addition of the N-S is helpful in improving the formation of C-S-H gels and accelerating the formation of CH crystals to reach saturation at a faster rate. This results in a denser hydration structure, which verifies the conjecture in the SEM (see Section 4).

The endothermic peak corresponding to the mass loss in stage in the $\mathbf{S}_I$-stage is not obvious. This is because the products of the $\mathbf{S}_I$-stage are AFt and C-S-H gels, while toner in the CRTS obstructs the wrapping between C-S-H gels and slows down the change of heat absorption and release.

5.3. Fourier Transform Infrared Spectroscopy

The results of the FTIR tests are presented in Figure 8.

As shown in Figure 8, the CRTS has similar characteristic bands compared to the cement grouting materials with toner. However, the characteristic peak intensity of hydration products in cement grouting materials with toner is lower. This indicates that the addition of toner not produced new products and hindered the hydration process of the materials.

The wide band centered on 3550 cm^{-1} is the stretching vibrations that are caused by the O-H adsorption of water molecules, while the characteristic peak of 3643 cm^{-1} represents the O-H stretching of the CH crystals. Moreover, the characteristic peak at 1640 cm^{-1} represents the bending vibration of water molecules in the C-S-H gels interlayer. The peak at 1480–1485 cm^{-1} is caused by the C-O tension of $CO_3{}^{2-}$ and the bending vibration of carbonate.

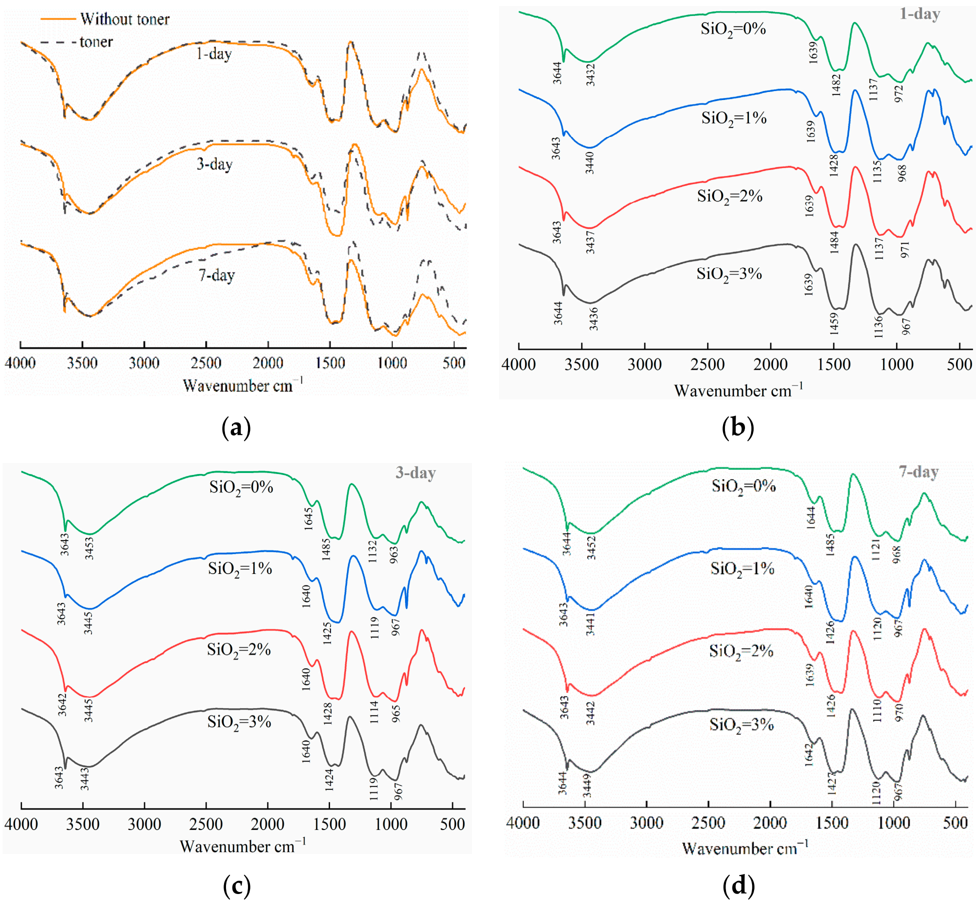


Figure 8. FTIR spectrums. (**a**) Cement grouting materials without the N-S, (**b**) CRTS at 1-day, (**c**) CRTS at 3-day, (**d**) and CRTS at 7-day.

6. Conclusions

In this study, the nano-SiO_2 (N-S) is used to improve the engineering properties of the CRTS used for the repair of reinforced concrete. Moreover, the effect mechanisms of the toner and N-S on the early strength of the CRTS under the different N-S contents and curing ages are revealed via the aforementioned four tests. The following conclusions can be drawn.

- The early strength (1-day and 3-day) of the CRTS decreases with the addition of the toner, while the fluidity accordingly increases, implying the toner brings a negative impact on the engineering properties of the CRTS. The N-S is able to reduce (but not eliminate) the negative impact. With the increase of every 1% N-S, the flexural strength of 1-day, 3-day, and 7-day average increased by 11.3%, 2.9%, and 0.9%, respectively, and the compressive strength of 1-day, 3-day, and 7-day average increased by 0.8%, 0.3%, and 0.1%.
- The toner and the N-S only influence the hydration progress of the CRTS rather than change the type of hydration products. Moreover, they both mainly act on the hydration process of the C_3S while presenting limited effects on the C_2S.
- The toner slows down the generation of the CH crystals and AFt in the early stage of the hydration process, especially for the CH crystals, so as to weaken the early strength of the CRTS. Although the N-S can expedite the conversion from generated CH crystals to C-H-S gels to partly improve the early strength, it is difficult to change the generation state of the CH crystals with the action of the toner. This is the reason why the N-S is unable to eliminate the negative impact of the toner on the early strength of the CRTS. In addition, overmuch the N-S will bring adverse effects on the hydration of the CRTS because of agglomeration.

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Conflicts of Interest: The authors declare no conflict of interest.

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