Article

Influence of Rubber Powder Modification Methods on the Mechanical and Durability Properties of Rubberized Magnesium Oxychloride Cement

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Abstract: In the present study, three modification methods, including water washing, sodium hydroxide (NaOH), and styrene–acrylic emulsion, were used to modify waste rubber powders. The influence of rubber powders on the mechanical properties and frost resistance of magnesium oxychloride cement was examined, and the different modification mechanisms were also analyzed. Based on the analysis of hydrophilic properties after modification, styrene–acrylic emulsion achieved the best modification effect, while water washing produced the least modification effect; regarding mechanical properties, magnesium oxychloride cement mixed with NaOH modified rubber powders achieved the best modification effect, in which the 28 d flexural strength and compressive strength increased by 41.2% and 59.6%, respectively. During the freeze-thaw cycles, the mass loss of specimens was reduced with an increase in the content of rubber powders. In addition, after 300 cycles, the relative dynamic modulus of elasticity of the blank sample was about 28.12%, while that of the magnesium oxychloride cement mixed with NaOH modified rubber powders was approximately 42.38%. In general, the properties of the modified rubber powder–magnesium oxychloride cement composite material can meet the requirements for engineering materials, which provides a theoretical basis and technical support for the application of rubberized magnesium oxychloride cement.

Keywords: rubber powder; magnesium oxychloride cement; surface modification; sodium hydroxide; styrene–acrylic emulsion

1. Introduction

Magnesium oxychloride cement is an inorganic cementitious material [1,2], which is widely used in various industries (e.g., agricultural appliances, wood production and home decoration, fire-fighting pipelines, etc.) due to its useful properties, such as being fast forming, providing superior mechanical properties early-stage, good adhesion and good fire resistance [3–7]. However, due to its shortcomings, for instance, low toughness, large capacity, and proneness to warping deformation, its applications are restricted [8–10]. At present, the primary method to improve the flexural toughness of magnesium oxychloride cement is to mix it with polypropylene fibers, polyethylene fibers, steel fibers, or other materials [11].

Rubber is a sort of elastic polymer. Adding it as a lightweight aggregate into cement paste can overcome the shortcomings of cement concrete and is a way of using waste rubber and so reducing the pollution caused by releasing it into the environment [12,13]. This discovery has attracted significant attention from scientific researchers; consequently, rubber cement-based composite materials came into being as a new type of composite material. It can be seen from numerous studies [14–16] that, after adding waste rubber powders as a lightweight filler into cement-based materials, the physical and mechanical...
properties, including compressive strength and flexural strength, of the resultant materials are significantly reduced. The decrease in strength is directly related to the amount and number of meshes of rubber powders that are mixed into cement-based composite materials. Eldin and Senouci et al. [17] reported that the compressive and splitting strengths of cement-based composite materials mixed with rubber in strip and granular shapes were lower than those of ordinary cement-based materials. Khabit and Topcu et al. [16] found that the compressive strength of cement-based composite materials mixed with fine rubber powders was much higher than that of cement-based materials mixed with thick rubber strips.

Plenty of earlier experiments [18–24] also suggested that it was possible to effectively improve cementation between rubber powders and cement and increase the flexural and compressive strength of rubber cement-based composite materials by giving specific treatments to the surface of rubber powders. For example, Senouci et al. removed the stains on the surface of rubber powders simply by soaking and rinsing; Rostami et al. soaked and cleaned the rubber powders with distilled water, CCl₄ reagent, and organic mixed emulsion. Their experimental results showed that, compared with the cement-based composite mixed with unmodified rubber powders, the strength of the cement-based composite material mixed with water-washed rubber powders was increased by about 15%, while that of the cement-based composite mixed with rubber powders modified by CCl₄ solution was increased by about 60% [25]. Z. Pavlik and O. Jankovsky et al. [26–29] maintain that adding elastic lightweight rubber particles instead of concrete aggregate can not only reduce adverse impacts on the environment, but also produce concretes with better physical and mechanical properties.

In this paper, the surface of rubber powders was treated in different ways to improve their compatibility with cement paste. Focusing on rubber–magnesium oxychloride cement, the influence of modified rubber powders on the mechanical properties and frost resistance of the cement was examined. The research findings can promote the comprehensive utilization of waste rubber and provide a theoretical basis and technical support for the application of engineering materials.

2. Raw Materials and Test Method

2.1. Raw Materials

The rubber powders used in the present study were 80-mesh rubber powders manufactured by Qingdao Huishang Rubber Co., Ltd. (Qingdao, China); the MgO powders were light-burned 85-mesh powders manufactured in Yingkou, Liaoning; the halogen flakes were manufactured in Haicheng, Liaoning, where MgCl₂ ≥ 60%, Na⁺ ≤ 0.004%, SO₄²⁻ ≤ 3%, Cl⁻ ≤ 0.05%, K⁺ ≤ 0.002%, and water-insoluble matter ≤ 0.18%; other chemical reagents (e.g., sodium hydroxide (NaOH) and styrene–acrylic emulsion) were all analytical reagents. Relevant parameters of rubber powder are shown in Table 1.

| Material  | Carbon Black Content % | Rubber Hydrocarbon Content % | Ash Content % | Tensile Strength MPa | Elongation at Break % | Bulk Density g/cm³ | Density g/cm³ |
|-----------|------------------------|------------------------------|---------------|----------------------|-----------------------|-------------------|---------------|
| 80-mesh   | 30                     | 56                           | 6.6           | 17.6                 | 55.8                  | 0.37              | 1.12          |

2.2. Sample Preparation

2.2.1. Modification of Rubber Powders

The surface of natural rubber powder represents the hydrophobic property. When using rubber powder in cementitious materials, proper treatment needs to be performed to modify the adhesion between rubber powder and the cement matrix. The surface impurities of rubber powder can be removed by washing the rubber powder with water. Zinc stearate on the surface of rubber powder can be at least partially removed in an
alkaline solid solution to improve the adhesion of the surface of the rubber powder to the cement group, while styrene acrylic emulsion could improve bonding between the inorganic material and the organic material. The particular process of rubber powder modification is as follows:

1. Washing: Soak the rubber powders completely in water for 24 h. After filtering the water, dry the rubber powders naturally in an outdoor environment for later use.

2. Modification by NaOH solution: Prepare saturated NaOH solution with a concentration of 10%. Mix the rubber powders with NaOH solution at a mass ratio of 1:4. Stir and soak the mixture for 24 h, and then rinse the rubber powders with clean water (pH = 7). Dry the rubber powders naturally for later use.

3. Modification by styrene–acrylic emulsion: Add the rubber powders and 15% styrene–acrylic emulsion into the aqueous solution. Stir the solution repeatedly until fully dissolved. Mix the rubber powders with the styrene–acrylic emulsion and make the mixture into a paste. Soak the mixture for 24 h. After drying at 80 °C, place the mixture into a ball mill for grinding to prepare modified rubber powders. Dry rubber powder can be obtained at 80 °C. The particle size of rubber powder remains almost the same as its original size after the grinding processes.

2.2.2. Preparation of Cement Specimens

In this experiment, the molar ratio of MgO:MgCl$_2$ was 6:1, the Baume degree (°Bé) of the MgCl$_2$ solution was about 26–28, and the amount of rubber powders added was determined based on the mass of MgO powders. The MgO powders and the MgCl$_2$ aqueous solution were weighed in proportion and added to the weighed unmodified/modified rubber powders. The mass ratio of rubber to magnesium oxide is 0–30%. The Baume degree of magnesium chloride solution of different samples remains unchanged, and the molar ratio of magnesium chloride to magnesium oxide remains unchanged. The mixture of the three was then stirred evenly and poured into prescribed molds, which were numbered accordingly. B–M represented the specimen of magnesium oxychloride cement without rubber powder; the specimen of magnesium oxychloride cement mixed with rubber powders was represented by R–M. More specifically, the unmodified rubber powder–magnesium oxychloride specimen was represented by NR–M; WaR–M represented the water-washed rubber powder–magnesium oxychloride specimen; the NaOH modified rubber powder–magnesium oxychloride specimen was represented by NaR–M; the styrene–acrylic emulsion modified rubber powder–magnesium oxychloride specimen was represented by SaR–M.

2.3. Experiment Methods

2.3.1. Hydrophilic Performance Test

The contact angle, also known as the wetting angle, refers to the angle formed by the tangent line and the solid-liquid boundary line, which is generally represented by the symbol $\theta$, as shown in Figure 1. It is used to reflect the tension of the interface, serving as an essential basis for judging the hydrophilicity of the cement specimen. In this paper, the image analysis method was used to measure the contact angle. For the specific procedure, a drop of liquid was dropped on the surface of the specimen first and the contact surface between the liquid and the specimen was photographed using a microscope with a camera. Then, the value of the contact angle was measured accordingly.

2.3.2. Mechanical Properties Tests

According to GB/T17671-1999 “Method of testing cements—Determination of strength (ISO Method)”, the samples were put into steel molds with dimensions of 40 mm × 40 mm × 160 mm, and three duplicated specimens were prepared for each group. The specimens were firstly cured in the air for 24 h before they were removed from the molds. Thereafter, the specimens were placed in a curing room (temperature 20 ± 1 °C, humidity above 95%) for 7 days and 28 days, until their testing date. Then, the flexural
and compressive strength tests were performed. During the flexural strength test, the side of the molding surface was taken as the contact surface, and the force application point was aligned with the centerline of the length direction of the test specimen. The load was applied uniformly in the vertical direction at a loading rate of 50 N/s until failure. During the compressive strength test, the side of molding surface was taken as the contact surface as well. The cement specimen was placed in the center of the press machine (with the centers aligned) and the load was applied uniformly at a loading rate of 2.4 kN/s until failure.

![Figure 1. Schematic diagram of the contact angle.](image)

2.3.3. Freeze-Thaw Durability Test

The frost resistance test was conducted following the quick-freezing method as specified in the “Standard for test methods of long-term performance and durability test method of ordinary concrete” (GB/T50082-2009). The size of the specimen was 40 mm × 40 mm × 160 mm, and three duplicated specimens were prepared for each group. First, the specimen was placed in an oven for drying until a constant weight was reached, and then weighed ($M_0$). Subsequently, a water soaking test was conducted for 72 h to render the specimen in a water-saturated condition. By remaining in the saturated condition, the specimen was then cooled at −20 °C for 3 h and thawed at 20 °C for approximately 1 h, which constituted a freeze-thaw cycle. The freeze-thaw cycle was repeated 50, 100, 150, 200, 250, and 300 times. After each freezing cycle, the specimen was dried to a constant weight and weighed ($M_1$) in order to calculate its mass loss and the relative dynamic elastic modulus. The freeze-thaw cycle test machine is shown in Figure 2.

![Figure 2. Freeze-thaw cycle equipment.](image)
2.3.4. Microstructural Properties

(1) Pore structure analysis: The porosity of the specimen was measured by the nitrogen adsorption method. In general, gas adsorption methods mainly utilize the phenomenon of capillary aggregation and the principle of volume equivalent substitution. On the premise that the shape of the pore is cylindrical and tubular, a capillary aggregation model is established so as to estimate the pore size distribution characteristics and the pore volume of the specimen. By measuring the amount of condensed gas of the specimen under different pressure conditions (pressure \( P \) and saturated pressure \( P_0 \)), the isotherm adsorption and desorption curves are drawn to obtain the pore volume and pore size distribution curves through theoretical methods.

(2) SEM microscopic test: A Quan-ta200 manufactured by the U.S. FEI company was used for the test. This equipment has a resolution of 1.0 nm, which is able to observe the microscopic morphology of hardened cementitious materials between coarse aggregates. By observing the interface bonding between rubber powders and the magnesium oxychloride cement through SEM, it is possible to understand the cementation between rubber powders and the cement.

3. Experiment Results and Discussions

3.1. Measurement of Contact Angle

The contact angle of the droplet on the rubber powder surface is formed by the tension balance between solid, liquid and gas. It conforms to the Young–Laplace Equation (1) as follows:

\[
\gamma_{sg} = \gamma_{sl} + \gamma_{lg} \cos \theta
\]

where \( \gamma \) refers to the tension between the interfaces; \( s, l, \) and \( g \) refer to solid, liquid, and gas, respectively; \( \theta \) refers to the contact angle formed by the tension balance between solid, liquid, and gas. When \( \theta < 90^\circ \), it indicates that the surface of the specimen is hydrophilic and can be infiltrated by water. The smaller the contact angle \( \theta \), the better the hydrophilicity. Conversely, when \( \theta \geq 90^\circ \), it indicates that the surface of the specimen is hydrophobic. The greater the contact angle \( \theta \), the less likely the liquid is to be absorbed by the specimen. Figure 3 shows the contact angle of unmodified rubber powders and rubber powders modified by different modes, where Figure 3a corresponds to the NR–M specimen, Figure 3b corresponds to the WaR–M specimen, Figure 3c corresponds to the NaR–M specimen, and Figure 3d corresponds to the SaR–M specimen.

It can be seen from the Figure 3a–d above that the contact angles of the NR–M specimen and the WaR–M specimen were 106.13\(^\circ\) and 97.31\(^\circ\), respectively, both greater than 90\(^\circ\). At this moment, the surface of the specimen was in a hydrophobic state. By comparison, the contact angles of NaR–M and SaR–M were 79.15\(^\circ\) and 54.07\(^\circ\), respectively, indicating that the surface of the rubber powder had changed from hydrophobic to hydrophilic. Overall, the contact angle of SaR–M was the smallest, implying that SaR–M has the best hydrophilic performance.

3.2. Mechanical Properties

Mechanical performance is the most significant indicator of the modification effect. Figures 5 and 6 show the influence of different rubber powder modification methods and rubber contents on the 7 d and 28 d flexural strength and compressive strength of magnesium oxychloride cement, respectively.

3.2.1. Flexural Strength

With the increase of rubber content, the flexural strength under all modification methods exhibited an increasing trend followed by a decreasing trend. The flexural strength of NaR–M was greater than that of B–M when the amount of rubber powders added was less than 20\%. The flexural strength of SaR–M was greater than that of B–M when the amount of rubber powders added was less than 15\%. From the perspective of modification method, NaR–M appeared to be the best option, followed by SaR–M, while
WaR–M and NR–M were not much different. When the amount of rubber powders added was 10%, the flexural strength reached the maximum value. Compared with NR–M, the 7 d and 28 d flexural strength of NaR–M had an average increase of 42.3% and 36.4%, respectively; those of SaR–M had an increase of 41.0% and 29.5%, respectively; while those of WaR–M had only an increase of 7.4% and 3.1% respectively (Figure 4).

![Figure 4](image_url)

**Figure 4.** The (a) 7 d flexural strength and (b) 28 d flexural strength of the R–M specimen showed basically the same trend.

3.2.2. Compressive Strength

It can be seen from Figure 5 that the 7 d and 28 d compressive strengths of the R–M specimen showed basically the same trend. With an increase of the amount of rubber...
powders added, the compressive strength under all modification methods exhibited an increasing trend followed by a decreasing trend. The compressive strength of NaR–M was greater than that of B–M when the amount of rubber powders added was less than 20%; the compressive strength of SaR–M was more significant than that of B–M when the amount of rubber powders added was less than 15%. From the perspective of the modification method, NaR–M appeared to be the best option, followed by SaR–M, while WaR–M and NR–M were not much different. When the addition amount of rubber powders was 10%, the compressive strength reached the maximum value. Compared with NR–M, the 7 d and 28 d compressive strength of NaR–M showed an average increase of 63.1% and 37.3%, respectively; those of SaR–M showed an increase of 29.5% and 23.5%, respectively, while those of WaR–M showed only an increase of 7.7% and 4.8%, respectively.

3.3. Frost Resistance
3.3.1. Apparent Analysis

Figure 6 shows the condition of the specimen with 10% of rubber powders added after 300 freeze-thaw cycles. (a) B–M specimen—the surface layer of cement had been completely frozen off and there were certain potholes; (b) NR–M specimen—the surface layer was partially frozen off; (c) WaR–M specimen—the surface layer was partially frozen off; (d) NaR–M specimen—only a small part around the edge was frozen off; (e) SaR–M specimen—only a small part around the edge was frozen off. From apparent analysis, it shows that adding a certain proportion of rubber powders is conducive to improving the frost resistance of magnesium oxychloride cement, and the frost resistance of NaR–M and SaR–M was better than that of NR–M and WaR–M.

3.3.2. Mass Loss Rate

Figure 7 shows the average mass loss rate of the specimen after every 25 freeze-thaw cycles in the 300-cycle test, which was calculated according to Equation (2):

\[
M_m = \frac{M_0 - M_1}{M_0}
\]

where \(M_m\) is the mass loss rate (%); \(M_0\) is the dry mass before freezing and thawing (g); \(M_1\) is the dry mass after freezing and thawing (g).
Figure 6. Photos of cement after 300 freeze-thaw cycles. (a) Blank specimen, (b) Unmodified specimen, (c) Modified by water-washing, (d) Modified by NaOH, (e) Modified by 15% styrene–acrylic emulsion.

Figure 7. Effect of different modified rubber powder on the quality loss of magnesium oxychloride cement.

After 300 freeze-thaw cycles, it can be seen in Figure 7 that the magnesium oxychloride cement exhibited good frost resistance—the mass loss of the B–M specimen was only 12.82%. With the addition of rubber powders, the frost resistance was improved to a certain extent;
the mass loss of NR–M and WaR–M was reduced to 11.01% and 10.78%, respectively. With the addition of modified rubber powders, the frost resistance was further improved; the mass loss of NaR–M and SaR–M was reduced to less than 10%.

3.3.3. Relative Dynamic Modulus of Elasticity

Figure 8 shows the relative dynamic modulus of elasticity of the specimen after every 25 freeze-thaw cycles in the 300-cycle test, which was calculated according to Equation (3):

$$P_i = \frac{f_{0i}}{f_{ni}} \times 100\%$$

(3)

where $P_i$ is the relative dynamic modulus of elasticity of the $i$-th specimen after $n$ freeze-thaw cycles (%), precision 0.01; $f_{0i}$ is the initial value of the fundamental transverse frequency of the $i$-th specimen before the freeze-thaw cycle test (Hz); $f_{ni}$ is the fundamental transverse frequency of the $i$-th specimen after $n$ freeze-thaw cycles (Hz).

![Figure 8](image_url)

**Figure 8.** Effect of different modified rubber powder on relative dynamic modulus of elasticity of cement.

After 300 freeze-thaw cycles, it can be seen from Figure 8 that the relative dynamic modulus of elasticity showed a decreasing trend with the increase of the number of freeze-thaw cycles. The NaR–M specimen had the highest relative dynamic modulus of elasticity, which was about 42.38%, followed by SaR–M, WaR–M and NR–M, while the B–M specimen had the lowest relative dynamic modulus of elasticity, which was about 28.12%.

The strength of magnesium oxychloride cement is easily affected by humidity, that is, its water resistance is poor, and it readily decomposes into magnesium hydroxide in the presence of water. However, after a freeze-thaw cycle, magnesium hydroxide reacts easily with carbon dioxide in the air to produce solid magnesium carbonate, which curbs the decline in strength. The addition of rubber enhances the surface tension of magnesium oxychloride cement, can generate more magnesium carbonate, and the rubber itself has a certain elasticity, which can disperse the stress caused by the change in temperature when the cement is frozen and the stress caused by expansion. Moreover, the modified rubber powder can improve its cementation with the cement, reduce the gap around the rubber powder and disperse the stress, inhibiting the formation and diffusion of cracks on the inner wall of pores when the cement is frozen, and reduce structural damage when the cement is frozen.
4. Microstructure Analysis

4.1. Pore Structure

Figure 9 shows the influence of modified rubber powders on the nitrogen adsorption isotherm of magnesium oxychloride cement. The comparison of nitrogen absorption can reflect the porosity in the cement structure. A higher porosity corresponds to less compactness in the structure and to reduced mechanical properties.

It can be seen in Figure 9 that R–M exhibited an increased porosity and reduced compactness compared with the B–M specimen. This is because the hydrophobicity of the surface of rubber powders leads to mutual repulsion between rubber powders and the cement paste, which reduces the cementation and the overall strength. The existence of pores improves frost resistance to a certain extent. When the cement is in a frozen state, the elasticity of rubber can disperse the stress caused by the temperature change and the stress caused by expansion, and inhibit the formation and spread of cracks on the inner walls of the pores, which can also improve the frost resistance. Compared with the NR–M specimen, WaR–M only had the impurities on the surface of rubber powders removed but was not substantially improved in terms of hydrophobicity; thus, the porosity of WaR–M was not much different from that of NR–M. The NaR–M and SaR–M specimens showed the lowest nitrogen absorption and therefore the lowest porosity.

4.2. SEM Analysis

In this section, the interface bonding between rubber powders and the magnesium oxychloride cement before and after modification was further analyzed through SEM observation (Figure 10).

It can be seen in Figure 10a that a large number of pores was associated with unmodified rubber powders. As rubber powders were embedded in the magnesium oxychloride cement, their contact area with the cement was limited. Meanwhile, the cement structure surrounding the rubber powders had a loose and porous structure and low compactness. This is the main reason for the significant reduction in flexural and compressive strength of magnesium oxychloride cement. It can be seen from Figure 10b that the addition of rubber powders did not affect the crystals of the magnesium oxychloride cement.

From Figure 10c, it can be seen that the pores around the water-washed rubber powders were slightly reduced, and their contact area with the cement was increased. Thus, the strength of the cement was slightly improved. However, there was still a great number of pores, and the porosity was not significantly improved. Further, as shown in
Figure 10d, the crystals of the magnesium oxychloride cement had not changed, suggesting that water-washing has a certain modification effect, but can only remove the impurities adsorbed on the surface of rubber powders, exposing the surface of rubber powders with increased surface roughness.

Figure 10. Cont.
Figure 10. SEM of magnesium oxychloride cement specimens mixed with rubber powders modified by different methods. (a,b) SEM of magnesium oxychloride cement mixed with unmodified rubber powders. (c,d) SEM of magnesium oxychloride cement mixed with rubber powders modified by water-washing. (e,f) SEM of magnesium oxychloride cement mixed with rubber powders modified by NaOH. (g,h) SEM of magnesium oxychloride cement mixed with rubber powders modified by styrene–acrylic emulsion.

From the 600× SEM image shown in Figure 10c, it can be seen that the NaOH modified rubber powders were tightly bonded with the magnesium oxychloride cement, leading to high compactness. Figure 10f shows that the crystals of the magnesium oxychloride cement remained basically unchanged. NaOH reacted with zinc stearate on the surface of the rubber powders to improve surface properties; meanwhile, strong alkali corrosion caused cracks on the surface of the rubber powders, which effectively improved their adhesion to cement.

From Figure 10g, it can be seen that the rubber powders modified by styrene–acrylic emulsion were tightly bonded with the magnesium oxychloride cement, with fewer pores and higher compactness exhibited. The styrene–acrylic emulsion created close contact with the cement by wrapping the rubber powders, which thereby increased the strength of the rubber–magnesium oxychloride cement. However, when the amount of styrene–acrylic emulsion was excessive, the styrene–acrylic emulsion film formed on the surface of rubber powders would absorb water and dissolve in the magnesium oxychloride cement paste. This would inhibit the growth of cement crystals during the hydration process. Consequently, the crystals would become thin and long, as shown in Figure 10h, leading to a reduction in the cement strength.

5. Conclusions

In this investigation, the effects of different modification methods on the comprehensive properties of rubberized magnesium oxychloride cement were evaluated. The results demonstrated that the proper modification method could improve the mechanical and microstructural properties and the durability of rubberized magnesium oxychloride cement. There are several conclusions that can be drawn:

1. In the WaR–M specimen, the impurities attached to the surface of rubber powders were removed. Compared with NR–M, this gave a slight improvement in mechanical properties and frost resistance, but the effect was not obvious.

2. The NaR–M specimen could chemically react with zinc stearate on the surface of rubber powders and corrode the surface of rubber powders to increase surface roughness. Thereby, the hydrophilicity of rubber powders was effectively improved, leading to tight bonding between rubber powders and the magnesium oxychloride cement. Compared with NR–M, the mechanical properties and frost resistance of NR–M were greatly improved.

3. The SaR–M specimen formed a hydrophilic film on the surface of rubber powders, which improved the hydrophilicity of rubber powders, leading to tight bonding between rubber powders and the magnesium oxychloride cement.
rubber powders and the magnesium oxychloride cement. Compared with NR-M, the mechanical properties and frost resistance of NR–M were greatly improved. However, excessive addition of styrene–acrylic emulsion would produce a certain inhibitory effect on the cement crystals, making the crystals thinner so as to affect the overall mechanical properties.

**Author Contributions:** Funding acquisition, Y.L.; investigation, D.Z.; resources, D.Z.; validation, Y.G.; visualization, D.Z. and S.W.; writing—original draft, D.Z.; writing—review and editing, D.Z., S.W., L.W., Y.L. and J.W. All authors have read and agreed to the published version of the manuscript.

**Funding:** This research was funded by National Key Research and Development Program of China: 2019YFC1907103, National Natural Science Foundation of China: 51672237, Primary Research and Development Plan of Jiangsu Province: BE2021684 and Jiangsu Agricultural Science and Technology Innovation Fund: CX(19)2041.

**Data Availability Statement:** All data included in this study are available upon request by contact with the corresponding author.

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

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