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Mechanical properties of magnesium potassium phosphate cement

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Keywords: magnesium potassium phosphate cement (MKPC), epoxy resin, glass fibre sheet, interface bonding performance of concrete.

Abstract. Magnesium potassium phosphate cement (MKPC) is a kind of phosphate hydrate formed by super-burning magnesia, soluble phosphate, a retarder and water in a suitable ratio and neutralised by acid-base neutralisation. A new type of environmentally friendly cementitious material, MKPC has received increasing attention in the field of civil engineering, especially in the field of building structural repair and reinforcement engineering. In this paper, the preparation process and compressive strength of MKPC were studied, and the mechanism of the compressive strength of MKPC was revealed. The shear strength characteristics of MKPC and epoxy resin in the interface between glass fibre sheets and concrete were compared. The results showed that when the water-cement ratio was 0.12 and the composite retarder ratio was 8 %, the compressive strength of MKPC increased to a maximum of 81.4 MPa. The formed BO33- covered the surface of $KMgPO_4 \cdot 6H_2O$ and hindered the continuation of the reaction, which resulted in a slow increase in the early compressive strength. MKPC can well replace the epoxy resin in the field of reinforced concrete structure and can significantly improve the interface bonding performance between glass fibre sheets and concrete.

1. Introduction

As the nation of China grows in economy, the number of offshore structures, such as sea-crossing bridges and large seaports, has been increasing. For example, the Hong Kong-Zhuhai-Macao Bridge has been designed and used for over 120 years, and the durability problem during structural service is apparent. If scientific methods can be used to accurately assess the damage degree of the structure, predict the evolution law of structural failure and adopt timely effective reinforcement techniques, the damage process of the reinforced concrete structure can be delayed, and the service life of the structure can be prolonged. At present, the techniques for reinforing concrete structures commonly used in engineering mainly include the increased section reinforcement method, outer steel plate reinforcement method, prestressed reinforcement method, shotcrete reinforcement method, additional member reinforcement method, and additional fulcrum reinforcement method [1]. These methods have certain defects and deficiencies, which inevitably increase the self-weight of reinforced concrete structures. In addition, the node processing of new and old structures is a difficult point. If the treatment is not good, it will have a more adverse effect on the reinforcement process and reinforcement effects. With the advancement of research, the application of fibre reinforced plastic (FRP) composite materials in civil engineering has gradually become a popular research topic at home and abroad.

Because building fires can cause enormous losses and impacts and occur frequently in people's lives, the high temperature at the time of occurrence severely restricts the carbon fibre fabric reinforcement technology in the event of a fire. Ordinary epoxy resin is not resistant to high temperature, and high-temperature-resistant epoxy resin is not only expensive but also needs to be adhered to the carbon fibre fabric in an ultra-high-temperature environment. Additionally, the ordinary epoxy resin is difficult to scale up in engineering processes. To improve the high-temperature resistance of the adhesive, the use of inorganic rubber instead of the organic epoxy adhesive is a promising method. Therefore, there is an urgent need to

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study a high-temperature-resistant inorganic cementitious material that is used simultaneously as an adhesive for concrete structures and to seal the anaerobic layer, but such research is relatively rare.

Magnesium phosphate cement is a kind of phosphate hydrate formed by super-burning magnesia, soluble phosphate, a retarder and water in a suitable ratio and neutralised by acid-base neutralisation. A new type of environmentally friendly cementitious material, magnesium phosphate cement has received increasing attention in the field of civil engineering, especially in the field of building structural repair and reinforcement engineering [1–2]. Compared with ordinary cement, magnesium phosphate cement has many advantages, such as fast coagulation speed, high early strength, strong adhesion to different materials, dry shrinkage, and good wear resistance [3–6].

According to the difference in soluble phosphate content, magnesium phosphate cement can be divided into three main types: phosphosilicate cement (SPC) [7], magnesium ammonium phosphate cement (MAPC) [8] and magnesium potassium phosphate cement (MKPC) [9–10]. Since ammonium dihydrogen phosphate ($NH_4H_2PO_4$) is used as the main reactant in the neutralisation reaction, MAPC will release irritating ammonia to the environment. Pollution has restricted the application of MAPC in the field of building structural reinforcement. Therefore, in this paper, potassium dihydrogen phosphate (KH_2PO_4) was used instead of ammonium dihydrogen phosphate ($NH_4H_2PO_4$) as the acidic component of MKPC. Additionally, potassium magnesium phosphate reinforced with glass fibre sheets was proposed, and the change regularity of the compressive strength of MKPC after strengthening was researched. The strengthening mechanism of glass-fibre-reinforced MKPC is discussed. This work will provide basic research for high-performance MKPC as an adhesive material instead of an epoxy resin-reinforced concrete structure and will expand the application of MKPC in civil engineering.

2. Methods

2.1. Materials and reagents

2.1.1. Cementitious material

High-purity magnesium oxide (ZH-V3L) was used in this study as the alkaline component in MKPC and was purchased from Zehui Chemical Group (Wuxi, China). The content of MgO in the high-purity magnesium oxide exceeds 98 %, and the relative molecular mass is 40.30. Potassium dihydrogen phosphate was used as the acidic component and was purchased from Haiteng Chemical Co., Ltd. (Suzhou, China). The density of potassium dihydrogen phosphate is 2.34 g/cm^3, and the relative molecular mass of KH_2PO_4 is 136.09.

Since the reaction rate of magnesium oxide and potassium dihydrogen phosphate is too fast to control, an appropriate amount of composite retarder was applied to slow the rate of the neutralisation reaction in this study. The composite retarder is composed of sodium tetraborate decahydrate, acetic acid and sodium polyphosphate, which were all purchased from Tianjin Yongda Chemical Reagent Co., Ltd. (Tianjin, China).

Through a number of pre-tested trials, the basic ratio of magnesium oxide, potassium dihydrogen phosphate and composite retarder is 64: 32: 4 by weight. The final setting time of MKPC generally does not exceed 20 minutes, which can result in good work performance.

2.1.2. Sand, stone and concrete mix ratio

In this study, a glass fibre sheet-concrete interface bonding test was carried out. The fine aggregate of the plain concrete is Songhua river sand (fineness modulus is 2.47), the coarse aggregate is granite gravel, and the cement is P.O. 42.50 Portland cement produced by Jilin Yatai Group. The mix ratio of the plain concrete (cement: water: sand: gravel) is equal to 360: 160: 625: 1275 by weight. The size of the plain concrete specimen was 100 mm × 100 mm × 400 mm and was maintained for 28 days under standard curing conditions. The measured value of the cubic compressive strength is 32.82 MPa.

2.1.3. Glass fibre sheet and epoxy resin

The glass fibre sheet is a CWR90-90 type alkali-free glass fibre roving fabric produced by Sanxing Glass fibre Co., Ltd. (Zigong, China) and has an areal density of 800 g/m^2 and a thickness of 2.4 mm. To compare the interfacial adhesion properties of MKPC, the epoxy resin-bonded glass fibre sheet was used in this study. A diglycidyl ether of bisphenol-F (DGEBF) epoxy resin with the brand name NPEF-170 was purchased from Nan-Ya Plastic Corporation (New Taipei, China). The epoxide equivalent is 163.80 g/eg, and the density is 1.19 g/cm^3. The hardener is methyl hexahydrophthalic anhydride (MeHHPA), provided by Qing Yang Chemistry Co., Ltd. (Jiaxing, China). The preparation of DGEBF and MeHHPA is 100:80 by weight.

2.2. Mix design and preparation of magnesium potassium phosphate cement

The magnesium oxide and potassium dihydrogen phosphate were taken and placed in a dry cement mortar mixer vessel. Then, the composite retarder was gradually added under stirring at 1000 rpm until completely incorporated, and the stirring was continued for approximately 2 minutes. Finally, distilled water was added twice, 90 % distilled water was stirred at 500 rpm, and the last 10 % distilled water was stirred at 1000 rpm. The mixture was stirred in a fast gear position, stirred for another 3 minutes, and the slurry was completely uniform, thereby obtaining the MKPC cementitious material. The composition ratio of MKPC is shown in Table 1. Sample No. MKPC-1, MKPC-2, MKPC-3, MKPC-4 and MKPC-5 were designed to research the effect of water cement ratio on the performance of MKPC, and sample No. MKPC-6, MKPC-7, MKPC-8, MKPC-9 and MKPC-10 were designed to research the effect of composite retarder mixture on the performance of MKPC.

Table 1. Composition ratio of MKPC (kg/m^3).

Sample No.	MgO	KH_2PO_4	Compound retarder	Distilled water	Water cement ratio
MKPC-1	1500	750	75	186	0.08
MKPC-2	1500	750	75	232.5	0.10
MKPC-3	1500	750	75	279	0.12
MKPC-4	1500	750	75	325.5	0.14
MKPC-5	1500	750	75	372	0.16
MKPC-6	1514.3	852.2	2 % MgO = 30.2	283.9	0.12
MKPC-7	1496.7	842.3	4 % MgO = 59.8	280.6	0.12
MKPC-8	1478.5	832.1	6 % MgO = 88.7	277.2	0.12
MKPC-9	1459.0	821.1	8 % MgO = 116.7	273.6	0.12
MKPC-10	1433.4	806.7	10 % MgO = 143.3	268.8	0.12

2.3. Characterisation

2.3.1. Compressive strength test

After a standard maintenance period of 28 days, the cured MKPC sample was subjected to a standard compressive strength test. The size of the MKPC sample was 100 mm × 100 mm × 100 mm. The compressive strength test was performed at room temperature in a computer-controlled universal mechanical testing machine (JQ03A, Zhongchen Data Technical and Equipment Co., China), according to GB 50010-2010 (code for the design of concrete structures). The loading speed was 1 mm/min, and five specimens were tested for each MKPC sample.

2.3.2. Effect of different cementitious materials on the bonding properties of glass fibre sheets and concrete

The surfaces of all the specimens were roughened by a needle scaler to expose coarse aggregates. A manual lay-up procedure was conducted to bond the glass fibre onto the surface of the concrete substrates. A bonding graph of the glass fibre sheets and concrete is shown in Figure 1. One layer of glass fibre sheet with a width of 50 mm was bonded on one side of a plain concrete prism along the axial direction with MKPC or epoxy resin. All specimens had a bonded length of 100 mm, which was longer than the effective bond length estimated from the previous model. The specimens were cured for 3 days, 7 days, 14 days and 28 days to compare the effect of different cementitious materials on the bonding properties of glass fibre sheets and concrete. The bonding properties of the glass fibre sheet and concrete were evaluated at room temperature in a computer-controlled universal mechanical testing machine (JQ03A, Zhongchen Data Technical and Equipment Co., China), and the loading model graph is shown in Figure 1.

3. Results and Discussion

3.1. Influence of different water-cement ratios and retarder dosage on the strength of MKPC

The compressive strength of MKPC under different water-cement ratios (0.08, 0.10, 0.12, 0.14, and 0.16) was analysed, as shown in Figure 2. The data are expressed as the mean ± SEM and were analysed with SPSS 13.0 software. Statistical comparisons between two groups were performed using Student’s t-test. Statistical comparisons among multiple groups were examined using analysis of variance (ANOVA). A two-tailed $P < 0.05$ was considered statistically significant.

With the increase in the water cement ratio, the compressive strength of MKPC increases first and then decreases, showing a parabolic change law. When the water cement ratio reaches 0.12, the compressive strength of MKPC reaches a maximum value of 73.8 MPa. When the water cement ratio of MKPC exceeds 0.12, the compressive strength of MKPC is greatly reduced, and the fluidity is too large. Thus, MKPC is not easily applied in subsequent engineering processes, such as bonding of glass fibre sheets. To some extent, the application of MKPC is limited.

Several studies have shown that the optimal molar ratio of over-burnt magnesium oxide to potassium dihydrogen phosphate is between 5:1 and 7:1, and the strength and fluidity of MKPC will reach optimum [11–12]. Therefore, the relationship between the content of composite retarder (2 % M, 4 % M, 6 % M, 8 % M, or 10 % M) and the compressive strength of MKPC was analysed when the molar ratio of over-burnt magnesium oxide to potassium dihydrogen phosphate was 6:1 and the water cement ratio was 0.12, as shown in Figure 3.

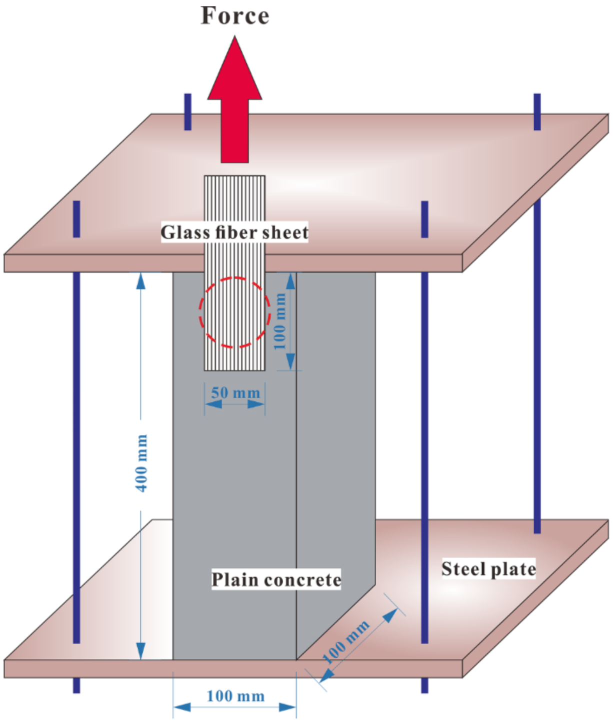

Figure 1. Bonding graph of glass fiber sheet and concrete.

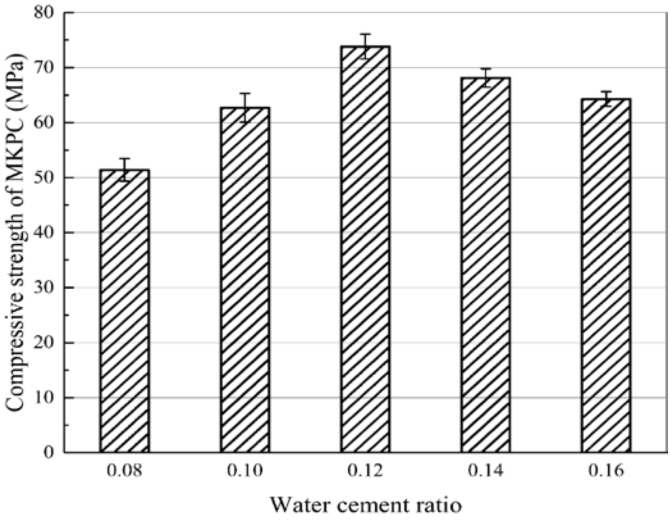

Figure 2. Relationship between water cement ratio and compressive strength of MKPC.

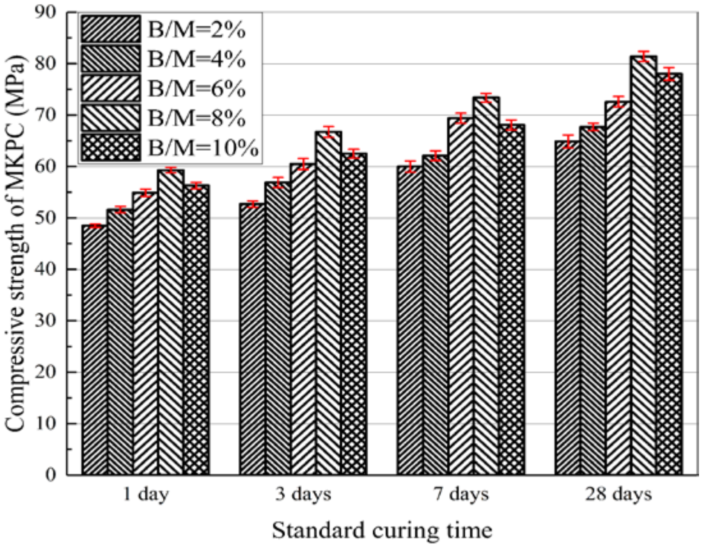

Figure 3. Relationship between compound retarder content and compressive strength of MKPC.

In Figure 3, the data are expressed as the mean ± SEM and were analysed with SPSS 13.0 software. Statistical comparisons between two groups were performed using Student's t-test. Statistical comparisons among multiple groups were examined using analysis of variance (ANOVA). A two-tailed $P < 0.05$ was considered statistically significant.

As the proportion of composite retarder increased from 2 % to 10 %, the compressive strength of MKPC first increased and then decreased. When the mass of composite retarder added was approximately 8 % that of the over-burnt magnesium oxide, the compressive strength reached the maximum value (81.4 MPa).

The main reason for this change is that the fluidity of MKPC increases with increasing water cement ratio. When the water cement ratio is 0.08, the fluidity is low and the workability is poor, and the initial setting time is too short to be suitable for engineering applications. According to the main chemical reaction in MKPC processing (related formula as shown below), when the water cement ratio exceeds the optimal water cement ratio of 0.12, excess water will rapidly prompt the hydration products towards the direction of the potassium magnesium phosphate complex ($KMgPO_4\,6H_2O$), which will lead to a decrease in the compressive strength of MKPC [13–15]. At the same time, excess water will lead to excessive fluidity, which is not conducive to engineering applications [16, 17].

$$KH_2PO_4 \rightarrow PO_4^{3-} + 2H^+ + K^+$$

$$MgO + 2H_2O \rightarrow Mg^{2+} + 2OH^-$$

$$KH_2PO_4 + MgO + 5H_2O \rightarrow KMgPO_4 \cdot 6H_2O$$

The composite retarder was applied as an additive in MKPC. The main component of borax ($Na_2B_4O_7 \cdot 10H_2O$) is formed in the hydration process [18–20]. The hydrolysis reaction is shown in the following formula.

$$Na_2B_4O_7 \cdot 10H_2O \rightarrow Na_2B_4O_7 + 10H_2O$$

$$Na_2B_4O_7 + 3H_2O \rightarrow 2NaBO_2 + 2H_3BO_3$$

$$2NaBO_2 + 4H_2O \rightarrow 2NaOH + 2H_3BO_3$$

The surface of the potassium magnesium phosphate complex ($KMgPO_4\,6H_2O$) was covered by the generated BO_3^{3-}, which hindered the continuation of the reaction. Therefore, the early compressive strength of MKPC increases slowly, and the addition of excess composite retarder will reduce the reaction rate. The hydration of borax was completed by increasing the curing time of MKPC, and the hydration of potassium dihydrogen phosphate is a dynamic equilibrium process, which leads to the rapid increase in the compressive strength of MKPC in the later period. However, excess composite retarder will still offset the growth trend of the compressive strength of MKPC.

3.2. Influence of different cementitious materials on the bonding properties of the glass fibre sheet-concrete interface

In recent years, FRP composite-reinforced concrete has been widely applied in concrete structure repair and has achieved remarkable reinforcement effects. This method of reinforcing concrete involves pasting or wrapping the FRP composite material on the outside of the concrete beam to improve the bearing capacity. The main advantages are high strength, low quality, convenient construction and good corrosion resistance.

In this paper, a glass fibre sheet was attached to the surface of ordinary concrete using MKPC ($W/M + K = 0.12$, $B = 8$ % M) and epoxy resin materials, as shown in Figure 1. After the paste was completed, all the test specimens were placed in a laboratory environment and were maintained for 3 days, 7 days, 14 days and 28 days. Then, a standard single-shear test was performed, and the shear strength of the interface between the glass fibre sheet and concrete is shown in Table 2.

Table 2. Shear strength of the interface between the glass fibre sheet and concrete (MPa).

Standard curing time	MKPC	Epoxy resin
3 days	2.08	1.67
7 days	2.34	1.92
14 days	2.51	1.94
28 days	2.72	1.94

According to the variation in the shear strength of the glass fibre sheet-concrete interface bonded with different cementing materials with curing time, with the gradual increase in the curing age, the interfacial shear strength of the glass fibre sheet bonded with MKPC and ordinary concrete is greater than that of the epoxy tree at the same curing age.

When the curing age reaches 28 days, the shear strength of the bonding interface reaches 2.72 MPa, which is the maximum value of all the test groups. The maximum failure mode is the delamination tearing of the glass fibre sheet (as shown in Figure 4), and the MKPC layer is not damaged (as shown in Figure 4). This mode indicates that MKPC plays a very good role as cementing material in the interface area between glass fibre sheets and concrete.

The shear strength of the bonding interface between MKPC and epoxy resin at different curing ages was compared and analysed, as shown in Figure 5. With increasing curing time, the shear strength of the glass fibre-concrete interface bonded with MKPC increased gradually and slowed down gradually after 14 days, and the growth rate slowed down significantly after 28 days. The shear strength is expected to continue to increase with curing time, but the growth rate is relatively slow. When the epoxy resin was applied, the shear strength increased rapidly at 3 days and then stabilised gradually. After 14 days, the shear strength increased slightly, which can be considered as no longer increasing.

The main reason for these different observations is that the strength evolution laws of the two cementitious materials are different. MKPC is a kind of fast-hardening and high-strength material. Its strength develops rapidly in the early stage and has great growth potential. Epoxy resin is a typical thermosetting polymer composite. Its strength hardly changes significantly with the external environment after curing, and its stability is good. From the above analysis, the MKPC prepared in this paper can better replace the epoxy resin to reinforce concrete structures, and the interfacial shear performance of MKPC has been significantly improved. Additionally, ammonia is no longer released during construction, thus expanding the application of MKPC in civil engineering.

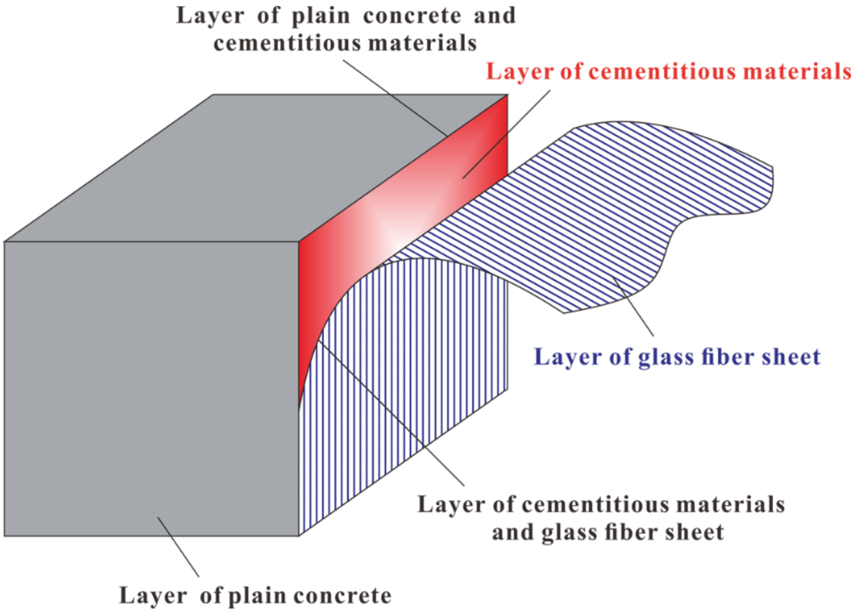

Figure 4. Structural relationship of glass fiber sheet and concrete bonded with cementitious material.

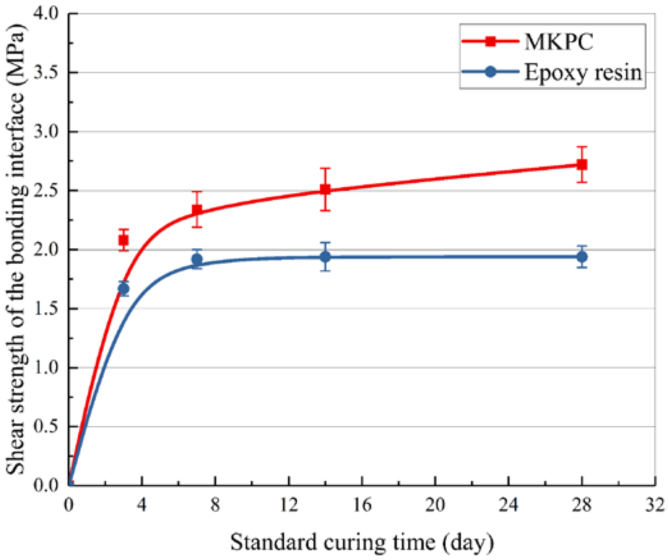

Figure 5. Shear strength of interface between glass fiber sheet and concrete.

4. Conclusions

This paper presented the influence of the water cement ratio and retarder content on the compressive strength of MKPC and its variation law. The BO_3^{3-} formed by hydration caused the compressive strength of MKPC to increase first and then decrease, and excess composite retarder hindered the solidification of MKPC. MKPC can better replace epoxy resin to reinforce concrete structures and significantly improve their interfacial shear strength. Additionally, ammonia was no longer released during construction, thus expanding the application of MKPC in civil engineering.

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