Minireview Article

Research Progress of Magnesium Phosphate Cement Repair Material

ABSTRACT

Magnesium phosphate cement (MPC) is produced by the hydration reaction of magnesium oxide and phosphate, which is a new type of cementitious material combined by chemical bonding, and has a short setting time and high early strength while reducing the amount of CO_2 emission, and has a broad prospect in the airport runway, tunnels, mines, and other civil buildings and national defense projects such as emergency repair. However, the hydration speed of magnesium phosphate cement is extremely fast, and the setting time is very short, resulting in some engineering construction can not be carried out. In view of the problems demanded by magnesium phosphate cement in engineering applications, the preparation and hydration process of magnesium phosphate cement was studied by using the method of literature review, focusing on the research of setting time, mechanical properties, durability and volume stability of magnesium phosphate cement, and the related problems were reviewed and discussed.

Keywords: Magnesium phosphate cement, setting time, mechanical properties, volume stability, durability

1. INTRODUCTION

"Industrial decarbonisation is becoming increasingly popular as concerns about global warming grow. The cement industry" [1] is an important area of carbon neutrality and is one of the main sources of CO_2 emissions in China. "A recent UN report paints a grim picture of the environmental situation, stating that the construction industry alone accounts for 36 per cent of the world's total energy consumption and 37 per cent of its CO_2 emissions. It is estimated that for every tonne of Ordinary Portland cement (OPC) produced, an equivalent amount of CO_2 is emitted into the atmosphere. It has been estimated that in 2021, India's cement manufacturing emitted 149 million tonnes of CO_2, making it the world's Preparation of magnesium phosphate cement second-largest polluter of cement manufacturing. If no action is taken to reduce carbon emissions from the construction sector, emissions will increase given the significant expansion of the sector expected, especially in developing countries like India" [2] "The construction industry is also transitioning to cleaner technologies, materials and adhesives. Existing technologies to reduce CO_2 emissions in the cement industry include raw material substitution by replacing certain components of raw meal with low-carbon raw materials or industrial wastes; fuel substitution by applying cleaner fuels with low carbon emissions to cement production; clinker substitution by adding gel-active materials to concrete to save on clinker use; methods and technologies to improve energy efficiency in the application of electricity and the use of fuels; and the promising, but not yet large-scale, application of carbon capture, storage and transfer[3-5]. Carbon Capture and Storage (CCS), which is promising but not yet applied on a large scale"[6-7]. "Raw material substitution is the most effective low-carbon production method, magnesium phosphate cement is one of the representatives" [8]. "Magnesium phosphate cement is a new type of cementitious material, compared with Portland cement, magnesium phosphate cement through a series of physicochemical effects of the raw material itself to produce: high speed of setting; high early strength; as low as minus 20 ℃ of the setting and hardening ability; high bonding strength; durability, and magnesium phosphate cement in the hydration process micro-expansion characteristics can be effective in the process of hydration, but not yet large-scale application of carbon capture and storage (CCS)" [9-10]."The micro-expansion characteristics of the hydration process can effectively reduce the shrinkage rate, can prevent the magnesium phosphate cement hardened slurry cracking" [11] ,"while the coefficient of thermal expansion of magnesium phosphate cement and ordinary concrete is closer to the repair and concrete interface with better compatibility" [12] . However, magnesium phosphate cement is currently in use in the process of the following problems[13] : Firstly, set hardening fast, inconvenient construction, adding retarder, strength decline; Secondly, magnesium phosphate cement water resistance is poor, water after the reduction of structural compactness, serious loss of strength; Thirdly: the cost of cost is expensive, and is currently only used in key projects.

This paper briefly describes the preparation and hydration process of magnesium phosphate cement, focuses on the research on setting time, mechanical properties, bonding properties and durability of magnesium phosphate cement, and discusses related issues.

2. PREPARATION OF MAGNESIUM PHOSPHATE CEMENT

Magnesium phosphate cement is generally an environmentally friendly cementitious material formed by mixing magnesium oxide (MgO), phosphates and admixtures such as retarders in a certain proportion and undergoing an acid-base neutralisationreaction. Phosphates are mainly ammonium dihydrogen phosphate ($NH_4H_2PO_4$) and potassium dihydrogen phosphate (KH_2PO_4). Taking KH_2PO_4 as an example, Magnesium potassium phosphate cement (MKPC) is able to react rapidly when mixed with water to form phosphates with gelling properties. It has been shown[15-19] that some silica minerals and solid wastes are easy to form new phosphates when mixed with MKPC. Due to its low solubility, the network structure of MKPC hydration products has the characteristics of high strength, good stability, low porosity, and high solidification rate of hazardous components, which also provides a new way of thinking for the treatment of solid wastes and radioactive hazardous pollutants.

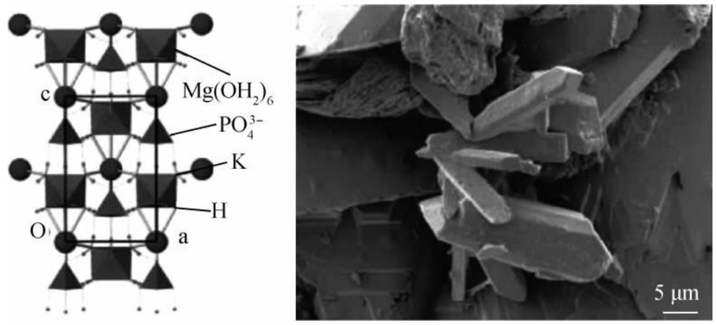
3. HYDRATION MECHANISM OF MAGNESIUM PHOSPHATE CEMENT

The hydration reaction of magnesium phosphate cement (MKPC) is mainly the process of acid-base neutralisation reaction between magnesium oxide and phosphate to release a large amount of heat, which is an environmentally friendly cementitious material. Xu[20], Meng[21], Chau[22]and Ding cast[23] studied the hydration reaction process of MKPC, and the results show that the main hydration product of MKPC is $MgKPO_4$-$6H_2O$ (MKPC), and the crystal structure of the MKPC is composed of "PO^{2-}"tetrahedra, MgO -$6H_2O$ octahedra and "K^+"combined, and the

hydration reaction equation is shown in (1):

$$MgO+KH_2PO+5H_2O \rightarrow MgKPO_4\text{-}6H_2O(1)$$

In addition it has been found that the hydration product MKPC morphology of MKPC is mainly in the form of short rods. See Figure 1.



(a) Crystal structure (b) SEM image

Fig. 1 Schematic diagram of MKPC[24]

At present, there are two main theoretical explanations for the hydration and hardening of magnesium phosphate cement: local chemical reaction mechanism and solution diffusion mechanism. Most scholars agree with the former, and the solution diffusion mechanism divides the hydration and hardening process of magnesium phosphate cement into three main stages[25-26] :

The first stage: dissolution of phosphate and borax. When the magnesium phosphate cement is mixed with water, the water-soluble phosphate and borax dissolve first, releasing "H^+","PO_4^{3-}"and "$B_4O_7^{2-}$",forming a low pH value phosphate aqueous solution;

Stage 2: Dissolution of magnesium oxide. The dissolution rate of magnesium oxide is much slower compared to phosphate, so the dissolution process of magnesium oxide takes place in an acidic phosphate aqueous solution, gradually releasing "Mg^{2+}", which exists in aqueous solution in the form of "$Mg(H_2O)_6^{2+}$";

The third stage: the formation of magnesium phosphate cement stone. "Mg^{2+}" dissolved in large quantities, "$H_2PO_4^-$","HPO_4^{2-}"constantly ionised"H^+"and "PO_4^{3-}", with the continuous consumption of "H^+" , the system pH gradually rises, the solution "$Mg(H_2O)_6^{2+}$","PO_4^{3-}"and"NH_4^+","K^+" and other ions began to react to generate hydration products, and ultimately the hydration products and the un-reacted particles of magnesium oxide are mutually adhesive, and the system is rapidly coagulated and hardened, the formation of magnesium phosphate cement stone.

4. CONDENSATION TIME

Magnesium phosphate cement is prepared by heavy burning magnesium oxide, potassium dihydrogen phosphate and retarder as raw materials, and its raw materials will affect the setting time of the cement to a certain extent, mainly by changing the rate of hydrolysis of MgO and thus affecting the rate of hydration reaction of magnesium phosphate cement and changing its setting time.

Ichraf[27] found that the setting time of magnesium phosphate cements became longer as the sintering temperature of magnesium silicate increased, and slurries prepared from magnesium silicate calcined above 700°C did not harden and there was no way to obtain a specific setting time[28]. These results, as shown in Table 1, indicate that there is no way for magnesium silicate calcined at this temperature to react with KH_2PO_4 , which may be due to the crystalline state of magnesium silicate. On the other hand, for the magnesium silicate calcined below 700 °C the solidification time gradually becomes longer with the increase of the calcination temperature and reaches a maximum value of 68 min at 700°C[29-30].

Calcinationtemperature (℃)	Handling	Setting time(min)
As-synthesized	Reacts quickly and forms a hardened cement	4.5
500	Reacts more slowly and forms a hardened cement	14
550	Reacts more slowly and forms a hardened cement	18
600	Reacts more slowly and forms a hardened cement	21
650	Reacts more slowly and forms a hardened cement	25
700	Reacts too slowly and forms a dry crumbled mixture	68
750	The mixture is dry after 2 days but it does not set intohardened cement	-
800	The mixture is dry after 2 days but it does not set into hardened cement	-

Table1 Setting time of MKPC mortar prepared by calcining magnesium silicate at different temperatures[27]

In addition to the influence of the activity of raw materials itself on the setting time, the fineness of raw materials also greatly affects the reaction rate and thus the setting time of cementitious materials. Liu Jin[31] found that “the setting and hardening time of magnesium phosphate cement is greatly affected by the particle size of MgO. The more MgO particles below 30um particle size, the shorter the setting time of cement paste and the higher the mobility; in addition, the particle size of MgO particles seriously affects the development of crystal lattice inside the hydrationy products of cement and has a greater impact on the early strength of magnesium phosphate cement, the smaller the particle size of MgO particles, the lower the early strength of magnesium phosphate cement. The smaller the Mg0 particle size, the lower the early strength of MKPC. Therefore, in order to ensure that the magnesium phosphate cement has both constructability and early strength and fluidity, the particle size of MgO particles should be reasonably controlled”.

In order to improve the setting time of cementitious materials, adding retarding agents is usually a very effective way, but the performance of the materials may be affected after the retarding agents are added. [32]Through the research in recent years, it has been found that the main retarders of magnesium phosphate cement are borax, boric acid, sodium tripolyphosphate and so on. Magnesium phosphate cement mainly reduces the overall exothermic rate and peak exothermic value of the cement by adding retarder[33], so as to reduce the hydration rate of the cement and prolong the setting time of the cement.[34] D.V. Ribeiro[35] found that the setting time of the magnesium phosphate cementitious material increased gradually with the increase of the concentration of boric acid under the same conditions. Sarkar[36] thought that it was the formation of a layer of magnesium borate compounds on the surface of the MgO particles after adding boric acid that had affected the performance of the material. He concluded that the addition of boric acid resulted in the formation of a layer of magnesium borate compounds on the surface of MgO particles[36], which encapsulated and covered the MgO particles, preventing the reaction of MgO with phosphate and further affecting the setting time of the cementitious material.Luo[37] investigated the effect of Borax (BS) and Sodium Gluconate (SG) retarder on the performance of Magnesium Phosphate Cement, and found that the setting time increases with the increase in the content of BS. The detailed results are shown in Fig.2.The prolongation of setting time after the addition of the composite retarder indicates that both BR and SG components of the retarder can play a role in regulating the setting time, which may be attributed to the effect of BR and SG components on the hydration reaction of the MKPC system. It is worth noting that the solidification time mainly increases with the increase of BR content and has little relationship with the decrease of SG, indicating that BR plays a greater role in prolonging the solidification time. Jun.[38] reported that the addition of glacial acetic acid can prolong the solidification time and improve the mechanical strength of the hardened specimens by increasing the concentration of “H^+”in the pore solution. As shown in Fig. 3, the setting time of

MKPCmortar without glacial acetic acid incorporation was 14 min at a W/C mass ratio of 0.12. Other conditions being constant, the setting time increased to 29 min, 34 min and 48 min when the concentration of glacial acetic acid was significantly increased to 3%, 6% and 9%, respectively.

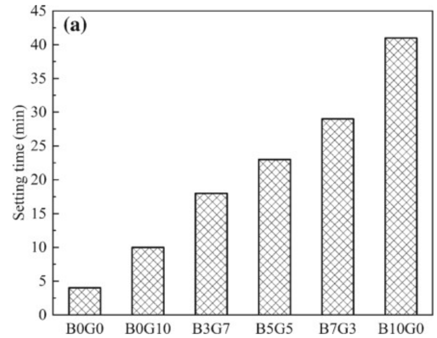


Fig. 2 Effect of different contents of BS and SG on MKPC coagulation time[37]

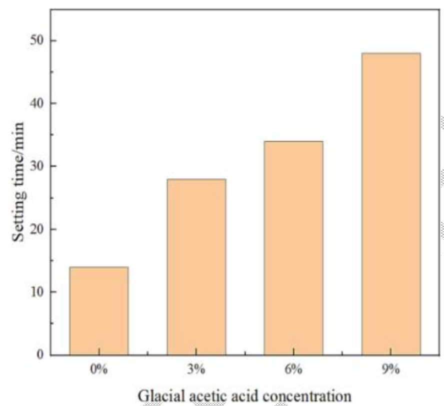


Fig. 3 Effect of glacial acetic acid concentration on solidification time[38]

Admixture is also the main factor affecting the setting time of MKPC, add active strong admixture, it will first participate in the reaction, thus reducingthedissolutionrateofMgO,affectingthesettingtimeofMKPC. Or the addition of dopants changed the pH value of magnesium phosphate cement mortar, making its original weak acid environment changed, thus reducing the rate of hydrolysis, affecting the setting time of magnesium phosphate cement.Xu[39] showedthatthesettingtimeofpotassiumphosphatecementincreasedwiththeaddition of silica fume (SF), and SF can significantly delay the setting of the MSPPC system.When the SF content in MKPC was 20%, the initial and final setting time of the mixture increased from 5.75 min and 6.5 min to 16.5 min and 36 min, respectively.This result suggests that SF may be involved in the hydration reaction of MKPC.Liu[40] found that the initial setting time of the MKPC mortar increased from 5 min to 16.5 min (control FF) when the FA content was increased from the addition of 5%, 10%, 20%, and 30% of MKPC mortar from 14 min (control group with 0 FA content) was slightly extended to 14.5, 15, 16 and 18 min The test results showed that the aluminosilicate glass phase in FA could hardly participate in the vigorous acid-base reaction of MKPC. This may be due to the low reactivity of FA and hence the addition of FA can retard the hydration process of MKPC.Haque[41] explored the setting time of specified MKPC compositions with the addition of different dopants and the observations are shown in Fig. 4. The final hardening time of the control MKPC-0 paste was about 10 minutes. Samples containing FA, SF, BX, PG, and As showed higher final hardening times compared to MKPC-0, with the SF-containing paste showing the highest final hardening time at around 17 minutes. Interestingly, the MKPC paste samples containing SNP showed the lowest final coagulation time of about 6 minutes compared to the other tested matrices.

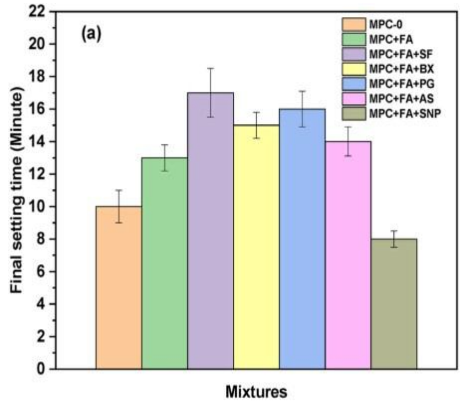


Fig. 4 Effect of different admixtures on final setting time of MKPC[41]

5. MECHANICAL PROPERTIES

The strength of magnesium phosphate cements is affected by MgO fineness,magnesium-to-phosphate ratio(M/P) substance ratio, and W/B. Reducing MgO fineness will promote the development of early compressive strength, but will not have much effect on the final strength. When the amount ratio of M/P substance is low, the unreacted phosphate is more, which iseasy to cause weathering, water erosion and other problems, which is not conducive to the maintenance of strength and durability in the later stage; but when the amount ratio of M/P substance is too high, the hydration reaction is too fast and a large amount of heat is given off, which may destroy the structure of the hydration products, and may cause the problem of insufficient amount of hydration products, etc. Le[42] found that the relationship between the M/P of magnesium phosphate cements and compressive strength is shown in Fig. 5, with the increase of M/P and W/B, the final strength is not affected. The relationship is shown in Fig.5, with the increase of M/P increases firstly and then decreases, when M/P is lower there is little effect on the compressive properties of different slurries. When M/P is equal to 5 its compressive property reaches the highest value.

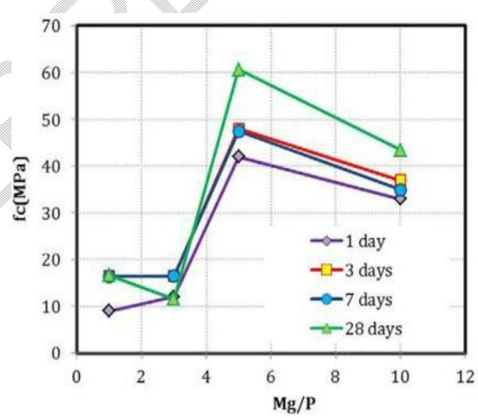


Fig. 5 Effect of M/P on compressive strength of MKPC[42]

Bilginer[43] investigated "the effect of M/P and M/B on the strength development of magnesium phosphate cement pastes, the test found that higher M/P increases the rate of early strength development, this situation produces we can also observe from the effect of M/P on setting time, the greater the M/P the shorter the setting time of magnesiumphosphatecement".Rouzic[44]foundthatby"varyingtheM/Pratio,itis possible to influence the reaction product type and hydration reaction rate, thus producing a denser microstructure and improving strength".Fan[45] found that "controlling the M/P ratio could regulate the reaction rate and the amount of hydration products of MKPC, thus affecting its mechanical properties.Yang[46] found that MgO activity and its specific surface area also had an effect on the strength of MKPC. WiththeincreaseofMgOactivityandspecificsurfacearea,theearlystrengthincreased sharply, andthestrengthremainedstableafter7dof age. Inadditiontotheamountofwater affecting the mechanical properties of MKPC, the type of water also affects the strength of MKPC". Yu[47] used seawater to prepare MKPC and found that seawater can prolong the condensation time of MKPC and reduce the compressive strength of MKPC. Ambient temperature also has a certain effect on the strength of composites. Wang[48] and Yang[46] found that the compressive strength of MKPC, especially the early strength, gradually increased with the increase of ambient temperature,andthestrengthofMKPCcouldreach17MPaafter3hofmaintenance at -20℃.

Zheng[49] prepared and calculated the relationship between the ratio of phosphatetothesumoffreewaterandwaterofcrystallisationandtheone-dimensional compressivestrengthusingthepotassiummagnesiumphosphatesystemasanexample, as shown in Fig.6. It converts different W/C values, M/P values and water of crystallisation in borax to actual W/P values. From Fig.6, it can be seen that the 1 d compressive strength of MKPC cementitious materials shows a trend of increasing and thendecreasing,buttheW/Pcorrespondingtotheinflectionpoint(maximumstrength) isslightlylowerthanthetheoreticallycalculatedvalue(W/P1/40.662).Qin[50]attributed this difference to the fact that the degree of reaction of phosphate isaffected by the nature and dosage of MgO and borax and so on to establish a magnesium phosphate cement strength model.

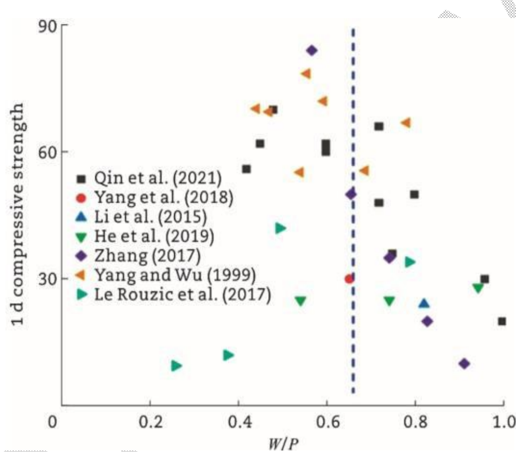


Fig. 6 1 d compressive strength of potassium magnesium phosphate cement paste[51-57]

6. VOLUME STABILITY

Ithasbeen studied[58-59]Theresultsshowthat theshrinkagedistortionof OPCis in the range of 2000-3000 μm, while the shrinkage distortion of MKPC is reduced by an order of magnitude with respect to that of OPC. Qiao[60] found that the reaction of MgO with KDP in MKPC resulted in a volume shrinkage of about 2.09%. However, the actual chemical shrinkage of MKPC was always less than 2.09% due to the presence of excess MgO and a small amount of unhydrated phosphate. In addition, the volumetric stability of MKPC is affected by various factors such as M/P ratio, retarder content, water-cement ratio, MgO activity, etc. Jiang[61] investigated the effect of different mineral admixtures on the volumetric stability of MKPCmortar.They pointed out that the kinematics of the effect of ultrafine fly ash on the volume deformation of MKPC is different from that of fly ash or silica fume. As the dosage of ultrafine fly ash in mortar increases, the volume expansion of MKPC decreases; conversely, it increases as the dosage of fly ash increases.Wu[62] found that the curing conditions have a certain effect on the volume stability of MKPC. They pointed out that MKPC cured in air is the best curing condition, but excessive moisture curing should be avoided. Microanalysis showed that the volume expansion of MKPC was related to hydration products, and the volume shrinkage was caused by drying shrinkage due to the evaporation of internal water.

7. CRACK REPAIR

Fan Yingru[63] found that the preparation of magnesium phosphate cement by mixing potassium dihydrogen phosphate and ammonium dihydrogen phosphate in a certain proportion can reduce the internal pores of the cement, reduce the shrinkage of the cement, and enhance the bonding performance of magnesium phosphate cement. Aili Yang[64] found that polymer emulsion can effectively inhibit the development of internal cracks in magnesium phosphate cement, enhance the strength of the cement, and enhance the cement bonding performance and inhibit the deformation of the cement.Qin[65] found that there exists a dense microstructure between the MKPC and the ordinary paste, and it is difficult to differentiate the transition zone. As shown in the figure 7, the transition zone at the interface between MKPC and OPC presents a highly irregular or rugged fracture surface, with cracks in the matrix area and no visible gaps or microcracks at the interface, indicating that the microstructure is well developed and the two surfaces are tightly bonded, and the magnification of the display reveals that the interface zone has a high degree of integrity and densification. The mechanism[66] : MKPC slurry interlocked with OPC matrix after hardening and formed high bond strength, which was attributed to the important role of phosphoric acid in the hydration process. On the one hand, phosphoric acid provides the slurry solution with a large amount of "H^+" , which dissolves the unhydrated cement particles and gel phase on the surface of the OPC matrix, leading to the formation of chemical bonds by surface etching. On the other hand, the solution containing a large number of ions ("H^+" ,"H_2PO^-" , "HPO^{2-} ", etc.) can easily penetrate into the OPC matrix through microcracks and pores to fill the pores and enhance the microstructure of the matrix.

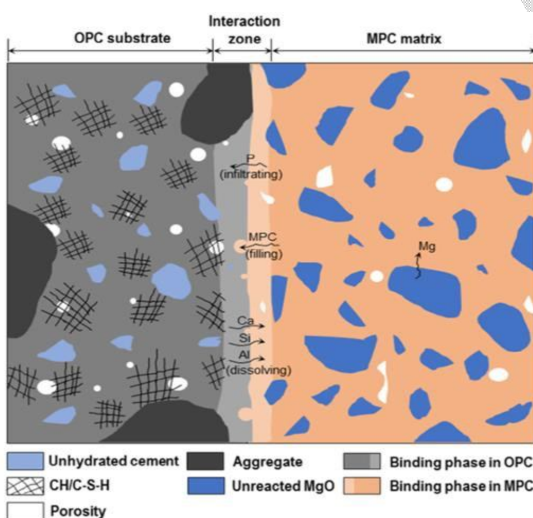


Fig. 7 Mechanism of interaction between MKPC slurry and OPC[65]

8. DURABILITY

Durability is an important factor in ensuring the safety and stability of MKPC structures. Once the durability of MKPC fails to meet the requirements, under the influence of external conditions, its mechanical properties will be significantly reduced or even completely lost. This is one of the difficulties that restrict the popularisation and application of MKPC. At present, the research on the durability of MKPC is mainly carried out from the aspects of its water resistance, abrasion resistance, air permeability, corrosion resistance, high temperature resistance and so on.

Sarkar[67] found that the strength of MKPC specimens cured naturally for 28 days decreased by 20% after being re-cured in water for 90 days. Li[68] found that the proportion of raw material composition has a great influence on the water resistance of MKPC, which can be improved by adding mineral admixtures and optimising the ratio. In addition to water resistance, Li[69] found that simultaneous immersion of MKPC in different solutions had different degrees of influence on its strength, and the degree of influence was as follows: water>NaCl solution>Na_2SO_4 solution. Yang[70-71] found that the strength of MKPC remained at 93% of the original strength after 28 days of natural curing and 360 days of immersion in seawater; the strength of MKPC remained at 93% of the original strength after 360 days of immersion in Na_2SO_4 solution; the strength of MKPC remained at 93% of the original strength after 360 days of immersion in Na_2SO_4 solution; the strength of MKPC remained at

93% of the original strength after 360 days of immersion in Na_2SO_4 solution. solution, its strength even slightly increased after 360 days of immersion in Na_2SO_4 solution. From the above results, it can be seen that the high salt resistance of MKPC may be due to its unique hydration product (guanoite) and corresponding dense microstructure with high resistance to salt solution.Li[72] also investigated the high-temperature osmotic resistance of MKPC. By heating the specimens to 130, 500 and 1000◦ C, they found that the strength of MKPC decreased significantly when the temperature exceeded 130◦ C. This was due to the fact that the rate of decrease in the strength of MKPC became smaller as the temperature increased.

CONCLUSION :

MKPC is a new type of green inorganic cementitious material. Due to its advantages of fast hardness, early strength, strong adhesion, good volume stability and other advantages and attention, in order to make MKPC can better meet the different conditions of use, the study found that by adjusting the particle size of raw materials, the ratio, mineral admixture, and other measures to improve the workability, mechanical properties, durability of MKPC. However, at present, China's research on MKPC is still in the initial stage, and there is still a big gap with developed countries, so it is urgent to strengthen the in-depth research on MKPC. Comprehensively analysing the research status at home and abroad, in order to better apply MKPC, the following issues deserve attention:

(1) Water conservancy project is a major project.To be widely used, MKPC must solve the problem of its poor water resistance. Therefore, researchers can further study how to solve the problem of insufficient water resistance of MKPC. At the same time, scientific methods can be used to evaluate the water resistance of MKPC more systematically,For example, comparing the strength changes under natural and water curing conditions, measuring the volumetric expansion rate of magnesium phosphate cement after curing in water, observing the microstructural changes of magnesium phosphate cement by using scanning electron microscope (SEM) and other techniques to analyze the morphology and distribution of hydration products as well as changes in the pore structure, and so on.

(2) The use of raw material selection, proportion and mineral admixture and other factors to enhance the mechanical properties of potassium magnesium phosphate cement, it is still difficult to fundamentally improve the service performance of magnesium phosphate cement, through the adjustment and optimisation of the hydration components of potassium magnesium phosphate cement is an effective way to enhance its service performance.For example, the addition of fly ash, slag, metakaolin, silica fume, etc. can participate in the hydration reaction of MPC, improve the fluidity and compressive strength of MPC slurry, improve abrasion resistance, and reduce costs..

(3) The high cost of raw materials of magnesium phosphate cement, especially the cost of re-fired MgO, largely affects the large-scale engineering application of magnesium phosphate cement at the present stage, and it is hoped that cheaper raw materials can be developed to replace or partially replace them in the future.

(4) At present, the application and research on the durability of MKPC is still in its infancy, and some aspects are still in a blank state. Durability is an important performance indicator to ensure that MKPC can be used stably for a long time under the influence of various environmental factors. Therefore, durability (extreme cold, heat, seawater, etc.) aspects need to be studied more systematically and comprehensively for magnesium phosphate cements under extreme conditions.

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Author(s) hereby declare that NO generative AI technologies such as Large Language Models (ChatGPT, COPILOT, etc.) and text-to-image generators have been used during the writing or editing of this manuscript.

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