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EFFECTS OF SODIUM CHLORIDE ON THE PROPERTIES OF MAGNESIUM POTASSIUM PHOSPHATE CEMENT (MKPC) USING MAGNESIA WITH DIFFERENT REACTIVITIES

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In order to control the reaction rate of magnesium potassium phosphate cement (MKPC) and to obtain a hardened material with good mechanical properties and prolonged setting time, sodium chloride was added as a set retarder. In addition to that, and for an economical construction material, this work focused on the combined effect of sodium chloride and magnesia characteristics in order to obtain the less calcined magnesia that gives with sodium chloride the desired material. The results showed that sodium chloride can reduce the reaction heat release and delay the setting time. The addition of NaCl in MPKC pastes prepared with magnesia calcined at 800°C and 900°C has a little influence on the retarding of the reaction and the enhancement of the compressive strength. However, the significant retarding effect was obtained using magnesia calcined at temperatures higher than 1000°C. The testing results demonstrate that the best correlation between higher compressive strength and an adequate setting time was obtained using magnesia calcined at 1100°C. For this sort of magnesia, the compressive strength development was investigated and the possible retarding mechanism was discussed.

Keywords: Magnesium potassium phosphates cement (MKPC)-magnesium oxide (MgO) - sodium chloride- calcination- compressive strength-setting time

1. Introduction

Chemically Bonded Phosphate Ceramics (CBPCs) are artificial stones made by acid - base reactions to form insoluble phosphate hydrates [1-3]. One of the most industrially known CBPSs is magnesia phosphate cement MPC mortar prepared by the reaction between magnesium oxide and a phosphate salt with the addition of inert filler such as sand [4, 5]. Such material was exhibited a great superiority in his properties versus to Portland cement.

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In fact, MPC can be used in the rapid repair of concrete structure because of the very quick setting, the high early strength and the good durability [6, 7]. Recently, it also proved to be good in management and stabilization of toxic and radioactive wastes because they can be chemically bond by MPC and form a stable and strong matrix that stores the hazardous wastes as insoluble phosphates [8-10]. For the preparation of MPKCs it is possible to use different phosphate salts mainly potassium and ammonium phosphate [11-13]. With ammonium salt, the reaction could release some amount of ammonium gas which is an unpleasant odor [14]. Ding and Li. suggested that ammonium dihydrogen phosphate could be replaced by potassium dihydrogen phosphate (KH_2PO_4) and proved that the main product was magnesium potassium phosphate hexahydrate $MgKPO_4.6H_2O$ which is also known as K-struvite due to its struvite isostructure [14-15]. The principal chemical reaction that gives this product can be expressed as follows:

$$MgO+KH_2PO_4+5H_2O \quad = MgKPO_4.6H_2O \qquad (1)$$

The hardening process of magnesium potassium phosphate cement MKPC is exothermic and rapid, which can be reflected by shortened the setting time and decreased the mechanical properties [16]. Therefore, to form practical cements, one must decrease the reaction rate either by pre-treatment of magnesia or by the addition of chemical agents as retarders [17, 18]. The pre-treatment of magnesia involves calcination at high temperature. It has been found that MgO, called dead burned magnesia, must be calcined at a temperature above 1300°C. In fact, the higher the calcination temperature of magnesia, the less reactivity and the lower reaction rate [19-21]. The chemical method involves the addition of a small amount of retarders. The most used and effective retarders are boric acid (H_3BO_3) and borax ($Na_2B_4O_7$, $10H_2O$) [22-24]. Unfortunately, the large quantity usage of these retarders would not lead only to excessive boron ions in the matrix but also increase the cost of the material. Therefore, a new generation setting retarder should be studied.

Previous researches demonstrated that sodium chloride can disturb the crystallization of struvite and ensure retardation of the magnesium phosphate precipitate [25,26]. Besides, Soudée and Pera [27] focused on the effect of sodium chloride on magnesium ammonium phosphate cement and considered that the retardation of acid-base reaction might be due to the sodium and chloride ions which obstruct the dissociation of the magnesia. Thereby, even the retarding effect of sodium chloride is confirmed, mechanical properties and retarding mechanism must be more cleared. The optimum NaCl dosage for assure the highest strength can vary for different sorts of magnesia and depends mainly on the reactivity of magnesia particle. In this study, the combined effect of sodium chloride and magnesia calcined at temperatures between 800°C and 1200°C was studied, in order to found suitable magnesia that, with sodium chloride addition, gives cement with appropriate setting time and higher compressive strength. It was also pursued to provide a better understanding of sodium chloride effect on the hardening mechanism of MKPC paste.

2. Methods and materials

The raw materials used in the study were magnesium oxide (MgO), potassium dihydrogen phosphate (KH_2PO_4) and sodium chloride (NaCl). The magnesium oxide produced by HiMedia Laboratories in India (purity higher than 98%) was calcined at different temperatures from 800°C to 1200°C for 2h. The potassium dihydrogen phosphate was produced by Scharlab in Spain (purity higher than 99%) and the sodium chloride was produced by SIGMA-ALDRICH (purity higher than 98%). It was added in quantities of 2, 4, 6 and 8% by weight of cement. Aggregates were not used in this research to prevent additional disruption from the impurities of aggregate.

To make sure the workability of the specimens was around the same level, the water included in the mixes was adjusted in accordance with the chemically bound water by magnesia reaction and it has been given in Table 1. The magnesium oxide to phosphate mass ratio was MgO/KDP = 1:1 and the sodium chloride were incorporated into the cement powder before mixing with water.

Table 1

Amount of water added (W/B) as a function of calcinations temperature and the magnesia reactivity

Magnesia	Calcination temperature (°C)	Reactivity index (seconds)	W/B
M_1	800°C	50	0,32
M_2	900°C	70	0,32
M_3	1000°C	180	0,30
M_4	1100°C	420	0,29
M_5	1200°C	480	0,29

Depending on the composition, the cement sample can set in seconds, which not useful for practical purposes. Therefore, a hand mixing process was tried and the samples were prepared in cylindrical plastic molds (20mm diameter× 40mm length). All specimens were demoulded after 24 hours of curing in laboratory air (25 ± 2°C). The reactivity of magnesia has been evaluated by the particle size and the neutralization method. The particle size distribution of magnesia powders was characterized with a laser particle size analyzer as marketed by Malvern Instruments.

On the other hand, the neutralization test consists in measurement of the time elapsed between the addition of citric acid and the formation of reddish color due to the phenolphthalein indicator [17, 28]. The reactivity is smaller as higher is the time.

The setting time of MKPC pastes was determined using a Vicat needle according to ASTM standard C191. Because the cement could set very fast and the initial setting is close to the final setting, only final setting was measured. The compressive strength of the hardened MKPC specimens was measured by a mechanical tester according to ASTM standard C348-02 using a LLOYD EZ50 universal testing machine. A set of 3 samples was tested for each composition at a crosshead speed of 1 mm/min. Magnesia that with sodium chloride gave a strong material with higher compressive strength and prolonged setting time was selected as magnesium oxide with the best reactivity. Therefore, specimens with various sodium chloride dosages were prepared with this magnesia and their crystalline phases after 28-day of MKPC pastes hardening were identified by X-Ray diffraction. The analyses were recorded on PANalatycal (Philips) X' Pert diffractometer spectrometer. The XRD patterns were obtained by a scanning rate of 1° per min for $2\theta = 5°$ to 80° and steps of $2\theta = 0.04°$.

Finally, in order to a better understanding of the formation mechanisms of MKPC containing NaCl, a temperature sensor was inserted into the paste to record its maximal temperature. Moreover, the pH of the reaction was studied in a dilute solution. It was recorded by a pH meter (Metrohm 827 pH lab). The mixture was composed of an equimolar mixture of KH_2PO_4 (200g/L) and magnesium oxide powder with the addition of sodium chloride in different percent.

3. Results and discussions

3.1. Influence of sodium chloride on the properties of MKPC using magnesia with different reactivities

With a fixed magnesia weight, the shorter time obtained to reach the neutral pH, signified the higher reactivity. As it can be seen in Table 1, reactivity index of the different magnesia increases with the increase in the calcination temperature from 50 seconds for M_1 to 480 seconds for M_5. Concerning M_4 and M_5, the reactivity index was increased almost four times compared to the three others. This result indicates that M_4 and M_5 present lower reactivities compared to M_1, M_2 and M_3. To justify this test results, particle size distribution of these samples was conducted. For the M_1, M_2 and M_3, the Fig. 1 shows that there is almost no change in the particle size distribution. For magnesia calcined at 1100°C and 1200°C (M_4 and M_5) the fine particles disappear, the smaller particles under 2µm melt and coarser particles above 10µm are obtained. Therefore, the diminish in reactivity of MgO is mainly appeared for magnesia calcined at 1100°C and 1200°C.

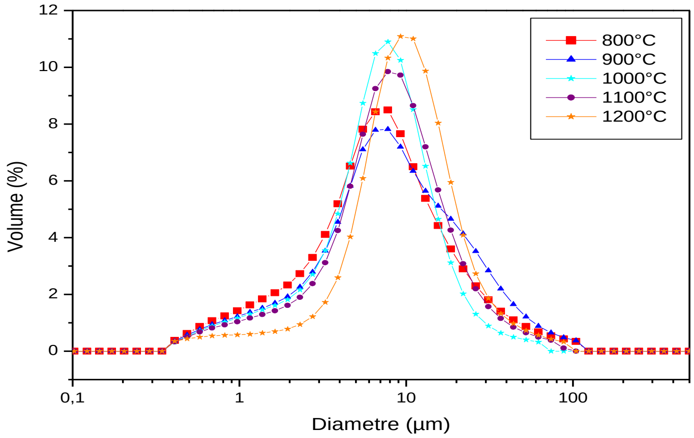

Fig. 1. Effect of calcination temperature on the particle size distribution of MgO

Fig. 2 displays setting time of MKPC pastes as a function of MgO calcination temperature and for different sodium chloride dosages.

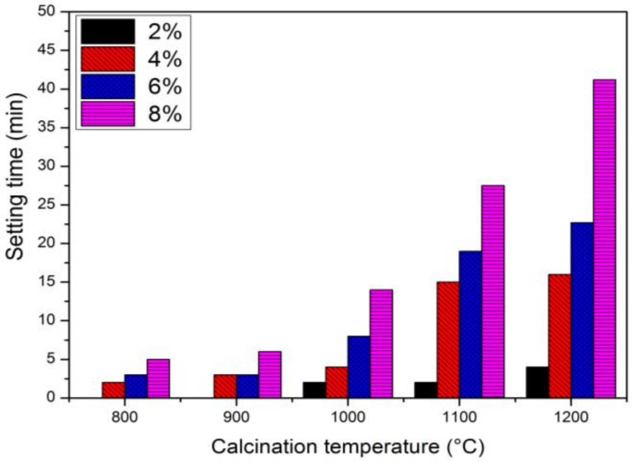

Fig. 2. Effect of calcination temperature on the setting time of MKPC with different NaCl dosages

The results show that the addition of NaCl could extend the setting time of MKPC pastes. For the samples prepared with MgO calcined at 800°C and 900°C (M_1 and M_2), the reactions were strongly exothermic and the setting time doesn't exceed 5 min. At these temperatures, samples with 2% of NaCl set in seconds, so we have obtained a precipitate instead of a hardened specimen, thus it was impossible to determine the compressive strength. The samples prepared with magnesia calcined at a temperature above 1000°C (M_3, M_4 and M_5) presented a substantially delayed setting time mainly with an addition of sodium chloride higher than 4%. With the magnesia calcined at 1200°C (M_5), the setting time of the cement with an addition of 8% sodium chloride exceeds 40 min. A similar behaviour was reported by Soudée and Perra [27], who observed an exponential increase in the setting time with dead burned magnesia which reached 85 min for 6% NaCl. In fact, as was shown in previous researches, the binding properties of the magnesia phosphate binder are affected by the calcination temperature of magnesia. This is why rapid setting time is observed for M_1 and M_2 binders due to high reactivity of the magnesia [21].

The compressive strength results of the magnesia phosphate binders with different sodium chloride dosages as function of calcinations temperature after 7 days of hardening are shown in Fig. 3.

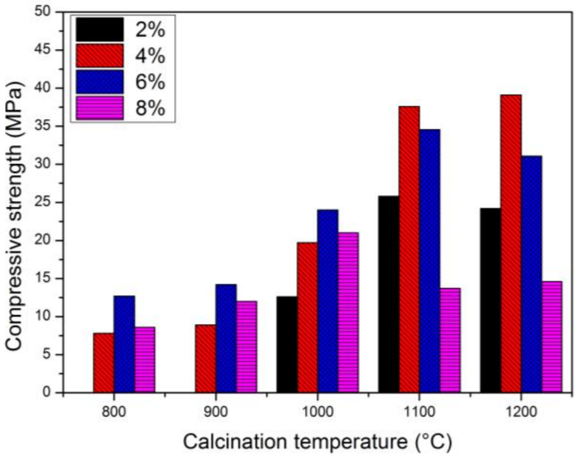

Fig. 3. Effect of calcination temperature on the compressive strength of MKPC with different NaCl dosages, after 7 days

As can be seen, the compressive strength of hardened pastes increased steadily with increase of calcination temperature. The lowest compressive strength values were obtained with M_1 and M_2 which can be attributed to the over fast reaction rate of the binders. However, cement pastes prepared with magnesia calcined at temperature higher than 1000°C developed higher compressive strength that exceeds 28MPa mainly for the addition of sodium chloride of 4 and 6%. This improvement of the strengths is mainly due to smaller reaction rate, with a better formation of MKP hydrates and a more compacted microstructure [28-30]. For the increase of calcination temperature from 1100°C to 1200°C, the addition of NaCl does not affect the strength very much. At these temperatures, the highest 7-days strength was obtained for sample with 4% NaCl. The addition of 8% NaCl results in a decrease of the compressive strength. Based on these observations, it can be seen that high NaCl addition could greatly extend the setting time of MKPC paste but it results in noticeable strength reduction. It can suppose that this drop is related to the excess of sodium and chloride ions that react with phosphate salt which occurred to the formation of new phases and reduces the main binding phase K-struvite.

Obviously, the balance between a sufficient long setting time and reasonable high strength can be achieved using magnesia calcined at 1100° and 1200°C. But, for economical reasons, the optimum calcination temperature can be chosen as 1100°C. Microstructural analysis was undertaken to verify its low reactivity. Fig. 4 shows that magnesia calcined at 800°C presents many corners and steps.

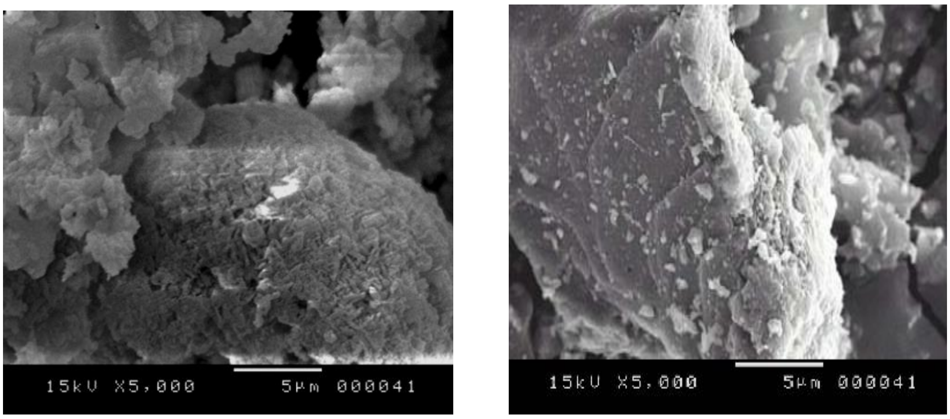

Fig. 4. Surface state of the magnesium oxide: a) calcined at 800°C; b) calcined at 1100°C

Calcination of magnesia at 1100°C reduces the disordered surface and an important reorganisation of the surface is observed [20, 29]. Therefore, magnesia calcined at 1100°C presents low reactivity compared to magnesia calcined at 800°C. Therefore, it was interesting to study the strength development after different days of curing for the binders prepared with magnesia calcined at 1100°C.

3.2. Effect of sodium chloride on the properties of MPC with magnesia calcined at 1100°C

Table 2 displays the compression strength development of the samples made with magnesia calcined at 1100°C.

Table 2

Compressive strength of MKPC pastes with magnesia M_4 calcined at 1100°C, after different hardening periods of time

%NaCl	CS 2days (MPa)	CS 7days (MPa)	CS 28days (MPa)
2	18.63	25.81	32.33
4	27.52	37.59	38.67
6	32.5	34.56	27.02
8	21.07	13.45	12.58

CS: Compressive strength

The addition of sodium chloride in amount of 2 and 4% related to the binder appears to determine the development of good compressive strengths, for all periods of curing (2-28 days). However, the addition of 6 and 8% NaCl determined the development of good compressive strengths at early ages; these cannot be sustained for long term. Especially, the strengths after 28 days are affected. For the composition with 8% NaCl this is less than 22MPa.

X-ray diffractograms of the pastes with NaCl contents ranging from 2% to 8% hardened for 28 days are presented in Fig. 5. It was found that the main binding phases are $MgKPO_4.6H_2O$ (MKP) and $MgHPO_4.3H_2O$, beside of unreacted MgO which still exist. The intensity of the main peaks on XRD patterns can be used to semi-quantitatively evaluate the content of MKP. Therefore, it can be seen that with the increase of NaCl addition from 2% to 4%, the intensity of the characteristic peaks of MKP (at $2\theta = 16.621°$, $21.100°$, $21.707°$, $27.526°$, $30.988°$, $33.399°$, $34.088°$) are strengthened and then weakened for the samples with 6 and 8% NaCl. Other phases detected, were potassium chloride (KCl) and sodium magnesium phosphate ($NaMgPO_4$). Unlike results presented by Soudée and Péra [27], sodium chloride was not detected in the cement hardened pastes. The probable explanation for the difference with other research is that the reaction mechanism or process may be different for the MKPC prepared with different phosphates.

On the other side, for the addition of 6 and 8% NaCl, $MgHPO_4,3H_2O$ (Newberyite) appears and coexists with $MgKPO_4.6H_2O$. It is probably that appearance of this new phase is related to a decrease in the crystallization of the reaction binder product MKP, which may be due to the important consumption of potassium ions consumed in KCl form.

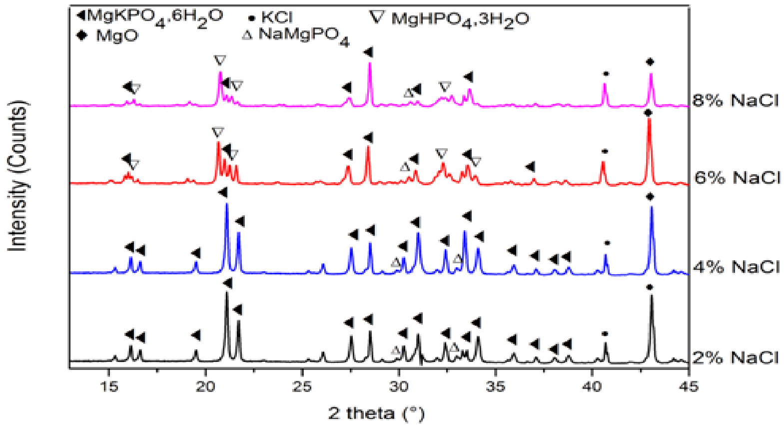

Fig. 5. X-Ray diffractograms of MKPC pastes with different dosage of NaCl.

Very approximately appreciation. It is recommended the consideration of peaks intensity by the impulses number and their graphically representation!

3.3. Retarding mechanism of sodium chloride in MKPC.

The chemical reaction between magnesia and potassium dihydrogen phosphate is essentially an acid-base reaction with strong heat release. The pH and temperature variation throughout the reaction process could provide useful information to disclose the different steps in the reaction. Temperature variation curves of MKPC pastes with different sodium chloride dosage within the first 30 min after mixing are shown in Fig. 6.

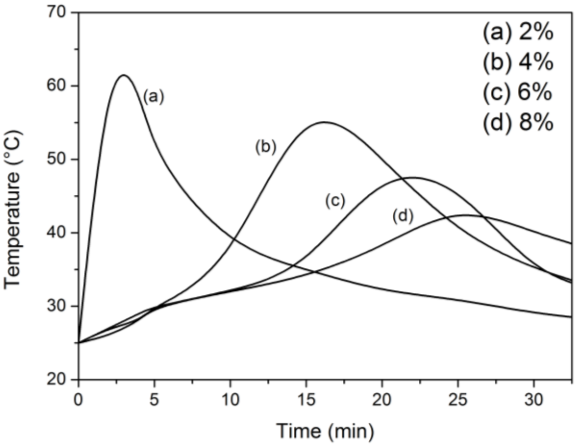

Fig. 6. Temperature rise curves of MKPC pastes with different NaCl dosages

It can be seen that the increase of NaCl dosage causes a decrease in the temperature of the mixture. Besides, as NaCl dosage is increased from 2 to 8%, the early temperature rise is slowly and the time for achieving the maximum temperature becomes longer. For example, the time for MKPC paste with 4% NaCl to achieve the maximum temperature (56.3°C) is 15 min, and time for MKPC paste with 8% to achieve the maximum temperature (42.4°C) is 25 min. In order to better understand the effect of sodium chloride on the reaction mechanism of MKPC, the reaction process was studied in a dilute system by measuring pH of the solutions. As is shown in Fig. 7, the addition of 2% NaCl determines a rapid reaction of MPK binder and the pH increases fast to around 8. But a slower evolution rate of pH is observed for the additions of 4, 6 and 8% NaCl. For the additions of 6 and 8% NaCl, the pH is kept between 6.5 and 7.

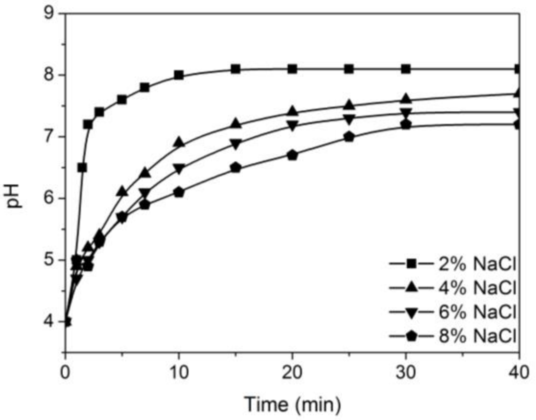

Fig. 7. Evolution of pH for MKPC pastes with different NaCl dosages

According to XRD, pH and temperature variation response, two reasons confirm the retarding effect of sodium chloride on MKPC pastes hardening. Firstly, the presence of sodium and chloride ions causes a decrease in the dissolution rate of magnesia. As proposed by SOUDEE and PERA [27], the sodium chloride changes the ionic strength of the solution, so the solubility product of the magnesia and phosphate acid was decreased. The drop of the concentrations of protons (H^+) in the solutions causes deceleration of passing into solution of the magnesia and a decrease in the pH of the solution. A pH value under 7.5 and 8, limits the formation of K-struvite, the most important phase responsible for good bonding of MKPC [31]. Secondly, it could suppose that the formation of new crystalline products, $NaMgPO_4$ and KCl leads to the retardation of the reaction process of MKPC. After the mixing of water with powders, sodium chloride and the potassium dihydrogen phosphate dissolve to form an acidic liquid containing Na^+, K^+, Cl^-, $H_2PO_4^-$, HPO_4^{2-} and PO_4^{3-}. The resultant acidic medium induces the dissociation of MgO. At first, potassium ions K^+ will react with chloride ions to form KCl. At the same time, sodium ions will form NaOH and consume OH^- ions in the solution which decreases the pH of the solution and facilities the formation of Newberyite. The decrease of OH^- and K^+ ions will markedly retard the growth of MKP. NaOH will dissolve at the end of the reaction and form $NaMgPO_4$ phase.

6. Conclusions

In this study, the effect of sodium chloride on the MKPC pastes setting time and compressive strength has been investigated. It was observed that as NaCl amount in the binder increases, there is a retarder in the final setting of the pastes. The retarding effect of sodium chloride appears significantly for the binders with magnesia calcined at temperature higher than 1000°C. For economical reasons, magnesia calcined at 1100°C having an appropriate reactivity to form with potassium phosphate and sodium chloride addition, a MKPC paste with slow setting and good mechanical strength (for 2 and 4%) was considered. The addition of NaCl in quantities of 2% and 4% appears to enhance the compressive strengths at all ages but a higher addition has a negative effect, especially at long ages. According to XRD results, pH and temperature variation, the retarding effect of sodium chloride could be explained by the change in the ionic strength of the mixture and therefore the solubility product of MgO and KH_2PO_4. When the solubility product of the reactants decreased, the rate of K-struvite precipitation also decreased and new phases (KCl and $NaMgPO_4$) are forming.

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