# Effect of heavy metals and water content on the strength of magnesium phosphate cements

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### A R T I C L E   I N F O

### A B S T R A C T

In this paper the mechanical properties of magnesium potassium phosphate cements used for the Stabilization/Solidification (S/S) of galvanic wastes were investigated. Surrogate wastes (metal nitrate dissolutions) were employed containing Cd, Cr(III), Cu, Ni, Pb or Zn at a concentration of 25 g dm$^{-3}$ and different water-to-solid (W/S) ratios (0.3, 0.4, 0.5 and 0.6 dm$^3$ kg$^{-1}$) have been employed. Cements were prepared by mixing hard burned magnesia of about 70% purity with potassium dihydrogen phosphate. Compressive strength and tensile strength of specimens were determined. In addition the volume of permeable voids was measured. It was found that when comparing pastes that the volume of permeable voids increases and mechanical strength decreases with the increase of water-to-solid ratio (W/S). Nevertheless pastes with the same material proportions containing different metals show different mechanical strength values. The hydration products were analyzed by XRD. With the increase of water content not previously reported hydration compound was detected: bobierrite.

## 1. Introduction

Magnesium phosphate cements (also called Magnesium Phosphate Ceramics included into the category of Chemically Bonded Phosphate Ceramics or CBPC) are highly crystalline materials and can thus be considered as room-temperature-setting ceramics developed for use in structural materials [1]. Furthermore, potassium magnesium phosphate cements are often used for the Stabilization/Solidification (S/S) of mixed wastes and low level nuclear wastes [2–4]. They are also employed for the rapid repair of pavements [5] together with magnesium ammonium phosphate cements [6].

The review of literature on potassium magnesium phosphate cements indicates that their main reaction product is magnesium potassium phosphate hexahydrate or k-struvite (MgKPO$_4$·6H$_2$O) [7], which is formed by an acid–base reaction between magnesia and potassium dihydrogen phosphate in the presence of water according to the following reaction [3]:

$$MgO(s) + KH_2PO_4(s) + 5H_2O \rightarrow MgKPO_4 \cdot 6H_2O(s) \qquad (1)$$
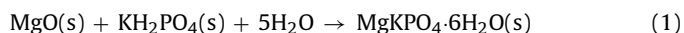
The acid–base reaction involved is highly exothermic. First, phosphate dissolves in water and an acid solution is obtained. Afterwards phosphate reacts with magnesia to form the reaction products. In order to reduce the reaction rate often hard burned magnesia is employed, because it has a lower surface area than pure magnesium oxide [8].

Related to mechanical properties of stabilized wastes compressive strength test is used in order to assure a minimum strength of the monoliths during transport and further disposal in landfills [9]. The USEPA recommends a minimum value of 0.35 MPa at 28 days for specimens immersed in water [10]. The Wastewater Technology Center of Canada has adopted a higher value of 3.45 MPa [11]. The same value was taken as a criterion by the US Nuclear Regulatory Commission for low level nuclear wastes [12]. Regarding the use of stabilized wastes as base of roads, in the United Kingdom four categories were stated according to the quality of the cement bound material (CBM): CBM1, CBM2, CBM3 and CBM4, which should have a minimum cube compressive strength at 7 days of 4.5, 7, 10 and 15 MPa respectively [13]. The cements used for construction in Europe are classified in different resistance classes according to the minimum compressive strength at 28 days: 32.5, 42.5 or 52.5 MPa [14]. Ding et al. [5] found that compressive strength of magnesium potassium phosphate cements containing 40% in weight of fly ashes was inversely proportional to the quantity of water at the specimen (for water-to-solid ratios between 0.16 and 0.21) with a maximum value of 58 MPa at 28 days. For magnesium ammonium phosphate cements Yang and Wu [15] found the same effect for specimens with water-to-solid ratio between 0.09 and 0.012, with a maximum strength value of 84.2 MPa at 28 days.

On the contrary the use of tensile strength test is not common in waste management, due to the simplicity of performing a compressive strength test for fragile materials and to the lack of standards

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Nomenclature

CBM        cement bound material
K_p        solubility product constant
USEPA      United States Environmental Protection Agency
W/S        water-to-solid ratio (dm³ kg⁻¹)
XRD        X-ray diffraction

Greek letters
σ_c        compressive strength (MPa)
σ_t        tensile strength (MPa)
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that define minimum tensile strength values for the disposal of a waste at a landfill site. Nevertheless, in view of using magnesium phosphate cements for structural applications, it is of interest to determine the tensile strength of the cements for two reasons:

(i) In structural applications the material can receive tensile or flexural loads as well as compressive ones.
(ii) Tensile strength is needed to perform life estimations using fracture mechanics concepts. This approximation is widely used for ceramic materials and allows estimating the structural behaviour of the material from the size and morphology of the preexisting defects (in this case pores) [16].

Pure uniaxial tensile test is not recommended for cements because of the difficulty in manufacturing the normalized specimens by conventional machining. It is also complicated to fix the specimen with jaws without damaging the material [17]. Therefore in this paper indirect tensile test (Brazilian test) is proposed for the measurement of tensile strength of magnesium phosphate cements. The test consists of applying a diametral compressive load to a disk-shaped specimen without fixing it to the test machine [18,19]. Although until now no indirect tensile strength values for magnesium potassium phosphate cements have been reported, magnesium calcium phosphates used in surgery and odonthology show tensile strength values at 24 h between 0.25 and 2.8 MPa (for different formulations) [20]. When performing bending tests to prismatic specimens of magnesium ammonium phosphate cement a reduction of the strength was observed as the water-to-solid ratio (W/S) increased: strength was reduced from near 11 MPa for a water-to-solid ratio of 0.06 to around 2 MPa for a water-to-solid ratio of 0.12 [21].

In this work the relationship between water content and mechanical strength (both compressive and tensile strength) was studied for magnesium potassium phosphate cements containing different heavy metals. In addition, the volume of permeable voids was determined in order to establish whether there is a direct correlation between volume of voids and strength when the cements contain heavy metals at significant concentrations, simulating stabilized hazardous wastes.

## 2. Experimental

### 2.1. Materials

Hard burned MgO used in this work was supplied by the Spanish company Magnesitas Navarras S.A. MgO is extracted from the air filters of the electric furnaces used for the calcination of magnesite at about 1100 °C during magnesia production process. Its composition and granulometry are shown in Table 1 [8]. The $KH_2PO_4$ extra pure was provided by Scharlau. In addition, heavy metal nitrate dissolutions were prepared to be used as surrogate wastes by using the following salts: $Cd(NO_3)_2 \cdot 4H_2O$, $Cr(NO_3)_3 \cdot 9H_2O$, $Cu(NO_3)_2 \cdot 3H_2O$, $Ni(NO_3)_2 \cdot 6H_2O$, $Pb(NO_3)_2$ or $Zn(NO_3)_2 \cdot 6H_2O$, and with a metal concentration of 25 g dm⁻³ in all cases.

### 2.2. Preparation of samples

The solid materials content of the samples is presented in weight with respect to the total solid content. In all cases the pastes were obtained by mixing MgO powder and $KH_2PO_4$ powder at 50% in weight. Later on the metal nitrate dissolution was added at different water-to-solid ratios (W/S): 0.3, 0.4 or 0.5 dm³ of dissolution in each kg of solid. For Cr(III) it was not possible to obtain a paste with a water-to-solid ratio of 0.3 because of the quick solidification of the mix. Therefore a paste with a water-to-solid ratio of 0.6 was prepared instead.

After the mixture slurry was stirred for 30 min it was transferred to cylindrical moulds of 30 mm diameter. The specimens were removed from the moulds after 3 weeks and left for curing for 10 months inside plastic bags at the laboratory ($25 \pm 2$ °C and $50 \pm 5$% of relative moisture).

The pastes were named by means of the metal chemical symbol (or either bl when the sample does not contain a heavy metal) followed by the water-to-solid ratio (W/S).

### 2.3. Test methods

#### 2.3.1. Compression tests

Compressive strength of all samples cured for 10 months was measured using an Instron 5585 Universal Machine. Specimens were cut to a length of 60 mm by means of a band saw. Three replicates were performed of each experiment. The speed of the plates was 2.5 mm min⁻¹.

#### 2.3.2. Brazilian tests

Tensile strength of all samples cured for 10 months was measured using an Instron 5585 Universal Machine. Specimens were cut to a length of 10 mm by means of a band saw. Three replicates were performed of each experiment. The speed of the plates was 2.5 mm min⁻¹.

#### 2.3.3. Volume of permeable voids

The volume of permeable voids was determined by immersion in water according to standard ASTM C642 [22]. The curing time of the samples was 3 weeks.

#### 2.3.4. Powder X-ray diffraction (XRD) analysis

The crystal phases in the samples were identified using a X BRUKER D5005 diffractometer with Cu Lα radiation. The curing time of the samples was 3 weeks. They were previously crushed to a size <0.5 mm.

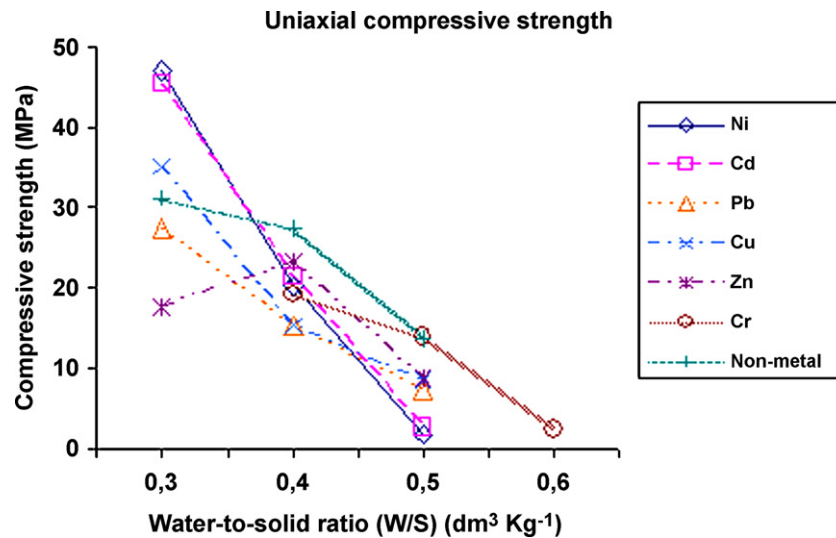**Uniaxial compressive strength**



**Fig. 1.** Effect of water-to-solid ratio (dm$^3$ kg$^{-1}$) and metal on compressive strength (MPa).

## 3. Results and discussion

### 3.1. Compressive strength

All the strength values determined are well above the 0.35 MPa limit defined by the US EPA (Fig. 1). Moreover, all the pastes with a water-to-solid ratio of 0.3 and 0.4 dm$^3$ kg$^{-1}$ exceed the limit of 3.45 MPa defined by the Wastewater Technology Centre of Canada. Pastes Ni0.3 and Cd0.3 show compressive strengths above 32.5 MPa which is the minimum strength required for construction cements in Europe. The strength of pastes with water-to-solid ratio (W/S) of 0.3 or 0.4 dm$^3$ kg$^{-1}$ is well above the requirements of 15 MPa stated in the United Kingdom for the use of materials as road base.

The results show that for pastes containing the same heavy metal, generally compressive strength is inversely proportional to the cement water content. This effect had been previously reported by other authors for magnesium potassium phosphate cements not containing heavy metals at high concentrations [5].

On the other hand it could be observed that for high water-to-solid ratios (W/S), compressive strength of samples containing metals was lower than that of the samples not containing metals.

This suggests that the addition of metals would produce defects at the crystalline matrices and weaken them. Especially, the pastes Ni0.5 and Cd0.5 have a compressive strength below 3.45 MPa. On the contrary, for low water-to-solid ratios (W/S) the presence of the metal would increase the strength of the matrices in some cases (pastes Cd0.3, Ni0.3 and Cu0.3). This could be due to the fact that if the specimen contains too little amount of water some of it will be used as coordination water of the metallic cations. Therefore it would remain less water free at the specimen, the actual water-to-solid ratio (W/S) would be lower than expected and the compressive strength would be higher.

### 3.2. Tensile strength

For a water-to-solid ratio of 0.3 dm$^3$ kg$^{-1}$ all the pastes exceed the 2.8 MPa that many formulations of magnesium calcium cement present (Fig. 2). Especially paste Pb0.3 shows a tensile strength above 5 MPa.

It can be observed that, for pastes containing the same heavy metal, in general tensile strength is proportional to the water content of the cement, as it was pointed out previously for compressive strength.
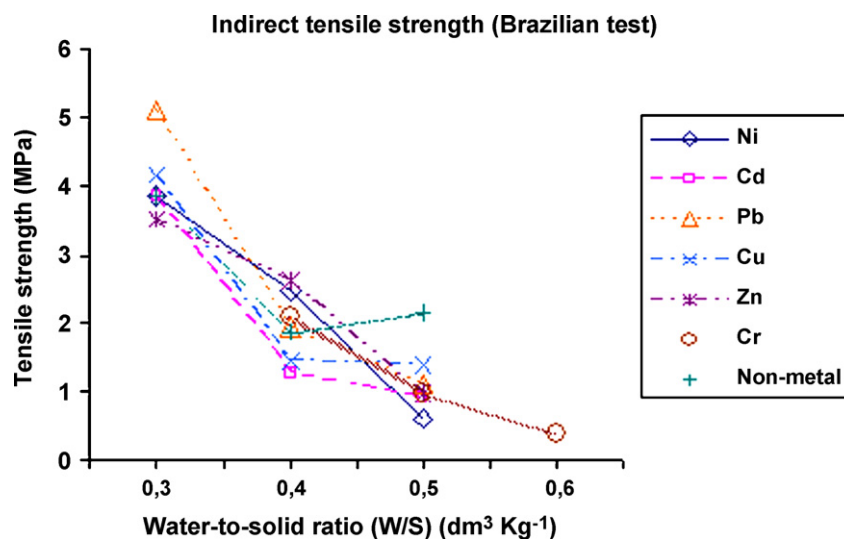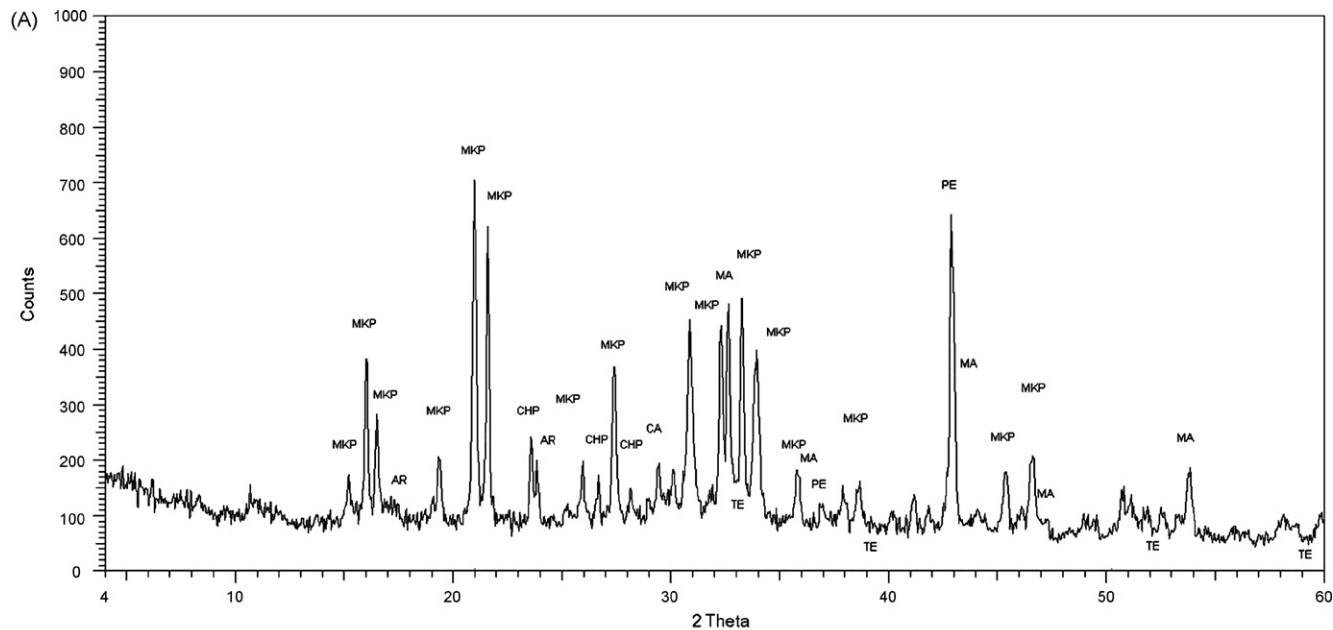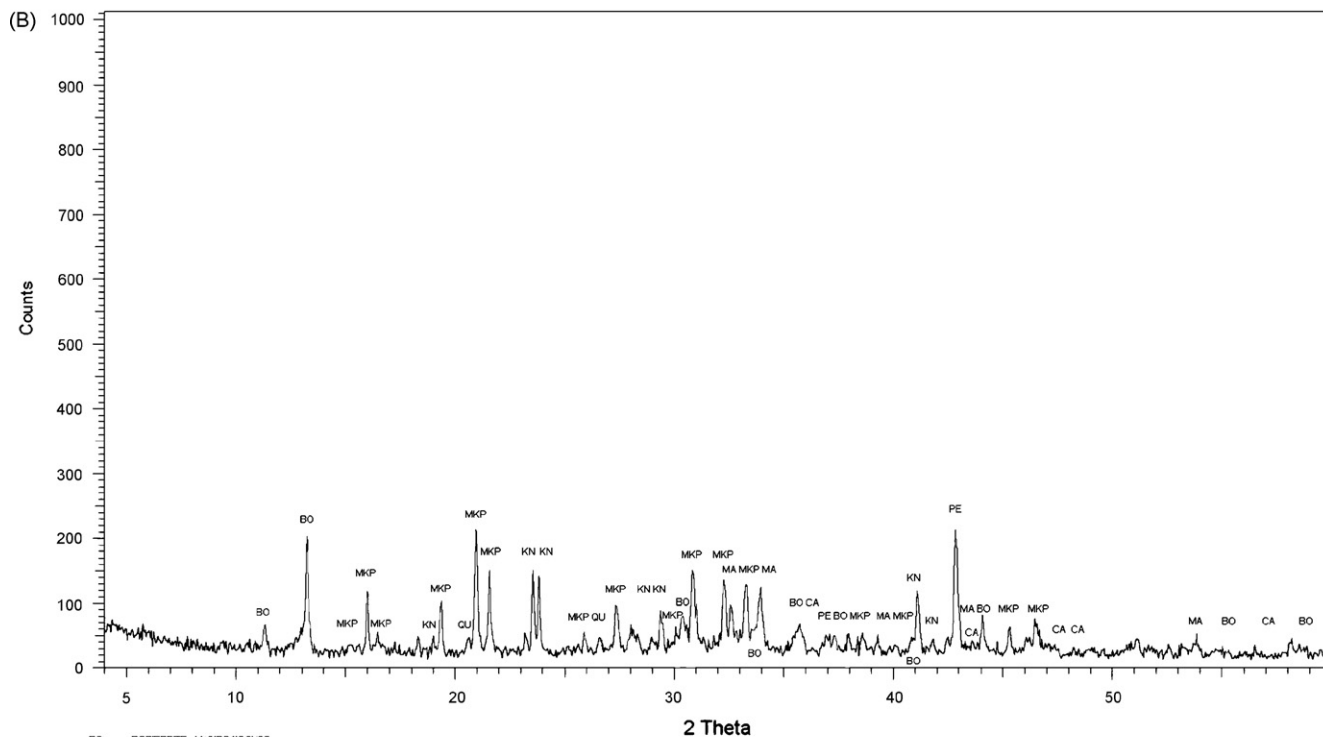
**Indirect tensile strength (Brazilian test)**



**Fig. 2.** Effect of water-to-solid ratio (dm$^3$ kg$^{-1}$) and metal on tensile strength (MPa).

(A)

MKP: MAGNESIUM POTASSIUM PHOSPHATE HYDRATE (K-STRUVITE) - MgKPO4·6H2O
PE: PERICLASE - MgO
MA: MAGNESITE - MgCO3
AR: ARCHERITE - KH2PO4
TE: TEOPHRASTITE - Ni(OH)2
CHP: CALCIUM HYDROGEN PHOSPHATE - CaH2P2O7
CA: CALCITE - CaCO3

(B)

BO: BOBIERRITE - Mg3(PO4)2·8H2O
QU: QUARTZ - SiO2
CA: CALCITE - CaCO3
PE: PERICLASE - MgO
MA: MAGNESITE - MgCO3
MKP: MAGNESIUM POTASSIUM PHOSPHATE HEXAHYDRATE - MgKPO4·6H2O
KN: POTASSIUM NITRATE - KNO3

**Fig. 3.** X-ray powder diffraction patterns of (a) paste Ni0.4 and (b) paste Ni0.5.

**Table 2**
Volume of permeable voids of pastes according to ASTM C642.

| Paste | Volume of permeable voids (%) |
|-------|-------------------------------|
| Ni0.3 | 40.0 |
| Ni0.4 | 45.0 |
| Ni0.5 | 48.3 |
| Cd0.3 | 39.9 |
| Cd0.4 | 48.2 |
| Cd0.5 | 49.7 |
| Pb0.3 | 43.9 |
| Pb0.4 | 50.9 |
| Pb0.5 | 53.8 |
| Cu0.3 | 40.4 |
| Cu0.4 | 44.3 |
| Cu0.5 | 51.1 |
| Zn0.3 | 38.9 |
| Zn0.4 | 49.1 |
| Zn0.5 | 48.5 |
| Cr0.4 | 43.3 |
| Cr0.5 | 50.4 |
| Cr0.6 | 49.3 |
| Bl0.3 | 45.7 |
| Bl0.4 | 50.3 |
| Bl0.5 | 55.5 |

In the same way that has been previously reported for compressive strength (Section 3.1) the presence of heavy metals at the cement has a negative effect on tensile strength for high water-to-solid ratios (W/S), while for low water content some pastes exceed the strength of the pastes not containing heavy metals.

### 3.3. Volume of permeable voids

For pastes containing the same metal in most cases the volume of permeable voids increases when the water-to-solid ratio (W/S) increases (Table 2), as it has been observed for magnesium potassium phosphate cements not containing heavy metals [5].

### 3.4. Powder X-ray diffraction (XRD) analysis

In a first step, samples with water-to-solid ratio (W/S) of $0.4 \, \mathrm{dm^3 \, kg^{-1}}$ containing the different heavy metals were analyzed. Phases containing heavy metal phosphate were not found, probably because their concentration was below the detection limit of the method. Nevertheless a nickel compound was observed in sample Ni0.4: nickel hydroxide ($Ni(OH)_2$) as teophrastite. Consequently, the sample Ni0.5 with higher water content was later analyzed (Fig. 3).

The results for sample Ni0.4 (Fig. 3a) show some unreacted magnesia according to the literature [7]. The main hydration product is k-struvite as expected. Some authors have studied the effect of increasing the water-to-solid ratio on the crystal phases of magnesium potassium phosphate cements for water-to-solid ratios (W/S) between 0.16 and 0.21 [5]. They reported an increase of the peak signal of k-struvite as the water content of the samples increased. On the contrary in this work for sample Ni0.5 (Fig. 3b) a new hydration product was detected: $Mg_3(PO_4)_2 \cdot 8H_2O$ or bobierrite whereas the signal of k-struvite decreased.

When a paste is synthesized from hard burned magnesia (which contains a relatively important mix of other mineral phases) with potassium dihydrogen phosphate several new products are quickly formed. The phases that will be formed will follow Ostwald's steps rule. According to this rule, among all the possible phases (k-struvite or $MgKPO_4 \cdot 6H_2O$, newberyite or $MgHPO_4 \cdot 3H_2O$, bobierrite or $Mg_3(PO_4)_2 \cdot 8H_2O$ and cattiite or $Mg_3(PO_4)_2 \cdot 22H_2O$), the most soluble ones will mainly form (in this case k-struvite ($K_{\mathrm{p_{struvite}}} = 2.4 \times 10^{-11}$)) [23]. At longer elapsed time and under favourable conditions, ageing of the samples could lead to mineral transformations towards more stable phases [24] like either bobierrite ($K_{\mathrm{p_{bobierrite}}} = 6.31 \times 10^{-26}$) [25] or cattiite ($K_{\mathrm{p_{cattiite}}} = 7.94 \times 10^{-24}$) [23]. Although newberyite is a usual compound of magnesium phosphate cements prepared with phosphoric acid and magnesium oxide [26,27] it has not been reported to appear in magnesium potassium phosphate cements probably because it only precipitates at pH below 6 [28], well below the usual pH of the leachates of the magnesium potassium phosphate cements (above 10).

It is well known the formation of cattiite by slow conversion of struvite ($MgNH_4PO_4 \cdot 6H_2O$) in the presence of water [29]. The transformation of k-struvite to cattiite was also observed by X-ray diffraction when applying dry–wet cycles to magnesium potassium phosphate cements [30]. Kanazawa et al. studied the conversion of cattiite to bobierrite at $25\,^{\circ}C$ in alkaline water (pH around 9 by addition of sodium carbonate) [31]. Other authors have observed the transformation of cattiite to bobierrite below $100\,^{\circ}C$ while above $500\,^{\circ}C$ bobierrite would lose hydration water and form anhydrous $Mg_3(PO_4)_2$ [32]. Thus, the presence of excess water enhances the transformation of k-struvite (most soluble phase) to cattiite and afterwards bobierrite (more stable phases).

The effect of bobierrite on mechanical strength needs to be investigated in the future.

### 4. Conclusions

This study showed that heavy metals have obvious effect on compressive and tensile strength of magnesium potassium phosphate cements. The main conclusions of the paper are as follows:

(1) Compressive and tensile strength of magnesium potassium phosphate cements containing heavy metals decrease with the increase of the water content of the specimen as is usual in cements without heavy metals.
(2) The presence of high concentrations of heavy metals noticeably influences the strength of the matrix regardless the volume of permeable voids. For high water content the strength of pastes containing heavy metals is lower than that of pastes without heavy metal. Thus, the presence of heavy metals would produce defects at the crystalline lattices which would weaken them.
(3) For some formulations containing a little water the presence of a heavy metal in the matrix enhances its mechanical strength, probably because some of the water is employed as coordination water of the cations leaving less free water and leading to a lower actual water-to-solid ratio.
(4) For high water-to-solid ratios a new hydration compound was observed: bobierrite, which is formed by slow conversion of k-struvite in the presence of water.

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## Glossary

*Cement bound material:* cement stabilized material used as roadbase.

*Chemically bonded phosphate ceramics:* all inorganic materials that are consolidated into a hard mass by chemical reactions and not by sintering. They include magnesium phosphate ceramics.

*Magnesium phosphate cements:* cements obtained by mixing magnesium oxide with either phosphoric acid or an acidic phosphate. They include magnesium potassium phosphate ceramics and magnesium ammonium phosphate ceramics among others. Magnesium phosphate cements are also called magnesium phosphate ceramics.

*Magnesium potassium phosphate cements:* magnesium phosphate cements obtained by mixing magnesium oxide with potassium dihydrogen phosphate in the presence of water.