# INFLUENCE OF CERTAIN COMPOSITION FACTORS ON THE MAGNESIUM PHOSPHATE MORTARS PERFORMANCES

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*Masele magneziano-fosfatice cu funcţie de utilizare în lucrări de reparaţii rapide ale unor suprafeţe ce nu pot fi scoase din uz decât pentru un timp limitat, trebuie să prezinte un timp de priză scurt, rezistenţe iniţiale bune în primele ore de întărire (minim 15MPa după o oră) şi o bună aderenţă la materialul care trebuie reparat. Lucrarea prezintă informaţii cu privire la influenţa unor factori compoziţionali precum natura sării fosfatice, proporţia întârzietorului de priză (borax), natura şi granulaţia fillerului, raportul apă/solid asupra performanţelor unor mortare magneziano-fosfatice: timp de rigidizare, rezistenţă la compresiune, aderenţă la suport din beton.*

*The magnesium phosphate masses used in the quick reparation works of certain surfaces which cannot be taken out of use but for a limited time, must have a short setting time, good initial strengths within the first hours of hardening (minimum of 15MPa after an hour) and a good adherence to the material which needs fixing. This paper presents information regarding the influence of certain composition factors such as the nature of the phosphate salt, the proportion of the setting time retarder (borax), the nature and granulation of the filler material, and the water/solid ratio on the performances of some magnesium phosphate binders: hardening time, compressive strength, adhesion to the concrete support base.*

**Keywords:** magnesium phosphate binder, setting time, compressive strength, adherence.

## 1. Introduction

Magnesium phosphate binders, which consist of sintered magnesite and the water solution of a phosphate salt, harden rapidly in air, as a result of acid base reactions, and they can be the base of certain composite type materials and used such as quick repairs to works which cannot be taken out of use except for a limited amount of time. Such materials need to display high initial compressive strengths, as well as adhesion to the concrete being repaired, a sufficient hardening time to put in work and a good strength [1-10]. The fulfilment of these

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requirements depends on a series of factors such as: the reactivity of MgO, the nature of the phosphate salt, the presence of a powder material having the role of filler, as well as of an additive with the effect of retardation of the chemical reaction, the binder/filler ratio, the water/solid ratio etc.

In this context, the objective of the experimental works was the investigation of the properties of some magnesium phosphate binding masses, with rapid setting and hardening, which condition the utilisation of these in the quick repair works of certain surfaces, that cannot be taken out of use but for a short time (in the order of a couple of hours), in correlation with intrinsic and extrinsic influencing factors (the nature of the phosphate salt, the variance characteristics of the sintered magnesite, the nature and granulation of the filler and correlated with this, the water/solid ratio, and the proportion of the setting time retarder).

## 2. Experimentals

For the achievement of the magnesium phosphate masses, the following materials and substances were considered:

- sintered magnesite (from China), characterized by a 95% MgO content, ground to a fineness, expressed as a Blaine specific surface, of 2890 $cm^2/g$, a particle content of under 32μm representing 91,3%.
- Mono ammonia phosphate, $NH_4H_2PO_4$;
- Sodium tripolyphosphate $Na_5P_3O_{10}$,
- Borax, $Na_2B_4O_7.10H_2O$;
- Quartz sand granulation 0.1-1 mm
- Ground quartz sand (Blaine specific surface of 3560 $cm^2/g$);
- Fly ash (Blaine specific surface of 2530$cm^2/g$);

Masses of magnesium phosphate binders were achieved by homogenising the solid state components through the addition of water at execution.
The characteristics of the composition of these are presented in Table 1.

The behaviour at hardening was investigated by determining the setting time with the help of the Vicat apparatus, using a modified truncated conical form, of the compressive mechanical strengths, on the microprobe (15x15x60 mm), and of adhesion to the concrete support base, with the use of the DYNA Z16 apparatus.

*Table 1*

**Characteristics of composition of the magnesium phosphate composites**

| Sample symbol | MgO/ phosphate salt | Binder/filler ratio: | | | Borax (%) | Water/solid ratio |
|---|---|---|---|---|---|---|
| | | Sand ($3560 cm^2/g$) | Sand (0.1-1mm) | Fly ash ($2530 cm^2/g$) | | |
| Phosphate salt = $NH_4H_2PO_4$ | | | | | | |
| A1 | | 1:1 | - | - | 4 | 0.17 |
| A2 | | 1:1 | - | - | 4 | 0.13 |
| A3 | 1:1 | | 1:1 | - | 4 | 0.13 |
| A4 | | | 1:1 | - | 4 | 0.10 |
| A5 | | | | 1:1 | 4 | 0.19 |
| A6 | | 1:1.5 | | | 4 | 0.16 |
| A7 | 1:1 | | 1:1.5 | - | 4 | 0.10 |
| A8 | | | | 1:1.5 | 4 | 0.24 |
| A9 | | 1:1.5 | - | - | 6 | 0.13 |
| A10 | 1:1 | - | 1:1.5 | - | 6 | 0.10 |
| A11 | | | - | 1:1.5 | 6 | 0.24 |
| Phosphate salt = $NH_4H_2PO_4+Na_5P_3O_{10}$ (9:1) | | | | | | |
| A12 | | 1:1.5 | | | 4 | 0.15 |
| A13 | 1:1 | | 1:1.5 | | 4 | 0.09 |
| A14 | | | | 1:1.5 | 4 | 0.24 |

## 2. Results

*Hardening time* of the magnesium phosphate binders is influenced by the nature of the phosphate salt, the binder/filler ratio correlated with the water/solid ratio, as illustrated in Table 2.

*Table 2*

**Hardening time of binders prepared with a phosphate oxide/salt ratio=1 and 4% borax**

| Binder symbol | Phosphate salt | Binder/filler ratio | | | Water/solid ratio | Hardening time | |
|---|---|---|---|---|---|---|---|
| | | Sand ($3560 cm^2/g$) | Sand (0.1-1 mm) | Fly ash ($2530 cm^2/g$) | | Start (min, sec) | Finish (min, sec) |
| A6 | $NH_4H_2PO_4$ | 1/1.5 | - | - | 0.16 | 2-30 | 6-30 |
| A7 | | - | 1/1.5 | - | 0.10 | 4-00 | 9-00 |
| A8 | | - | - | 1/1.5 | 0.24 | 2-15 | 6-00 |
| A12 | $NH_4H_2PO_4+Na_5P_3O_{10}$ (9:1) | 1/1.5 | - | - | 0.15 | 7-00 | 16-30 |
| A13 | | - | 1/1.5 | - | 0.09 | 7-30 | 17-00 |
| A14 | | - | - | 1/1.5 | 0.24 | 5-30 | 10-30 |

The investigated masses are characterised by a rapid setting time depending on the nature of the used phosphate salt, as well as on the nature and granulation of the filler. The association of the mono ammonia with sodium tripolyphosphate, in a proportion of 9:1 leads to a lengthening of the start time of binder hardening from approximately 2-4 minutes up to approximately 5-7 minutes, even when the water/solid ratio is diminished – as in the case of the use of sand fillers, which proves the fluidizing effect of the sodium tripolyphosphate on the composite type magnesium phosphate mixes. Likewise, the duration of the end time of the binder hardening is increased from 6-9 minutes to 9-16 minutes. The hardening speed of the binder differs, depending on the nature and granulation of the filler. Therefore, in the presence of the same type of filler, the period of workability of the binder that contains finer quartz sand is somewhat shorter than that of the coarser sand. A similar behaviour was observed in the case of the coarser siliceous filler and of Soudeé [7], compared to the finer filler. The author's view was that the coarser, siliceous filler absorbs a smaller quantity of water, leaving a lot more water available for the unfolding of the chemical reactions. The presence of the fly ash shortens the setting time, which is something that Yang and collaborators also discovered [5].

*Mechanical strengths* of the binder masses are influenced in great measure by the nature of the phosphate salt, the nature of the filler and the degree of fineness thereof, the binder/filler ratio, the water/solid ratio, the proportion of the addition of a setting time retarder.

The influence of the nature of the phosphate salt is illustrated in Fig. 1 for masses containing quartz sand of a 0.1-1 mm granulation. The association of the sodium tripolyphosphate with the mono ammonia phosphate has a positive influence on the development of the strengths, especially on short term, leading to values of approximately 17 MPa after an hour of hardening, and 50 MPa after 48 hours respectively.

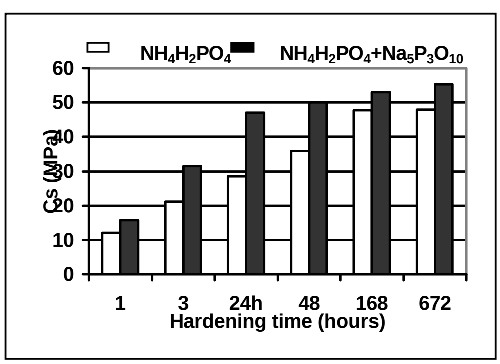
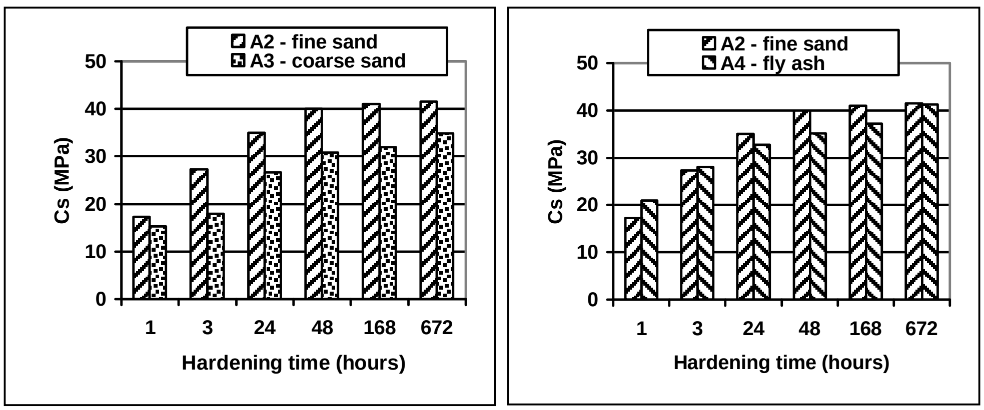



Fig. 1 Compressive strength of A7 and A13 in presence of different phosphate salts masses vs. time

The influence of the nature and grinding fineness of the material used as filler is brought into evidence in Fig. 2. It is realised that if the ratios of the binder/filler (1/1) and water/solid (0.13) are kept constant, the mass containing the fine sand develops better mechanical strengths compared to the mass containing sand of a 0.1-1 mm granulation. The use of fly ash as a filler in magnesium phosphate composites imposes a rise in the water/solid ratio to 0.19 in order to ensure a workability that permits operability. In this case an important rise in compressive strength is detected within the first hours of hardening compared to the mass that contains sand, a fact which correlates with the hardening speed thereof.




a) b)

Fig.2 Compressive strengths vs. time depending on:
a) filler fineness (a/s=0.13); b) nature of filler

The influence of the water/solid ratio on the development of mechanical strengths of the magnesium phosphate binders is brought to evidence in Fig. 3 for masses containing fine quartz sand and having a binder/filler ratio of 1. It is determined that the mass having a water/solid=0.17 develops inferior compressive strengths within the first hours of hardening compared to the mass with a water/solid=0.13. A similar behaviour is explained by the better compaction of the mass that has a smaller water/solid ratio, which at the same time ensures a quicker hardening of the mass so that increases in strength after 2 days are insignificant.

The influence of the binder/filler ratio is illustrated in Fig. 4 in the case of quartz sand of a 0.1-1 mm granulation at the preparation of magnesium phosphate binders which contain mono ammonia phosphate, characterised by a water/solid ratio= 0.10 and 4% borax.

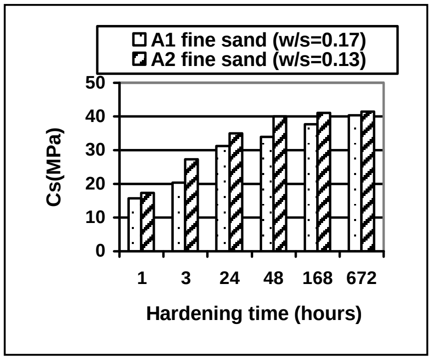



Fig.3 The influence of the water/solid ratio on the compressive strength of masses with a binder/filler ratio = 1/1

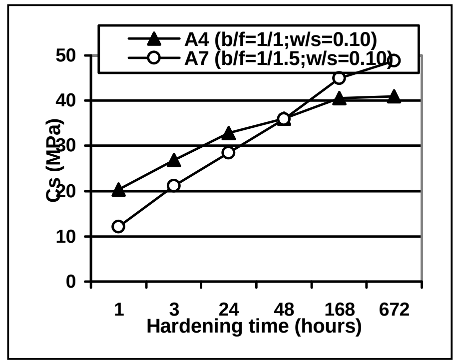



Fig.4 The influence of the binder/filler ratio on the evolution of the compressive strength of magnesium phosphate composites with a quartz sand of 0.1-1 mm granulation.

The experimental data evidence a rapid hardening reflected in the important rise in compressive strength with a rise in the binder/filler ratio. Even though the best initial strengths (up to 24 hours) develops their composition with a binder/filler ratio = 1, these are nevertheless not very different from those of a composition with more filler, which constitutes an advantage from an economical point of view.

It is noteworthy that the mass with the greater filler content presents significant rises in compressive strength on a longer term basis (7-28 days); this is probably due to a slower hardening rate.

For the study of the influence of the borax proportion on the speed of the reaction in the $MgO-NH_4H_2PO_4-H_2O$ filler system, borax was added to some of the masses in a proportion of 4-6% in view of the binder. In Fig. 5, the evolution of compression for magnesium phosphate masses with 4-6% borax containing quartz sand with a 0.1-1 mm granulation, is comparatively illustrated.

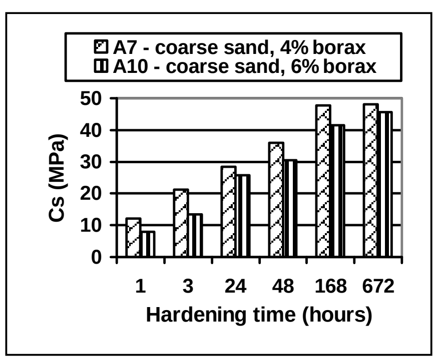



Fig. 5 The influence of borax on the compressive strengths of the magnesium phosphate binders prepared with sand of a 0.1 – 1mm granulation

*The adhesion of the magnesium phosphate binders to the concrete support* was achieved selectively with masses containing only mono ammonia phosphate as a source of phosphate, or associated with sodium tripolyphosphate. The data regarding the adhesion of magnesium phosphate mortars to the concrete support, expressed as a bonding strength, are presented in Table 3.

*Table 3*

**Adhesion of composite magnesium phosphate masses to the concrete support, expressed as bonding strength**

| Binder symbol | Bonding strength * ($N/mm^2$) | | Way in which failure takes place |
|---|---|---|---|
| | 1 day | 7 days | |
| A6 | 0.22 | 1.68 | Failure at the composite support interface |
| A7 | 4.80 | > 8 | Failure from the support |
| A8 | 0.16 | 2.90 | Failure at the composite support interface |
| A12 | 1.4 | 5.6 | Failure at the composite support interface |
| A13 | 7.8 | > 8 | No failure took place |
| A14 | 1.1 | 6.7 | Failure at the composite support interface |

*Bonding strength is calculated by the following formula: $R_s = \frac{F}{A}$,

where F is the bonding force applied to the mortar-concrete support, as a whole, in N, and A is the surface on which the bonding force is applied and is measured in, $mm^2$

Bonding strength of the mortar-concrete as a whole is different depending on the nature and granulation of the filler. The best behaviour from this point of view, is recorded in the case of the mortar prepared with quartz sand that is coarser (fraction 0.1-1 mm); the value of the bonding strength after a day is of 4.8 $N/mm^2$ and after 7 days, over 8 $N/mm^2$ with evulsion taking place in the concrete support. In the case of finer sand (3560 $cm^2/g$), the bonding strength values are much smaller (0.22 $N/mm^2$ – after a day, with 1.68 $N/mm^2$ – after 7 days respectively), and evulsion takes place at the interface of the magnesium phosphate mortar/concrete. This greatly different behaviour under the bonding strength condition is probably due to the fact that in the case of the coarser filler, the older concrete, not being moistened in advance, caused an adsorption on its surface, of a part of the water utilised in the preparation of the binder. This happened in the conditions in which an inert, finer, filler adsorbs a greater quantity of water than a coarser one and a smaller quantity thereof remains for the $MgO-NH_4H_2PO_4$ reaction which is probably insufficient for a complete reaction. Another possible argument for the very good behaviour of a mass with coarser sand had to do with the roughness of the filler particles, with the coarser sand ensuring a better contact between support and binder. The combined use of mono ammonia phosphate with sodium tripolyphosphate has a beneficial influence on the adhesion of the magnesium phosphate binders to the concrete support.

## 3. Conclusions

Magnesium phosphate binding masses, with adequate usage for the purpose of quick repairs by means of controlling compositional factors, can be obtained.

Mechanical strengths and duration of putting into operation of magnesium phosphate binders are favoured by the association of mono ammonia phosphate with sodium tripolyphosphate, as is the favouring of adhesion of these to the concrete support. Based on the experiments carried out, we consider that magnesium phosphate masses with quartz sand of a 0.1-1 mm granulation presents the best performance from a setting, compressive strength and adhesion point of view.

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