

# EFECTUL PLUMBULUI ȘI NICHELUI ASUPRA PROCESELOR DE ÎNTĂRIRE ȘI A PROPRIETĂȚILOR UNOR CIMENTURI FOSFATICE

## EFFECT OF LEAD AND NICKEL ON THE HARDENING PROCESSES AND PROPERTIES OF PHOSPHATE CEMENTS

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*This paper presents the influence of oxide component (magnesia or calcined dolomite) on the setting time and compressive strengths of phosphate cements in which the phosphate precursor was sodium dihydrogen phosphate. These cements were used for the immobilization of two simulated wastes with Ni or Pb content. The presence of Ni or Pb in these cements determines a decrease of the early compressive strengths (after 1 day of hardening). The leaching test (SR EN 12457-4) performed on the cements with waste content hardened for 28 days shows the immobilization of these two heavy-metals in both types of phosphate cements (based on magnesia or calcined dolomite); nevertheless, for the phosphate cements based on calcined dolomite (with/without Ni or Pb waste) an important strength loss is recorded at longer periods of time (28 days, 1 year) most probably due to the delayed CaO hydration with volume expansion. X ray diffraction was used to identify the crystalline compounds formed in hydrated cements and Scanning Electron Microscopy (SEM) coupled with EDS (Energy Dispersive Spectroscopy) to assess the microstructure and elemental composition of identified phases.*

*Lucrarea prezintă influența exercitată de natura componentului oxidic (magnezită calcinată sau dolomită calcinată) asupra timpului de priză și a rezistențelor la compresiune ale cimenturilor fosfatice în care s-a folosit ca precursor fosfatic  $\text{NaH}_2\text{PO}_4$ . Aceste cimenturi au fost folosite pentru imobilizarea a două compoziții simulate de deșeuri cu conținut de Ni sau Pb. Prezența Ni sau Pb în aceste sisteme liante determină o scădere a rezistenței la compresiune la perioade scurte de întărire (o zi). Testul de levigare (SR EN 12457-4), realizat pe cimenturi cu conținut de deșeuri întărite timp de 28 zile, a arătat că se realizează o imobilizare a acestor metale grele în cele două tipuri de cimenturi fosfatice studiate; cu toate acestea, în cazul cimentului fosfatic pe bază de dolomită calcinată (cu/fără deșeu cu Ni sau Pb) se constată o scădere importantă a rezistenței la compresiune la perioade mai lungi de întărire (28 zile, 1 an) datorată foarte probabil hidratării întârziate a CaO cu creștere de volum. Analiza de difracție de raze X s-a utilizat pentru a identifica compoziții cristaline formate în cimenturile hidratate și microscopia electronică de baleiaj cuplată cu spectroscopia cu raze X pentru a evalua microstructura și a realiza o analiză elementală a fazelor identificate.*

**Keywords:** magnesia, calcined dolomite, sodium phosphate, nickel, lead, waste

### 1. Introduction

Phosphate cement also known as chemically bonded ceramics are formed by an acid-based reaction between an oxide and phosphoric acid or phosphate salts [1-5]. Magnesium phosphate cements (MPCs) were extensively studied in the past two decades, due to several superior properties as compared to ordinary portland cement (OPC), such as rapid hardening, high early strength, long term durability, low drying shrinkage and high deicer scaling resistance [6-11]. Therefore, MPCs can be used for the rapid repair of deteriorate concrete highways, bridge decks, airport runways, as a grout for the enhancement of tunnel structure or for the solidification/stabilization of various hazardous waste streams [1,9, 12-15].

The main precursor of MPC is MgO which is obtained by the calcination at high temperatures (above 1400°C) of magnesite ( $\text{MgCO}_3$ ) which can become in next future a critical raw material [16]. Calcined dolomite obtained by the thermal treatment of dolomite (sedimentary rock that contains calcium

and magnesium carbonates) can be used in the manufacture of phosphate cements [17]. The most frequently used phosphate salts for the manufacture of magnesium phosphate cements are ammonium and potassium dihydrogen phosphates. The reaction products, resulted in the interaction of magnesia with these two phosphates, are crystalline struvite ( $\text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O}$ ) or K-struvite ( $\text{KMgPO}_4 \cdot 6\text{H}_2\text{O}$ ) and a variable amount of glassy (amorphous) phase [1,6,17,18]. When sodium dihydrogen phosphate is used as phosphate precursors in MPC, the resulting cement consists mainly of an amorphous (glassy) phase [1,18]. Phosphate cements based on mixtures of magnesia (with/without fly ash) and high dosage of sodium dihydrogen phosphate were studied by Colorado and Singh [19]. They reported the formation as a main crystalline phase of  $\text{Mg}_3(\text{PO}_4)_2$ , along with sodium-magnesium phosphate ( $\text{NaMgPO}_4$ ), trisodium phosphate ( $\text{Na}_3\text{PO}_4$ ), sodium phosphate hydrate ( $\text{Na}_3\text{PO}_4 \cdot \text{H}_2\text{O}$ ) and sodium hydroxide ( $\text{NaOH}$ ).

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According to Chun et al. [5], the use of disodium hydrogen phosphate as precursor in magnesia phosphate cements (MPC) reduces the heat released in the initial reaction period and increases the fluidity and setting time of MPC.

Phosphate reactions can be used for the solidification/stabilization of heavy-metal contaminants - the reaction between the heavy metal compounds and the acid solution, followed by physical encapsulation within the newly formed, dense and durable phosphate matrix, can effectively stabilize these contaminants [1]. It was also reported that highly soluble nitrates of heavy metals can be converted to insoluble phosphates therefore reducing their leaching and forming an adequate final waste form [14, 15, 20].

This paper studies the possibility to obtain a new category of sodium phosphate cements based calcined dolomite and sodium dihydrogen phosphate and to assess their ability to effectively immobilize lead or nickel present in two simulated wastes. The influence of oxide component (magnesia or calcined dolomite) along with the influence of Ni and Pb on the setting time and compressive strengths were also assessed.

## 2. Materials and method

### 2.1. Materials

The precursors used in this study were:

- Magnesia (M) obtained by the calcination of magnesite at 1500°C (industrial product);
- Calcined dolomite (D) obtained by the thermal treatment of dolomite at 1400°C for 3 h; according to the information obtained from the supplier, dolomite has a content of 47% CaCO<sub>3</sub> and 37.5% MgCO<sub>3</sub>;
- sodium dihydrogen phosphate (NaH<sub>2</sub>PO<sub>4</sub>·2H<sub>2</sub>O – NP), chemical reagent;
- simulated waste with nickel or lead content of 2.71 wt.%; these wastes were prepared by grinding sand with nickel sulphate (NiSO<sub>4</sub>·6H<sub>2</sub>O) or lead sulphate (PbSO<sub>4</sub>). The sand had a fineness corresponding to 95% passing through the 0.5 mm sieve. The main crystalline compounds, assessed by XRD (Fig. 1), are SiO<sub>2</sub>, NiSO<sub>4</sub>·6H<sub>2</sub>O and PbSO<sub>4</sub> respectively.

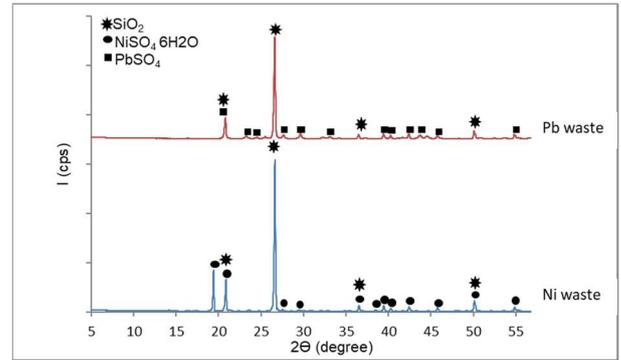


Fig. 1 - XRD patterns of nickel waste and lead waste / Analize DRX pentru deșeurile de nichel și de plumb.

The compositions of the studied phosphate cements are presented in Table 1. For all compositions water to solid ratio was 0.2.

### 2.2. Methods

In order to assess the efficiency of this type of phosphate cements to immobilize the nickel and lead wastes, a leaching test was performed on the phosphate cements hardened in air for 28 days, according to the method presented in the standard SR EN 12457-4 [21]. The hardened specimens were crushed until the particles become smaller than 10 mm and then mixed with water (water to solid = 10). The suspensions were stirred (using an orbital shaker) at a rate of 10 rotations/minute for 24 hours; the suspensions were filtered and nitric acid was added to leachate to achieve a pH<2. The concentration of heavy metals in leachate was assessed with an atomic absorption spectrometer, using graphite technique with transverse heated graphite furnace, with single and double beam mode and with automatic 6-lamp turret.

The setting time of phosphate pastes was determined with Vicat apparatus using a conical mould with the following dimensions: height 30 mm, top opening 30 mm and bottom opening 39 mm. Sodium dihydrogen phosphate was mixed water and the solid components (magnesia/calcined dolomite with/without Ni or Pb waste) were dry mixed. The initial setting time (Ti) of paste represents the time elapsed from the moment when the mixture of solid components was mixed with the mixture of sodium dihydrogen phosphate and water, until the Vicat needle stops at 3 mm above

Table 1

Compositions of phosphate cements based on magnesia (M) and calcined dolomite (D) and sodium phosphate (NP) with / without Ni or Pb waste/ Compozițiile cimenturilor fosfatice pe bază de magnezită calcinată (M) și dolomită calcinată (D) și fosfat de sodiu (NP) cu / fără deșeu de Ni sau Pb

Sample code	MgO (M) wt. %	MgO.CaO (D) wt. %	NaH <sub>2</sub> PO <sub>4</sub> ·2H <sub>2</sub> O (NP) wt. %	Ni waste* wt. %	Pb waste* wt. %
MNP	49	-	51	-	-
DNP	-	49	51	-	-
MNP_Ni	37	-	51	12	-
DNP_Ni	-	37	51	12	-
MNP_Pb	37	-	51	-	12
DNP_Pb	-	37	51	-	12

\*) The Ni or Pb wastes were used to partially substitute magnesia or calcined dolomite

Table 2

Heavy metals content assessed in leachates of phosphate cements with Ni or Pb wastes, after 28 days of curing / Conținutul de metale grele în levigatele cimenturilor fosfatice cu conținut de nichel și plumb, întărite timp de 28 zile

Heavy metal	Max. regulated concentration in leachate Order 95/2005 (mg/kg)	Concentration (mg/kg)			
		MNP_Ni	DNP_Ni	MNP_Pb	DNP_Pb
Ni	40	16.2	25.62	1.13	0.37
Pb	50	0.58	0.064	3.93	5.02

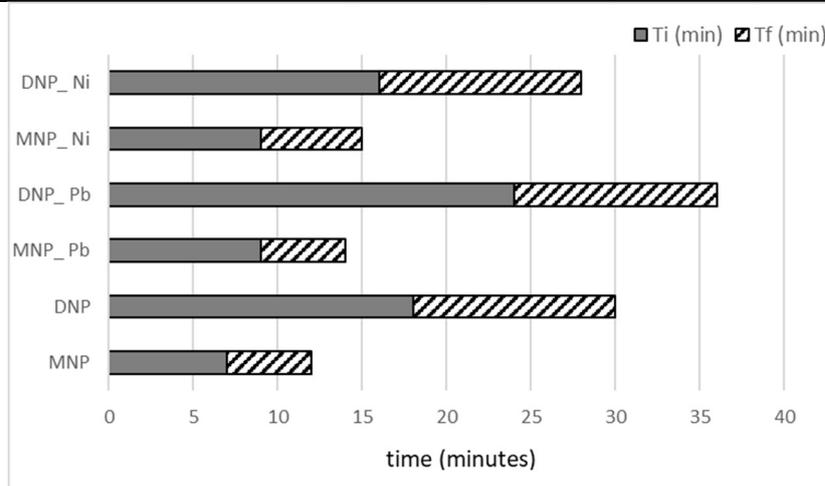


Fig. 2 - The initial setting time (Ti) and final setting time (Tf) of phosphate cements based on magnesia or calcined dolomite with / without Ni or Pb wastes / Timpul de început de priză (Ti) și de sfârșit de priză (Tf) al cimenturilor fosfatice pe bază de magnezită calcinată sau dolomită calcinată cu / fără conținut de deșeuri cu Ni sau Pb

the bottom of the mould; the final setting time (Tf) represents the time elapsed from the moment when the mixture of solid components was mixed with the sodium dihydrogen phosphate and water until the Vicat needle forms only a fine mark on the paste surface.

The compressive strengths were assessed on cement specimens - cuboids (15x15x60 mm) - hardened in air at 20±2°C for 1, 28 and 360 days. Minimum four specimens cured in similar conditions were used to assess the compressive strengths on a Tonitech testing machine. The loading was performed with a rate of 5 mm/min.

X ray diffraction (XRD) analyses were carried on a Shimadzu diffractometer XRD 6000-Ni-filtered CuK $\alpha$  ( $\lambda=1.5406\text{\AA}$ ) radiation, scanning speed of 2°/min in 2 $\theta$  range of 5-60°.

The microstructure of the studied materials was assessed using a Quanta Inspect F scanning electron microscope (1.2 nm resolution) coupled with EDS. The specimens used for SEM analyses were coated with gold.

### 3. Results and discussions

In order to evaluate the possibility to use these type of phosphate cements for the immobilization of wastes with high nickel or lead content, the leaching test presented in the norm SR EN 12457-4 [21] was performed for phosphate cement pastes with waste content, hardened for 28 days. The results are presented in Table 2.

As it can be seen from Table 2, when Ni and Pb wastes are immobilized in the phosphate cements based on magnesia or calcined dolomite,

the concentration of Ni or Pb in leachates are below the maximum regulated values [22,23]. The sodium phosphate cements based on magnesia seems to be more effective in the immobilisation of both Ni and Pb as compared to the compositions based on calcined dolomite.

The influence of Ni and Pb on the setting time values of the studied phosphate pastes are presented in Figure 2.

The setting time of compositions based on magnesia is shorter as compared with the setting time of composition based on calcined dolomite (Fig.2). The partial replacement of magnesia with the simulated Pb or Ni wastes determines a short delay of both initial and final setting times. In terms of practical application, a longer initial setting time is recommended [24].

The compressive strength of sodium phosphate cements based on magnesia or calcined dolomite with/ without Ni or Pb wastes are presented in Figure 3. After one day of hardening, the composition based on calcined dolomite (DNP) has a higher mechanical strengths as compared with the composition based on magnesia (MNP); this can be due to different reaction products formed in these two systems, as well as to the rapid setting and hardening of MNP as compared with DNP, which could generate internal stress.

As expected, the substitution of magnesia or calcined dolomite with Ni or Pb wastes reduces the compressive strengths assessed after one day of hardening. This could be due to the decrease of the active component (magnesia or calcined dolomite – Table 1) which reacts with sodium

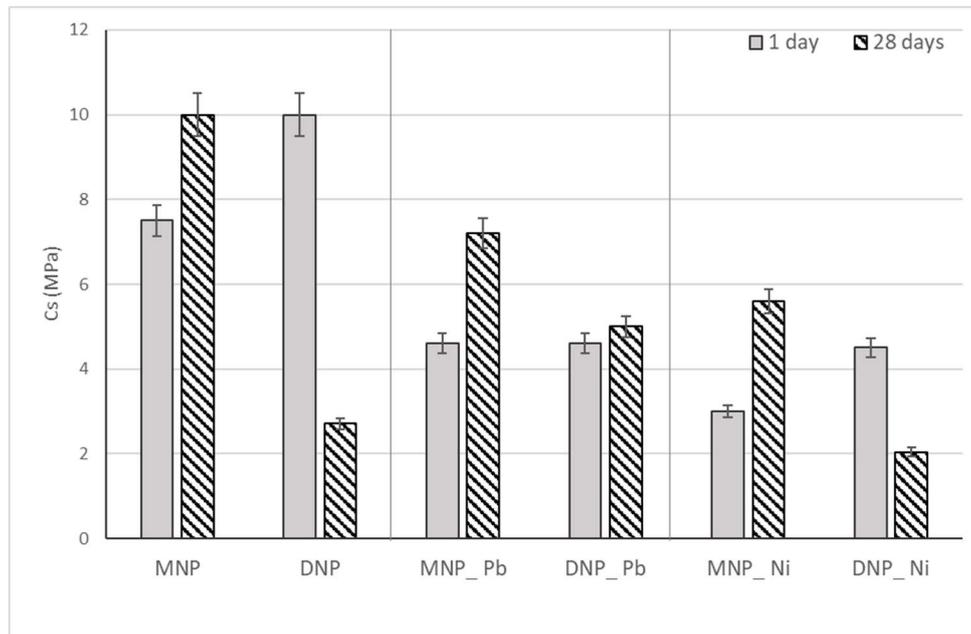


Fig. 3 - The compressive strength of phosphate cements based on magnesia or calcined dolomite with/without Ni or Pb wastes / *Rezistențele la compresiune ale cimenturilor fosfatice pe bază de magnezită calcinată sau dolomită calcinată cu/fără conținut de deșeuri cu Ni sau Pb.*

phosphate and forms the binding matrix.

The compressive strength of phosphate cements based on magnesia (with/without wastes content) increases from 1 to 28 days, opposite to the compressive strengths of the compositions based on calcined dolomite; in this case, for DNP and DNP\_Ni, an important decrease of compressive strengths at 28 days is recorded. Due to this phenomenon we decided to assess the mechanical properties at longer curing times for the phosphate cements based on calcined dolomite i.e. after 360 days of hardening; after this long period of curing, at the surface of all specimens (with/without wastes), deep cracks were visually assessed and the values of compressive strengths are below the detection limit of the testing machine.

In order to better understand the processes which determines this evolution of compressive strengths, X-ray diffraction analyses were performed on phosphate cements hardened for different periods of time (Figures 4 and 5). On the XRD patterns of phosphate cements based on magnesia hardened for 3 days (Fig.4a) one can assess the presence of MgO and possibly of NaMgPO<sub>4</sub>·7H<sub>2</sub>O [14]. According Wagh [1], the main reaction product resulting from the interaction of MgO with NaH<sub>2</sub>PO<sub>4</sub> is a Na-glassy phase; one can notice a broad halo between 2θ=25-35° which could be associate to a phase with a low crystallinity degree. The XRD patterns of the magnesia-based phosphate cements with simulated wastes with Ni and Pb content, do not permit the assessment of compounds such as nickel phosphate, lead phosphate or pyromorphite, which could explain the good immobilization of these heavy-metals in this

type of cement [14,15]. This could be due to the low intensities of XRD peaks associated with these compounds which are shielded by the XRD peaks with high intensities associated to crystalline compounds such as quartz and magnesia.

The increase of hardening period up to 28 days (Fig.4b) do not essentially modify the nature of crystalline phases assessed in these systems. For this period of time, on the XRD patterns one can assess peaks with low intensities which can be attribute to Na<sub>2</sub>HPO<sub>4</sub>·2H<sub>2</sub>O; this suggests the conversion of phosphate ions from H<sub>2</sub>PO<sub>4</sub><sup>-</sup> to HPO<sub>4</sub><sup>2-</sup> and PO<sub>4</sub><sup>3-</sup> (depending on the pH) [18], and their incomplete consumption in the formation of NaMgPO<sub>4</sub> and sodium glassy phase. For longer curing periods one can presume an acidic attack or conversion of Na-glassy phase with Na<sup>+</sup> release and Na<sub>2</sub>HPO<sub>4</sub> formation and crystallisation.

The XRD patterns of phosphate cements based on calcined dolomite with/without heavy metals wastes are presented in Fig. 5. For a short hardening period (3 days – Fig. 5a) one can assess the presence of MgO and CaO (from unreacted calcined dolomite) as well as XRD peaks with low intensities specific for Ca(OH)<sub>2</sub>. Some of the peaks can be also associated with the presence of sodium magnesium phosphate and sodium calcium magnesium phosphate. At the surface of specimens hardened for 360 days (insert in Fig. 5c) one can assess the presence of deep cracks; based on the increase of the intensities of Ca(OH)<sub>2</sub> peaks associated with the reduction of the intensities of CaO peaks, one can explain the important strength loss assessed at longer hardening periods (28 and 360 days) to a delayed hydration of CaO with a corresponding volume expansion.

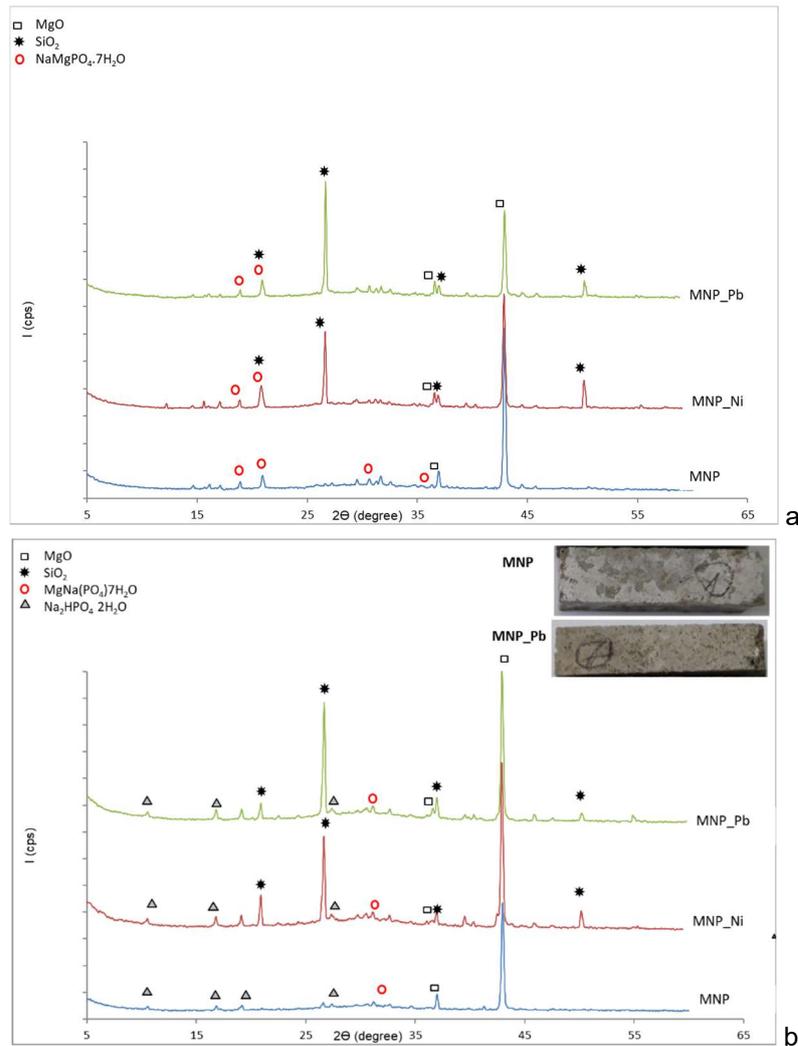


Fig. 4 - XRD patterns of phosphate cements based on magnesia with / without Ni or Pb wastes, hardened for: a) 3 days, b) 28 days / Analize DRX ale cimenturilor fosfatice pe bază de magnezită calcinată cu / fără conținut de deșeuri de Ni sau Pb, întărite timp de: a) 3 zile; b) 28 zile

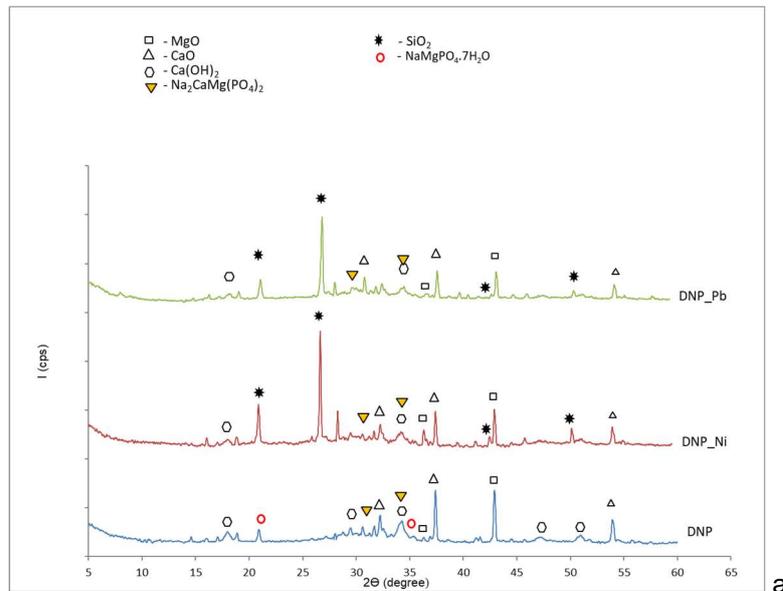


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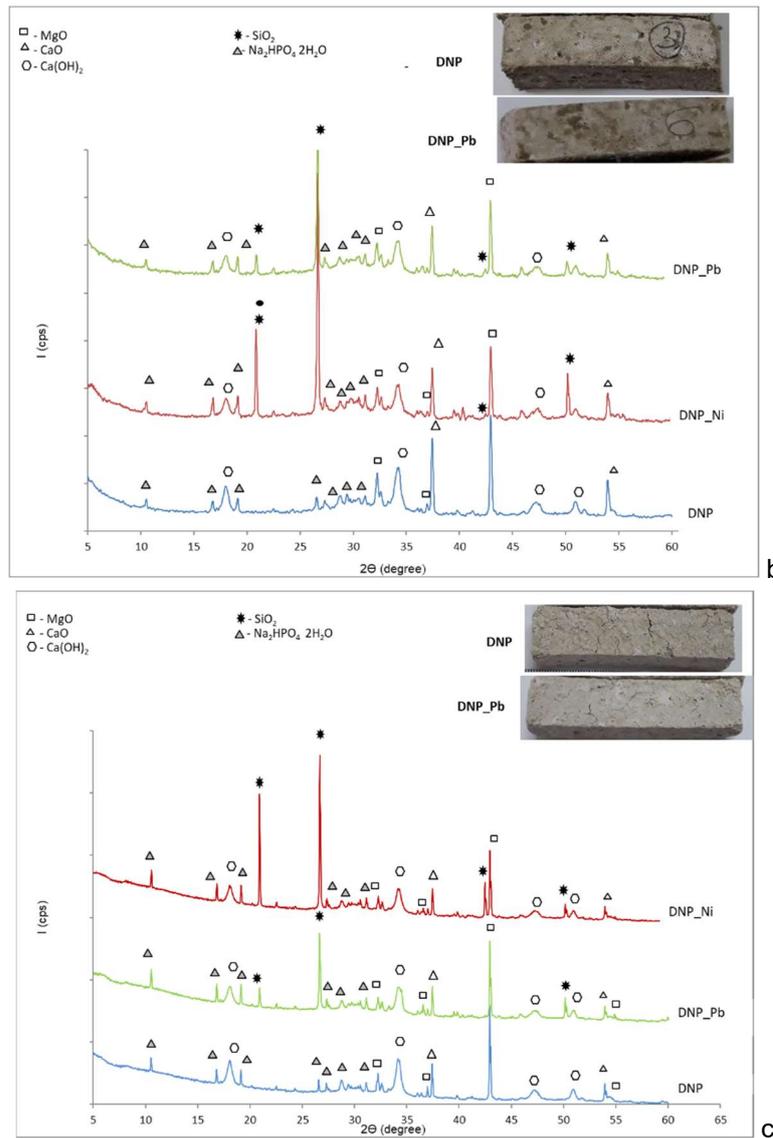


Fig. 5 - XRD patterns of phosphate cements based on calcined dolomite with/without Pb or Ni waste, hardened for: a) 3 days; b) 28 days; c) 360 days/ Analize DRX pentru cimenturile fosfatice pe bază de dolomită calcinată cu/ fără conținut de deșeu de Ni sau Pb, întărite timp de : a) 3 zile; b) 28 zile; c) 360 zile.

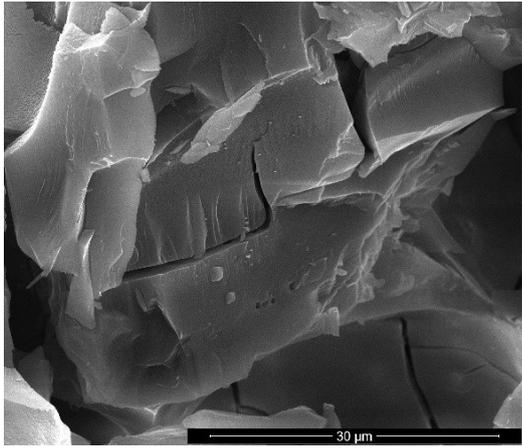
The SEM images of phosphate binders based on magnesia with/without simulated Pb and Ni waste are presented in Figure 6-7.

The SEM images show mainly the presence of a glassy phase, associated with sodium or magnesium phosphates [1,19]. The EDS spectrum of the zone presented in the insert from Fig. 6i, show a high content in P and Na along with a smaller content in Mg in this phase. The presence of Pb and Ni wastes seems to not modify the microstructure of these cements.

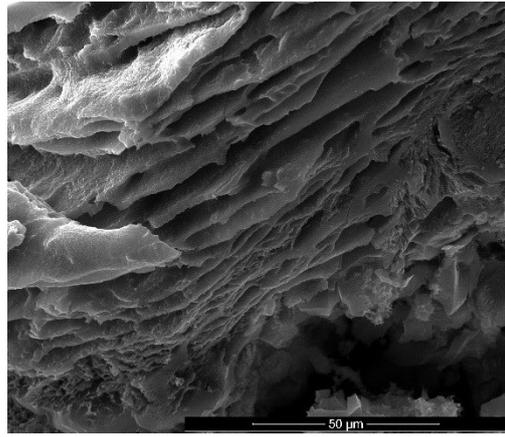
The SEM images of phosphate cements based on calcined dolomite (DNP) hardened for

28 days (Fig. 7a-e) shows the presence of a glassy phase along with a needle like phase. This last phase is present at the surface of glassy phase and according to the EDS spectrum (presented in Fig. 7e) contains mainly P and Na and a small amount of Ca.

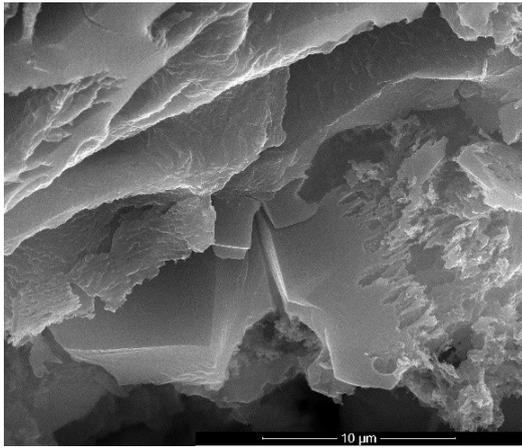
These two phases can be also assessed in the cements with Ni waste (Fig. 7 f-i) or Pb waste (Fig. 7 j-m). Interesting to note, on the SEM image of DNP\_Pb (Fig. 7m) one can assess the presence of a phase with a different morphology i.e. small lamellar plate-like particles which could be associated to  $\text{Na}_2\text{HPO}_4$  [25].



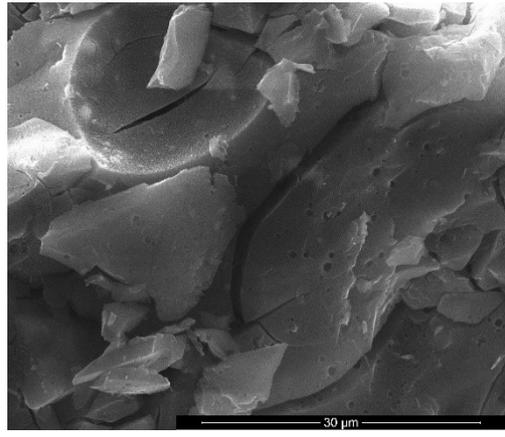
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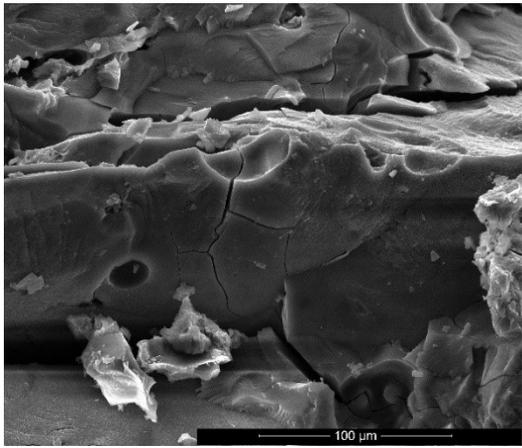
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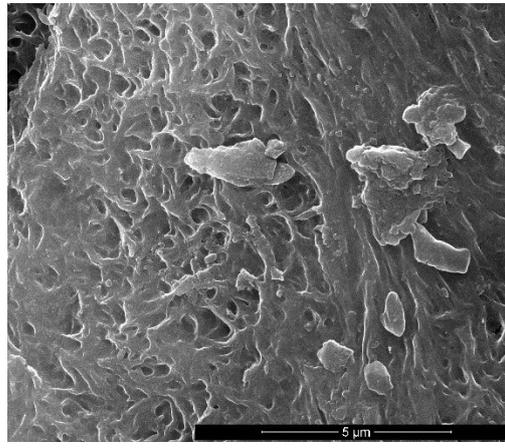
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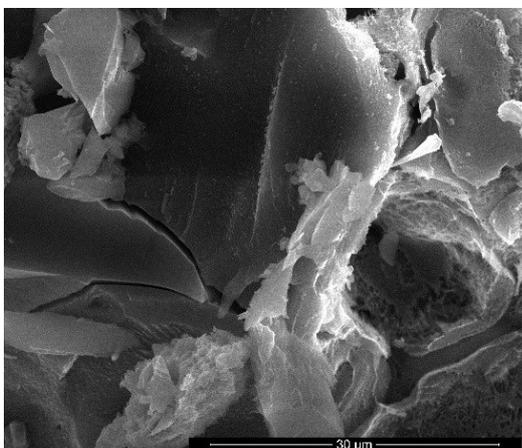
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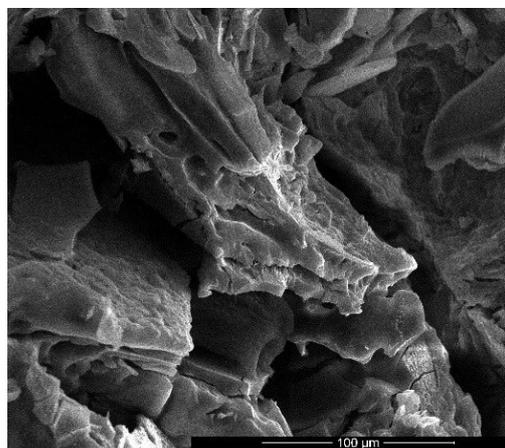
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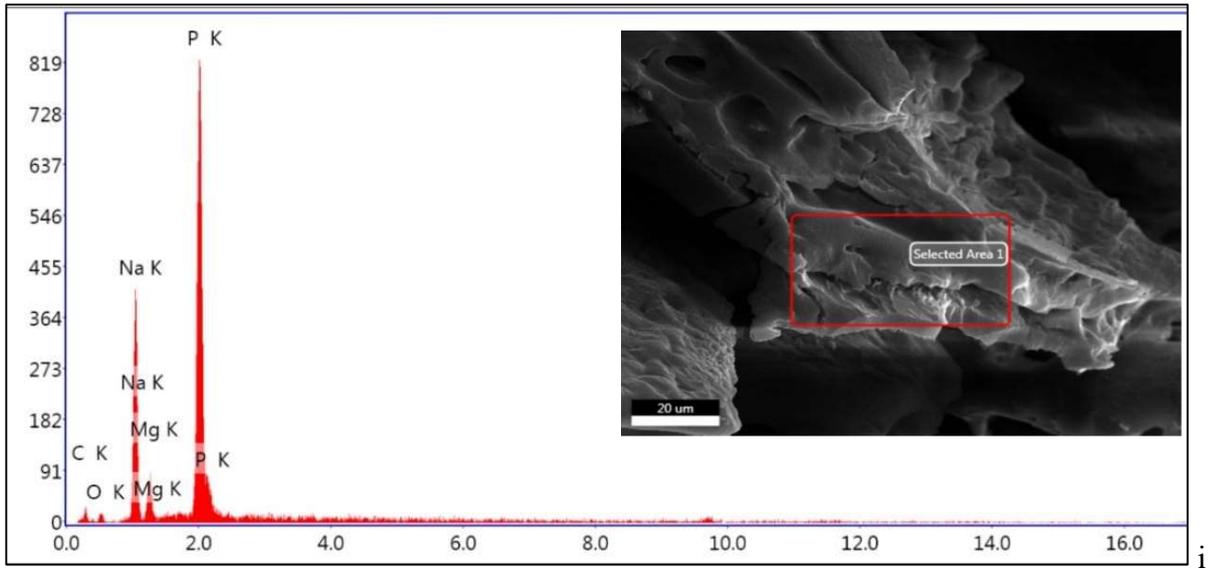


Fig. 6 - Scanning electron microscopy images and EDS spectrum of phosphate cements based on magnesia with/without Ni or Pb wastes: (a-c) MNP; (d-f) MNP\_Ni; (g-i) MNP\_Pb/ Imagini de microscopie electronică de baleiaj și spectrul EDS pentru lianții fosfatice pe bază de magnezită calcinată cu/ fără conținut de deșeuri cu Ni sau Pb: (a-c) MNP; (d-f) MNP\_Ni; (g-i) MNP\_Pb.

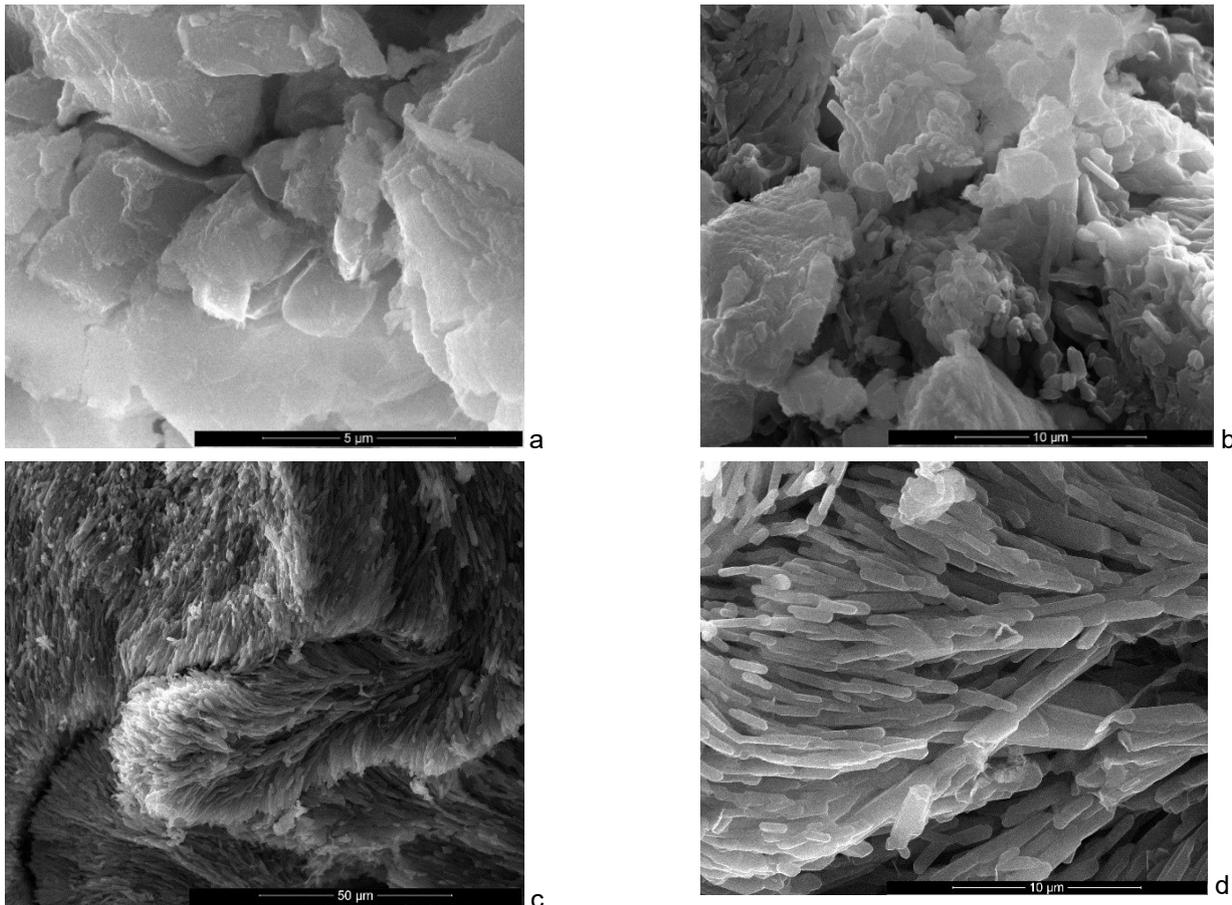


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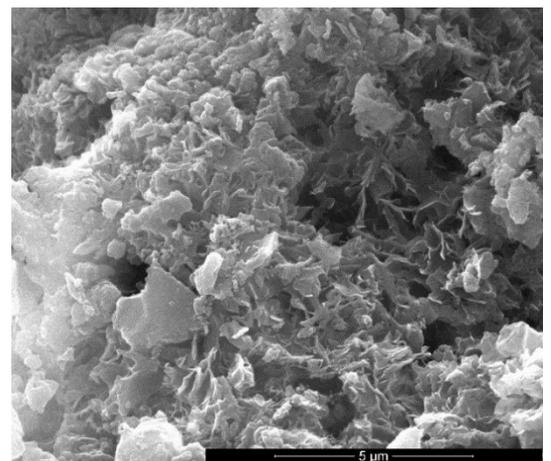
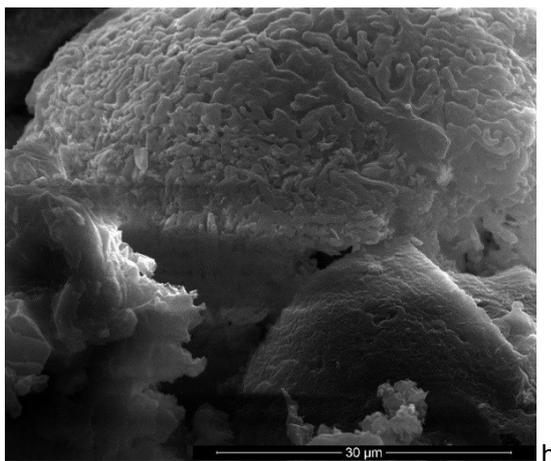
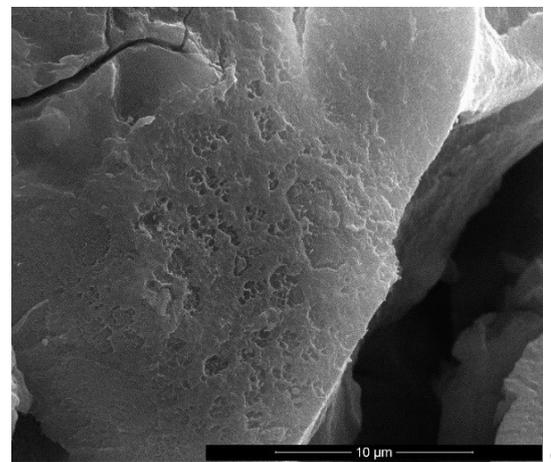
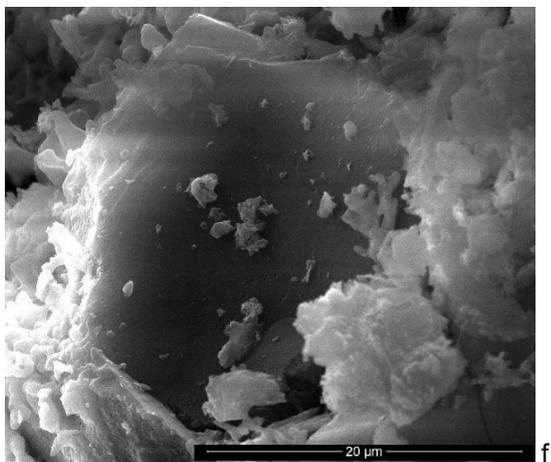
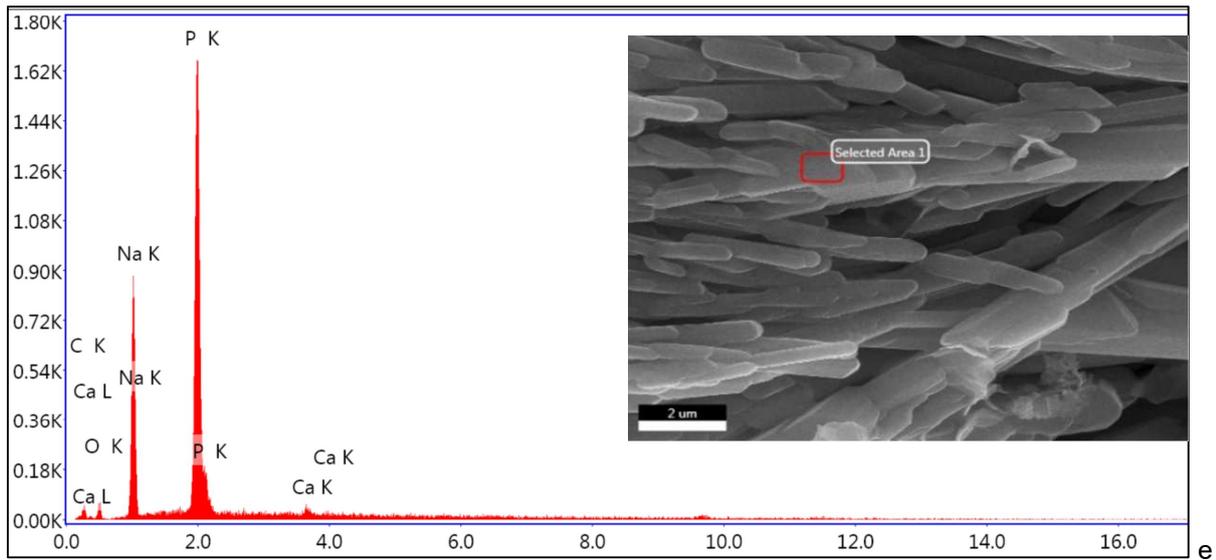


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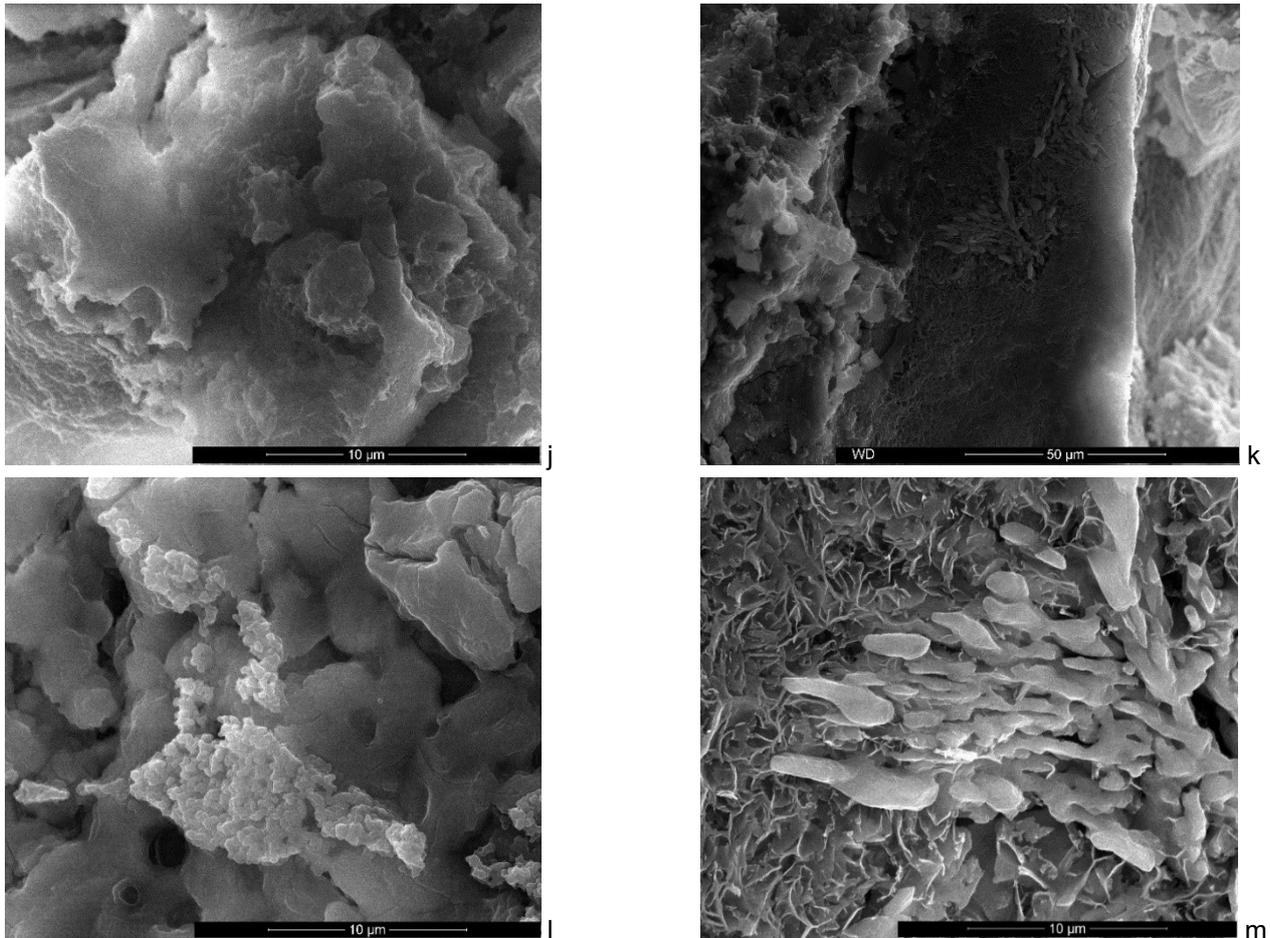


Fig. 7 - Scanning electron microscopy images and EDS spectrum of phosphate cements based on calcined dolomite with/without Ni or Pb waste: (a-e) DNP; (f-i) DNP\_Ni; (j-m) DNP\_Pb/ *Imagini de microscopie electronică de baleiaj și spectrul EDS pentru cimenturile fosfatice pe bază de dolomită calcinată cu/fără conținut de deșeuri cu Pb sau Ni: (a-e) MNP; (f-i) MNP\_Ni; (j-m) MNP\_Pb.*

#### 4. Conclusion

Phosphate cements, based on magnesia or calcined dolomite and sodium dihydrogen phosphate, were obtained and studied in order to assess their feasibility to immobilize two heavy-metals i.e. Ni or Pb.

The following conclusion can be drawn:

- The concentrations of Ni and Pb leached from the phosphate cements with waste content cured for 28 days, are below the maximum regulated values (Order 95/12 feb. 2005). The sodium phosphate cements based on magnesia seems to be more effective in the immobilisation of both Ni and Pb as compared to the phosphate cements based on calcined dolomite.

- Setting time of phosphate cement based on magnesia is shorter than the setting time of the composition based on calcined dolomite. The partial replacement of the magnesia with the simulated Pb or Ni wastes determines a short delay of both initial and final setting times.

- The substitution of magnesia or calcined dolomite with Ni or Pb wastes decreases the early compressive strengths (after one day of hardening);

this could be due to the decrease of the dosage of active component (magnesia or calcined dolomite), which reacts with sodium phosphate and forms the binding matrix. For longer curing times (28 days, 1 year) an important decrease of the compressive strengths of cements based on calcined dolomite is recorded; this is most probably due to the delayed CaO hydration with volume expansion. These data confirm the need to monitor the properties of this type of cements at longer curing times, especially when their intended use is for the immobilization of various waste streams.

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