THE IMPACT OF INDUSTRIAL SOLID WASTES ON HEAVY METAL SOLIDIFICATION/STABILIZAYION OF MAGNESIUM POTASSIUM PHOSPHATE

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Abstract:

In order to examine the impact of admixture amount on the heavy metal solidification/stabilization effect of Magnesium Potassium Phosphate cement (MKPC), industrial dusts, iron ore tailings and slag were added into Magnesium Potassium Phosphate containing 5g/kg of heavy metals at the ratios of 0, 5%, 10%, 20% and 30% respectively. Mixtures were firstly conserved for 1d, 7d, 15d and 28d respectively at conditions of 20±1℃ and humidity ≥95%. Then, Toxicity Characteristic Leaching Procedure (TCLP) method was adopted to measure the leaching concentrations of solidified heavy metal ions, and to examine the impact of admixtures on MKPC solidified heavy metals. The results showed that admixtures at the ratio of 30% would reduce the strength of solidified heavy metals by approximately 70%, while extension of conservation time would improve the strength so as to meet the requirements of landfill processing. After conserving the samples containing 30% of admixtures for 28d, the ion concentration of Zn, Cu and Pb detected by TCLP method were lower than the TCLP standard regulated by the US Environmental Protection Agency (EPA). It shows that MKPC can be mixed with an appropriate amount of admixtures and conserved for a certain period of time before heavy metal solidification/stabilization.

1 Introduction

With the rapid development of China's economy, heavy metal pollution has become a serious problem that draws increasing attention. Based on existing findings, in 2006, about 150 million mu of arable land were polluted by heavy metals in China, accounting for 8.3% of the country's total arable land, and 50 million mu of arable land encountered serious pollution [1-3]. Over the past decade, heavy

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metal pollution incidents were reported almost every year; typical cases include: cadmium pollution in Shaoguan of Guangdong Province in 2005; cadmium pollution in Xiangjiang River in 2009; copper pollution of Zijin Mining in 2010; and cadmium pollution in the Longjiang River of Guangxi Province in 2012 [4-6]. Since heavy metals can only be transferred between different carriers but not degraded, treatment of heavy metal pollution is particularly difficult [7].

Solidification/stabilization technology is one of the commonly used methods for the treatment of heavy metal wastes, wherein ordinary concrete is a kind of commonly used curing material. In view of the poor acid resistance of ordinary concrete after curing heavy metals, its applications in heavy metal treatment are greatly limited. MKPC is a new type of inorganic cementitious material with phosphate hydrate as the binder phase. It is generated through acid-base chemical reaction under acidic conditions after mixing dead burned magnesia with additive agents, soluble potassium phosphate and mineral admixtures at certain ratios. Compared with other materials, MKPC is featured with high early-phase strength, excellent fiber bonding properties, as well as some major advantages of ceramic materials [8]. In some Western developed countries, MKPC has been largely used in the rapid repair of bridges, runways and road construction projects since 1980 s [9], which proved its excellent durability. In 1997, the US Argonne National Laboratory developed a kind of magnesium phosphate with potassium dihydrogen phosphate as the acid component, and successfully applied it into solidification of radioactive and toxic wastes, as well as solidification of geothermal areas and frozen deep wells [10]. Earlier studies have already demonstrated the excellent performance of MKPC in heavy metal curing. Buj, et al [11] used MKPC to solidify/stabilize Cd, Cr, Cu, Ni, Pb and Zn, and measured the leaching concentrations by applying simple batch test (EN 12457-2), equilibrium leaching test, availability test (NEN 7371) and acid neutralization capacity test (ANC) respectively; the results were compliant with current standards, and the curing performance on Pb and Cr remained satisfactory under acidic and neutral conditions. Ma Baoguo, et al [12] suggested that lead nitrate had limited effect on the coagulation time of magnesium phosphate cement, and no effect on the product types ($MgKPO_4 \bullet 6H_2O$ and $Mg_3 (PO_4)_2$) when its concentration is <2.5%, but the product morphology varied. He found that the leaching concentration was one order of magnitude lower than the national standard (5 mg/L), and Scanning Electron Microscope-Energy Dispersive Spectrometer (SEM-EDS) indicated that Pb existed in the cement solidification products. While applying magnesium phosphate cement to solidify Hg, Randal, et al [13] observed that the leaching concentration would exceed 0.2 mg/L when the additive ratio was increased to 70 wt% (weight percent), which did not meet the Resource Conservation and Recovery Act disposal requirements. Meanwhile, MKPC can be mixed with some industrial wastes (such as low-quality fly ash, industrial dust, muddy seabed sediments, etc.) to exhibit high strength, and with wood wastes to form dense composite materials. Thus, a variety of wastes can be transformed into useful construction materials [14]. For general hazardous wastes, after the solidification process using inorganic cementitious materials (such as cement, MKPC, etc.), the solidified products can be stored in barrel, land-filled, used as construction landfill or as construction materials depending on the solidified product strength. Solidification/stabilization, as a mature technology, has been successfully applied into the disposal of a variety of wastes; it is particularly suitable for the disposal of heavy metal pollutants and hazardous wastes [15]. Out of the 57 types of disposal technologies for hazardous waste identified by the US "Resource Conservation and Recovery Act (RCRA)", solidification/stabilization is regarded as the most effective one [16]. However, when solid wastes contain special salts, the strength of the solidified product, as well as the follow-up process will be impacted. Meanwhile, the use of inorganic cementitious materials in the solidification process also can increase the volume and mass of the solid wastes. Therefore, the research related to the solidification/stabilization technology should focus on resource optimization and improvement of solidification effect [15].

To sum up, since MKPC can be either mixed with industrial solid wastes to produce high-quality construction materials, or used to cure heavy metals, radioactive and toxic substances, this study considered adding industrial solid wastes into MKPC and using the mixture product to solidify/stabilize heavy metal wastes. The purpose was to examine its material properties and heavy metal leaching performance, in order to provide a theoretical basis for the application of MKPC in solid wastes and heavy metal wastes treatment.

2 Material and methods

2.1 Material

2.1.1 Dead-burned magnesium oxide (MgO)

Dead-burned MgO powders were obtained by grinding magnesia clinker after being melted in electric arc at 1500-1700℃. Its physical and chemical properties are shown in Table 1.

2.1.2 Industrial solid wastes

Industrial dusts consist of coal dusts from a power plant in Yancheng (70%) and dusts from a textile plant in Yancheng (30%). Iron ore tailings were taken from an iron ore plant in Jiangsu Province, and slag was taken from a power plant in Yancheng. Iron ore tailings and slag were both ground into powers. Initial metal content in each one of the 3 wastes studies are shown in Table 2.

Table 2. Initial metal content of Industrial Solid Wastes

Metal content (mg/kg)	Industrial dusts	Iron ore tailings	Slag
Zn	73.1	100.2	89.7
Cu	168.9	112.6	98.6
Pb	262.6	80.7	288.4
Ni	1346.9	1009.8	1423.6

Table 1. Physical and chemical properties of dead MgO

Name	Composition and content (%)	Surface area (m^2/kg)	Mesh number	Bulk density (g/cm^3)
MgO powders	MgO: 96.0%, CaO:1.33%. SiO_2: 1.92%, Fe_2O_3: 0.34%, Al_2O_3: 0.16% and Other：0.25%	189	120	3.45

Potassium dihydrogen phosphate (KH_2PO_4), natrium boricum and heavy metal nitrates are analytical reagents.

2.2 Methods

2.2.1 Preparation of MKPC containing heavy metals

The basic mixture ratio of MKPC is as follows: nMgO:nMPP = 5:1, mNB:mMgO = 10%, W/C = 0.08, mMgO:mW = 5:1 [Note: MPP: potassium dihydrogen phosphate; NB: natrium boricum; W: water; C: Cement (solid); n:n is molar ratio; m:m is mass ratio]. The additive ratios of heavy metals are 1 g/kg, 3 g/kg, 5 g/kg, 7 g/kg respectively; industrial dusts, iron ore tailings and smelting slag are added into the mixture according to the weight ratios of 0, 5, 10, 20 and 30% respectively (when the ratio of heavy metal is 7 g/kg, W/C = 0.11). Add water and stir rapidly, and then leave the mixture in cement maintainer for 1, 7, 15 and 28 d respectively under the conditions of temperature 20±1 ℃ and humidity ≥95%.

2.2.2 Heavy metal leaching test

To simulate the initial leaching status of the worst situation when curing potential hazardous wastes and municipal solid wastes simultaneously, the US Environmental Protection Agency (EPA)'s Toxicity Characteristic Leaching Procedure (TCLP) was adopted to measure the leaching concentration. Refer to literature [17] for the specific method. Then, extract and filter supernatant, and apply Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) to detect the concentration of heavy metal ions.

The strength of the solidified product was measured according to GB/T17671-1999 “Test Method for Strength of Hydraulic Cement Mortar (ISO)”.

3 Results and analysis

3.1 The impact of different industrial solid waste admixtures on MKPC strength

Table 3 shows the changes of MKPC strength corresponding to different industrial solid waste admixtures

Table 3. Influence of industrial solid waste on compressive strength

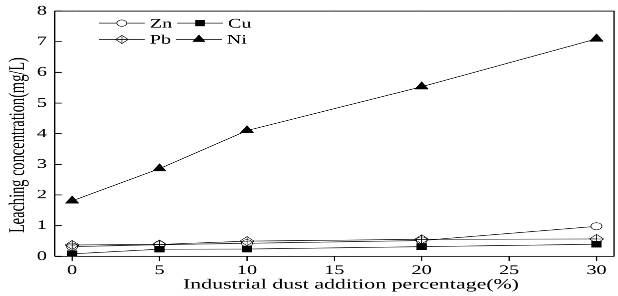
Curing time (d)	No addition	Industrial dust addition percentage (%)				Iron ore tailings addition percentage (%)				Slag addition percentage (%)			
		5	10	20	30	5	10	20	30	5	10	20	30
		Compressive strength (MPa)											
1	32.2	20.8	16.5	12.1	10.7	19.4	15.2	10.5	9.4	20.1	16.1	11.4	8.9
7	48.8	25.2	22.8	16.6	15.5	23.8	19.7	16	14.4	24.2	21.2	15.1	13.8
15	57.8	37.1	30.3	25.8	24.2	31.3	28.6	24.2	23.2	34.8	29.5	23.9	22.7
28	65.8	41.8	35.8	33.2	30.4	38.8	32.2	30.5	28.7	40.6	34.9	32.8	29.6

As shown in Table 3, the strength of MKPC was reduced after adding industrial solid wastes. Different industrial wastes showed different impacts. The impact of industrial dusts on MKPC strength was relatively smaller, while the impact of mining tailings was relatively larger. With the ratio of waste admixture increasing from 5 to 30%, the strength reduction of the solidified product increased from 35 to 70%. Extension of conservation time could significantly improve the strength of the solidified product. Generally, the strength can be increased by 20-30% after 7d conservation, by 40-50% after 15d conservation, and by 50-70% after 28 d conservation.

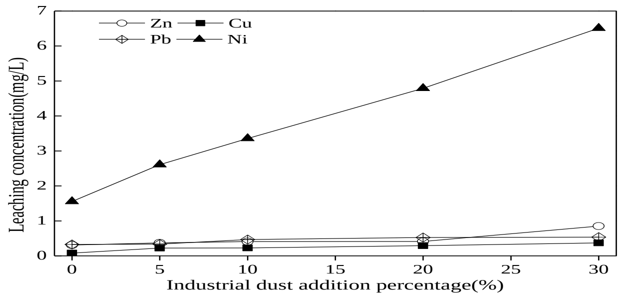
3.2 The impact of different industrial solid waste admixtures on heavy metal solidification of MKPC

3.2.1 The impact of industrial dusts on heavy metal solidification of MKPC

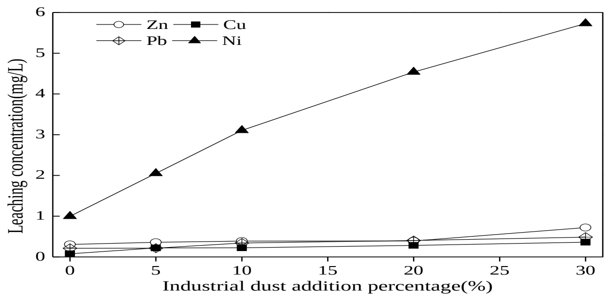
Fig. 1 shows the leaching concentrations of solidified heavy metals containing various ratios of industrial dusts (the ratio of Zn, Cu, Pb and Ni is 5 g/kg each; the conservation time is 1, 7, 15 and 28 d respectively).



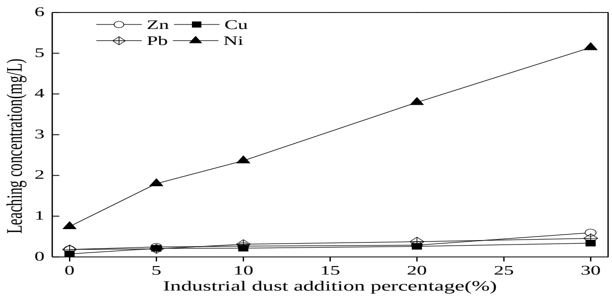
(a) Curing time 1 d



(b) Curing time 7 d



(c) Curing time 15 d



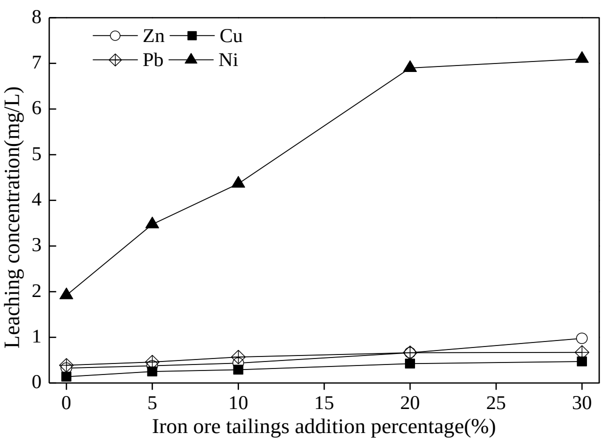
(d) Curing time 28 d

Figure 1. Impact of industrial dusts on heavy metal leaching concentration.

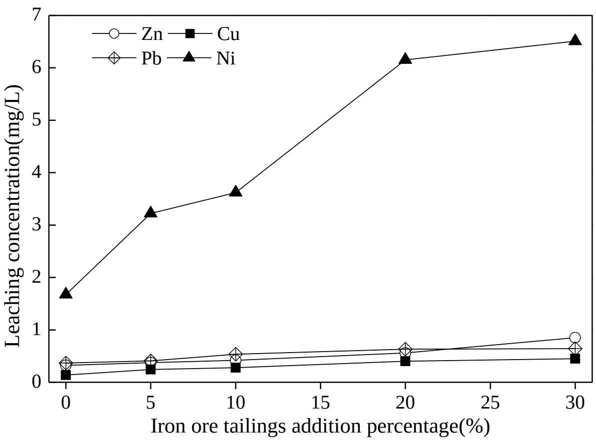
As shown in Fig. 1, the leaching concentration rose with an increase in the ratio of industrial dusts. Out of the four types of heavy metal (Zn, Cu, Pb and Ni), the leaching concentration of Ni ions was the highest, while the leaching concentrations of other three heavy metal ions were relatively low and close to each other. When the ratio of industrial dusts reached 30%, the leaching concentration of Ni exceeded 5 mg/L, showing a poor solidification effect. Extension of conservation time could improve the solidification effect. When the ratio of industrial dusts was equal to 30%, the leaching concentration of Ni ions dropped from 7.096 mg/L to 5.142 mg/L with the conservation time increasing from 1 to 28 d, and the solidification rate was improved by 27%. The leaching concentrations of Zn, Cu and Pb ions dropped from 0.312, 0.0787 and 0.37mg/L to 0.183, 0.0747 and 0.182 mg/L respectively, and the solidification rates were improved by 41, 5 and 51% respectively.

3.2.2 The impact of iron ore tailings on heavy metal solidification of MKPC

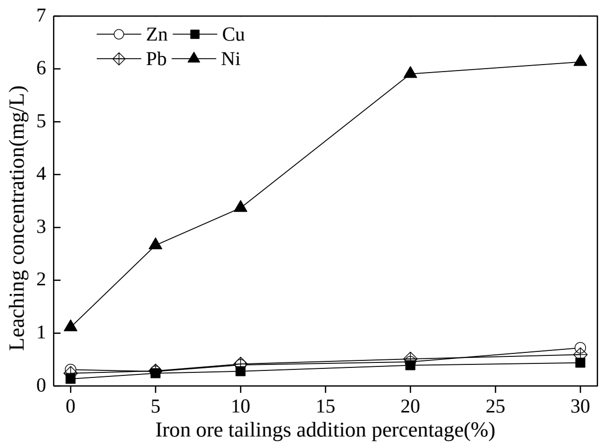
Fig. 2 shows the leaching concentrations of solidified heavy metals containing various ratios of iron ore tailings (the ratio of Zn, Cu, Pb and Ni is 5 g/kg each; the conservation time is 1, 7, 15 and 28 d respectively).



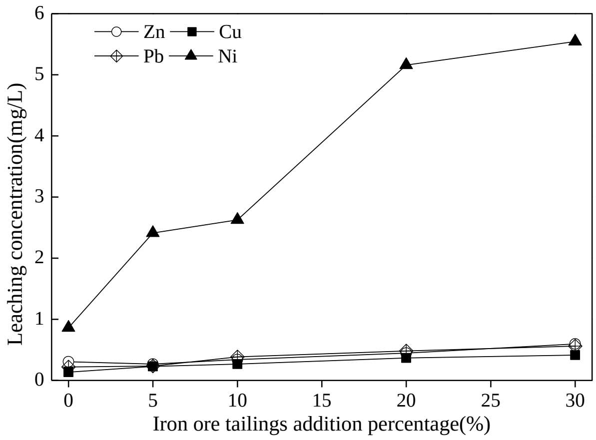
(a) Curing time 1 d



(b) Curing time 7 d



(c) Curing time 15 d



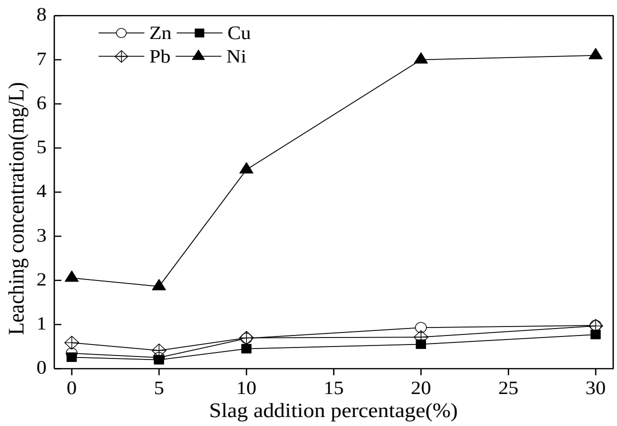
(d) Curing time 28 d

Figure 2. Impact of iron ore tailings on heavy metal leaching concentration.

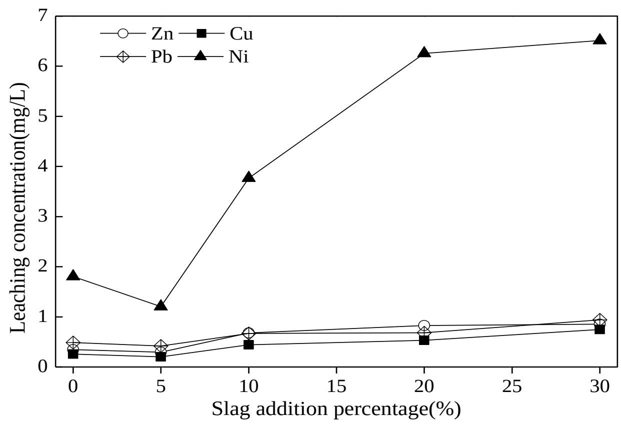
As shown in Fig. 2, the regularity of the heavy metal leaching concentration with the admixture of iron ore tailings was similar to that with industrial dusts. Out of the four types of heavy metal (Zn, Cu, Pb and Ni), the solidification effect of Ni is the poorest. When the additive ratio reached 30%, the leaching concentration of Ni was 5.544 mg/L after 28 d conservation. It showed that, with the increase in the ratio of iron ore tailings, the solidification effect of Ni dropped significantly. Extension of conservation time could improve the solidification effect. When the ratio of iron ore tailings was equal to 30%, the leaching concentrations of Zn, Cu, Pb and Ni ions dropped from 0.977, 0.472, 0.672 and 7.098 mg/L to 0.597, 0.416, 0.562 and 5.544 mg/L respectively, and the solidification rates were improved by 39, 12, 16 and 22% respectively.

3.2.3 The impact of slag on heavy metal solidification of MKPC (decreasing at 5%)

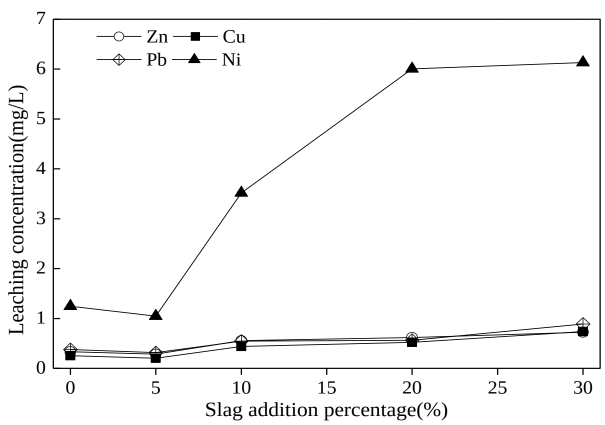
Fig. 3 shows the concentration distribution after 1, 7, 15 and 28 d conservation when the ratio of heavy metal is 5 g/kg.



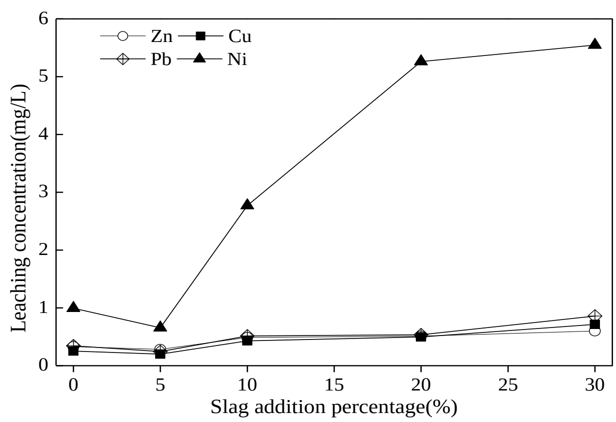
(a) Curing time 1 d



(b) Curing time 7 d



(c) Curing time 15 d



(d) Curing time 28 d

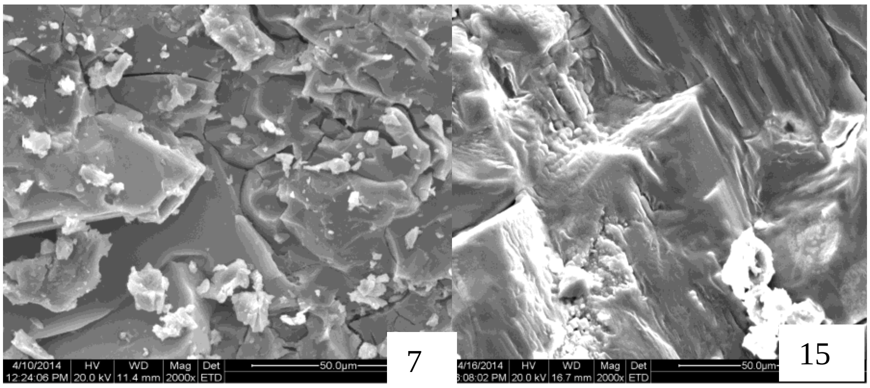
Figure 3. Impact of slag on heavy metal leaching concentration.

As shown in Fig. 3, when the additive ratio of slag was equal to 5%, the solidification effect of MKPC was improved. Out of the four types of heavy metal (Zn, Cu, Pb and Ni), the leaching concentration of Cu was the lowest (0.201 mg/L). Meanwhile, the solidification effect of Ni was again the worst; when the additive ratio reached 30%, the leaching concentration of Ni became 5.546 mg/L after 28 d conservation. It showed that an increase in the ratio of slag could significantly reduce the solidification effect of Ni. On the other hand, extension of conservation time could improve the solidification effect. When the ratio of slag was equal to 30%, the leaching concentrations of Zn, Cu, Pb and Ni ions dropped from 0.979 mg/L, 0.772 mg/L, 0.97 mg/L and 7.1 mg/L to 0.599 mg/L, 0.716 mg/L, 0.86 mg/L and 5.546 mg/L respectively, with the conservation time increasing from 1 to 28 d; the solidification rates were improved by 39%, 7%, 11% and 22% respectively.

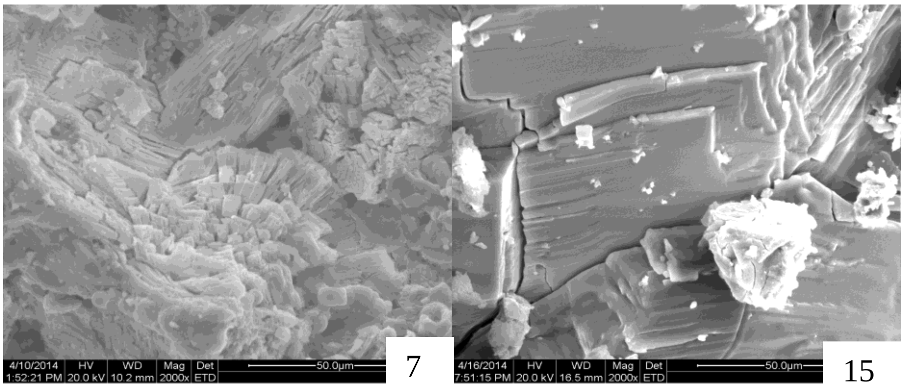
4 Discussion

4.1 Strength of the solidified product

The strength of the solidified product is a key factor for measuring the curing effect. A low level of strength cannot meet the transportation, landfill and toxic leaching requirements, so for the various processing procedures, the solidified product must meet appropriate strength requirements. The strength of MKPC will be heavily impacted after adding industrial solid wastes. The power plant coal dusts and slag used in this study showed similar features as fly ash: there could be porous sponge particles, impurities that inhibit MKPC hydration [18] and some unburned carbon. These impurities can form porous hydration products or produce an impact on the hydration process, which will in turn reduce the strength of cement [18, 19]. The iron ore tailings contain a large amount of calcium and iron oxides, which can react with potassium dihydrogen phosphate and therefore consume phosphate. As a consequence, the insufficiency of phosphate during MKPC hydration will affect the reaction between magnesium and phosphate. Meanwhile, calcium and iron oxides transform into calcium and iron phosphate hydrates, the strength of which is lower than $MgKPO_4 \cdot 6H_2O$, and thus the strength of the final solidified product is also reduced. Hence, when using MKPC to solidify/stabilize heavy metal wastes, the ratio of industrial solid wastes should be carefully controlled. The hydration process of MKPC is an acid-base neutralization process in nature. $H_2PO_4^-$ is acidic, while metal oxides are alkaline; the reaction between the two generates phosphate gel, which then undergoes crystallization and solidification continuously to form the solidified product eventually. Thereby, extension of conservation time can increase the strength of the mixture so as to compensate the strength degradation of MKPC caused by industrial solid wastes. The solubility of magnesium oxide in H_2PO_4 acidic solution is much higher than alumina and iron oxide, so its reaction rate with phosphate is also much faster. The reaction of the two can get hardened in a few minutes, while some other oxides, such as magnesium, calcium and iron oxides, have not participated in the reaction yet. In order to ensure complete reaction, later stage conservation is very critical. Favorable conservation conditions allow reactants to continue reaction slowly, and in turn to improve the strength of the solidified product continuously. The Scanning Electron Microscope (SEM) graph for no industrial solid wastes and mixing with 30% of industrial dusts, undergoing a conservation time of 7d and 15d respectively, are shown in Figure 4 (conservation temperature: 20±1℃; relative humidity: 95%). As shown in Figure 4, the solidified product after 7d conservation showed more cracks and smaller crystals, while the product after 15 d conservation showed fewer cracks and larger crystals, indicating that the hydration reaction of MKPC continued slowly with the extension of conservation time and the internal structure transformed from porous sponge particles into a more compact structure. Thus, the strength of the solidified product was improved gradually.



(a) no addition



(b) industrial dust addition 30%

Figure 4. The SEM of MKPC under different curing time.

4.2 The impact of industrial solid wastes on the toxicity leaching of the solidified product

A small ratio of industrial solid wastes produces insignificant impact on the heavy metal solidification effect of MKPC, and sometimes, the effect may even be strengthened. For example, the heavy metal solidification effect of MKPC can be improved after being mixed with an appropriate ratio of fly ash, because the dissolution of fly ash produces an acidic condition. Existing research shows that neutral and slightly acidic conditions are most favorable for the absorption of heavy metals [20], so fly ash can improve the solidification effect. In this study, the leaching concentration was lowest when adding with 5% slag (Fig. 3); then with the gradual increase of the additive ratio, the leaching concentration of heavy metal grew slowly. For industrial dusts and tailings, the leaching concentration of heavy metal grew consistently with the increase of the additive ratio. This is perhaps directly related to their constituents. The composition of slag is similar to fly ash, containing active oxides such as silicon and aluminum. Similar to magnesium, silicon and aluminum can also react with KH_2PO_4 to generate stable hydrated phosphate precipitation. During the initial reaction stage, there are PO_4^{3-} ions in the phosphate solution; after reacting with alkaline oxides, PO_4^{3-} undergoes polymerization to generate $[PO_4]^{2-}$ ions, which turns into the "endpoint" of the chain polymerization. During the middle stage of the reaction, $[PO_4]^{2-}$ ions turn into "middle state" $[PO_4]^{-}$ ions, and the ends join together to form a series of linear polyphosphates. During the late stage of the reaction, $[PO_4]$ is uncharged; it forms "branches" and develops rapidly into the space to form a three dimensional network [21]. This network has a curing and adhesive effect. It can not only further accelerate the hardening process of cement, but also solidify part of the heavy metal ions to achieve heavy metal solidification/stabilization purposes. However, a large amount of impurities in industrial dusts and tailings will hinder above reaction and spoil the formation of the linear and reticular structure. As a result, the mixture cannot be fully solidified, and the solidification effect is weakened. Meanwhile, by continuously increasing the admixture ratio, the solidification effect becomes worse.

The solidification effect of MKPC varies for different heavy metals. Buj et al. [11] added heavy metal $Cu(NO_3)_2 \cdot 4H_2O$, $Cr(NO_3)_3 \cdot 9H_2O$, $Cu(NO_3)_2 \cdot 3H_2O$, $Ni(NO_3)_2 \cdot 6H_2$, $Pb(NO_3)_2$, or $Zn(NO_3)_2 \cdot H_2O$ of 25 gdm^{-3} into MKPC and adopted the Availability test method for leaching test, the liquid-to-solid ratio is 50 $dm^3\ kg^{-1}$. The leaching concentrations of Cd, Cr, Cu, Ni, Pb and Zn are 0.04, 0.54, <0.02, 0.18, 0.02 and <0.10 $mgkg^{-1}$, respectively. Thus, it shows that the solidification effect of MKPC for 5 types of heavy metal is apparently different. The addition amount of heavy metal can also affect the solidification effect of MKPC on heavy metals. Randal et al. [22] used magnesium phosphate cement containing Hg and $HgCl_2$ to solidify simulated pollutants. When the addition amount is equal to 50 wt%, the leaching concentration of Hg and $HgCl_2$ meets the standard of landfill, but when the addition amount is increased to 70 wt%, the leaching concentration exceeds the standard of 0.2 mg/L.

In view of the above reasons, the solidification ability of MKPC is different for different heavy metals. The concentration of Ni in the 3 types of industrial solid waste is relatively high, so any extra addition of Ni will lead to an excessive level of Ni concentration. Thus, part of Ni cannot form insoluble phosphates in the solidification product. Meanwhile, the addition of the 3 types of industrial solid waste into MKPC can hinder the formation of the poly phosphate network structure, which will further reduce the solidification effect of MKPC on heavy metals. Therefore, the leaching concentration of Ni appears higher than other heavy metals.

5 Conclusion

This study examined the strength and heavy metal leaching toxicity of the solidified MKPC product containing industrial dusts, iron ore tailings and slag at the ratios of 0, 5, 10, 20 and 30% respectively, with various durations of conservation. The analysis about the impact of industrial solid waste admixtures on the heavy metal solidification/stabilization effect of MKPC reaches the following conclusions:

- when the admixture ratio of industrial solid wastes was no more than 30%, the solidification strength was always no less than 8.9 MPa after 1 d conservation, which meets the strength requirements for landfill barrel storage and building fill treatment processes. Extension of conservation time can improve the strength of the solidified product. Therefore, when MKPC is mixed with a certain ratio of industrial solid wastes before heavy metal treatment, the conservation time should be extended appropriately to meet the strength requirements of various treatment processes. Although the strength of the solidified product meets the processing requirements, when different disposal methods are applied, it is still necessary to be cautious about the re-release of heavy metals;
- Fly ash substances like slag could strengthen the heavy metal solidification effect when the additive ratio was 5%. However, if the ratio of the three types of industrial solid wastes was too large, the heavy metal solidification effect would drop. When the ratio of the three types of solid waste was equal to 30%, the leaching concentrations of Zn, Cu and Pb were lower than the standard limits regulated by TCLP of US EPA (Zn, 25mg/L; Cu, 15 mg/L; Pb, 5mg/L), indicating that MKPC has a good solidification ability. This paper did not concern the long-term leaching behaviors and leaching characteristics. Therefore, further studies should be conducted to evaluate the long-term cumulative leaching concentration of the solidified product.
- The solidification ability and leaching regularity of Zn, Cu and Pb were similar, but Ni was different from other heavy metals. Extremely high levels of nickel leaching concentrations may be due to the high initial nickel content in three kinds of industrial solid waste. Further research is needed to interpret specific reasons.

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