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The Effect Of Iron-Containing Slags On The Neutralization Processes Of Water-Soluble Po4-3 In Phosphogypsum Mixtures

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Abstract: The paper presents the results of studies on the neutralization of water-soluble PO4-3 ions in phosphogypsum, which have a negative effect on the properties of gypsum binder. The results of physical and chemical studies of phosphogypsum and metallurgical slag are presented. The use of iron-containing additives can also improve some physical properties of the final product.

It has been established that neutralization of water-soluble PO4-3 in phosphogypsum using Fe2+ and Fe3+ ions is a promising method that not only improves the quality of building materials but also solves environmental problems associated with phosphogypsum disposal.

Keywords: Phosphogypsum; ions; water-soluble; waste; slag; neutralization; mixtures; setting times; mobility; strength.

Introduction

It is known that phosphogypsum is a by-product formed during the sulfuric acid decomposition of apatite or phosphate ores to produce phosphate fertilizers. The operation of mineral fertilizer production enterprises is accompanied by the formation of large-tonnage waste, the volume of which reaches tens of millions of tons.In the sulfuric acid method of processing phosphate rock concentrate, depending on the raw materials and the technology used, 4.3–5.8 tons of phosphogypsum are formed per 1 ton of H3PO4 The waste occupies significant areas, sometimes even land suitable for agricultural work, and has a negative impact on the environment. The disposal of large-tonnage industrial waste is one of the most pressing environmental problems facing the global economy.

The Experimental Part

The use of phosphogypsum in the production of various building materials is of significant economic and environmental importance. However, to date, the utilization of phosphogypsum waste has been insufficient. The reason for the limited use of phosphogypsum lies mainly in the need to remove or minimize compounds containing P2O5, which negatively affect the strength of the material.

Based on the above, we set ourselves the task of investigating the possibility of improving the technological properties of phosphogypsum.

Researchers from various countries have conducted extensive studies on the use of phosphogypsum in various industries [1-7].

For this purpose, we used phosphogypsum and iron-containing slag waste from metallurgical production as research objects (Table 1). The results of chemical analysis showed that, in terms of chemical composition, phosphogypsum mainly consists of calcium oxides, sulfur, and silica (Table 1).

Table 1.
Results of chemical analysis of the raw materials studied

Name of samples	**The content of basic oxides (wt.%)**								LOI	∑
	SiO_2	Al_2O_3	Fe_2O_3 +FeO	CaO	MgO	R_2O	SO_3	P_2O_5		
Phosphogypsum	10,33	0,52	0,13	33,34	0,44	0,08	45,41	1,45	7,74	99,44
AGMK slag	34,31	6,85	51,55	3,04	1,8	1,3	0,01	-	-	98,86
Slag «Uzmetkombinat»	20,11	7,25	28,39	34,87	8,25	0,3	0,60	-	1,36	98,13

X-ray phase analysis of phosphogypsum (Fig. 1) showed the presence of diffraction reflections (d = 0.755; 0.379; 0.370; 0.287, and 0.189 nm), corresponding to the main effects of dihydrate gypsum CaSO4·2H2O, as well as effects (d = 0.334; 0.221; and 0.181 nm), corresponding to the mineral quartz.

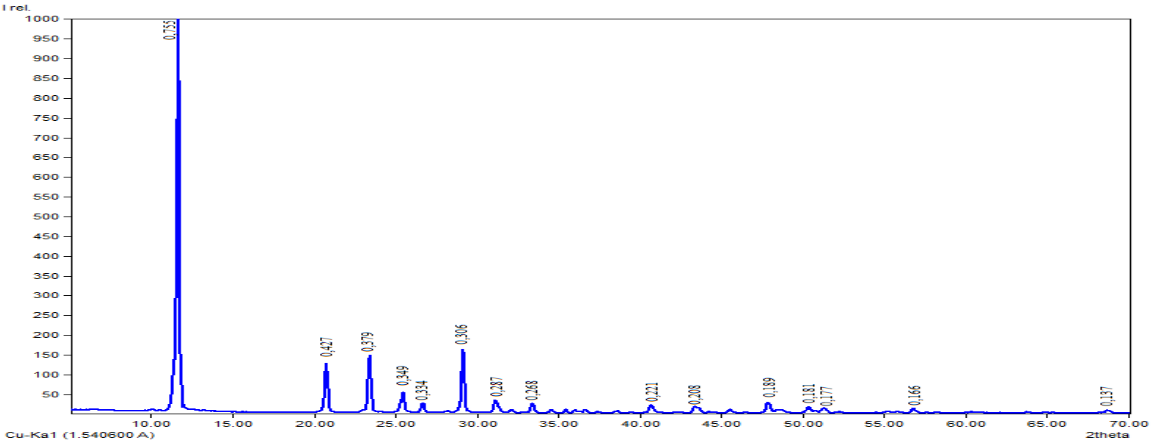

Fig. 1. X-ray image of phosphogypsum waste.

Differential thermal analysis of phosphogypsum samples (Fig. 2) showed that the dendrogram curves exhibit pronounced double endothermic dehydration effects of dihydrate gypsum in the temperature range of 120-200 oC, accompanied by weight loss of the test sample.

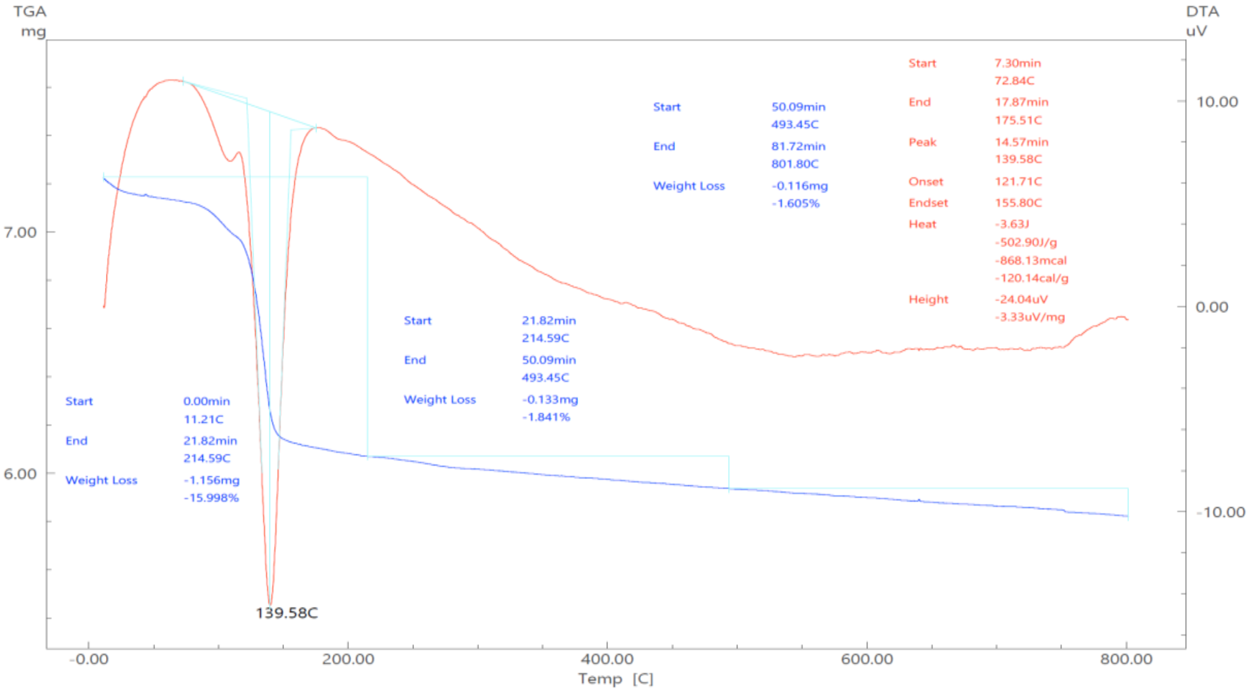

Fig. 2. Results of differential thermal analysis of phosphogypsum

SEM analysis of Fig. 3 performed on a JSM-IT210 device on a phosphogypsum sample showed that, in addition to dihydrate gypsum crystals, the phosphogypsum sample contained aggregated areas of clay and quartz minerals, which was also confirmed by chemical and X-ray phase analysis.

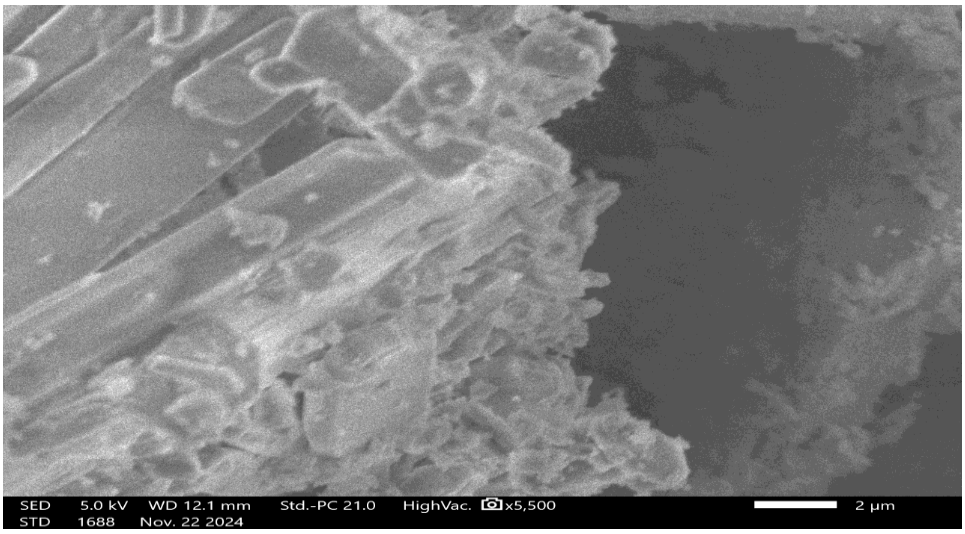

Fig. 3 SEM image of non-heat-treated phosphogypsum.

Iron-containing slags from metallurgical production mainly consist of the following basic oxides: SiO2; Al2O3; Fe2O3; CaO; MgO, etc. (Table 1).

The results of X-ray phase analysis (Fig. 2.9) showed that the X-ray diffraction pattern of the slag samples from the Almalyk Mining and Metallurgical Plant mainly contained diffraction maxima lines corresponding to the mineral fayalite (d=0.355, 0.282, 0.262, 0.256, 0.241, 0.230, 0.177, 0.152, 0.143 nm); hematite (d=0.251, 0.208, 0.184, 0.170, 0.148 nm); in insignificant quantities of minerals of wüstite (d=0.295, 0.253...nm) and quartz (d=0.334, 0.161 nm).

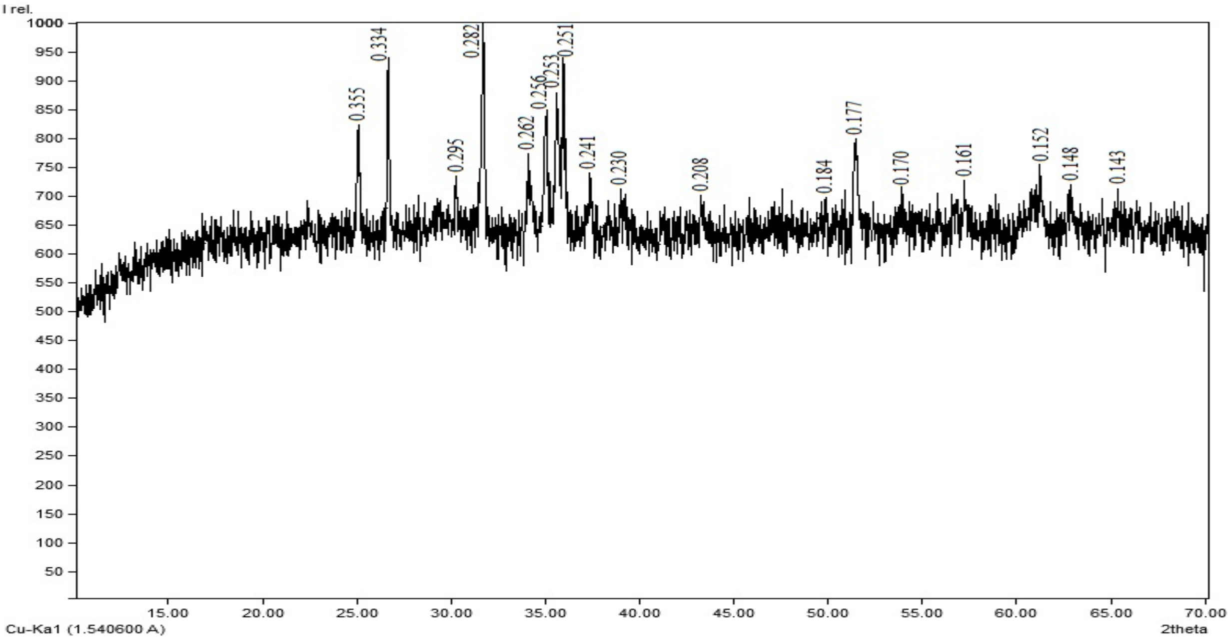

Figure 4. X-ray image of slag waste from the Almalyk Mining and Metallurgical Plant

The X-ray diffraction pattern of the slag from Uzmettkombinat JSC (Fig. 5) shows the presence of diffraction reflections corresponding to minerals, gelenite (d=0.423, 0.302, 0.284, 0.272, 0.212, 0.203, 0.197, 0.181, 0.137 nm); hematite (d=0.268,0.222, 0.187 nm) and quartz (d=0.334, 0.245, 0.227, 0.197 nm).

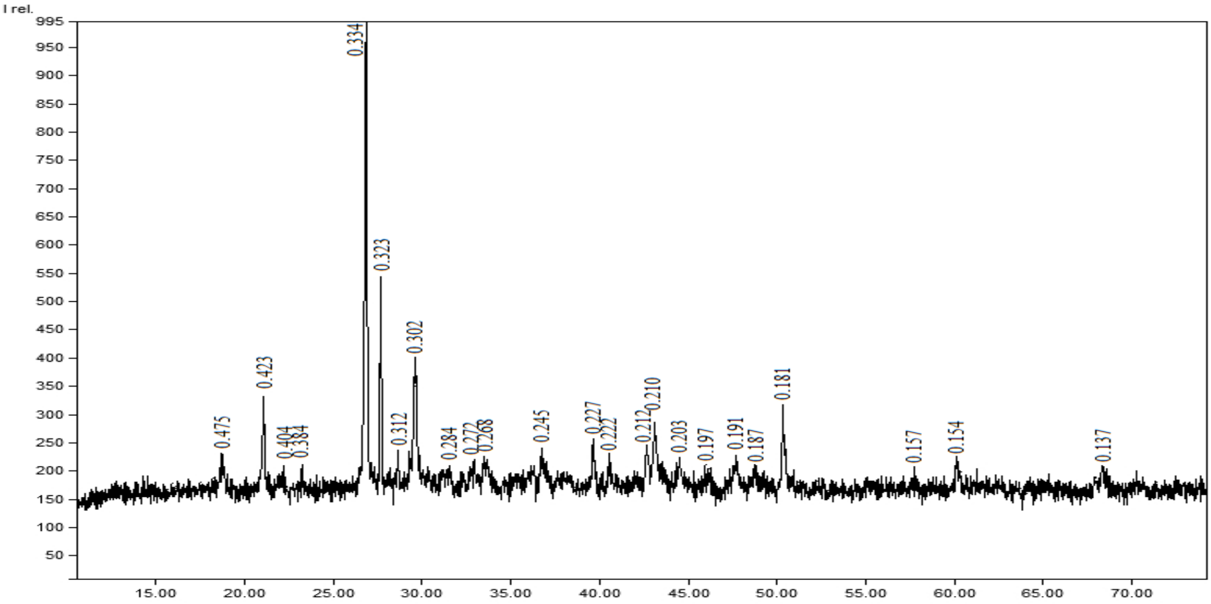

Figure 5 - X-ray image of slag waste from Uzmetsombinat JSC

Petrographic studies of slags from Uzmetsombinat JSC showed a dense structure with minor inclusions in the form of small iron particles, as well as the presence of minerals from the helenite series, such as ockermanite and melilite, which have a finely dispersed structure. In some places, minerals of the hematite group Fe_2O_3 are also present in the structure. Due to the slow cooling of the slag, a gamma form of dicalcium silicate is observed, and periclase and quartz are also present in the slag structure. In the porous and loose structure of the Almalyk GOK slag, mainly plates of phaylite Fe_2SiO_4, hematite Fe_2O_3, and hydrohematite $Fe_2O_3·H_2O$ are observed.

Literature sources indicate that the reason for the limited use of phosphogypsum is mainly the need to remove or minimize the water-soluble compound P_2O_5, which negatively affects the strength of silicate building materials [8-12].

Based on the above, we were tasked with investigating the possibility of improving the technological properties of phosphogypsum and incorporating it as a raw material in the manufacture of silicate building materials.

Currently, there are a number of developments dedicated to the processing of phosphogypsum into a suitable binding material. However, to date, it has not been possible to obtain a suitable raw material for gypsum binders that meet consumer requirements and existing standards, as well as sanitary norms for the maximum content of phosphorus compounds.

The authors [10] have developed a method for obtaining gypsum binder from phosphogypsum, including dehydration of phosphogypsum and grinding, which is different in that, in order to increase the strength of the binder and the possibility of adjusting its properties, crushing to a fraction of (10-500) mm is carried out, and dehydration is carried out in an ultra-high frequency electromagnetic field with a power of (0.5-50) kW for (10-60) minutes. The disadvantage of this invention is the high energy consumption for crushing and creating a high-frequency electromagnetic field.

There is also a known method [11] for processing phosphogypsum, which includes feeding phosphogypsum into a hopper, followed by its grinding and the addition of an additive, differing in that after entering the hopper, the phosphogypsum is fed into a hammer mill for grinding, then mixed with water and a sorbent - finely dispersed tuff in an amount of (0.1-3.0)% of the phosphogypsum mass, and the resulting suspension is fed by process pumps to vortex layer units for treatment in a rotating magnetic field with ferromagnetic particles, then enters settling tanks, where coagulation and precipitation of suspended particles occur. However, the complexity of this method lies in the complexity of the technology, which consists of many processing stages and the corresponding energy consumption to complete the process.

The authors [12] also developed a method for obtaining β-hemihydrate calcium sulfate from phosphogypsum. The method consists of repulping with the addition of a neutralizer, filtering the resulting pulp, and then drying it to a moisture content of (1-3) % and dehydrating it. The neutralizer used is the chemical reagent BaCl2 in an amount of (0.125-1.0) % of the dry weight of phosphogypsum. The disadvantage of this method is the use of BaCl2, which is a chemical reagent and has a certain cost, increasing the cost of production.

Analyzing the results obtained by many researchers [13-18], we conducted a series of experiments to neutralize residual P2O5 in phosphogypsum. The prerequisite for conducting the experiments is the ability of P2O5 in aqueous solution to react with Ag+, Ca+2, Ba+2, Zn+2, Fe+2, Fe+3, Al+3, and other ions to form an insoluble precipitate [19].

In this regard, slag waste from metallurgical production containing iron oxides (Table 2) is of interest. For this purpose, we prepared a series of compositions (phosphogypsum + AGMK slag) -FShA and phosphogypsum + Uzmetsombit slag (FShU) with different ratios of phosphogypsum and metallurgical slag with a water-cement ratio of 0.52 (Tables 2 and 3). The amount of water-soluble P2O5 and Fe+2 was determined by a photocolorimetric method using a KFK-3 photometer.

Table 2.

Effect of AGMK slag additive on phosphogypsum properties

Ingredients	Contents, mass, %		Setting time, min		Time of mobility	Compressive strength, MPa 3x3x3 cm cube samples	PO_4^{3-} content, mass %,
	Phosphogypsum	AGMK slag	beginning	end			
FShA -0	100	0	60,25	180,30	120,05	2,96	1,07
FShA -1	99	1	18,05	48,40	30,35	3,2	0,71
FShA -2	98	2	8,10	39.30	31,2	4,1	0,40
FShA -3	97	3	7.00	28,00	21,0	4,7	0,34
FShA -4	96	4	6.50	26.30	19,8	5,1	0,32
FShA -5	**95**	**5**	**5.10**	**23.55**	**18,45**	**5,5**	**0,30**
FShA -6	**90**	**10**	**4.50**	**22,35**	**17,85**	**7,06**	**0,26**
FShA -7	**85**	**15**	**4.20**	**20.15**	**15,95**	**10,42**	**0,18**
FShA -8	80	20	3.55	18.10	14,55	11,13	0,17
FShA -9	75	25	3.00	17.00	14,00	12,49	0,17

Apparently, the neutralization of water-soluble PO43- in phosphogypsum using Fe2O3 and FeO ions involves several stages associated with chemical reactions. P2O5 (phosphorus anhydride) is a phosphorus oxide that, in its water-soluble form, can form phosphate ions (PO43-). Although Fe2O3 (iron (III) oxide) is a compound that is poorly soluble in water, it can be a source of iron for reactions that may occur in the presence of phosphoric anhydride ions.

$$\mathrm{FeO} + \mathrm{H_3PO_4} = \mathrm{Fe_3(PO_4)_2} + \mathrm{H_2O}$$

$$3\mathrm{FeO} + 2\mathrm{H_3PO_4} \rightarrow \mathrm{Fe_3(PO_4)_2} \downarrow + 3\mathrm{H_2O}$$

The reaction result shows that, as a result of the interaction between iron oxide and orthophosphoric acid formed by the dissolution of PO+3 ions in water, an insoluble precipitate of Fe3(PO4)2 is formed.

Table 3.

Effect of slag additive from Uzmetsombinat JSC on the properties of phosphogypsum

Ingredients	Contents, mass, %		Setting time, min		Time of mobility	Compressive strength, MPa 3x3x3 cm cube samples	PO_4^{3-} content, mass %,
	Phosphogypsum	Slag Uzmetkombinat	beginning	end			
FShU-0	100	0	60,25	180,30	120,05	2,96	1,07
FShU -1	99	1	20,45	58,44	28,09	3,7	0,70
FShU -2	98	2	10,10	43.35	33,25	4,8	0,50
FShU -3	97	3	10,00	38,00	28,00	5,1	0,44
FShU -4	96	4	9,50	30,50	21,00	5,4	0,40
FShU -5	**95**	**5**	**7.20**	**28.50**	**21,3**	**5,9**	**0,33**
FShU -6	**90**	**10**	**5.50**	**24,30**	**18,8**	**6,8**	**0,29**
FShU -7	**85**	**15**	**5.00**	**22.15**	**17,15**	**9,62**	**0,21**
FShU -8	80	20	4.35	20,10	15,75	9,83	0,19
FShU -9	75	25	4.00	19,11	15,11	9,29	0,17

It should also be noted that the chemical composition of both slags (Table 1) also contains a certain amount of FeO2-, which can pass into solution and interact with P2O5 ions to form an insoluble iron phosphate salt. When P2O5 reacts with CaSO4·2H2O (phosphogypsum), calcium phosphates may initially form: 3CaSO4 + P2O5 = Ca3(PO4)2 + 3SO3. When slag is added, iron oxide can form a mixed iron and calcium phosphate: Fe2O3 + H2O + Ca3(PO4)2 = 2FePO4 + Ca (OH)2

Thus, neutralization of water-soluble P2O5 ions in phosphogypsum with Fe2O3 ions promotes the formation of more stable forms of iron phosphate, which is insoluble in water, and reduces its mobility.

The results of determining the setting times of phosphogypsum binder, which is one of its main characteristics (Table 2), showed that even a 1% addition of slag significantly affects the content of water-soluble P2O5, which leads to an improvement in the properties of phosphogypsum binder. The results presented in Tables 2 and 3 show that even a 1% addition, regardless of the type of slag added, sharply reduces the content of P2O5 ions in the phosphogypsum mixture.

The reduction in the content of toxic and undesirable elements makes phosphogypsum safer for use in construction.

The results in Tables 2 and 3 show that the strength of gypsum binder made from unmodified phosphogypsum exhibits low compressive strength of only 2.9 MPa, with initial and final setting times of 60.25 min and 120.05 min, respectively. The addition of even 1% slag sharply reduces the setting time to meet the requirements for industrial gypsum binders. The best results are achieved when adding slag in an amount of 10-15% of the phosphogypsum weight. At the same time, the amount of soluble P2O5 decreases to 0.18-0.21%, which improves the physical and mechanical properties of the phosphogypsum binder (Tables 2 and 3).

The resulting product can be used as a high-quality binder for building boards, plasters, and other building materials.

The use of iron-containing additives can also improve some of the physical properties of the final product.

Thus, the neutralization of water-soluble PO43- in phosphogypsum using Fe2+ and Fe3+ ions is a promising method that can not only improve the quality of building materials but also solve environmental problems associated with the disposal of phosphogypsum.

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