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Review

Possibility of Using Alkali-Activated Phosphogypsum from the Production of Orthophosphoric Acid for the Building Materials—A Review

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Abstract: This paper focuses on the possibility of using phosphogypsum, which is a residue from the production of orthophosphoric acid as an additional source of calcium and the use of spent caustic as an alkaline activator for production of ceramic materials in construction industry. The use of the above-mentioned waste will allow to increase fraction of calcium, sodium and silicate needed for the geopolymerization process and improve properties of material. This review presents a description of the geopolymerization process and the influence of alkaline activator on the reactions occurring in ceramic materials. Collected information, which confirm the possibility of using post-production waste from chemical industry as components for the production of building materials.

Keywords: alkaline activation; building materials; phosphogypsum; spent caustic

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Academic Editor: Andrey Voshkin

Received: 7 November 2024
Revised: 18 December 2024
Accepted: 26 December 2024
Published: 3 January 2025

Citation: Liczbińska, A.; Gębicki, J. Possibility of Using Alkali-Activated Phosphogypsum from the Production of Orthophosphoric Acid for the Building Materials—A Review. *Processes* **2025**, *13*, 97. https://doi.org/10.3390/pr13010097

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1. Introduction

The construction industry is one of the most environmentally burdensome industries, which not only generates greenhouse gas emissions, but is also extremely energy-intensive. It is estimated that the annual global production of bricks will increase to 2.5 billion pieces by 2030 [1], while approximately 2.07 millions tons of carbon dioxide are generated, and the energy needed for production ranges from 0.54 MJ to 3.14 MJ per kilogram of brick. These values depend on the type of brick, kiln and fuel used the weight of the brick may vary from 2 kg to 4 kg) [2,3].

The production of bricks is based on the creation of amorphous structures of aluminosilicates, which are formed in the dehydroxylation reaction of mineral compounds, creating matrix connections between SiO_2,– Al_2O_3 and CaO [4]. The pozzolanic reaction takes place in an alkaline environment at temperatures up to 500–700 °C [5,6]. The addition of active materials that can create pozzolanic bonds has a positive effect on the compressive strength, chemical corrosion, temperature shrinkage, but also on the setting time and heat of hydration of bricks and lime mortars [7–10].

For many years, in order to increase the amount of active ingredients in building materials, ashes from the incineration of e.g., sewage sludge or municipal waste have been used. It was noted that the addition of an appropriate amount of ashes has a positive effect on the mechanical and thermal properties of the obtained building materials [11–14]. In addition, their use allows to reduce the firing temperature. Taking into consideration the number of landfills and the amount of disposal wastes presented in Table 1, the use of waste as components in the production of building materials in the form of bricks or

binders can become a new way to manage them in accordance with the assumptions of the promoted circular economy [14–17].

Table 1. Number and capacity of recovery and disposal facilities for European Union (27 countries) [18].

2020	Disposal—Landfill (D1, D5, D12)	Disposal—Landfill for Hazardous Waste	Disposal—Landfill for Non-Hazardous Waste	Disposal—Landfill for Inert Waste	Disposal—Incineration (D10)	Recovery—Recycling and Backfilling	Recovery—Recycling
Facilities number	6.164	304	2.405	3.455	548	206.346	200.225
Rest capacity [km^3]	3.997	0.467	2.503	1.027	x	x	x

x—lack of data.

Studies were carried out to check the effect of alkaline substances used as activators of the mineral binding process in ceramic materials. The increase in durability is one of many examples of the positive impact of alkali, along with an increase in fire resistance or a reduction in firing temperature, which directly reduce the carbon dioxide emissions [19–21].

The last two years have been full of papers focused on the use of phosphogypsum as additives in the production of building materials. Due to European Commission Decision of 3 May 2000 replacing Decision 94/3/EC establishing a list of wastes pursuant to Article 1(4) Council Directive 75/442/EEC on waste and Council Decision 94/904/EC establishing a list of hazardous waste pursuant to Article 1(4) of Council Directive 91/689/EEC on hazardous waste, phosphogypsum is classify with code 06 09 01 [22]. According to the data available in the literature, only about 15% of phosphogypsum from chemical production is reused, mainly in construction and road construction [23–28] Additionally, given that in 2023 the total amount of this waste in landfills worldwide exceeded 6 billion tons, and an average of 300 million tons per year increases, this is a growing problem [29–32]. It is estimated that by 2050, the total amount of waste phosphogypsum in landfills will exceed 11 billion tons, taking into consideration that for every ton of phosphoric acid produced, there are 4 tons of waste phosphogypsum [29]. According to statistics, nearly 60% of waste phosphogypsum is stored in landfills, and about 25% is dumped in the seas and oceans [25,33]. Closed landfills are usually covered with a layer of soil, but the fact that the problem is not visible does not mean that it disappears [34,35]. This proves the need to find new ways to manage phosphogypsum on a larger scale and implement new solutions in line with the idea of circular economy.

Due to the limited number of papers summarizing the current state of knowledge about the use of phosphogypsum as composites in the production of building materials, it was decided to collect the available information and articles in the form of this literature review. To the best of the authors' knowledge, no such summary work has appeared in recent years, therefore this work may constitute a stimulation in the field of the use of phosphogypsum as building materials.

2. Pozzolanic Materials

Pozzolans consist mainly of silica or aluminosilicate structures that combine with calcium in the presence of water. The finer grain size, the better their binding properties. The content of amorphous silica also has an undoubted influence. In the reaction of calcium with silicate ions and the hydration of the resulting calcium silicates, the CSH phase, which is sparingly soluble in water, is formed in an amorphous form and tobermorite in a crystalline form [36,37]. Additionally, the formation of hydrated calcium aluminates (C_4AH_{13}, C_2AH_8), hydrogehlenites (C_2ASH_8) and hydrogarnets (C_3AS_3-C_3AH_6) can be observed. The example of formation is presented in Figure 1. It is important that the

reactions leading to the hardening of the mixture are initiated already at room temperature, however, they are relatively slow, so the material must "mature" to fully demonstrate its increased strength properties [8,37–39].

$$2Ca(OH)_2 + SiO_2 + H_2O \xrightarrow{800\,^{\circ}C} Ca_2O(SiO_2) \cdot 3H_2O \quad (1)$$

$$3Ca(OH)_2 + SiO_2 + 3H_2O \xrightarrow{800\,^{\circ}C} Ca_3O_2(SiO_2) \cdot 6H_2O \quad (2)$$

$$3Ca + Al_2O_3 + 6H_2O \xrightarrow{800\,^{\circ}C} Ca_3O(Al_2O_3) \cdot 6H_2O \quad (3)$$

$$CaSO_4 \cdot 2H_2O + Al_2O_3 + Ca_2O \xrightarrow{800\,^{\circ}C} 4CaOAl_2O_3 \cdot SO \cdot 2H_2O \quad (4)$$

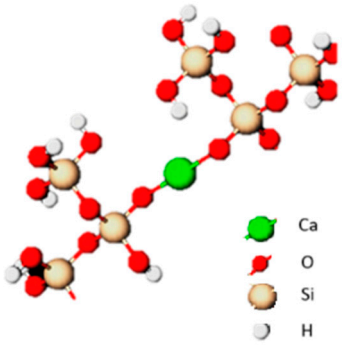


Figure 1. Schematic representation of the C-S-H gel.

Pozzolanic materials in the concrete and cement industry are used as an additional source of minerals, mainly silica. For many years, a popular trend has been the cooperation of enterprises with producers of ceramic building materials in the disposal of fly ash or micro silica remaining after combustion processes, e.g., municipal waste or biomass [40,41]. This is the result of researches on alternative routes of cement production to reduce pollution from the production of standard cements. It was found that wastes from the energy and chemical industry exhibit the properties of pozzolanic materials involved in the growth of the C-A-S-H phase (C–CaO, A–Al_2O_3, S–SiO_2, H–H_2O) due to the high content of calcium, silicon and aluminum [42,43]. The calcium content in pozzolanic material influents directly into the overall strength, the length of the material setting time and the temperature of the hydration process. Taking into consideration that all these conditions are conducive to the stability of the final product and its functional properties, in Table 2 the information how the addition of the considered waste as phosphogypsum and spent caustic improve the properties of the material, are shown [10,44–48].

Table 2. Summary of the dependence of selected parameters on the addition of waste.

Type	Improvement over Standard Bricks	Citation
Phosphogypsum	- compressive strength - water-saturated compressive strength - bending strength - lower water absorption (above 60% of phosphogypsum addition)	[49–52]
Spent caustic	- compressive strength - geopolymerization structures - N-A-S-H and C-A-S-H structures - efficiency of impurity immobilization - similar coefficient of improvements to NaOH	[53,54]

Even in antiquity, it was noticed that some substances can act as an activator of the geopolymerization reaction for pozzolanic materials. One of them is seawater, which, thanks to the content of chloride and sulfate ions, increases the temperature of the system, which promotes the hydration process [55–58]. For the same purpose, alkaline activators are used, which additionally promote resistance to cracking of the material or propagation of cracks.

Taking into consideration the possibility of simultaneous formation of C-A-S-H and N-A-S-H phases (n–Na_2O, A–Al_2O_3, S–SiO_2, H–H_2O), due to the use of a material containing calcium and an alkaline activator (most often NaOH), research is carried out to determine their compatibility. Depending on the amount of calcium and sodium, the phases form in different proportions and influence each other, what influences into thermodynamics, chemistry and efficiency of the entire process [59,60].

Due to the beneficial effect of fly ashes on the physicochemical properties of ceramic materials, many studies can be found in the literature focusing on the use of ashes not only from the energy industry, but also from the incineration of municipal waste and sewage, the management of phosphogypsum and other industrial and municipal waste, which contain large amounts of minerals in their composition, that can contribute to improving geopolymerization conditions [40,41].

3. Phosphogypsum as a Way to Increase the Mineral Fraction

One of the most problematic hazardous waste in landfills is phosphogypsum derived from the extraction method of orthophosphoric acid, otherwise known as the wet method. Orthophosphoric acid is an important raw material that is mainly used in the production of fertilizers, but also in medicine and the metallurgical industry. The annual global production of orthophosphoric acid has been at a level exceeding 60 million tons per year for many years [61,62]. The wet production method involves the extraction of phosphorus from phosphorus-containing minerals (mainly phosphates) using sulfuric acid. Thus, it uses the mechanism of the substitution reaction by replacing the acid residue of a weak acid with the acid residue of a strong acid. The main reactions in the process are shown below [63–65]:

$$Ca_{10}F_2(PO_4)_6 + 10\ H_2SO_4 + 20\ H_2O \rightarrow 6\ H_3PO_4 + 2\ HF + 10\ CaSO_4 \cdot 2H_2O \quad (5)$$

$$Ca_5F(PO_4)_3 + 5\ H_2SO_4 + 2\frac{1}{2}H_2O \rightarrow 3\ H_3PO_4 + HF + 5\ CaSO_4 \cdot \frac{1}{2}\ H_2O \quad (6)$$

The main component of phosphogypsum is calcium sulfate, but also calcium fluoride, calcium phosphate and silica. It is neutral to the environment in itself, however, taking into account the mineral composition of the raw materials used in the production of phosphoric acid, it may be contaminated with heavy metals (Cr, Zn, Cd, Pb) and radioactive elements (Ra-226, U-238) or residual amounts of orthophosphoric acid and hydrogen fluoride [65–71]. In addition to impurities in the form of oxides and other inorganic substances, phosphogypsum contains organic matter—including dioxins [64]. Impurities and significant acidity of phosphogypsum are the main problem that prevents it from being used as a substitute for natural gypsum [71–74].

Phosphogypsum also contains fractions that are desirable for their reuse. It is possible to determine the presence of calcium, iron and aluminum fractions, which undergo the following transformations in the extraction process:

$$CaCO_3 + H_2SO_4 \rightarrow CaSO_4 \cdot \frac{1}{2}H_2O + CO_2 + \frac{1}{2}H_2O \quad (7)$$

$$Fe_2O_3 + 2\ H_3PO_4 \rightarrow 2\ Fe^{3+} + 2\ PO_4{}^{3-} + 3\ H_2O \quad (8)$$

$$Al_2O_3 + 2\,H_3PO_4 \rightarrow 2\,Al^{3+} + 2\,PO_4^{3-} + 3\,H_2O \quad (9)$$

Depending on the technological regime, concentrations and temperature of the extraction process, it is possible to obtain three main varieties of phosphogypsum. The dependence of the resulting forms on the process conditions is shown in the Table 3 [75–80]:

Table 3. Dependence of the resulting forms of phosphogypsum on the parameters of the orthophosphoric acid production process.

Process Name	Gypsum (Dihydrate) Process	Hemihydrate Process	Anhydrite Process
Phosphogypsum form	$CaSO_4 \cdot 2H_2O$	$CaSO_4 \cdot \frac{1}{2}\,H_2O$	$CaSO_4$
Process Temperature	<107 °C	>107 °C	>120 °C
H_2SO_4 concentration	78%	>78%	>78%
H_3PO_4 concentration	50%	60%	68%

Thanks to the above parameters, it is possible to control the transformation of the resulting sulfates and the size of the formed phosphogypsum. From the perspective of production efficiency and the economics of the phosphoric acid production process, it is necessary to strive to obtain coarse crystalline phosphogypsum and anhydrite, which are easy to filter and do not require large amounts of water, what conduct to obtain high concentration of acid. For this reason, relatively high temperatures of around 110 °C are used in the process [77,81–83].

Bearing in mind that the annual global production of phosphogypsum reaches up to 280 Mt, the cleaning of phosphogypsum waste generates additional costs and does not eliminate the waste problem. In order to increase the efficiency of acid production and minimize the amount of waste, the technological principle of better use of the raw material is considered as the recrystallization of the resulting phosphogypsum and the use of raw materials of higher purity. The second idea is problematic due to the global crisis related to the depletion of phosphorus deposits and the fact that most of them are of medium or low purity. In order to maximize their use, research is carried out on the technology of extraction with other inorganic acids, or oxidation of phosphorus-containing minerals with various oxidizing agents, such as ozone, ultrasound, or microwaves [61,65,84,85]. The summary of the above information is presented in Figure 2.

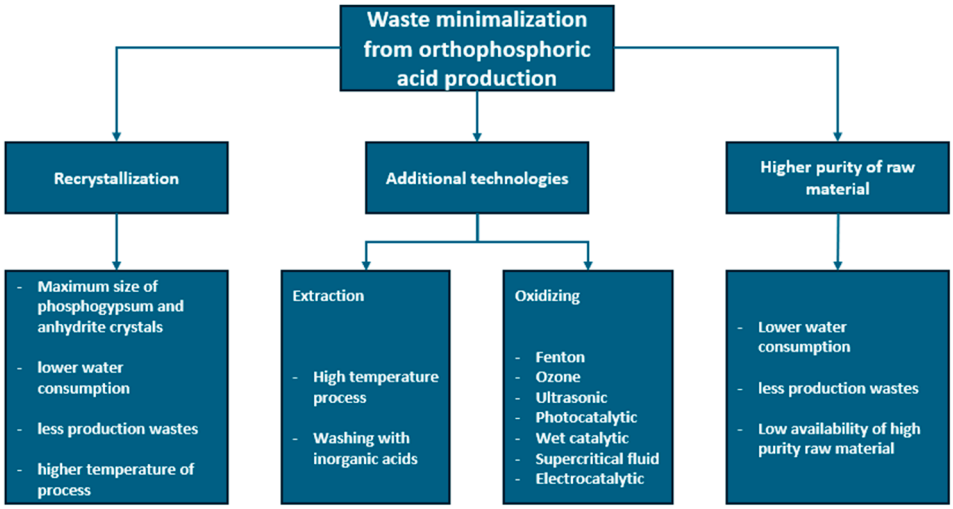


Figure 2. Methods of minimizing waste and intensifying of orthophosphoric acid production.

4. Alkaline Activation

Spent caustic is a waste stream generated mainly in refineries and petrochemical plants in the process of purifying olefins from sulfides, hydrogen sulfide and other sulfur compounds (e.g., mercaptans), carbon monoxide and organic substances [53,54]. It has been classified as hazardous waste, mainly due to its high alkalinity (pH > 12) and the content of radioactive and toxic elements. It consists mainly of NaOH and Na_2CO_3—their total content can be even more than 20%, so it has great potential to be a precursor of the bond formation reaction between aluminum-silicate anions and calcium or sodium cations [53–89].

Neutralization of the spent substance is usually done by wet air oxidation (WAO), which involves washing sulfur compounds and oxidizing them with high-pressure steam according to the following reactions [81,90,91]:

$$Na_2S + \frac{1}{2}H_2O + O_2 \rightarrow \frac{1}{2}Na_2S_2O_3 + NaOH \quad (10)$$

$$\frac{1}{2}Na_2S_2O_3 + NaOH + O_2 \rightarrow Na_2SO_4 + \frac{1}{2}H_2O \quad (11)$$

$$Na_2S + O_2 \rightarrow Na_2SO_4 \quad (12)$$

$$NaSH + 2O_2 + NaOH \rightarrow Na_2SO_4 + H_2O \quad (13)$$

$$CaSO_4 \cdot \frac{1}{2}H_2O + \frac{3}{2}H_2O \rightarrow CaSO_4 + 2H_2O \quad (14)$$

$$CaO \cdot Al_2O_3 \cdot 6\,H_2O + 3\,(CaSO_4 \cdot 2\,H_2O) + 20\,H_2O \text{ -> } 3\,CaO \cdot Al_2O_3 \cdot 3\,CaSO_4 \cdot 32\,H_2O \quad (15)$$

According to the available literature, the above properties allow the use of the spent caustic as a substitute for expensive pure NaOH [59,60,92–94].

The activation itself takes place by creating hydrates in the form of N-A-S-H or C-A-S-H, which take the form of a gel with properties that combine amorphous and zeolite structures [53,95–97]. Increasing the pH causes the separation of silicon and aluminum atoms from the solid form to the forming aluminosilicate matrix [97]. Due to the presence of sulfates in the composition of the substance, one of the crystalline forms is ettringite with the formula $Ca_6A_{l2}(SO_4)_3(OH)_{12}\cdot 26H_2O$, which allows to control the rate of hydration (binding) of the resulting mixture. For the same purpose, gypsum is added to the production of concrete [53,98].

Alkaline activators have become a popular direction for the development of building materials due to their relatively large impact on improving the mechanical strength, durability and thermal resistance of the resulting materials compared to the cost of their production [99]. Technologies involving the use of alkaline activators have been developed since around the 1970s. From the very beginning, the use of waste materials containing large amounts of silica or alumina, such as sludge or fly ash, was taken into consideration [53,99]. The biggest problem is the control of the setting process, which also depends on the ratio of water and caustic solution [99,100]. It was observed that the higher pH of the alkaline activator, the more drastic acceleration the material hydration, even in the first minutes after activator addition. The kinetic of the reaction is connected with the sudden heat release. Taking into consideration available literature, main product (alkali activated) could be formed even in 12 h, while the standard setting process could take up to several days [100,101]. At the moment, it is a common method of producing binders all over the world, by smaller and larger manufacturers, and the scale of use of these additives depends on their availability. The main advantages of using alcali activators are presented in Figure 3.

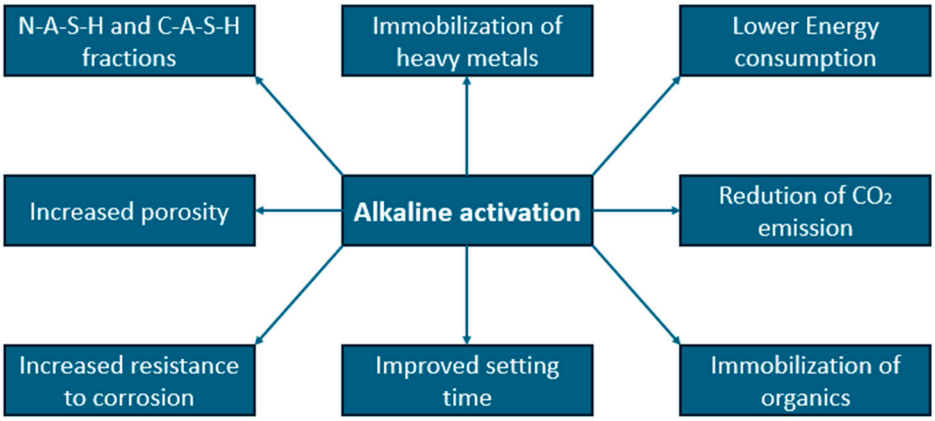


Figure 3. Presentation of selected parameters, which are positively influenced by alkaline activator.

5. Waste or Component

Taking into consideration the properties of both substances described above—phosphogypsum and spent caustic, there is the idea of using them as components in the production of building material.

According to studies published so far, both the presence of phosphogypsum and spent caustic have a positive effect on compressive strength. The fresh and aged samples showed better compressive strength properties than the samples without the additives in the form of these wastes. In addition, it was confirmed that a higher content of Na_2CO_3, which is one of the main components of the spent caustic, has a positive effect on the above properties, m.in. by extending the setting time [53,54,96].

Considering that the bonds form gel systems at room temperature, the energy consumption in the material manufacturing process can be significantly reduced. According to literature data, even samples that do not require firing have better strength properties than those that do not contain spent caustic or phosphogypsum. This is due to the activation of phosphogypsum with an alkaline solution, which reduces the grain size of phosphogypsum and increases the gelation activity and thus also the density [19,53,102–104]. In addition, tetrahedral aluminosilicate structures enable the immobilization of heavy metals through the so-called encapsulation of heavy metal ions, which consists in encapsulating them in microporous matrices [102,105]. Similarly, in the case of organic substances, the resulting zeolite structures increase the absorption surface area of the material and immobilize them [54].

Taking into consideration the above, the simultaneous use of both mentioned wastes opens up new paths of research on the possibility of managing post-production waste into building materials. They have the potential to create a new alternative with the same or better performance parameters than the building materials used so far, while meeting all environmental standards [104–106].

As it shows in Table 4 with summary of articles used in this paper due to the year of publication, it seems to be clear that the topic of using phosphogypsum as a component to building materials is more and more popular in recent years but it is still not the main path to develop. There are also works focusing on other directions of use, mainly phosphogypsum, such as fillers or environmental functional materials, but at the moment their use in the production of building materials seems to be the most promising [107–111].

Table 4. Summary of citations in scope of phosphogypsum used as a component to the building material production.

Additional Material	Alkaline Activator	Properties of Material Compared to Conventional Solutions	Citation
- Natural sand - Fly ash - Lime - Phosphogypsum	None	- improvement of compressive strength - durability in humid - curing temperature: 1000 °C (fired)	[50]
- Wade sand - Hydraulic lime - Cement - Phosphogypsum	None	- better binding action - increase of mechanical and compressive strength - lower water absorption - curing temperature: ambient (unfired)	[51]
- Biomass bottom ash - Phosphogypsum	NaOH Na_2SiO_3	- curing temperature: 100 °C (dried) - improvement of compressive strength	[52]
- Phosphogypsum - Ground granulated blast furnace slag - Fly ash	NaOH	- rapid setting - crystal phase - large pores - C-S-H phase in early hydratation stage - curing temperature: 45 °C (unfired)	[100]
- Phosphogypsum - Cement	Solution of NaOH, $CaCO_3$, MgO, sodium diphenylamine-4-sulfonate	- small grain size - high gelation activity - feasible unfired building materials - curing temperature: ambient (unfired)	[101]
- Phosphogypsum - Calcined clays	11.28 M NaOH	- improvement of compressive strength - no release of harmful substances - higher dense with lower porosity - curing temperature: 750 °C (fired)	[106]
- Phosphogypsum - Cement - Silica fume - Limestone powder	None	- improvement of compressive strength - improvement of hydratation kinetic - chemical shrinkage - possibility of ettering forming - curing temperature: ambient (unfired)	[104]
- Fly ash - Phosphogypsum	NaOH	- improvement of compressive strength - thermal shock resistance - curing temperature: 400/600/800/1000 °C	[102]
- Cement - Phosphogypsum - Fly ash - Sodium metasilicate	Citric acid Sodium silicate	- improvement of compressive strength - curing temperature: 105 °C	[11]

Referring to the table it could be stated phosphogypsum is never used as the only additive but mixed with other wastes used as additives enables the improvement of mechanical and chemical properties of the material and probably has influence of curing temperature. As there is no industrial-scale technology for the production of building materials with added phosphogypsum, the above information summarised in the Table 4 gives a broader spectrum of the advantages of using this waste.

Importantly, the table includes papers that did not use alkaline activator or used it in different concentrations. Any work that included the use of an activator represented the use of NaOH, or solutions with it.

As the work presented above focused on the use of several additives simultaneously with and without the use of an alkaline activator, it is difficult to know which component

had a direct effect on specific improvements. An important point to note is that no work was found that described the use of only phosphogypsum and an alkaline activator.

6. Conclusions

Taking into consideration the increasing demand for construction materials and the increased involvement of the industry in minimizing the harmful impact on the environment, new ways and solutions are being sought to manage waste, reduce emissions and energy consumption. Construction is one of the industries that has the largest share in greenhouse gas emissions and energy absorption, which is why the entire range of research focuses on the production of eco-building materials.

1. There are emerging trends in construction, that use waste, which properties favor the desired characteristics of building materials.
2. Researches are carried out on the use of alkaline substances as activators for the formation of N-A-S-H and C-A-S-H aluminosilicate forms combines gel and zeolite forms.
3. The addition of phosphogypsum and spent caustic affects the formation of gel forms, which results in an increased effect on the setting.
4. Because of the formation of tetrahedral forms of aluminosilicates, it is possible to immobilize heavy metals and develop an absorbent surface by creating a zeolitic fraction for the immobilization of organic substances.
5. The material binding stage begins at room temperature, so it is possible to reduce the energy consumption needed, e.g., for firing the material, due to the lack of such necessity.
6. Further researches are needed to determine the leaches of organic and inorganic substances from the material, its radioactivity and the selection of the optimal composition in order to minimize the negative characteristics of the material.

Taking into consideration given amounts of phosphogypsum in landfills (between 3 and 8 billion tons, depending on the data) and that annual production is more than 300 million tons, the use of this material should be considered not only because of its potential to improve the properties of building materials, but also because of its availability and the negative margin of the waste itself [25,111].

Author Contributions: Conceptualization, A.L. and J.G.; writing—original draft preparation, A.L.; writing—review and editing, J.G.; supervision, J.G. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Data Availability Statement: No new data were created or analyzed in this study.

Conflicts of Interest: The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper. This research received no external funding.

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