

# Valorization of phosphogypsum in cement-based materials: Limits and potential in eco-efficient construction

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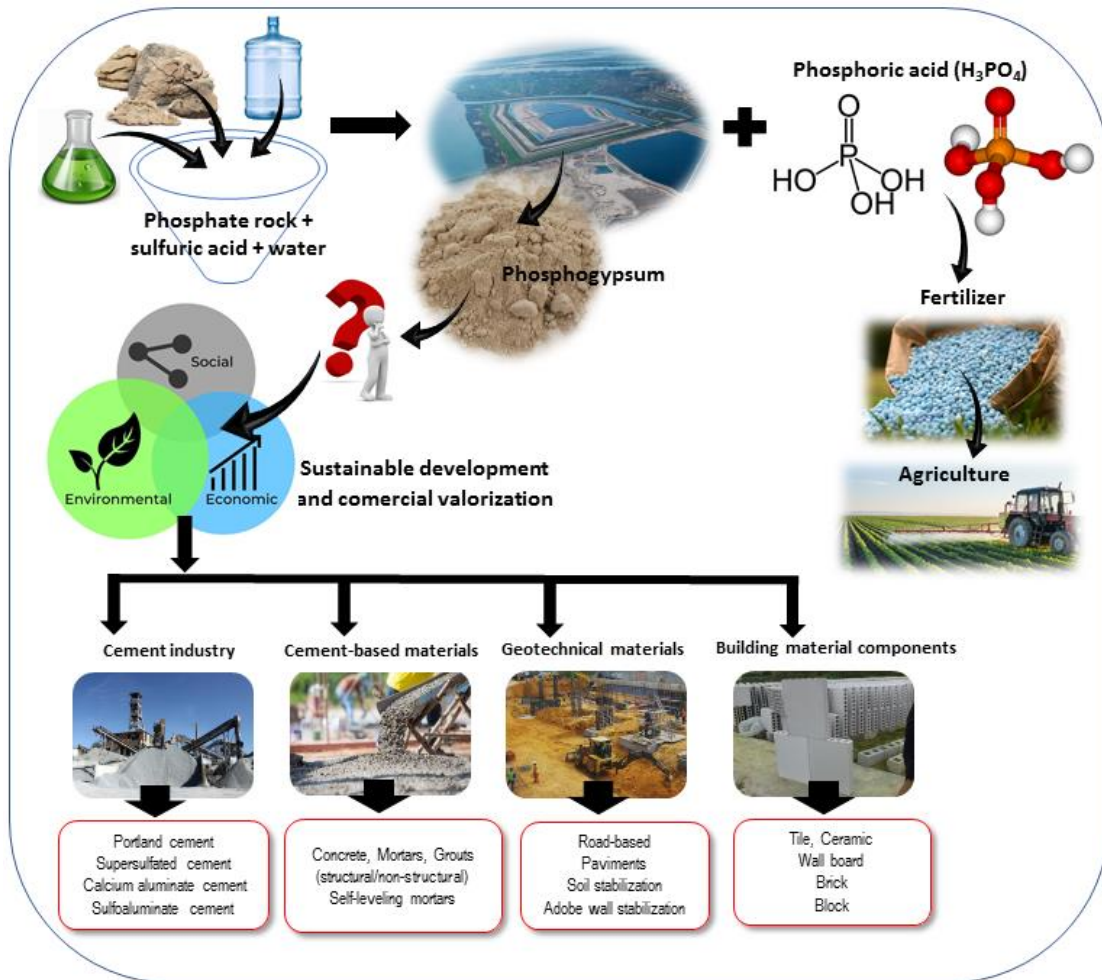
## *Abstract*

Phosphogypsum (PG) is a type of synthetic gypsum generated during the production of phosphoric acid. Each ton of phosphoric acid generates 5 tons of phosphogypsum. This industrial process has caused significant environmental problems worldwide. After an extensive literature review, it was possible to verify that both sedimentary and igneous PG can be used as in materials building components. The use of PG up to 50% meets the limits required for index I, with a radioactivity equivalent to radio-226 and a concentration of radon-222. The data found on bricks (0,11-0,41 Bq m<sup>-2</sup> h<sup>-1</sup>) and plate (0,16-0,41 Bq m<sup>-2</sup> h<sup>-1</sup>) show a lower exhalation rate. It is also found that calcination contributes to the reduction of PG impurities and to the of mechanical strength increase. Mortars and concretes produced with PG-based cementitious systems cementitious systems based on PG achieve high strength (60 MPa, 70 MPa, 80 MPa) and meet the technical feasibility criteria of European standards EN 998-1, EN 998-2, EN 13813, Eurocode 1992 and the American standard ASTM C270. The setting time is influenced by the mineral phase of the PG. It is concluded that cementitious materials produced from phosphogypsum are technically viable and achieve a satisfactory performance. It is possible to advance the following lines: PG-based concrete with high performance, PG-based hydration cement, stabilized mortar, PG-based concrete with high workability and studies about chloride ingress, carbonation, sulfate attack and acid attack in PG-based concrete.

## Keywords

Phosphogypsum – Eco-efficient cement-based materials – Cementitious systems –  
Valorization - Green building materials - Eco-friendly cement

## Graphical abstract



## Abbreviations

$\alpha$ - PG       $\alpha$ -hemihydrate  
A-PG      Anhydrite  
A-PG-II      Anhydrite-II  
A-PG-III      Anhydrite-III

LS      Limestone  
NG      Natural Gypsum  
PC      Portland cement  
PG      Phosphogypsum

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1	β- PG	B-hemihydrate	PL	Portland cement limestone
2	B	Binary	PS	Portland cement slag
3	BA	Bottom ash	QA	Quaternary
4	CAC	Calcium aluminate cement	QI	Quinary
5	CRS	Carbonate silica rock	RM	Red mud
6	CSA	Sulfoaluminate cement	SF	Silica fume
7	Di-PG	Dihydrate	SS	Steel Slag
8	EAFS	Electric arc furnace steel slags	SSC	Supersulphated cement
9	FA	Flay ash	T	Ternary
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11	FGD	Flue-gas desulfurization gypsum		
12	GGBFS	Ground granulated blast-furnace slag		
13	HCBPG	Hydraulic cement-based on phosphogypsum		
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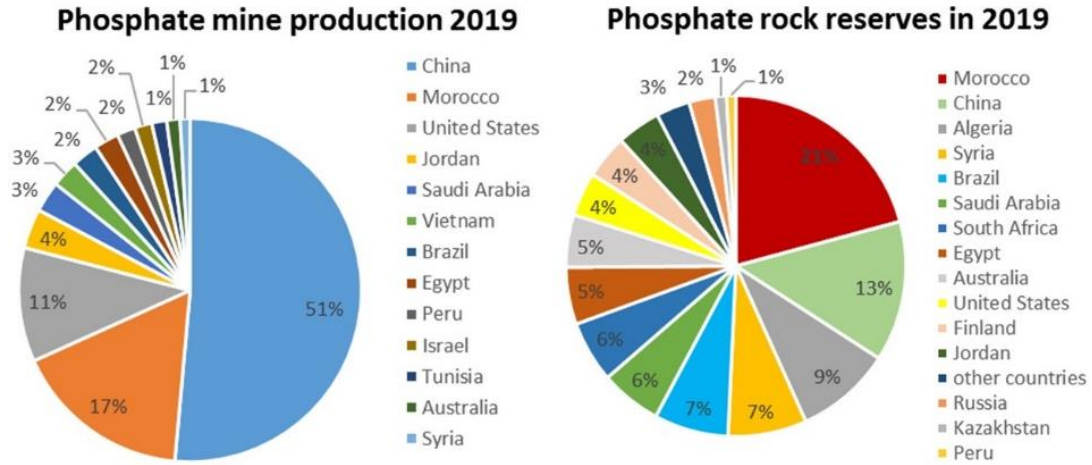
## 1 Introduction

Sustainability is based on economic development, social equity and environmental protection. Different nations under various international agreements have committed themselves to the goal of preserving the natural resources, and the construction of a sustainable built environment [1]. Nowadays, energy efficiency and climate change are worldwide discussed [2]. In the last decades, the European Union (EU) has consistently implemented public policies on those issues. The goal is to reduce the energy consumption, the foreign energy dependence, the carbon dioxide emissions, and to foster actions aiming at reducing both the climate change, and the industrial wastes [3].

By following the public policies that guide both the energy consumption economy and the industrial processes eco-efficiency, the civil construction sector, especially the cement industry, has adhered to sustainable production processes. In this way, it seeks for methods of using industrial waste to produce cement and technologies that minimize CO<sub>2</sub> emissions [4]. It is estimated that until 2050, there will be a 12-13% increase in cement production. The result will be an overall 4% CO<sub>2</sub> emissions increase. However, with the use of mineral additives and industrial by-products, to be used in the clinker/cement ratio, the CO<sub>2</sub> emission can be reduced by 37% [5]. Currently, the challenge is to find alternatives that allow for producing cement in a sustainable way. The use of alternative fuel and new technology by-products have increased, while the consumption of conventional raw material for manufacturing cement has been reduced [1], [6]–[8].

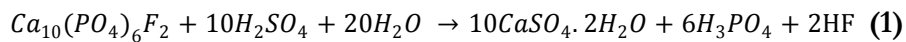
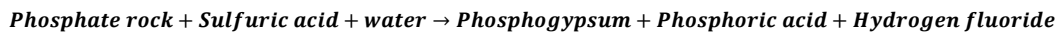
Phosphate rock is an abundant mineral in nature which can be found predominantly in the form of calcium phosphate (apatite), and it can be used with or without industrial process [9], [10]. The physical-chemical characteristics (mineralogical and chemical composition, impurities, grain size, etc.) depend on the geographic location of the phosphate rock [11].

Thus, 85% of phosphate rock sources are of sedimentary origin, and only 15% are igneous [12]. Among the sixteen largest phosphate rock reserves in the world, Morocco (23%) and China (13%) have the largest reserves (**Figure 1**). In 2019, China (110Mt), Morocco (36Mt) and the United States (23Mt) were the three largest producers of phosphate rocks [13].



**Figure 1: Mine production and reserves of phosphate rock in 2019. Adapted from US Geological Survey, 2020 [13]**

Through the chemical reaction (**Equation 1**) between phosphate rock (fluorapatite), sulfuric acid and water (wet chemical treatment), phosphoric acid is obtained and dihydrate phosphogypsum is generated [14]. For each 1 ton of phosphoric acid produced, 5 tons of phosphogypsum are generated [15], [16]. Approximately 85% of the generated waste is stored in areas near rivers or seas, in stacks containing millions of tons of waste, without purification treatments. Until now, only 15% of phosphogypsum is recycled, the remaining 85% are stored in areas near rivers or seas, in open air-piles, without any special treatment and causing great environmental impacts [17]. Phosphogypsum is classified as NORM waste (Naturally Occurring Radioactive Material), and its reuse can cause damage both to human beings and environment, in terms of radiation [18], [19]. Although phosphogypsum has NORM characteristics, over the years, research has been developed to make possible using it as building material component [20], [21] or as fertilizer [22].



Phosphogypsum is considered a synthetic gypsum [23], [24]. It can be used for the manufacture of coating mortars [25]. Besides, it can be also used as mineral additive in

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Portland cement, and in composite cement with blast furnace slag [25], [26]. And, it is also used to regulate the cement hardening time [27].

Valorization of phosphogypsum is an active research field, and therefore, constant advances are carried out to use it as recycled material, with technologically improved levels of radioactivity [28]. García-Tenorio et al [29] assessed the environmental impact generated by NORM wastes, analysing the environmental radiological safety associated to the possible recovery. García-Díaz [30] studied the valorization and characterization of NORM industrial wastes to be used in building materials. Campos et al. [31] focused on measuring radon expiration in slabs and bricks manufactured with PG generated in Brazil. In the research carried out by Saadaoui et al. [32] was investigated the potential uses and problems caused by PG.

The work by Zemni et al [33] provides a study on the generation of calcium silicate and sodium sulphate, through the chemical reactions between phosphogypsum and sodium silicate. The results showed that the solid produced during the reaction can sequester CO<sub>2</sub>. Rashad [24] assessed the effect of PG on properties such as workability, density, mechanical strength and durability within cementitious matrices. Ennaciri and Bettach [34] analysed the conversion of PG to calcite and lithium sulphate monohydrate for batteries, industrial and environmental processes. Huang et al. [35] investigated the possibility of producing Calcium belite-sulfoaluminate clinker from PG .

This research is organized as follows: The first part offers a brief discussion on sustainable development and the influence of the cement industry in increasing global warming, as well as aspects of plaster generation and ways of recovery. The second part shows the methodology developed for construction of this paper, informing the types of databases used, the data classification procedure and the standards studied. The third part provides a description of the mineralogical phases of phosphogypsum. The fourth part focuses on the physical-chemical behavior of PG, in terms of on fineness and particle distribution, major elements, minor elements and radionuclides. The fifth part discusses the rheological behavior, analyzing the normal consistency of the paste, spread flow test in mortars and the slump test in concrete. Then, the sixth part shows the morphology of PG with a focus on microstructure and impurities. The seventh part presents studies on the setting time in cement, plaster and Portland cement systems. The next section discusses the types of PG-based cementitious systems, focusing in paste, mortar and concrete. The ninth part discusses the mechanical resistance of the mortars and concrete, produced with PG-based

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1 cementitious systems. The tenth part compares the cement-based PG studies, then  
2 performance and technical feasibility are analyzed. The eleventh part discusses the durability  
3 aspects of cement-based PG, focusing on solubility, water resistance, shrinkage by drying  
4 and expandability. In the twelfth and thirteenth parts, other ways of enhancing  
5 phosphogypsum are shown, such as geotechnical materials.  
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9 The study of sustainable building materials contributes to the promotion of a circular  
10 economy. In this context, this paper was developed, with the objective of investigating and  
11 evaluating the performance and technical feasibility of different cementitious systems based  
12 on phosphogypsum. Based on the data provided, it is expected to contribute to its  
13 enhancement, offering a basis for taking the PG out of the classification of industrial by-  
14 product and transforming it into an input potentially capable of being used in cement-based  
15 without causing risks to human health.  
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## 22 **2 Methodology**

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24 This work provides a literature review from the most relevant advances on cement-based  
25 phosphogypsum. The data are analysed following these steps: (i) collection of the data, (ii)  
26 the classification of data according to the specific topic, (iii) the organization of data in the  
27 form of tables and graphs, (iv) the description of the topic, and (v) conclusions per item.  
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### 31 **2.1 Data Selection**

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33 The data were selected from a comprehensive literature review, focusing on research on the  
34 feasibility of formulating HCBPG (Hydraulic Cement Based on Phosphogypsum) and the  
35 application in building materials. The selected articles are obtained from scientific literature,  
36 including indexed journals related to cement, mortar, concrete and plaster. The following  
37 sources were reviewed: Science Direct, Scopus, Mendeley, Google Scholar, Springer Link,  
38 Taylor and Francis, International Journal of Innovative Science Engineering and Technology  
39 (IJSET), International Journal on Emerging Researches in Engineering Science and  
40 Technology, MATEC Web of Conferences, International Meeting of Laboratories and  
41 Matters Experiments, Construction and Listening Systems (RILEM), American Society for  
42 Testing and Materials (ASTM), and European Standard and American Standard.  
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### 51 **2.2 Classification of the literature**

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53 A review was performed based on the keywords phosphogypsum, mortar, cement-based,  
54 binder, concrete, durability, industrial by-product, raw material, supplementary cementitious  
55 materials, setting time, curing, filler, gypsum and synthetic gypsum. After that stage, the data  
56 were classified as follows: 1) Search; 2) Classification; 3) Creation of tables; 4) New  
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classification; 5) New tables; 6) Definitive tables. Table 1 shows the methodology used to classify the literature used in this work. **Table 1** summarizes the classification of the literature.

**Table 1: Classification of the literature**

Stage	Name	Result obtained
1	Search	Data search and grouping. Use of Mendeley and JabRef software to organize and create tables of the references found. The result was 192 publications related to phosphogypsum.
2	Classification	Classification of the data in fourteen study subjects based on the titles
3	Creation of tables	Creation of tables on concrete studies, binders, phosphogypsum and lime-based cements, pastes and mortars and sulphated cement.
4	New classification	Review of the tables and changes in the classification of themes based on step 1.
5	New tables	Creation of new tables based on cement systems, mortars, concrete, self-levelling mortars and chemical composition of phosphogypsum.
6	Definitive tables	Definition of tables on the subjects concerning chemical composition, paste, mortar, concrete and geotechnical construction materials and building components.

### 2.3 Standards used

To verify the potential ad usefulness of the reviewed data, European codes and American codes were taken as a basis for comparison. It was possible to compare the following properties: setting time of cementitious systems, mechanical strength and expandability. The codes and scientific works are provided in **Table 2**.

Properties	Used in	Type of binder	Specification or References used as parameter	Ref.
Consistency	Paste	Dihydrate	Singh (2002)	[25]
		Plaster	Lin (2019)	[36]
		HCBPG and Portland cement replacement by PG	Neville and Brooks (2010)	[37]
Setting time	Paste	Plaster	EN 13279-1 and ASTM C28	[38], [39]
		HCBPG	EN 15743, EN 14216, EN 14647, ASTM C91, ASTM C1600 and ASTM C 595	[40]–[45]
		Portland cement	EN 197-1 and ASTM C 150	[46], [47]
Compressive strength	Mortar	HCBPG and Portland cement replacement by PG	EN 998-1, EN 998-2, EN 13813, and ASTM C270	[48]–[51]
	Concrete	HCBPG and Portland cement replacement by PG	Eurocode 1992 and EN 206	[52], [53]
Expansibility (Soundness)	Paste	HCBPG	EN 15743	[42]
		Portland cement	EN 197-1	[46]
Shrinkage	Paste	Plaster, HCBPG and SAC/PC	Hewlett and Liska (2019) Lamond and Pielert, EN 1367-4, EN 12620 ASTM C270	[54]–[57] [51], [58], [59]
Spread flow	Mortar	HCBPG	EN 1015-2 and EN 13454-1	

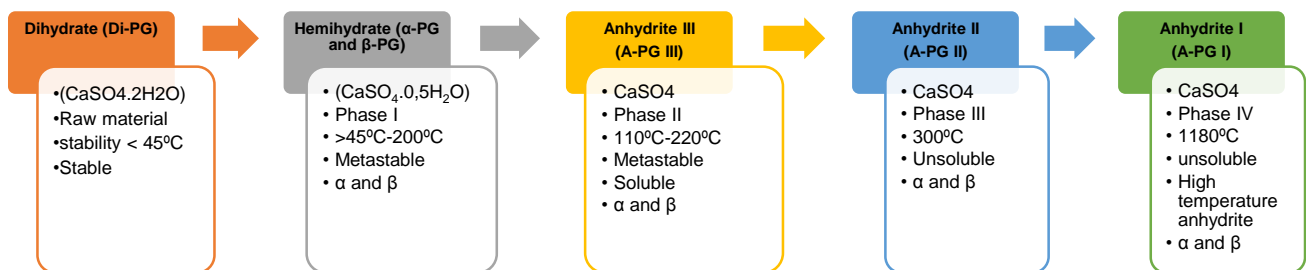
Slump	Concrete	HCBPG and Portland cement replacement by PG	EN 206 and Neville and Brooks (2010)	[37], [53]
Water-resistant	paste	Plaster and HCBPG	Kovler (2001), Selim et al (2020) and Piasta and Zarzycki (2017)	[60]–[62]

**Table 2: Reviewed Standards and scientific works**

### 3 Phosphogypsum: mineralogical phases description

Gypsum is the mineralogical term used for calcium sulphate [63]. It is defined as a non-hydraulic and air binder [64], [65]. Natural calcium sulphate is found in three different mineral phases, which are distinguished by the degree of hydration: Dihydrate ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ) hemihydrate ( $\text{CaSO}_4 \cdot 0,5\text{H}_2\text{O}$ ) and anhydrite ( $\text{CaSO}_4$ ) [66].

However, when the dihydrate (Di-PG) mineral phase is heated (calcination), dehydration process occurs changing the crystalline structure. The first mineral phase after dehydration is known as hemihydrate ( $\alpha$ -PG and  $\beta$ -PG). Anhydrite III (A-PG III) is the second mineral phase, anhydrite II (A-PG II) is the third mineral phase, and anhydrite I (A-PG I) is the fourth mineral phase [67]. **Figure 2** shows the dehydration process of calcium sulphate.



**Figure 2: Dehydration process of calcium system. Adapted from Kuntze [63] and Odler [67]**

Dihydrate mineral phase is gypsum in the raw state [68] and remains stable until the temperature of less than 45°C [67]. The Hemihydrate phase can occur in two ways:  $\alpha$ -hemihydrate due to the autoclaving process or  $\beta$ -hemihydrate through calcination. It has metastable thermodynamic behaviour and both processes occur at temperatures between 45°C-200°C and is popularly known as Plaster of Paris [69], [70].

The process of mineral phase changes to arise the anhydrite mineral phase, consuming a large quantity of energy. Anhydrite-III ( $\text{CaSO}_4 \cdot \text{III-}\alpha$  and  $\text{CaSO}_4 \cdot \text{III-}\beta$ ) occurs through the calcination process of the gypsum hemihydrate between 110-220°C and has metastable thermodynamic behaviour. Whereas, anhydrite-II ( $\text{CaSO}_4 \cdot \text{II}$ ) comes from the calcination of anhydrite III at 300°C. It has rhombic crystalline structure and thermodynamic stability. Anhydrite I is the last mineralogical phase that were the dihydrate gypsum arrives. The



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process occurs through the calcination of anhydrite II with a temperature higher than 1180°C [70]. It has a defined structure and exhibits thermodynamic stability.

It can be concluded that the dihydrate or hemihydrate phase consume low amount of energy than anhydrite II, II and III phases, during the heat treatment. The use of PG in phases A-PG III, A-PG II e A-PG I, in spite of contributing to the mechanical strength of cement-based materials, has a high production cost and impacts on the environment, due to the high energy required for calcination.

#### 4 Physical-chemical properties

Based on the literature review, the fineness and particle distribution and chemical composition of the phosphogypsum were analysed.

##### 4.1 Particle fineness and distribution

The fineness of the binders is a crucial property for cementitious materials. It is determined through the specific surface area and is directly related to cement hydration and the development of mechanical strength. The analysis of the particle size distribution is also important, as it helps to determine the total surface area of the particles [37], [71].

Raw phosphogypsum (Di-PG) when extracted from the piles is like wet sand (**Figure 3**). However, in the dry condition at room temperature, it is a material like silty sand (fine sand). The grains are soft, with uniform granulometry and are considered fine (less than 250 µm). It presents excellent compaction that, consequently, influences the compressive strength [72], [73]. It is worth mentioning that the granulometric distribution and the morphology of the grains, depends on the type of phosphate rock, beneficiation form, distribution of impurities [74], [75] and the time of the attack of sulfuric acid during the production of phosphoric acid [72], [73]. Furthermore, has an influence on the hardening time, water demand, fluidity, consistency, microstructure and resistance [76].

Di-PG particles can have a maximum size of 0.5mm (No. 40 sieve) to 1.0 mm (No. 20 sieve). The material passing through the 0.075 mm sieve (200 sieve) can range between 50-75% [72]. The silt present in the Di-PG can be classified as soil A-4 in the soil classification system AASHTO20. Generally, the phosphogypsum hemihydrate particle is larger in size than Di-PG [72]. PG particles can be almost 10 times thinner than GGBFS particles [77].



**Figure 3: Raw phosphogypsum**

The Uniformity Coefficient ( $C_u$ ) indicates the size of the soil particles and the uniformity level of the granulometric distribution curve. The value of  $C_u$  is found by using the ratio  $D_{60}$  and  $D_{10}$ . The coefficient of curvature ( $C_c$ ) Indicates the shape of the particle size distribution curve between  $D_{60}$  and  $D_{10}$ . The value  $C_c$  is found using the ratio  $(D_{30})^2 / (D_{10} \times D_{60})$ . It is known that  $D_{10}$ ,  $D_{30}$  and  $D_{60}$  are the particle diameters corresponding to 10%, 30% and 60% of the percentage that passes in the particle size distribution curve [78].

$C_u$  of phosphogypsum dihydrate can range between 5.28-13.67 [79], [80] while the  $C_c$  can range between 0.98-8 [79]–[81]. In addition, the effective diameter  $D_{10}$  can vary in size between 0.74-19.40  $\mu\text{m}$ . Particles of size  $D_{30}$  can vary in size between 5.50-54.78  $\mu\text{m}$ .  $D_{50}$  size particles can vary in size between 4.63-13  $\mu\text{m}$ . The diameter  $D_{60}$  can vary between 20.5-102.41 and particles of size  $D_{90}$  can be 19.98-65.01  $\mu\text{m}$  in size [77], [79], [80], [82].

Specific surface area of Di-PG phosphogypsum can range between 2.5 to 17.5  $\text{m}^2/\text{g}$  [83]–[85]. In general, phosphogypsum piles (dihydrate) can contain numerous particles with very fine granulometry and the specific area of the particles is considered large [86]. However, when Di-PG is subjected to heat treatment (calcination), the value of the specific surface area reduces [82].

Research on PG as calcium sulphate has an important role in the rheological behaviour of cement, as it influences the first hours of cement hydration. The fineness and size of the particles (fine, medium and coarse), influence the degree of dissolution and act on the rheological behaviour of the paste. The finer particles have a high degree of dissolution and therefore react faster. The greater the surface area of the particle is, the greater the effect on

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the viscosity of the paste is [87]. Therefore, focusing on HCBPG, the study of the fineness and distribution of PG particles is important. In fact, if the fineness of the binders is known, the reactivity degree of the particles can be determined, as well as the kinetics of hydration of HCBPG, the development of microstructure and the development of cement-based PG properties.

## 4.2 Chemical composition

From the literature review data, the major and minor elements and radionuclides that are present in the chemical composition of phosphogypsum were analysed by comparing the geological origin.

### 4.2.1 Major elements

In nature, pure calcium sulphate is rarely found. Calcium sulphate dihydrate and anhydrite are more easily found together, in addition to impurities [88]. The chemical composition of the natural dihydrate sulphate contains calcium oxide (CaO), sulfuric oxide (SO<sub>3</sub>), water (H<sub>2</sub>O), calcium carbonate (MgO), silica oxide (SiO<sub>2</sub>), aluminium oxide (Al<sub>2</sub>O<sub>3</sub>), iron oxide (Fe<sub>2</sub>O<sub>3</sub>), calcium carbonate (CaCO<sub>3</sub>) and magnesium carbonate (MgCO<sub>3</sub>) and anhydrite (CaSO<sub>4</sub>) [89], [90]. The type and amount of impurities that are present in calcium sulphate are related to its geological origin [91].

**Table 3** shows the chemical composition of phosphoric rock, phosphogypsum dihydrate and the variation of the main elements based on the origin of the phosphoric rock and the type of geological formation. It should be noted that the amount of phosphoric rock and phosphogypsum sources of sedimentary origin is higher than those of igneous origin [92], [93]. Because there are many studies published on sedimentary PG, the most representatives were selected. It is worth noting that there are few studies published on igneous PG.



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**Table 3: Comparison among the main elements of sedimentary origin and igneous origin (%)**

Component	Sedimentary								Igneous						
	Phosphate Rock		Phosphogypsum						Phosphate Rock		Phosphogypsum				
	USA (Florida) [94]	Togo [94]	Turkey [95]	India [96]	Morocco [97]	China [98]	Egypt [99]	South Africa [100]	Russia [94]	Brazil [94]	Finland [101]	Canada [102]	Russia [103]	Brazil (Cajati) [104]	Brazil (Imbituba) [20]
SiO <sub>2</sub>	5.5	8.0	3.87	0.90	1,09	5,05	2.27	1.37	1.2	2.1	0.2	6.38	4.8	1,35	0,22
Al <sub>2</sub> O <sub>3</sub>	1.07	1.5	0,20	0,06	0,075	0,66	-	0.23	≥ 0.4	0.47	0.3	0.24	0.26	2,10	0,16
Fe <sub>2</sub> O <sub>3</sub>	1.19	1.1	0,32		0,006	0,35	1.74	0.12	≥ 0.3	2.07	-	0.04	0.1	-	-
CaO	48.3	48.4	33,5	31.50	33.64	30,52	30.2	43.65	50.5	50.3	45.9	30.2	37.6	31,7	32,46
MgO	0.40	0.15	0,42	0.053	0,002	-	-	-	≥ 0.1	0.66	0.2	0.41	0.8	0,12	-
SO <sub>3</sub>	1.1	0.4	42,86	45,10	53,46	44,47	41.1	51.01	-	1.3	51.4	43.1	54	43,6	42,5
P <sub>2</sub> O <sub>5</sub>	33.0	33.8	0,48	0.52	0,48	0,81	1.56	1.28	38.9	35.2	0.6	1.3	1.7	1,13	0,16
F	3.7	3.8	1,12	0.253	1,18	0,26	1.39	1.1	8.9	1.5	-	-	0,7	0,10	-
Na <sub>2</sub> O	0.47	0.30	0,13	0.079	0,010	0,08	0.14	-	0.5	0.13	-	0.05	-	0,04	0,22
K <sub>2</sub> O	0.09	0.03	-	0.024	-	0,14	0.08	-	0.2	0.06	-	0.06	-	0,02	-
TiO <sub>2</sub>	-	-	-	-	-	0,07	0.4	-	0.5	0.92	-	0.06	-	-	-
LOI	-	-	17.10	19,80	-	18,29	20.8	-	-	-	-	-	-	-	20,02

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1 The chemical composition and mineralogical characteristics of phosphogypsum depend on: the  
2 nature of the phosphoric rock, the type of process to obtain phosphoric acid, the efficiency of the  
3 industrial process, and on the storage age and various contaminants that are added to the  
4 phosphogypsum during the industrial process [105]. Despite this, phosphogypsum contains  
5 chemical, physical and mechanical characteristics that are similar to natural calcium sulphate [68],  
6 [106], [107].  
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10 The igneous phosphoric rock, in addition to being considered better to produce phosphoric acid,  
11 has less radioisotopes population. In general, the phosphate rock from sedimentary origin has great  
12 concentration of radionuclides from the uranium series and low from the thorium series.  
13 Phosphate rock from igneous origin has low concentration of uranium and thorium. About 5% of  
14 uranium as element are left in phosphogypsum during the industrial process [108], [109], while  
15 clearly higher percentages great than 80% of Ra-226, Pb-210 and Po-210 remains in the PG.  
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22 Russia's phosphoric rock has a higher concentration of  $P_2O_5$  compared to phosphoric rock from  
23 Togo, USA and Brazil. And the phosphoric rocks of Togo and USA contain similar concentrations  
24 of  $P_2O_5$ . The fluoride (F) from USA and Togo (sedimentary), presented the same amount and  
25 among all the sedimentary phosphogypsum examples, Egypt has the highest fluoride level. Russia  
26 phosphoric rock has a higher fluoride contamination level than Brazil. However, in all the  
27 examples, the fluoride content of the igneous phosphogypsum is considerably low.  
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33  $P_2O_5$ , F,  $Na_2O$ ,  $K_2O$ ,  $TiO_2$  are considered as impurities in the phosphogypsum. The  $P_2O_5$  and F  
34 are found in three forms: (i) on the surface of phosphogypsum crystals with water-soluble  
35 compounds ( $H_3PO_4$ ,  $Ca(H_2PO_4)_2 \cdot H_2O$ , NaF), (ii) substituted in the lattice of phosphogypsum  
36 crystal (effectively solid solutions of  $CaHPO_4 \cdot 2H_2O$  or  $Na_2SiF_6$ ) in PG and (iii) as insoluble  
37 compounds ( $Ca_3(PO_4)_2$ ,  $CaF_2$ ). These impurities are generated during the reaction of phosphate  
38 rock with sulfuric acid to produce phosphoric acid [110]  
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44 Generally, phosphogypsum dihydrate can contain high levels of phosphorus oxide ( $P_2O_5$ ) and  
45 fluoride (F) [111]. The level of F varies between 0.57-2.5% and the level of  $P_2O_5$  can vary between  
46 0.41-2.0% [14], [17], [104], [112], [113]. When phosphogypsum undergoes processing and changes  
47 from the dihydrate phase to hemihydrate and anhydrite, the level of impurities decreases, mainly  
48 phosphorus oxide ( $P_2O_5$ ), fluoride (F) and alkalis. However, the amount of sulfuric oxide ( $SO_3$ )  
49 and calcium oxide (CaO) increases [114]–[116].  
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55 Phosphates and fluorides have an effect on hydration and setting time, while the compressive  
56 strength in the early ages of cement is reduced [117]. During the hydration reactions of Portland  
57 cement and Portland cement slag, the phosphoric compounds and fluoride compounds that are on  
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1 the surface of the phosphogypsum, react and release the  $\text{PO}_4^{3-}$  and F molecules. These molecules  
2 act as inactive substances on the surface of the grain cement during mixing. As a result, cement  
3 hydration is temporarily postponed [25].  
4

5 In Sulfoaluminate cement, the phosphate (CSA) that is present in the dihydrated phosphogypsum  
6 increases the water demand of the normal paste and inhibits the hydration of  $\text{C}_4\text{A}_3\text{S}$  (ye'elimate). In  
7 addition, it causes a decrease in the compressive strength during the first 6h of hydration of the  
8 CSA cement [112], [118]. Some types of phosphoric rock contains high level of  $\text{AlF}^{5-}$  (fluoride  
9 type) and have a negative effect on the formation of crystals of phosphogypsum (retard the growth  
10 of crystals, shape, particle size and uptake of impurities) [119].  
11

12 Phosphogypsum can be chemically treated and become more suitable for the use of cementitious  
13 systems in a large scale and in an economical way. Phosphogypsum purification methods include:  
14 (i) simple water washing or wet sieving, but all impurities are not removed; (ii) neutralization with  
15  $\text{Ca}(\text{OH})_2$ ; (iii) hot aqueous ammonium sulphate solutions; (iv) there is also the possibility of using  
16 heated ammonium sulphate; (v) calcination of the raw material and (vi) calcination is also used after  
17 appropriate treatment with sulfuric acid [120].  
18

19 The use of heat treatment can remove all impurities by subjecting the phosphogypsum to a  
20 temperature between 130-150°C [121]. Despite the existence of many treatments for the removal  
21 of plaster impurities, acid-soluble phosphates have a difficult removal and, therefore, influence the  
22 setting time and strength level of the cement to which plaster has been added [122].  
23

24 Considering the aforementioned issues of the natural gypsum and the synthetic gypsum, it is  
25 concluded that the chemical characteristics of phosphogypsum are quite like to those of the natural  
26 gypsum and, therefore, is a potential compound of cementitious matrices.  
27

28 It is also concluded that calcination is the most used treatment for the removal of impurities from  
29 phosphogypsum, as it modifies its speed of solubilization and chemical reaction [123]. The metals  
30 present in the phosphogypsum can be removed with water through the leaching procedure. Studies  
31 also show that the reduction of impurities influences the mechanical strength increase, the setting  
32 time and the morphology of the material. The use of synthetic gypsum as raw material substitution  
33 is nowadays a sustainability-oriented alternative strategy [124] [125], [126].  
34

#### 35 **4.2.2 Minor elements and radionuclides**

36 Minor elements (heavy metals) and radionuclides are potential toxic elements, present in  
37 phosphogypsum [115]. PG deposits worldwide contain generally high concentrations of  
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1 radioactive material [113], such as, polonium (Po) and radium (Ra) being consequently a source of  
2 gaseous radon (Rn).

3  
4 These radionuclides can be leached into underground aquifers and surface water systems, being  
5 potentially incorporated in the food chain, and suspended particles can be inhaled by workers and  
6 population. Consequently, they can be an external and internal source of radiation [127]. During  
7 the production of phosphoric acid, most of U and Th are dissolved, however, most of Ra is  
8 deposited in PG. Therefore, the radionuclide  $^{226}\text{Ra}$  is the most worrying element in PG [113].

9  
10 Heavy metals in phosphogypsum appear in different amounts and may contain rare earth elements  
11 (REE) [128]. The most common toxic heavy metals are: chromium (Cr), lead (Pb), cadmium (Cd),  
12 zinc (Zn), copper (Cu), arsenic (As) [129], manganese (Mn) [113]. The amount and type of minor  
13 elements in the composition of PG can range widely, and also depends on the origin of the  
14 phosphate rock [115].

15  
16 PG of sedimentary origin may contain a high concentration of heavy metals and REE, Cd, As, Sb,  
17 V, Cr, Zn, Cu, Ni. PG of igneous origin has low concentrations of Cd, As and Hg. These heavy  
18 metals are potentially harmful to human health, and through water and absorption by plants, they  
19 can be consumed directly or indirectly by humans [128].

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22 **In Table 4**, the concentration of heavy metals is provided. In the literature, PG of sedimentary  
23 origin is considered to have a moderate-high level of danger, whereas PG of igneous origin is  
24 considered to have a low level of danger [129]. Among the sixteen chemical elements with toxic  
25 characteristics, the PG of Morocco presented the largest amount (8) and the highest concentrations  
26 of As, Cd, Cr, Zn, Th, La, Sc and Y. The PG of Brazil presented high level of Ba concentration,  
27 while PG from USA (Idaho) showed high concentration of Pb. PG from Canada showed a high  
28 level of concentration of the elements Cu, Ni, Zr and U. It is noteworthy that data on the Po  
29 element were not found in the review. It is also observed that PG from Russia contains low levels  
30 of heavy metal concentration.

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33 **Table 5** shows the concentration of the most common radionuclides in phosphogypsum and the  
34 geological origin. Most of the data on radionuclides present in phosphogypsum were of  
35 sedimentary geological origin. And it is worth mentioning that, although the PG used in China is  
36 of sedimentary origin, in general, the concentration of radionuclides is low in comparison to other  
37 data of the same geological origin and in comparison, to the PG data of igneous origin.

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56 **Table 4: Comparison of heavy metals between Morocco, EUA, Canada, Brazil and Russia phosphogypsum.**  
57 **Adapted from [130], [131].**



Geological Origin	Heavy metals (ppm)													REE (ppm)			Sedimentary	Igneous	Ref.
	As	Ba	Cd	Cr	Cu	Ni	Pb	Sr	Zn	Zr	Th	U	Po	La	Sc	Y			
Morocco	-	98	6	20	21	<1	6.2	709	8	6.2	4.0	8.3	-	86	4.7	144	x		[132]
Morocco	11	-	30	291	22	-	7	-	345	-	-	-	-	-	-	-	x		[129]
USA (Florida)	-	43	<2	5	3.4	5	10	750	6.4	54	1.5	4.5	-	39.8	<0,05	71	x		[132]
USA (Idaho)	-	47	10.7	48	11.4	5	13	660	31	48	<0,5	7.3	-	73,1	<0.05	125	x		[132]
Canada (Alberta)	-	140	28	70	41.7	15	7	670	112	110	1	13.3	-	-	-	-		x	[113]
Brazil (Imbituba)	-	836	0.8	7	6	6	3	12090	15	-	-	-	-	-	-	-		x	[20]
Russia	1	-	0.1	23.3	30	-	3	-	19	-	-	-	-	-	-	-		x	[129]

**Table 5: Comparison of radionuclides between Australia, Brazil, China, Egypt, EUA, India, Indonesia, Spain and Sweden phosphogypsum. Adapted from [130], [131].**

Use in	PG rock origin	Radionuclides (Bq/Kg)					Geological origin		Ref.
		238U	226Ra	210Pb	210Po	230Th	Sedimentary	Igneous	
Australia	Numerous	10	500	-	-	-	x		[115]
Brazil	Brazil	42	695	894	677	560		x	[133]
Brazil	Brazil	49	744	1061	-	782		x	[134]
China	Keiyan	15	85	82	82	-	x		[135]
USA	Central Florida	130	1140	1370	1030	113	x		[115]
Egypt	Nile Valley rock	-	100	-	445	-	x		[135]
India	Vadorado	60	510	490	420	-	x		[135]
Indonesia	PT Petrokimia Gresik	43	473	480	450	-	x		[135]
Spain	Morocco	140	620	-	82	280	x		[16]
Sweden	Kola (Russia)	-	15	-	-	-		x	[115]

It is possible to verify the variety of heavy metals present in PG and the variation in concentration between PG of sedimentary origin and that of igneous origin. In general terms, the table shows that the Brazilian PG has a lower concentration of heavy metals, while the PG of Morocco presented the highest concentration of heavy metals. In relation to radionuclides, it appears that the PG generated in Russia has a lower concentration of 226Ra, while the PG generated in USA (Florida) contains the highest concentration of radionuclides. Although the PG of China is of sedimentary origin, the concentration levels of radionuclides are low compared to the other countries mentioned in this study.

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## 5 Rheological behaviour

1 Study of the rheological behaviour of cementitious materials in the fresh state is directly related to  
2 their efficiency [136]. Workability is a property that is associated with cement-based consistency  
3 [137], and therefore with the fluidity of concrete and mortars [138].  
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5

### 5.1 Normal consistency of paste

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7 The normal consistency of paste is the term used to define a standardized mixture between the  
8 binder (cement, plaster or lime) and water. This properties has direct influence on the rheological  
9 behavior of cementitious systems. In addition, the normal consistency of paste, interferes with the  
10 workability of cement-based materials [139]. It determines the appropriate degree of plasticity of  
11 the paste [140] as well as the amount of water that is required. In addition, it is related to the initial  
12 and final setting times, and to the expansibility of the cement paste [37]. Figures 4-6, provide the  
13 relation between the compressive strength and the normal consistency of the plaster, HCBGP and  
14 Portland cement, in the mineral phases dihydrate (Di-PG), hemihydrate ( $\alpha$ -PG and  $\beta$ -PG) and  
15 insoluble anhydrite (A-PG II and A-PG I).  
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18 In this review, the values of normal consistency of phosphogypsum dihydrate, plaster, HCBGP  
19 and Portland cement with replacement PG, were compared to the data from the literature. It is  
20 worth mentioning that it was not possible to find standardized data. The normal consistency of  
21 natural dihydrate plaster is 60% consistent [25]; while the normal consistency of the  $\alpha$ -PG plaster  
22 has a value of 30-45% and the  $\beta$ -PG plaster 65-85% [36]. The normal consistency of 26-33% [37]  
23 was used as reference in cementitious systems (HCBGP) and Portland cement with partial  
24 replacement PG.  
25  
26

27 Figure 4 shows the percentage of the normal plaster consistency in relation to the compressive  
28 strength. The analysed works include the following phases: mineral dihydrate (Di-PG), hemihydrate  
29 ( $\alpha$ -PG and  $\beta$ -PG), and insoluble anhydrite (A-PG II and A-PG I). In general, it is observed that  
30 there is a relation between the compressive strength, the mineral phase of the phosphogypsum and  
31 the normal consistency: if the value of normal consistency increases, the value of compressive  
32 strength decreases.  
33  
34

35 It is noted that the consistency of Di-PG is directly related to the purity of the material, and the  
36 use of retardant does not cause interference. Normal Di-PG consistency can range from 66% to  
37 80% [25], [96]. When the normal consistency of natural calcium sulphate dihydrate is compared,  
38 it is observed that both types (selenite and phosphogypsum) have similarities in the values of  
39 compressive strength and normal consistency [25].  
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The consistency of  $\alpha$ -PG without setting retardant is 58% consistent and its compressive strength is 15 MPa. However, the use of setting retarder reduces consistency and increases setting time. The use of 0.20% sodium succinate reduces the consistency to 35% and increases the strength to 25.58 MPa [141].

The consistency of  $\beta$ -PG without the use of setting retardants can vary between 60% to 66% [141]–[143]. Washed purified  $\beta$ -PG has a consistency of 64%, and the compressive strength is 13.5 MPa. A-PG I has a very low percentage of normal consistency (22.14%-23.53%) compared to other mineral phases. The compressive strength is high (35.1 MPa -37.2 MPa), regardless of the amount of setting retarder [144]. A-PG II calcined at 800°C, presented a low consistency of 26.14% and resistance to compression of 56 MPa.

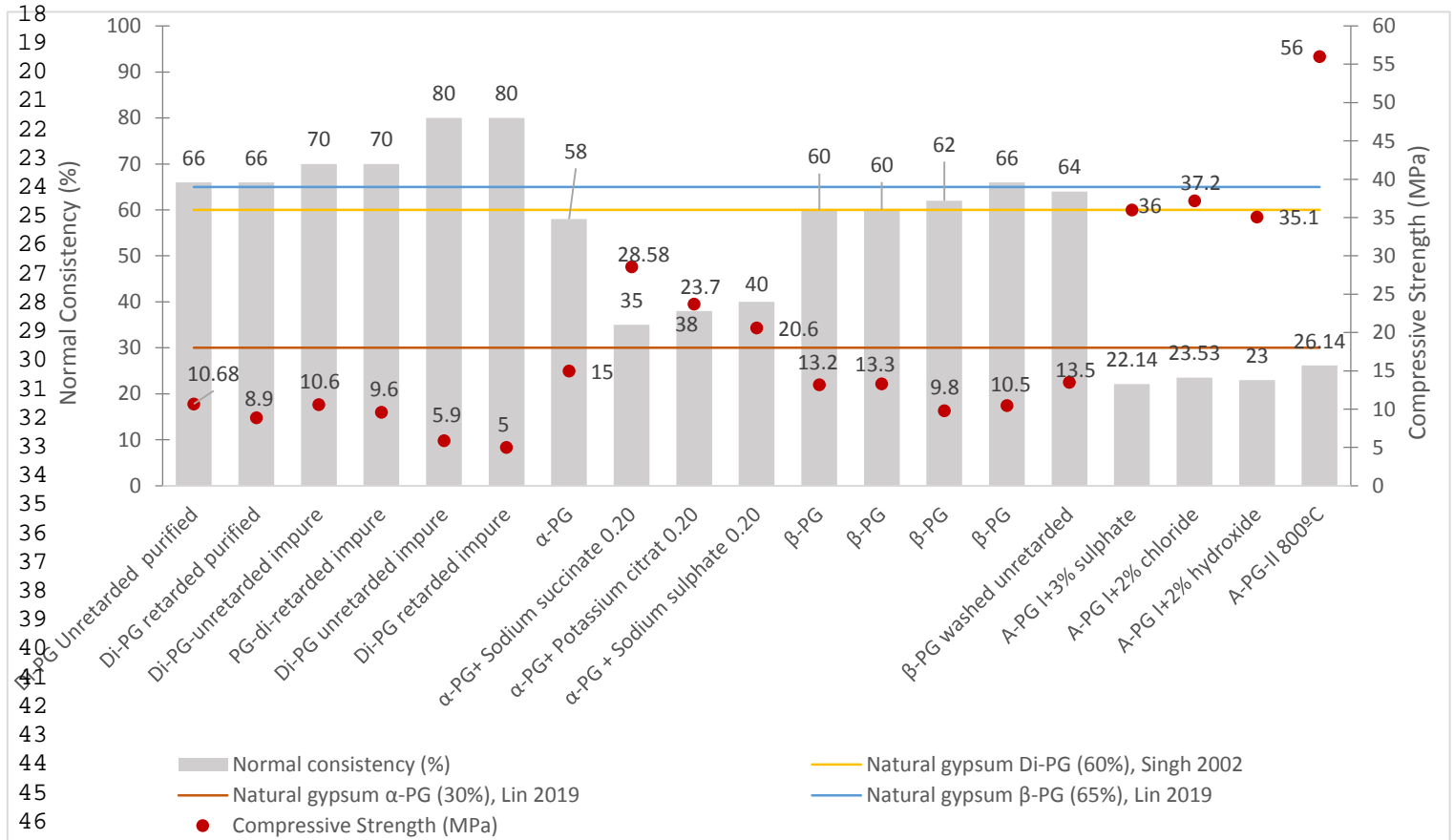


Figure 4: Normal plaster consistency

Figure 5 shows the relation between normal consistency of HCBPG and compressive strength. No results of normal HCBPG consistency were found in the literature for Di-PG, A-PG I and % A-PG III. The normal consistency of the  $\alpha$ -PG + FA + lime system is 44%, and the compressive strength is 26.6MPa when the HCBPG curing temperature is 50°C. The normal consistency of the  $\alpha$ -PG + GGBFS + marble dust + chemical activator system was 41% and the compressive strength

was 32.2 MPa, due to the curing temperature of 50°C [141]. Studies of the  $\alpha$ -PG + FA + lime + PC system (15% + 60 + 5% + 20%) showed that the normal consistency of 22% for the compressive strength of 35MPa [145].

Normal consistency of the  $\beta$ -PG + GGBFS + PC system is 36% and the compressive strength is 35MPa [143]. The  $\beta$ -PG + FA + PC system showed a 34% consistency and a compressive strength of 8.78MPa [146]. The  $\beta$ -PG + FA + 10% lime system has a normal consistency of 34% and a curing temperature of 50°C, and a compressive strength of 22.41MPa [147]. Standard consistency of the  $\beta$ -PG + FA + lime + clay system was 37% and the compressive strength was 27.2MPa [148]. The normal consistency of the 50% A-PG II + 50% GGBFS system was 27.42% and the compressive strength is 35 MPa [149].

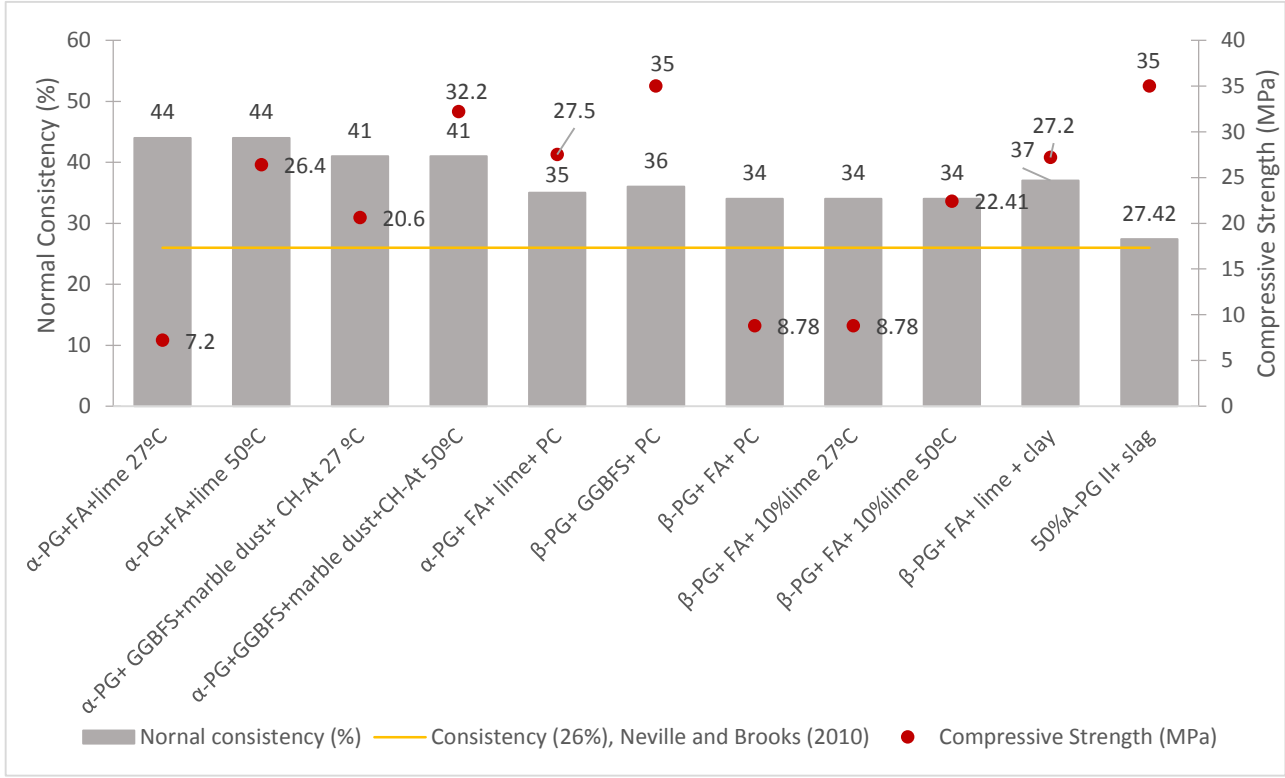
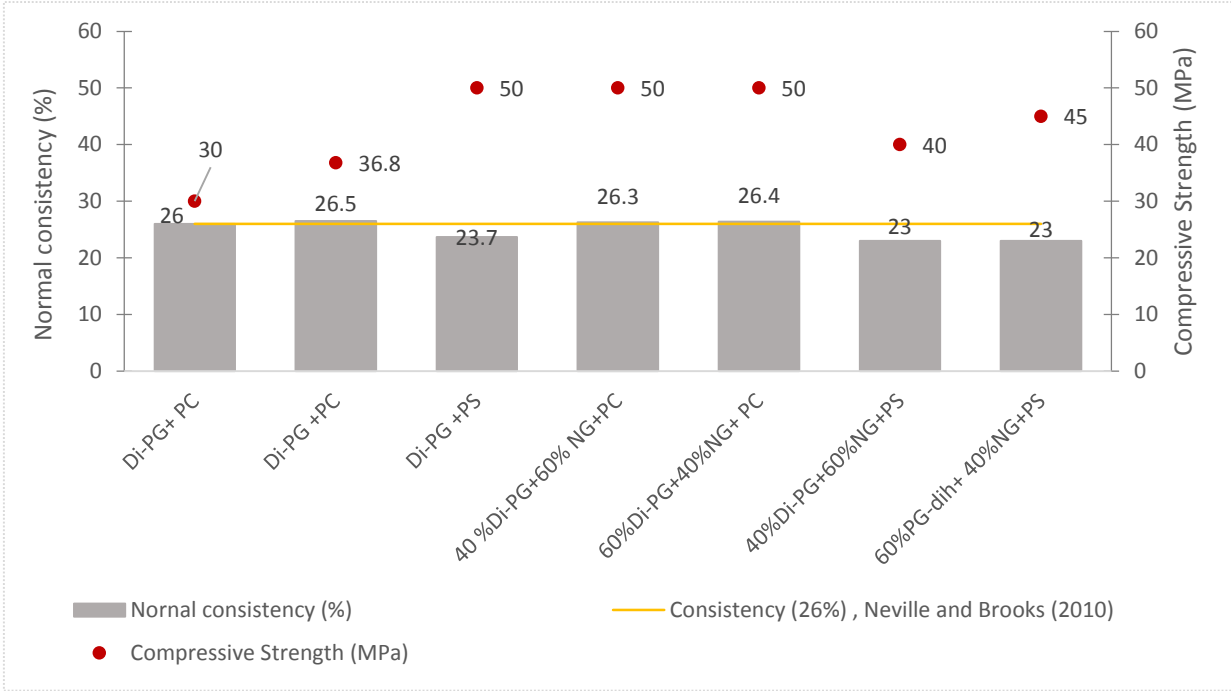


Figure 5: Normal HCBPG consistency

The **Figure 6** shows that studies on normal consistency with partial replacement of Portland cement were performed only with Di-PG. The binary Di-PG + PC system showed a normal consistency between 26% -26.5% and the compressive strength of 30MPa-36.8MPa respectively [27], [150]. The Di-PG-PS binary system had a normal consistency of 23.7% and a compressive strength of 50MPa [150]. The normal consistency of both systems 40% Di-PG + 60% NG + PC and 60% Di-PG + 40% NG + PC is 26.3% and the compressive strength is 50 MPa, despite the percentage of natural plaster used and phosphogypsum, are different in both systems [150].The normal consistency for both systems 40% Di-PG + 60% NG + PS and 60% PG-dih + 40% NG + PS is 23%. However, the compressive strength of the 60% PG-dih + 40% NG + PS system is 45 MPa [150].

**Figure 6: normal consistency in Portland cement with partial PG replacement**



In view of the normal consistency data with PG, it can be concluded that the normal consistency value of natural gypsum [25] is close to the value of the Di-PG phosphogypsum paste and the  $\beta$ -PG plaster. The normal consistency in Di-PG with the presence of impurity, increases the normal consistency. The treated  $\alpha$ -PG plaster has a consistency value close to the value of the untreated  $\beta$ -PG plaster and the reference value [36].

The use of the sodium succinate, potassium citrate and sodium sulphate retardants has an influence on reducing the normal consistency of the  $\alpha$ -PG plaster and on increasing the resistance to compression, in addition, the values found for normal consistency are close to the normal

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consistency value of the reference [36]. References to the normal consistency of phosphogypsum A-PG II and A-PGI was not found, however, it is concluded that the normal consistency is lower when compared to Di-PG,  $\alpha$ -PG and  $\beta$ -PG. It is also concluded that the consistency, curing temperature and mineral phase can influence the mechanical strength of the paste.

The normal consistency of the HCBPG can vary between 37-44% and for all the analysed results, it can be verified that they are above the reference value (26%) [37]. HCBPG with  $\alpha$ -PG have higher values of normal consistency compared to HCBPG with  $\beta$ -PG and A-PG II. It is worth mentioning that the PG in the mineral phases  $\alpha$ -PG and A-PG II and A-PG I has low porosity [67], [143], [149] and therefore the normal consistency will be lower in comparison to the porosity of the mineral phases Di-PG and  $\beta$ -PG [67].

The normal consistency of studies with partial replacement of Portland cement by phosphogypsum varied between 23-26.5%, and the compressive strength of the paste varied between 30-50 MPa. All the studies have values close to the reference [37].

The compressive strength values are directly related to the normal consistency. It is also concluded that the temperature during the curing period and the mineral phase of PG influences the mechanical strength of the paste. It should be noted that the normal consistency with low water requirement, influences the reduction of porosity and the production of cement-based with high performance [151].

## **5.2 Spread flow and slump test**

The Spread flow is a test that assess the ability to deformation a mortar under the action of loads [152]. In the laboratory, spread flow is evaluated with the aid of the flow table and the cone trunk mold (Spread flow test) [153]. The slump test is performed on concrete to assess its fluidity [154]. There are few studies that explain the relation between the components of mortars, their fluidity and slump [155]. It is important to note that the current state of the research has scattered information regarding the flow spread and the slump test. Thus, the found results were of mortars produced with Di-PG,  $\alpha$ -PG,  $\beta$ -PG and A-PGII.

Spread flow data were compared with the American standard ASTM C270 [51] which requires the mortar spread to be between  $110 \pm 5$  mm. The European standards EN 1015-2 [50] relates the density of the mortar in the fresh state with the spreading and requires the spread flow value between 120-175mm.

The standard EN 13454-1 [59] requires the following spread flow: fluid (greater than or equal to 220mm), highly plastic (150-220mm) and thick (110-140mm).

The test slump data were compared with the standard EN 206 [53] and the Neville and Brooks reference [37]. Table 6 and Table 7 shows the data summary referring to the spread flow and the Slump test, respectively.

**Table 6: Summary of the spread flow data**

Mineral phase	Composite binder	Spread Flow (mm)	Ref.
Di-PG	10%Di-PG + PC	300	[156]
	50%Di-PG + PC	175	[156]
	Di-PG + PC+ 1%Lime	301	[157]
	Di-PG +PC + 4% Lime	247	[157]
	Di-PG + FA + lime + PC	342-375	[158]
$\alpha$ - PG	33% $\alpha$ -PG + FA + lime+PC	342-375	[158]
	52% $\alpha$ -PG + FA + lime+PC	190-270	[159]
$\beta$ -PG	$\beta$ -PG+GGBFS+PC	105	[160]
	$\beta$ -PG+FA+PC	105	[160]
	$\beta$ -PG+FA+Clay+lime	100-105	[148]
A-PG III	unkown	unkown	unkown
A-PG II	10%A-PGII + PC	300	[156]
	50%A-PGII + PC	250	[156]
	A-PGII + FA + lime + PC	342-375	[158]
A-PG I	unkown	unkown	unkown

**Table 7: Summary of the Slump test**

Mineral phase	Composite binder	Slump (mm)	Ref.
Di-PG	15%Di-PG+PC	80	[161]
	5%Di-PG+PC <sup>1</sup>	85.45	[162]
	10%Di-PG+PC <sup>1</sup>	80	[162]
	2,5%Di-PG+PC <sup>2</sup>	72.30	[162]
	5%Di-PG+PC <sup>2</sup>	78.5	[162]
	5%Di-PG+PC <sup>3</sup>	68	[162]
	10%Di-PG+PC <sup>3</sup>	62	[162]
	Di-PG + GGBFS + Lime	155	[163]
$\alpha$ - PG	unkown	unkown	unkown
$\beta$ -PG	unkown	unkown	unkown
A-PG III	unkown	unkown	unkown
A-PG II	A-PG II + GGBFS + Lime	55	[163]
A-PG I	unkown	unkown	unkown

<sup>1</sup> Concrete produced with Class of strength M20

<sup>2</sup> Concrete produced with Class of strength M25

The studies show that mortars produced with the  $\beta$ -PG+GGBFS+PC,  $\beta$ -PG+FA+PC and  $\beta$ -PG+FA+Clay+lime systems can achieve a flow between 100-105mm. It was not possible to find the percentage of each component in the literature [148], [160].

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1 Mortars with the formulations Di-PG + FA + lime + PC,  $\alpha$ -PG + FA + lime+PC and A-PGII +  
2 FA + lime + PC had a flow variation between 342-375mm. Each mixture was composed of 33%  
3 PG, 42% FA, 20% PC and 3% lime [158]. Mortar made with the formulation  $\alpha$ -PG + FA + lime +  
4 PC with 52% PG, 33% FA, 10% PC and 3% lime showed that the flow can vary between 190-  
5 270mm [159].  
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9 Mortars with cement replacement by PG, were carried out with Di-PG and A-PGII + PC. The  
10 variation used for PG, for both studies, was between 10-50%. Mortars made with 10% Di-PG had  
11 an approximate flow of 300 mm, while mortars with 50% Di-PG had an approximate flow of 175  
12 mm. Mortars made with 10% A-PG II had a flow of 300 mm, while mortars made with 50% A-  
13 PGII had an approximate flow of 250 mm [156].  
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18 The study with Di-PG + PC + lime showed that the flow can vary between 274-320 mm. The  
19 study consisted of varying the percentage of lime by 0-4%, with 1% substitution by 1%. Mortars  
20 with 1% lime showed a flow of 301 mm, while mortars with 4% lime showed a flow of 274 mm  
21 [157]. The partial replacement of Portland cement by Di-PG at 5% provides a slump of 100 mm  
22 and with 15% of Di-PG the slump can reach 80 mm [161].  
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28 The concrete grades can also be tested for partial replacement of PC with PG. Class M20 concrete  
29 with 5% Di-PG replacement reaches a slump of 85.45 mm, while 10% Di-PG reaches a slump of  
30 80 mm. Class M25 concretes with 2.5% Di-PG can have a slump of 72.3 mm and the use of 5%  
31 Di-PG influences a slump of 78.5 mm. M30 grade concretes with 5% Di-PG can have a 68 mm  
32 slump and with 10% Di-PG the slump can reach 62 mm [162].  
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38 The concrete produced with the Di-PG + GGBFS + Lime system with 17% PG, 82% GGBFS  
39 and 1% lime has a 155 mm slump and 2.5% incorporated air. The A-PGII + GGBFS + Lime  
40 system with 11% PG, 81% GGBFS and 8% lime has a 55 mm slump and the incorporated air  
41 content is less than 2% [163].  
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45 In view of the spread flow data, it can be concluded that: most of the data found on spread flow  
46 comply the standard EN 13454-2 requirements. Mortars can be classified as fluid (great than 220  
47 mm) and used as self-leveling mortars. It was verified that only mortars produced with  $\beta$ -PG +  
48 GGBFS + PC,  $\beta$ -PG + FA + PC and  $\beta$ -PG + FA + Clay + lime cementitious systems, accomplish  
49 the ASTM C270 requirements. Whereas from the EN 1015-2 standard requirements, only, mortar  
50 produced with 50% Di-PG + PC accomplishes the spread flow requirements. It is also verified  
51 that this mortar can exhibit light density (great than 1200kg/m<sup>3</sup>), in the fresh state [152].  
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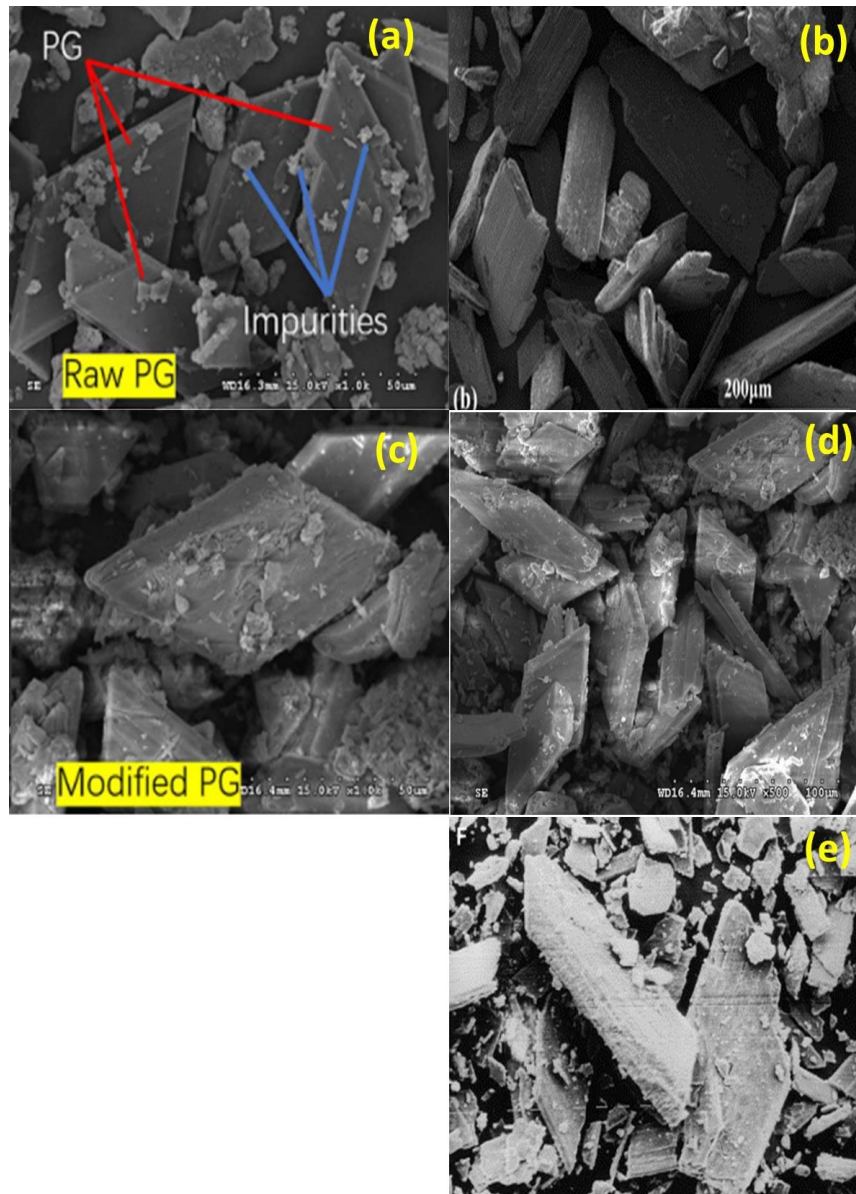


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All the data about slump test of concrete produced with partial replacement of Portland cement by PG and cementitious systems based on PG, are in accordance with the EN 206 standard. Most studies on concretes showed a slump between 55-85.45 mm. These data, according to the consistency class S2 (50-90 mm), are related to the standardized consistency and can be applied in foundation, slabs and general structures. Only the concrete produced with the Di-PG + GGBFS + Lime cementitious system reaches the consistency class S4 (160-210 mm), due to its 155 mm slump. This type of concrete can be used in foundations, slabs, pumping and piling concretes. Furthermore, it is considered a concrete with high workability, due to its fluid consistency, released and compacted with little or no effort and with sufficient cohesion to be handled without segregation or exudation [164].

## 6 Phosphogypsum morphology

Scanning electron microscopy (SEM) has been used to study the morphology of phosphogypsum. By using this technique, it is possible to identify the shape and growth of the crystals. In addition, the formation of the PG microstructure can be monitored [146]. SEM can also evaluate the mineralogical formation of PG [165]. In **Figure 7**, the phosphogypsum in the dihydrate, hemihydrate, anhydrite III, anhydrite II and anhydrite I can be observed.



**Figure 7:** (a) Dihydrate. Reprinted from Gong et al. [157] with permission from ASTM International; (b) Hemihydrate ( $\alpha$ -PG e  $\beta$ -PG). Reprinted from Ma et al. [166] with permission from Elsevier; (c) Anhydrite III. Reprinted from Gong et al. [157] with permission from ASTM International; (d) Anhydrite II. Reprinted from Gong et al. [156] with permission from ASTM International and (e) Anhydrite I [167]. Reprinted from Singh and Garg with permission from Elsevier.

**Figure 7-a** represents phosphogypsum in the dihydrate phase. Raw PG is composed of monoclinic crystals in tubular shape. The small solid particles are adsorbed on the surface. On the surface of the PG crystals, organic matter can be observed, as well as phosphorus and soluble fluorine. These impurities are granular in shape and can affect plaster strength [157].

**Figure 7-b** shows the morphological form of PG hemihydrate ( $\alpha$ -PG and  $\beta$ -PG). The crystalline structure is orthorhombic [67]. After calcination ( $\beta$ -PG) or autoclaving ( $\alpha$ -PG) the impurities present on the surfaces of the Di-PG, become imperceptible. In addition, it is possible to observe some layers in the structure, due to the presence of water molecules that alternate with calcium

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1 sulphate molecules [166]. The crystalline structure of  $\alpha$ -PG requires low water demand and high  
2 mechanical resistance, and this influences the high performance of products that use this mineral  
3 phase. However, it demands the high cost of preparation, which limits its use on a large scale [166].  
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5 In **Figure 7-c**, the morphology of PG in the soluble anhydrite III phase (A-PG III) can be seen.  
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7 The crystalline structure of the A-PG III is hexagonal [67] porous and fluffy. Crystals of A-PG III  
8 are smaller compared to crystals of PG hemihydrate. With calcination, A-PG III reduces porosity  
9 and improves mechanical properties [157].  
10

11  
12 **Figure 7-d** shows the morphology of PG in the insoluble anhydrite II phase (A-PG II). The  
13 crystalline structure of A-PG II is rhombic [67]. According to the literature, despite calcination at  
14 high temperature, small particles of PG dihydrate may still exist. The existing PG Dihydrate  
15 particles are related to the dehydration process. Once PG reaches the last mineral phase (A-PG I),  
16 Di-PG particles are no longer identified. The calcination of A-PG II also contributes to the  
17 reduction of porosity, increases the density of the material and reduces the size of the particles. All  
18 these modifications contribute to the mechanical efficiency of the cement-based PG [156].  
19

20  
21 **Figure 7-e** shows the morphology of insoluble PG anhydrite I (A-PG I). The crystalline structure  
22 is cubic [67]. The stability is reached between 800°C to 1000°C, due to the extreme reduction of  
23  $P_2O_5$  and F. The crystalline structure of A-PG I at 1000°C changes from euhedral to anhedral. The  
24 crystals reduce in size until they reach the form of microcrystals. The definitive modification of the  
25 crystalline structure from A-PG III to A-PG I can be considered a decisive factor for the optimal  
26 development of the mechanical strength of cement-base PG. In addition, a significant increase in  
27 density, specific surface area and pH can be seen in A-PG I. However, the loss of mass by ignition  
28 is reduced [167].  
29

30  
31 In view of the data found in the literature, it is concluded that the PG impurities can be seen  
32 through scanning electron microscopy, and the purification can also be detected, as the PG  
33 modifies the mineral phase. According to the review data calcination also influences the crystalline  
34 structure modification in each mineral phase. It is not possible to identify such changes in the  
35 figures presented in this work. It is suggested that the microscopy analysis was performed with the  
36 PG in the powder aspect. However, if the PG was hydrated, changes in the crystalline structure,  
37 due to the influence of temperature, could be identified, as occurred in the study by Geraldo et al.  
38 [168].  
39

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### 7 Setting Time

Setting time is the term used to define the time that cementitious materials (e.g. paste, mortar and  
concrete) take to change from the fresh (fluid) to the hardened state [37]. The presence of 0.1%

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1 fluoride in the cement influences the setting time by approximately 60 minutes. In addition, the  
2 use of fly ash and blast furnace slag, in the cement-based composition, can affect the setting time  
3 increase. However, the addition of lime can cause slightly shorter hardening time [169].  
4

5 Figures 8, 9, 10, show the initial and final setting time of the plaster, HCBPG, and the partial  
6 replacement of cement by phosphogypsum, respectively. It was not possible to compare the setting  
7 time of phosphogypsum dihydrate (Di-PG), because in the literature the natural plaster dihydrate  
8 is considered a mineral in the raw state and it is not commonly used in civil construction [91]. The  
9 existing specifications correspond to that of construction gypsum plaster ( $\beta$ -PG), which undergoes  
10 the calcination process [36], [89].  
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12 The plaster setting time was compared with the setting time described in the European standard  
13 EN 13279-1 [170] and the American standard ASTM C28 [39]. The European standard EN13279-  
14 1 considers setting time only in the plaster and determines setting time manual (great than 20 min)  
15 and setting time mechanical (great than 50 min), and makes no reference to the final setting time  
16 [38]. While the American standard ASTM C28 considers that the setting time shall be neither less  
17 than 20 nor more than 40 minutes in no retarded Gauging Plaster. In Gauging Plaster with retarded,  
18 the initial setting time shall be not less than 40 minutes.  
19

20 The standards EN 15743 [42], EN 14216 [41], EN 14647 [40], ASTM C91 [43], ASTM C1600 [45]  
21 and ASTM C 595 [44] were used as a reference to compare the setting time of cementitious systems.  
22

23 The standard EN 15743 refers to Supersulphated cement and classifies the setting time of the  
24 according to compressive strength. Following the classification: (i) the compressive strength greater  
25 than or equal to 32MPa and 52 MPa must have a setting time greater than or equal to 75 minutes.  
26 (ii) the compressive strength greater than 42.5 MPa and 62.5 MPa must have a setting time greater  
27 than or equal to 60 minutes. (iii) the compressive strength greater than or equal to 52.5 MPa must  
28 have a setting time greater than or equal to 45 minutes.  
29

30 The standard EN 14216 refers to Low heat cement and classifies the setting time according to the  
31 nominal compressive strength. The compressive strength between 22.5-42.5 MPa must have an  
32 initial initial setting time equal to 75 minutes. While standard EN 14647 refers to Calcium aluminate  
33 cement and classifies the setting time according to compressive strength and initial time of the  
34 hardening of cement. The standard require that the initial setting time should occur in 90 minutes.  
35 In addition, considers the compressive strength greater than 18 MPa in 6 hours and greater than  
36 40 MPa in 24 hours.  
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38 The standard ASTM C91 refers to Masonry cement and classifies the setting time according to  
39 three different types of cement. The Cement N (initial setting time not less than 120 me and final  
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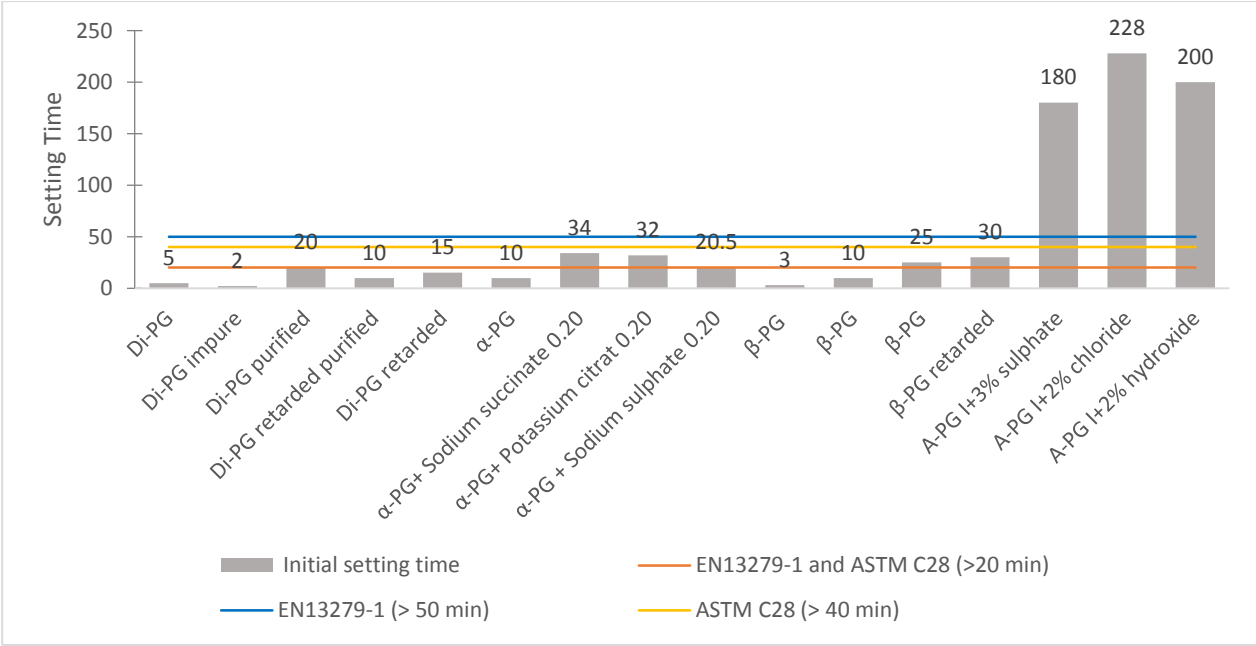
setting time not more less 1000mm) and the cements S and M (initial setting time not less than 90 me and final setting time not more less 1000mm).

The standard ASTM C1600 refers to Rapid hardened hydraulic cement and accept that the setting time of ranges from 10 to 45 min. The standard ASTM C 595 refers to Blended hydraulic cements and determines that the setting time not less than 7 minutes and not more than 45 minutes.

The European standard EN 197-1 [46] and the US standard ASTM C150 refers to Portland cement. Both standard accept the initial setting time in 45 minutes and only standard ASTM C150 [47] determines the final setting time in 375 minutes

**7.1 Setting time of plaster**

In **Figure 8**, it is possible to observe the initial and final setting time of the plasters Di-PG,  $\alpha$ -PG,  $\beta$ -PG and A-PGI and A-PGII. When comparing the setting time of natural calcium sulphate dihydrate (NG) with synthetic calcium sulphate (PG), the initial setting time of PG is less than the initial setting time of NG. It is noteworthy that only the data for the initial setting time were found.



**Figure 8: Plaster of setting time**

According to the literature, Di-PG has an initial setting time between 2 and 5 minutes in tthe case of absence of retarder and impurities [25], [171]. Di-PG without purification treatment and retarder increases the setting time by 20 minutes. The initial setting time of Di-PG with retarder and purified

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is 10 minutes [25], while the initial setting time of Di-PG with retarder and without purification is 15 minutes [96].

In  $\alpha$ -PG the initial setting time is 10 minutes, however, the use of retarder, promotes an increase in the setting time. With 0.20% sodium succinate, the initial uptake time of  $\alpha$ -PG is 34 minutes. A-PG with 0.20% potassium citrate the initial setting time is 32 minutes and  $\alpha$ -PG as 0.20% sodium sulphate the initial setting time is 20.5 min [141].

The initial setting time of  $\beta$ -PG can vary between 3 minutes and 25 minutes [96], [141], [143], [172]. The initial setting time of the  $\beta$ -PG with retarder is 30 minutes [96]. It should be noted that A-PG I (calcined at 1000°C), has the longest initial setting time, among all the studied mineral phases. It can be seen that the initial setting time for A-PG I was 228 minutes, with 2% chloride. In addition, it appears that the initial setting time of the A-PG I can vary between 180 and 228 minutes.

It can be concluded that when Di-PG has impurities in its chemical composition, it has a very fast initial setting time (2-5 minutes), making difficult its use. Di-PG without impurities or with setting retarder, increases both setting time and efficiency. It is important to comment that the data found on the Di-PG setting time cannot be compared due to the absence of data on the setting time of natural plaster.

Only the data related to the purified Di-PG,  $\alpha$ -PG (sodium and potassium),  $\beta$ -PG (with and without retarder) accomplish the requirements. A-PG I (with retarder) does not accomplish the prescriptions as the initial setting time is higher than that of recommended by the standards.

## **7.2 Setting time of hydraulic cement-based on phosphogypsum (HCBPG)**

The setting time of phosphogypsum-based hydraulic binders (HCBPG) was studied. Ternary, quaternary systems and cement formulations for Supersulphated cement were identified. Most of the mineral phases used in the composition of PGHB were: Di-PG,  $\alpha$ -PG,  $\beta$ -PG and A-PG II. In addition, different types of initial and final setting times can be noted, and three categories were created to understand the hardening speed of PGHB. The categories are: fast (7-45m), medium (60-75m) and low (90-120m). The catch time data for the rapid and medium categories can be viewed in figure 9, while figure 10 shows the catch time data for the slow category.

It can be seen in **Figure 9** that the Di-PG + FA + PC system presented initial and final setting time of 45 and 90 minutes respectively [160]. For the  $\alpha$ -PG + FA + lime and  $\alpha$ -PG + GGBFS + marble dust + chemical activator systems only the initial setting times were found, being 24 and 17 minutes, respectively [141].

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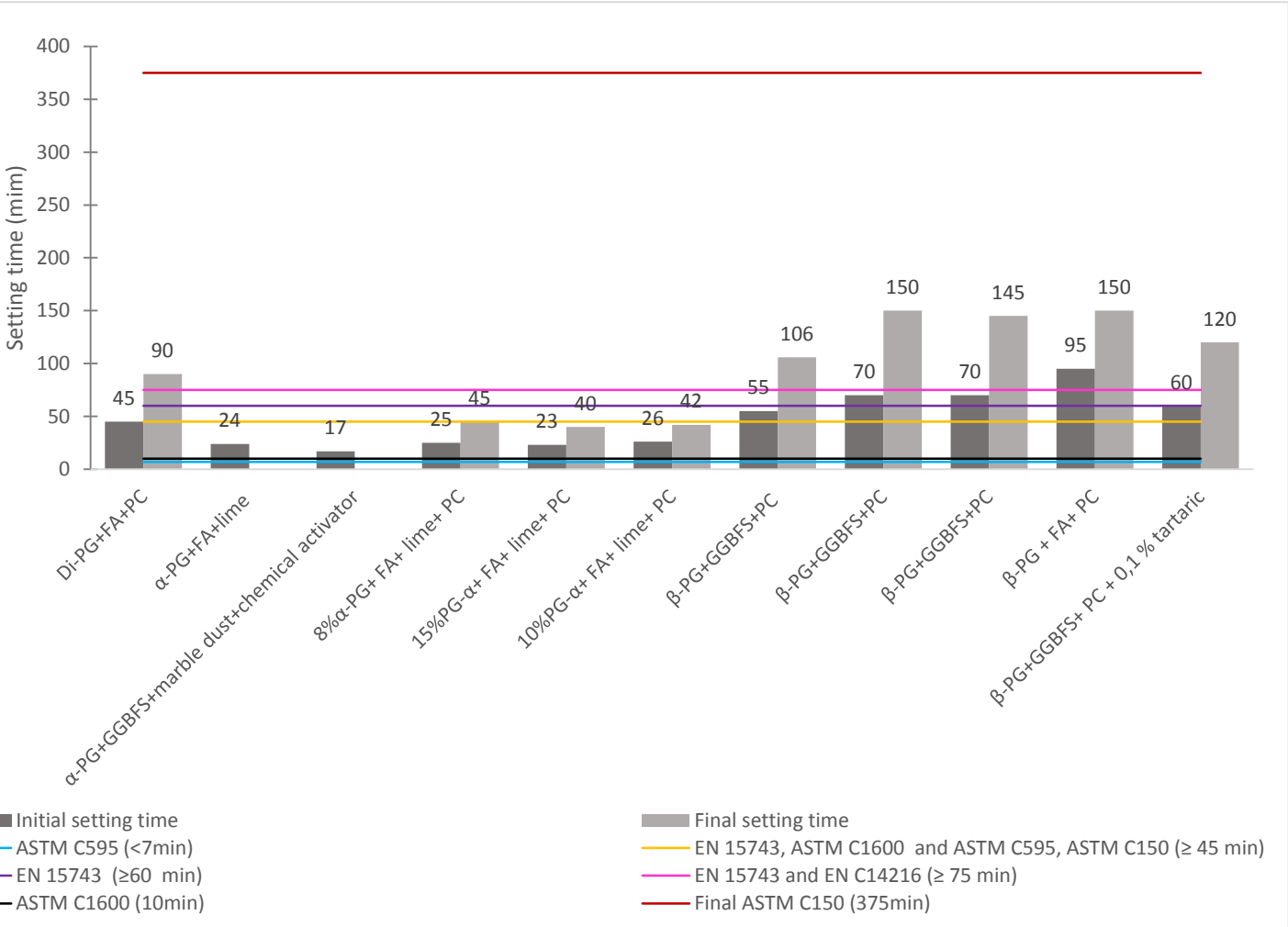
The  $\alpha$ -PG + FA + lime + PC system has the lowest initial setting time when using 15%  $\alpha$ -PG, and the longest final setting time when using 8%  $\alpha$ -PG [145]. In the  $\beta$ -PG + GGBFS + PC system, the initial setting time can vary between 55 minutes and 70 minutes and the final setting time can vary between 106 minutes to 150 minutes [143], [160], [173], [174]. The  $\beta$ -PG + GGBFS + PC + 0.1% tartaric system has an initial setting time of 60 minutes and a final setting time of 120 minutes.

It can be observed in figure 9 that don't have cementitious systems meet the initial setting time requirements of standard ASTM C595 (<7 min). The cementitious systems  $\alpha$ -PG + FA + lime,  $\alpha$ -PG + GGBFS + marble dust + chemical activator, 8%  $\alpha$ -PG + FA + lime + PC, 10%  $\alpha$ -PG + FA + lime + PC meet the standards ASTM C16000 (10-45min). In addition, the aforementioned cementitious systems can be classified as agglomerates with a fast setting time (10-45 min).

The initial setting time of the cementitious systems Di-PG + FA + PC,  $\beta$ -PG + GGBFS + PC,  $\beta$ -PG + FA + PC and  $\beta$ -PG + GGBFS + 0.1% tartaric acid are according to the requirements of the standards EN 15743, ASTM C1600, ASTM C595 and ASTM C150. However, the final setting time of the cementitious systems only meets the standards EN 15743 and EN 14216. It should be noted that the cementitious systems can be classified as a binder with an medium setting time (60-75

mim). And the final setting time of all the cementitious systems meet the requirements of the standard ASTM C150 (375 mim).

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**Figure 9: Fast and medium setting time of hydraulic cement-based on phosphogypsum**

It can be seen in **Figure 10** that the A-PG II + GGBFS system (calcined at 800°C) has an initial time of 210 minutes and a final setting time of 360 minutes [142]. It should be noted that the initial and final setting times of A-PG II + GGBFS system (calcined at 750°C) may vary depending on the proportion of PG and GGBFS used. The longest setting times occur with 70% A-PG II and 24% GGBFS. The shortest initial and final setting times occur with 50% A-PG II and 50% GGBFS [149].

Besides, it possible verify that the A-PG II + Slag + lime system [175] present an initial setting time of 259 min and a final setting time of 550 min. The Di-PG + slag + Lime system [175] presents the initial setting time of 365 min and the final setting time of 580 min. The 10% A-PG II + 2% Di-PG + slag + lime system [175] has an initial setting time of 330 min and a final setting time of



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555 min. While the Di-PG + Steel Slag + limestone system [176] has an initial setting time of 485 min and a final setting time of 717 min.

The use of alkaline activators in the composition of cementitious systems and its the influence on the initial and final setting time are also depicted in **Figure 10**. The Di-PG + SS + LS + NaOH system [177] has an initial setting time of 129 min and a final setting time of 252 min. The Di-PG + GGBFS + Alkali activator + sodium silicate + sodium hydroxide system [178] has an initial setting time of 48 min and a final setting time of 222 min. The Di-PG + GGBFS + Alkali activator + Sodium hydroxide system (27.8) [178] has an initial setting time of 90 min and a final setting time of 318 min, while the Di-PG + GGBFS + Alkali activator + Sodium hydroxide (18.5) [178] has an initial setting time of 108 min and a final setting time of 300 min.

The initial setting time with HCBPG was compared to the requirements of the European standard EN 15743 [179] as the Supersulphated cement is composed of calcium sulphate, Portland cement and blast furnace slag. In addition, the American standard ASTM C 595 [44] was also used in the comparison, due to its specifications about ternary systems.

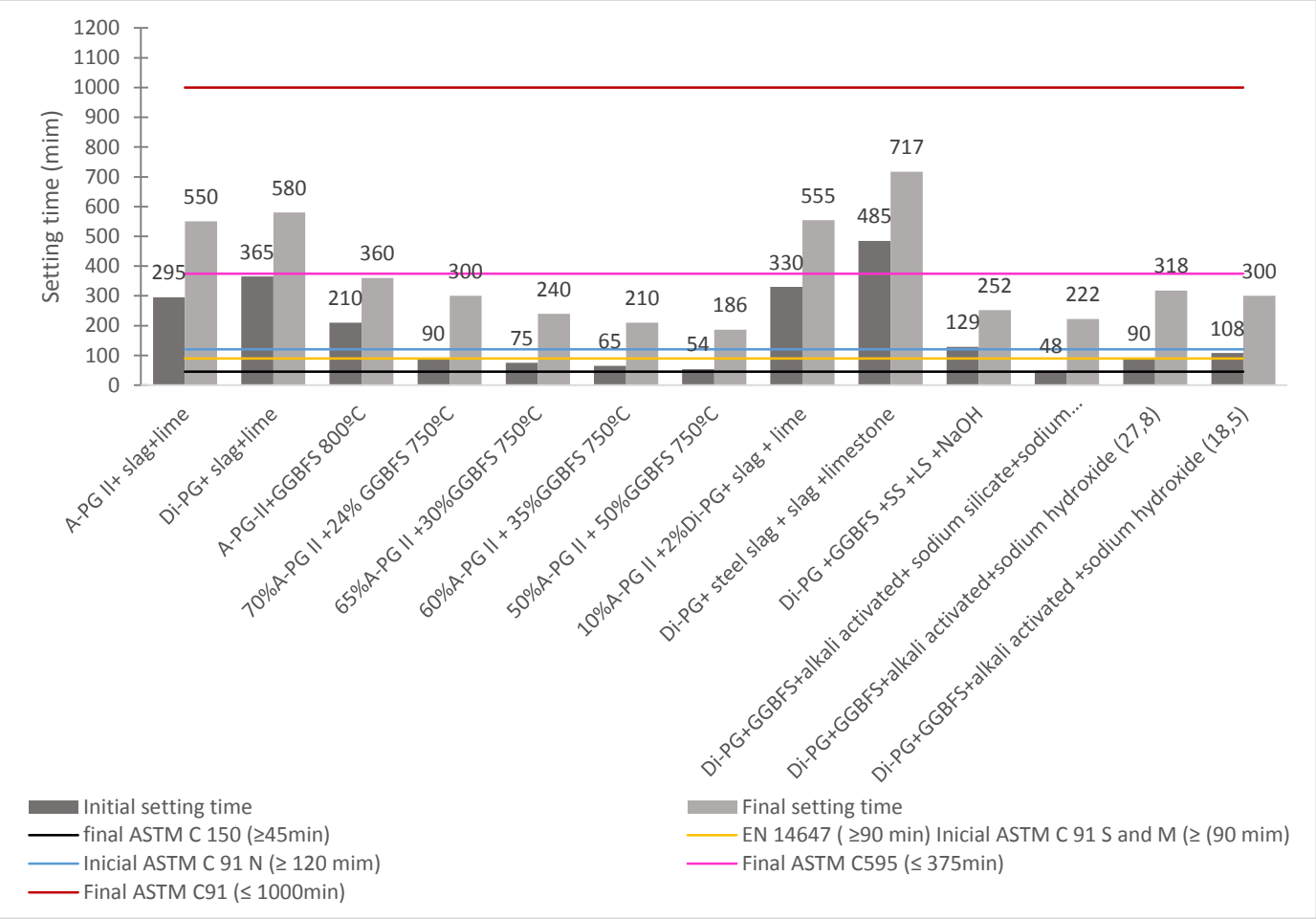
It can be seen that all the data found on the HCBPG initial setting time are in accordance with the ASTM C 595 standard [44] requirements. It can be concluded that the systems  $\alpha$ -PG + FA + lime,  $\alpha$ -PG + GGBFS + marble dust + chemical activation, 8%  $\alpha$ -PG + FA + lime + PC, 15%  $\alpha$ -PG + FA + lime + PC, and 10%  $\alpha$ -PG + FA + lime + PC do not accomplish the requirements of the initial setting time prescribed by the standard EN 15743 [179]. Most cement systems accomplish the final setting time of ASTM C 595, overtaking the established limit.

it is important to mention that all cementitious systems can be classified as a binder with an low setting time. Besides, only the initial setting time of cementitious systems A-PG II + slag + lime, Di-PG + slag + lime, A-PG II + GGBFS 800°C, 10% A-PG II + 2% Di-PG + slag + lime, Di-PG + steel slag + slag + limestone meet the requirements of the standard ASTM C91 (great equal to 120 min). In addition, can be seen that the initial setting time of cementitious systems 70% A-PG II + 24% GGBFS 750°C, 65% A-PG II + 30% GGBFS 750°C, 60% A-PG II + 30% GGBFS 750°C, 50 % A-PG II + 50% GGBFS 750°C and Di-PG + GGBFS + alkali activated + sodium silicate + sodium hydroxide (20) (Supersulphated cement) do not meet the requirements of EN14647 and ASTM C91 (great equal to 90 min).

Only the final setting time of cementitious systems A-PG II + slag + lime, Di-PG + slag + lime + 10% A-PG II + 2% Di-PG + slag + lime, Di-PG + steel slag + slag + limestone ASTM C150 requirements. It should be noted that all cementitious systems can be classified as agglomerates

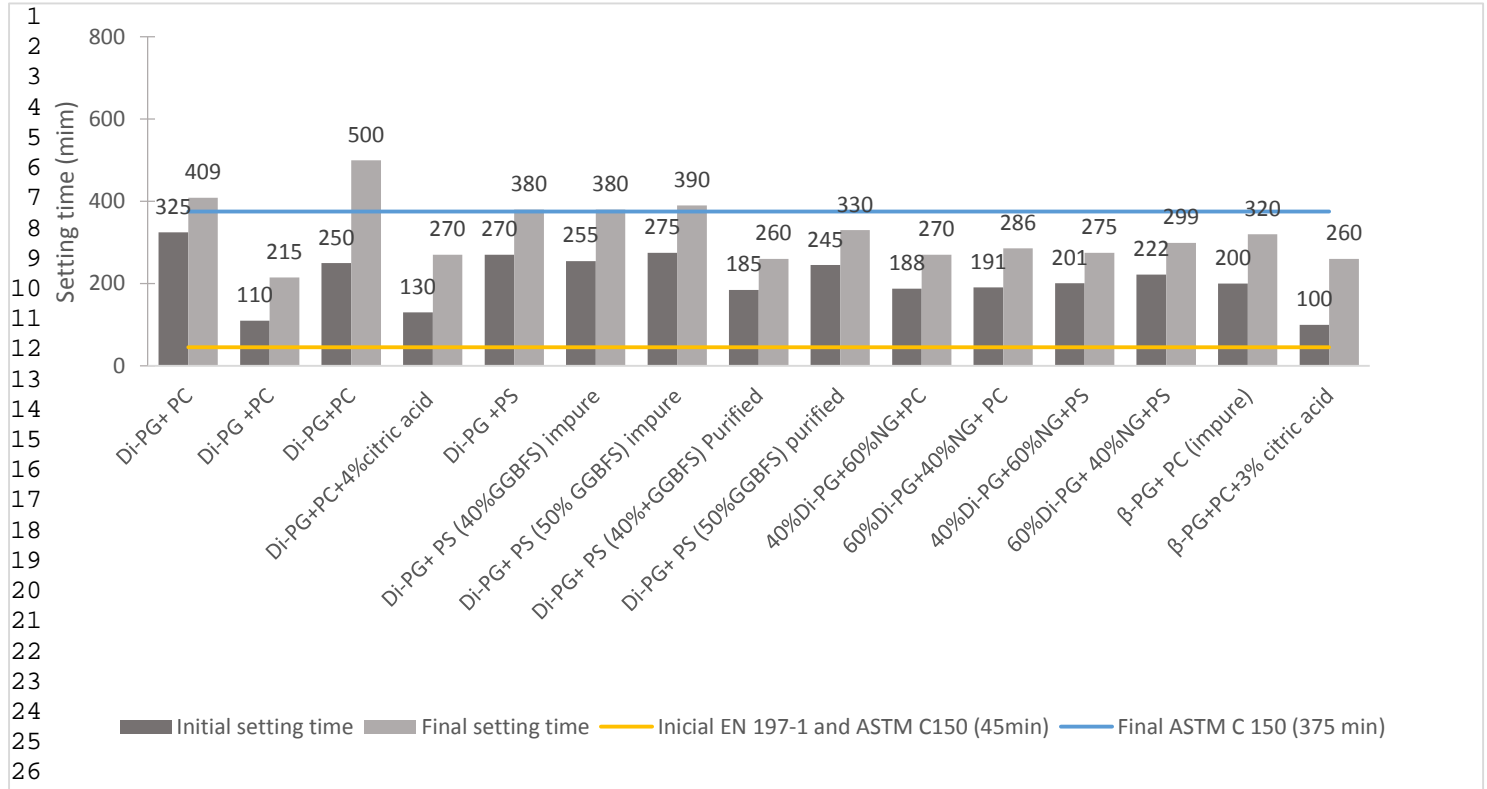
with a slow setting time and meet the final setting time of the ASTM C91 standard (less than 1000 min).

**Figure 10: Slow setting time of hydraulic cement-based on phosphogypsum**



**7.3 Setting time of Portland cement**

Figure 11 shows the initial setting time and the final setting time of Portland cement with partial replacement of PG. It is observed that the mineral phase found in most studies was Di-PG, and the percentage of substitution used by researchers was 5% PG [25], [150], with the exception of the Di-PG system + PC that used only 1% replacement of Portland cement with PG [27].



**Figure 11: Setting time of cementitious systems with Portland cement**

It is noted that the initial setting time can vary between 110 and 325 minutes [25], [27], [150]. While the final setting time can vary between 215 and 500 minutes. Most Portland cements were produced with five percent phosphogypsum. Only the PC with an initial setting time of 110 m and a final setting time of 215 min used one percentage of phosphogypsum in the composition.

Di-PG treated with 4% citric acid has an initial setting time of 130 minutes and a final setting time of 270 minutes [25]. In the Di-PG + 40% GGBFS + 60% Clinker system (Di-PG + PS), the initial setting time was 255 minutes and the final setting time was 380 minutes [150]. The Di-PG + 50% GGBFS + 50% Clinker system (Di-PG + PS) presented the initial setting time of 275 minutes and the final setting time of 390 minutes. It appears that the Di-PG + OS system has a longer setting time and a final setting time, if compared to the Di-PG + PC system.

Purification of Di-PG + 40% + GGBFS + 60% Clinker (Di-PG + PS) and Di-PG + 50% GGBFS + 50% Clinker (Di-PG + PS) systems with citric acid causes a reduction in the initial and final setting time. The Di-PG + 40% + GGBFS + 60% Clinker system had an initial setting time of 185 minutes and a final setting time of 260 minutes. Meanwhile, the Di-PG + 50% GGBFS + 50% Clinker system presented an initial setting time of 245 minutes and a final setting time of 330 minutes [25]. It is noted that the purification of the systems and Di-PG + 40% + GGBFS + 60%

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1 Clinker and Di-PG + 50% GGBFS + 50% Clinker contributed to the reduction of the initial setting  
2 time and the final setting time.

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4 Data of the mixture of PG + NG were also found in the literature. The initial setting time of the  
5 40% Di-PG + 60% NG + PC system was 188 minutes, and the final setting time was 270 minutes.  
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7 The 60% Di-PG + 40% NG + PC system has an initial setting time of 191 minutes and a final  
8 setting time of 286 minutes. It is suggested that the increase in the percentage of PG in the system  
9 may influence the increase in the initial and final setting times [150].  
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13 The 40% Di-PG + 60% NG + PS system has an initial setting time of 201 minutes and a final  
14 setting time of 275 minutes. While the 60% Di-PG + 40% NG + PS system presented an initial  
15 setting time of 222 minutes and a final setting time of 299 minutes [150]. The increase in the initial  
16 and final setting times may occur due to the increase in the percentage of PG. In addition, the  
17 addition of GGBFS in the cement can also influence the increase in the initial and final setting time  
18 [169].  
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23 The  $\beta$ -PG + PC system presented the initial setting time of 200 minutes and the final setting time  
24 of 320 minutes [25]. Although the  $\beta$ -PG plaster has a initial setting time between 5-8 minutes and  
25 the final setting time is less than 30 minutes [36] and being considered a material of fast hardening,  
26 it appears that there was no influence on the reduction of the initial and final setting time of the  $\beta$ -  
27 PG + PC system. However, it can be observed that the treatment with 3% citric acid, can contribute  
28 to the reduction of the initial and final setting time [25].  
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34 Regarding the initial setting time of Portland cement with PG replacement, the obtained data are  
35 in accordance with the American standard ASTM C150 [47] and the European standard EN 197-  
36 1[46]. Di-PG + PC, Di-PG + PS, Di-PG + 50% GGBFS + 50% Clinker, Di-PG + 40% GGBFS  
37 + 60% Clinker systems do not accomplish the ASTM C150 standard final setting time requirements  
38 [47].  
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## 45 **8 Phosphogypsum-based cementitious systems**

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47 Cementitious systems can be composed of binary, ternary or quaternary mixtures [180]. Binary,  
48 ternary or quaternary designations indicate the number of materials or fillers that are included in  
49 the cement mixture, without indicating its nature or content [181]. During cement production, the  
50 clinker may be partially replaced by materials such as fly ash, blast furnace slag and limestone to  
51 produce composite cement. Construction industry constantly seeks to develop cement mixtures  
52 and ways to standardize mixtures. In addition to seeking material availability by region [182], [183].  
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1 International standards for the development of non-cement Portland have been studied by the  
2 RILEM Technical Committee 224-AAM, in partnership with business and society to achieve  
3 satisfactory results [184]. Investigations on calcium sulphate-based cement systems have become a  
4 promising alternative to produce cement and concrete at room temperature, due to the availability  
5 of raw materials.  
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9 In addition, combining hemihydrate or anhydrite calcium sulphate with moderate amounts of  
10 calcium aluminate or silica contributes to stabilize the cementitious system. High calcium sulphate  
11 cements are better suited for work on fiberglass reinforcement than steel reinforcement [185].  
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14 Thus, it is observed that the study of calcium sulphate-based cement systems is a promising field  
15 and contributes to the production of cements with greater eco-efficiency.  
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### 18 **8.1 Studies on phosphogypsum pastes**

19 Most studies focused on phosphogypsum are related to the control of the setting time and the  
20 microstructure properties [77], [117], [186]–[188]. In view of this fact, a comprehensive literature  
21 search was conducted to list the different possibilities of producing Hydraulic Cement Based on  
22 Phosphogypsum-based (HCBPG) applied in paste application. The results obtained can be seen in  
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27 **Table 8- Appendix A.**  
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29 As far as cementitious formulations are concerned, it is observed that almost all the studies were  
30 developed by using ternary (T), quaternary (QA) and Supersulphated cement (SSC) systems,  
31 although Belz et al. [189] and Hua et al. [190] developed binary systems. Regarding the use of  
32 phosphogypsum, dihydrate (Di-PG) and  $\beta$ - hemihydrate ( $\beta$ -PG) phases are found in almost all the  
33 works. The anhydrite phase (A-PG) appears only in the works of Singh and Garg [142], Erdem  
34 and Olmez [191], Singh and Garg [149], [173] and Michel et al. [192]. However, PG  $\alpha$ - hemihydrate  
35 ( $\alpha$ -PG) was not found in any paste study.  
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38 As aforementioned, there are impurities in the chemical composition of PG dihydrate, and because  
39 of this, the mechanical performance of HCBPG might be compromised. The quality of materials  
40 produced with PG dihydrate can also be influenced by their porosity, as PG dihydrate is composed  
41 of interconnected crystals, with porosity up to 70% and its mechanical properties are influenced  
42 by porosity [193].  
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45 The works with PG  $\beta$ -hemihydrate show better performance compared to PG dihydrate, due to  
46 modifications of its crystal structure [66]. Furthermore, its brittleness after heat treatment can be  
47 attenuated by reducing its porosity, increasing the specific area of the particles, and increasing the  
48 speed of mechanical development [194]. Meanwhile, PG  $\alpha$ -hemihydrate, with autoclaving, will  
49 present a higher degree of inter-crystalline bonding and lower porosity, bearing a higher degree of  
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1 resistance [195]. Anhydrite phase can react with water and convert to plaster, and therefore to be  
2 used in building materials. Although the setting time is slow, its reactivity can be increased  
3 considerably after grinding, by increasing the specific area and adding activators [196].  
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5 The fly ash (FA) and blast furnace slag (GGBFS) were the most by-products used in PG-based  
6 cementitious systems [197], [198]. The silica fume (SF) [199]–[201], bottom ash (BA) [202] and  
7 carbonate rock (CSR) [200] were in some studies.  
8

9 The use of ordinary Portland cement (OPC) has been found in almost all the works. However, the  
10 work by Liu and Wang [203] uses the PC with blast furnace slag, while Gaiducis et al. [200] used  
11 the PC with limestone filler. Replacing OPC with industrial by-products during cement production  
12 contributes considerably to reducing CO<sub>2</sub> emissions, because less clinker will be consumed [204].  
13

14 Between 2017-2019, most publications are related to the development of Supersulphated cements  
15 (SSC). This fact reflects that researchers have shown interest in defining the appropriate content  
16 of phosphogypsum in cementitious systems, such as SSC [179], [205]–[208]. Sulfoaluminate cement  
17 (CSA) and Calcium aluminate cement (CAC) have been used also in recent research [209],  
18 [210]. These two types of cements are also considered more eco-efficient materials due to their low  
19 energy consumption and low carbon dioxide emissions [185], [211].  
20

21 Research on HCBPG shows that it is possible to develop ternary cement systems without the  
22 presence of Portland cement [73]. Different authors have developed formulations based on  
23 phosphogypsum (PG), fly ash (FA) and/or blast furnace slag (GGBFS), hydrated lime and/or  
24 alkaline activating solutions ([103], [142], [148], [212]–[215]). Due to the use of alkaline activators,  
25 the cementitious systems also acquire pozzolanic characteristics [216], [217].  
26

27 It is concluded that most of the studies were with ternary, quaternary and Supersulphated cement  
28 systems. PG dihydrate and PG  $\beta$ -hemihydrate were the most used mineral phases. The use of PG  
29 dihydrate influences material quality due to impurities and porosity. PG  $\alpha$ -hemihydrate has lower  
30 porosity compared to PG  $\beta$ -hemihydrate and, therefore, HCBPG may have higher mechanical  
31 resistance. HCBPG with anhydrous PG have higher setting time. The use of Portland cement  
32 predominated, however, studies were also performed with Calcium aluminate cement (CAC),  
33 Calcium sulfoaluminate cement (CSA). Fly ash (FA) and blast furnace slag (GGBFS) were the  
34 supplementary cementitious materials used in all the studies. Also, it can be noted that lime was  
35 the most used alkaline activator.  
36

## 37 **8.2 Studies on phosphogypsum mortars**

38 Cement-based mortars are cementitious materials characterized by the mixture of cement paste  
39 and sand [218]. They can be used in different applications such as outer coating, inner coating or  
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consolidation injections. Each type of application has different requirements despite of using the same building materials [219].

Research on the application of phosphogypsum in mortars aimed at identifying the study parameters (water/binder, binder/sand and spread) used for the preparation of the product, as well as the most used mineral phases, the type of cement, the presence of cementitious systems and the mixing ratio. The results obtained are presented in **Table 9-Appendix B (from 1992 to 2020)**.

Based on the information found in the literature, various industrial by-products with phosphogypsum can be combined to produce mortars. However, it is observed that the percentage of used materials can vary considerably, especially the percentage related to PG, OPC, FA and GGBFS. PG ranges from 10% to 90%, GGBFS ranges from 1% to 92%, FA 1.5% to 60%, PC 3% to 90%, Lime 1% to 50%, and SS 5% -13, 6%. Moreover, the works by Singh and Garg [160] and Wang et al. [220] did not provide data on formulations. And Singh and Garg [174] reported only the percentage of PG that was used.

The review has also shown that several types of phosphogypsum mineral phases have been studied. Dihydrate phosphogypsum (Di-PG) was used in almost all the works, followed by the phases  $\beta$ -hemihydrate (calcined) and  $\alpha$ -hemihydrate (autoclaved). Ren [159] and Hyung [221] employed phase II anhydrite. Mun et al [175] used PG anhydrite II. The study by Mun et al.[222] evaluated the effect of PG on dihydrate, anhydrite (II and III) and  $\beta$ -hemihydrate phases for all the manufactured cement systems. No studies have been found with anhydrite I. The fact that PG dihydrate appears in most mortar studies suggests an attempt to value PG through more economical techniques, since dehydration (calcination, autoclaving and semi-drying) is an expensive process [223].

Most cementitious systems were composed of ternary and quaternary systems, including the use of blast furnace slag (GGBFS) and/or fly ash (FA), hydrated lime and/or Portland cement. The works by Mun et al. [175], Huang and Lin [176], Hyung et al. [221], Li et al [224] and Wang et al. [220] developed ternary systems for phosphogypsum-based hydraulic binders, without clinker. Gong et al. [156] produced mortars using binary systems (PG-PC).

Around 330Mt/year of GGBFS and 900Mt/year of FA are generated [211]. Due to the high global availability, the low cost and the high-quality control FA and GGBFS are considered basic raw materials for the formulation of eco-efficient cement and cement systems [225], [226]. Portland cement was the only hydraulic binder used in cement-based mixtures. Despite the diversity of cement types on the market [204], due to the standardization of research on Portland cement (PC), other types of cement are no longer used.

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Therefore, it can be concluded that the formulations used in mortars have very different formulations. PG dihydrate is present in most studies, however studies have also been performed with anhydrite (II and II) and hemihydrate ( $\alpha$ -PG and  $\beta$ -PG). The analysed cementitious systems contain the following compositions: (I) without Portland cement: PG-FA-lime; PG-GGBFS-lime; PG-GGBFS-FA-lime, (II) with Portland cement: PC-PG-FA-lime; PC-PG-GGBFS-lime; PC-PG-GGBFS-FA-lime. The other systems found were binary (PC-PG) or systems developed for Supersulphated cements (GGBFS-PG-PC). Only Portland cement was used, while fly ash (FA) and blast furnace slag (GGBFS) were the supplementary cementitious materials used in all studies.

### 8.3 Studies on phosphogypsum concretes

Concrete is a building material produced from the mixture of fine aggregate, coarse aggregate and cement. Mineral additions and chemical additives are also used to improve concrete performance [227]. Constant research is carried out in order to develop more eco-efficient concretes, and for this, industrial waste and agro-industrial waste are used [228]–[230].

**Table 10-Appendix C** provides relevant concrete studies that used PG. Between 1999-2019, 14 articles were published and most of them used Portland cement as raw material. Almost all the studies are related to the partial replacement of Portland cement by phosphogypsum [231], [232]. The results demonstrated that the properties of synthetic gypsum cement are similar to those of obtained by using natural gypsum [233].

Although PC prevails in the concrete production, the study by Tian et al. [234] highlights from an eco-efficient framework, as it manufactures concrete with less environmental impact. By using phosphogypsum-based Sulfoaluminate cement (CSA), they produced a material that consumes less energy, emits less CO<sub>2</sub> and has a lower cost. That is due to a greater incorporation of gypsum (15-25%) [46], [107], [235] compared to Portland cement which incorporates only 5% [107], [236].

The works by Yang et al. [237], Ding et al. [238], Buhari e Raju [161] and Sukaman et al. [239] show that the production of concrete with ternary cement systems are possible, while Yang et al. [237] proposed a quaternary system. The concretes developed by these authors used phosphogypsum-based hydraulic binders and cementitious supplements such as blast furnace slag and fly ash. Ding et al. [238] proposed a quinary system with PG, PC, GGBFS and SS to develop a Supersulphated cement.

The use of binary systems is more common in concrete production than ternary systems [229]. However, the possibility of creating cementitious systems with 3 or more materials may contribute to improve the concrete properties [240]. PG dihydrate is present in almost all the works. However,



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1 in the work by Smaddi et al. [241] was observed the use of  $\beta$ -PG and A-PG, while the study  
2 developed by Yoo et al. [163] formulated HCBPG with the simultaneous presence of Di-PG and  
3 A-PG, and did not use PC in the formulation of the cement system  
4

5 Based on the literature review, the use of PG in PC, CSA and HCBPG is observed. Furthermore,  
6 different cementitious systems, with different types of raw materials and study parameters were  
7 used. It is important to highlight that only Reddy et al. [242] reported the whole mix preparation  
8 data (%W, water/binder ratio, optimal water/binder ratio, fineness modulus) as well as the effect  
9 on compressive strength with the use of phosphogypsum.  
10

11 It can be concluded that many studies use PG to partially replace cement, however, there are also  
12 studies that used PG in concrete, with and without Portland cement. PG in the dihydrate phase  
13 was the most used, although some studies have also used PG hemihydrate ( $\alpha$ -PG and  $\beta$ -PG).  
14 Portland cement is the most used cement, but the use of calcium sulfoaluminate cement is also  
15 observed. The ternary system with GGBFS is present in most studies. The studies with the  
16 application of PG in concrete have different values in the material production parameters. The  
17 water/cement ratio varied between 0.4-0.65, the optimal water/cement ratio varied between 0.4-  
18 0.6. The percentage of phosphogypsum considered as excellent varied between 4.5%-55%, the  
19 fineness module of the fine aggregate varied between 2.73-3.71, while the fineness module of the  
20 coarse aggregate showed a variation of 3.87- 7.2. The effect of compressive strength in relation to  
21 the optimal percentage of PG varied between a 20-25% increase.  
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## 34 **9 Mechanical strength of mortar and concrete with phosphogypsum**

35 Studies related to the mechanical strength of cementitious matrices are directly linked to safety and  
36 structural stability. The compressive strength is one of the properties that could indicate possible  
37 quality variations. Based on the literature review, it is evident that a main objective of the studies  
38 on the application of PG in mortars and concretes is the investigation on the mechanical properties.  
39 The obtained results were aimed at analyzing compressive and flexural strengths.  
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42 The studies also provide the microstructure of mortars and concretes through tests such as:  
43 Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS), X-ray  
44 Fluorescence (XRF) and X-ray Diffraction (XRD). These tests provide the chemical  
45 characterization, including the pozzolanic reaction, as well as the formation of ettringite and C-S-  
46 H. and consequently, the resistance increase.  
47  
48

49 Thermo-gravimetric and differential thermal analysis (TG-DTA) tests were used to verify at which  
50 temperature the PG modified the mineral phase (dihydrate to  $\alpha$ -hemi,  $\beta$ -hemi, Anhydrite-III,  
51 Anhydrite-II and Anhydrite). The phosphogypsum in the mineral phase  $\beta$ -hemi is present in almost  
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all the studies. In addition, TG-DTA in hydraulic binders contributes to identifying the peak endothermic reaction (145-790°C) related to the formation of ettringite and CSH, and the gain of mechanical strength [143].

This item is structured as follows: (i) effect of PG-based cement systems without cement Portland on mortars; (ii) effect of PG-based cement systems with cement Portland on mortars; (iii) effect of PG-based cement systems on concrete; (iv) effect of the replacement of Portland cement by PG on concrete; and (v) considerations about PG mineral phase.

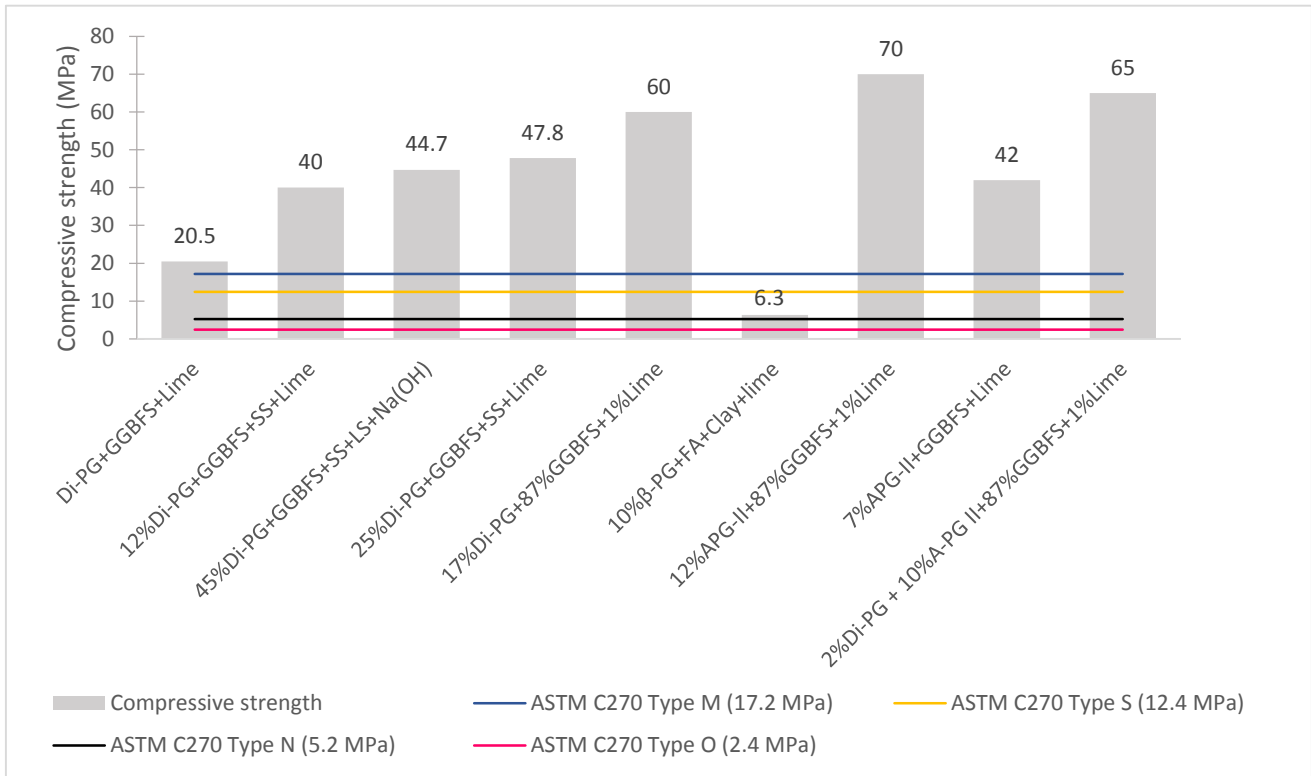
To evaluate the technical feasibility of mortars and phosphogypsum concretes, the American standard ASTM C270 [51], and the European standards EN 998-1 [48], EN 998-2 [49], EN 13813 [50] and EN 1992-1-1 [52] were used as reference. The ASTM C 270 standard requirements refer to mortars produced in the laboratory, being that the reason why it was chosen to compare with the compressive strength data of mortars, found in the literature. The following types of mortars were found: M (17,2MPa), S (12,4 MPa), N (5,2 MPa) and O (2,4 MPa). The respective uses are: masonry foundation, masonry foundations wall, exterior and interior wall.

The standard EN 998-1 classifies coating mortars as CS I (0,4-2,5MPa), CS II (1,5-5,0 MPa), CS III (3,5-7,5MPa), CS IV ( $\geq 6,0$  MPa). The standard EN 998-2 classifies structural mortars as M1 (1 MPa), M2,5 (2,5 MPa), M5 (5 MPa), M10 (10 MPa), M15 (15 MPa), M20 (20 MPa) and Md (d). The minimum compressive strength required by Eurocode EN 1992-1-1 must be 25MPa for cylindrical samples and 30 MPa for cubic samples.

The standard EN 13813 refers to self-leveling mortars based on calcium sulphate and based on Portland cement. According to this Standard, the following compression strength classes may be required: C5 (5MPa), C7 (7MPa), C12 (12MPa), C16 (16MPa), C25 (25MPa), C30 (30MPa), C35 (35MPa), C40 (40MPa), C50 (50MPa), C60 (60MPa), C70 (70MPa) and C80 (80MPa).

### ***9.1 Effect of PG-based cement systems without cement Portland on mortars***

**Figure 12** shows the most significant results of compressive strength in mortars produced without Portland cementitious systems. This analysis was performed to find out prevails in those studies that present the highest compressive strengths, as well as the possible influence of the phosphogypsum mineral phase.



**Figure 12– Compressive strength in mortar no Portland Cement.**

The following mortars with cementitious systems were evaluated: 12%A-PGII+87%GGBFS+1%Lime, 10%A-PGII+2Di-PG+87%GGBFS+1%Lime, 17% Di-PG+87%GGBFS+1%Lime and 11%A-PGII+81%GGBFS+8%Lime. The compressive strength values were 70 MPa, 65 MPa, 60 MPa and 65MPa, respectively [175].

Other studies with the cementitious system PG + GGBFS+ Lime was made. During the studies, the influence of the mineral phases of PG and lime was also tested. The highest values of compressive strength achieved were 7% A-PG II + GGBFS + Lime cementitious system (42 MPa) and 12% Di-PG+ GGBFS+ Lime cementitious system (40MPa) [221].

The mixtures of 25%Di-PG+GGBFS+SS+Lime and cementitious system 45%Di-PG+GGBFS+SS+LS+Na (OH) were also analysed. PG dihydrate was used in both studies and the compressive strength values achieved were 47MPa and 44.7 MPa, respectively. It is observed that the exchange of Lime for Na (OH) and the use of limestone in the cement system (PG+ GGBFS+ SS + LS + Na(OH)) influence the reduction of compressive strength [176], [177].

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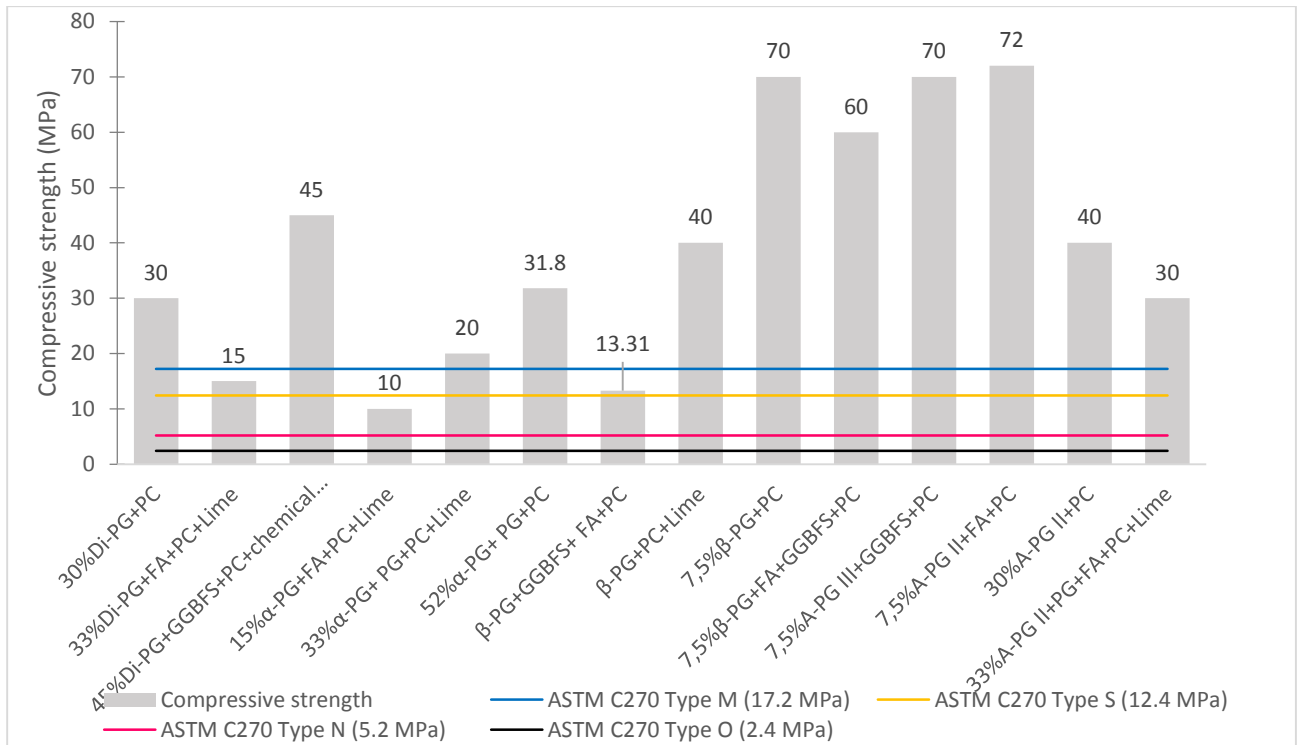
1 The mortar with the lowest compressive strength was produced with a cementitious system  
2 produced with  $\beta$ -PG+ FA + clay+ lime sand with a fineness modulus of 1.26. The result was 6.3  
3 MPa [148].  
4

5 Comparing the data with the American standard ASTM C270 requirements, it can be concluded  
6 that: only the mortar with the Di-PG + GGBFS + lime system does not accomplish the  
7 requirements for type M mortar, and therefore cannot be used in foundation or masonry  
8 foundation wall. The mortar with the with 10% $\beta$ -PG+ FA + clay+ lime system (6.3 MPa) matches  
9 the requirements of type O mortar, and therefore it can only be used indoors, without loading.  
10

11 According to the European standard EN 998-1, all mortars meet the requirements by compressive  
12 strength of the categories CS I, CSII, CS III and CS IV. Only the mortar with 10% $\beta$ -PG+ FA +  
13 clay+ lime system (6.3 MPa) meet the of categories M1, M2.5 and M5 to standard EN 998-2. The  
14 results of other mortars accomplish the compressive strength required requirements. The standard  
15 requires that mortars with values above 20 MPa, must be declared by the manufacturer. Based on  
16 the compressive strength data found in the literature, it appears that mortar with cement systems  
17 without Portland cement, can be used to produce self-leveling mortar, according to the  
18 compression strength requirements (standard EN 13813).  
19

## 20 *9.2 Effect of PG-based cement systems with cement Portland on mortars*

21 **Figure 13** shows the most significant results on the compressive strength in mortars produced  
22 with Portland cementitious systems. This analysis was also performed to find out the study that  
23 provides the highest compressive strength result, as well as the possible influence of the  
24 phosphogypsum mineral phase.  
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**Figure 13 – Compressive strength in mortar with Portland Cement.**

The use of PG above 7.5% influences the reduction of the compressive strength, regardless of the PG mineral phase. Mortar produced with 7.5%A-PG II+FA+PC system reached 72MPa. Mortar prepared with 7.5%β-PG+FA-GGBFS+PC system gave a value of 60 MPa. Mortar with A-PG III+GGBFS+PC system provided 70 MPa. The mortar with the β-PG+PC system reached 70 MPa [222].

The compressive strength results show that PG mineral phase influences the water resistance. The mortar produced with 33% A-PG II + FA + Lime system reached 30MPa, however, the mortars with 33%α-PG+ FA + Lime system and 33%Di-PG + FA + Lime system reached 20 MPa and 15 MPa, respectively [158].

The studies show that temperature and autoclaving time of PG influence the compressive strength. The result of the mortar with 52%α-PG + FA + PC + Lime system pre-treated at 180 °C during 6h in autoclave reached 31.8MPa [159]. The studies show that mortars produced with 45%Di-PG+48%GGBFS + 7%PC + 1% alkaline activator cementitious system reach compressive strengths of 45MPa [243].

Mortars produced with 30%A-PG II+ PC cementitious system reach a value of 40 MPa, while that mortars produced with Di-PG+PC cementitious system reach 30 MPa. The data showed that the calcined PG at 800 °C improved the performance of mortar and increase the compressive

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1 resistance [156]. Moreover, the effect of lime content on the mechanical properties of mortars was  
2 analysed. The results showed that PG  $\beta$ PG+PC and 2% lime mortars reach 36.71 MPa. A 2% lime  
3 content reduces the compressive strength [157].  
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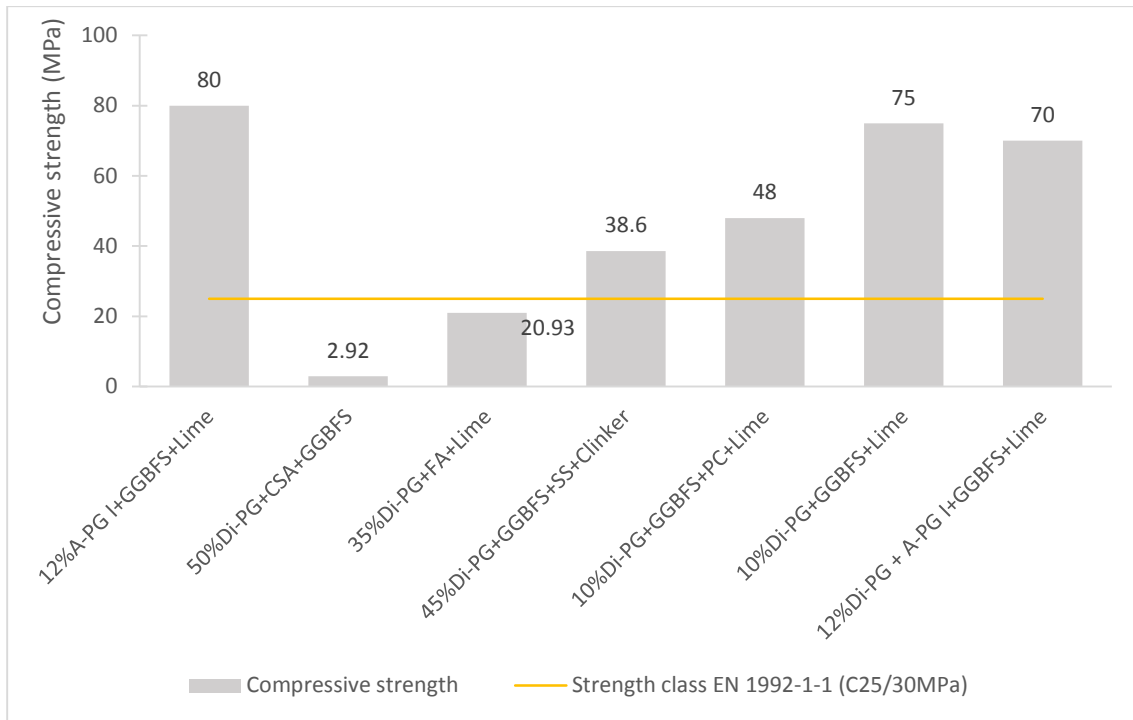
5 Mortars produced with  $\beta$ -P+ GGBFS+FA + PC cementitious system (calcined at 60 °C) and 1:2  
6 binder/sand ratio reaches 13.31 MPa [160]. In the case of mortar with 1:3 binder/sand ratio,  
7 fineness modulus 1.90 and  $\alpha$ -PG +FA + Lime + PC (autoclaving 130°C) 10 MPa were obtained  
8 [145].  
9

10 The Portland cement-based mortars show a variation in the compressive strength between 10-70  
11 MPa. Comparing the data found with the requirements of the American standard ASTM C270, it  
12 can be concluded that: mortars with 33% Di-PG + FA + PC + lime and  $\beta$ -PG + GGBFS + FA  
13 + PC cementitious systems are only compatible with mortars type O, N, S. The analysed mortars  
14 can be used in indoor areas, foundations external and masonry, respectively. The mortar with  
15 15% $\alpha$ -PG + FA + PC + Lime cementitious system accomplish the requirements of type O and N  
16 mortars and it could be used in internal and external locations, respectively.  
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19 All the data on Portland cement-based mortars are in accordance with the European standard EN  
20 998-1 and are compatible with the classifications CS I (0.4-2.5MPa), CS II (1.5-5.0 MPa), CS III  
21 (3.5-7.5MPa), CS IV ( $\geq$  6.0 MPa). When comparing the data with the standard EN 998-2  
22 requirements, it can be concluded that all the mortars studied accomplish the compressive strength  
23 requirements (M1, M2.5, M5, M10, M15 and M20). However, the Standard requires that mortars  
24 with values above 20 MPa, must be declared by the manufacturer.  
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### 26 **9.3 Effect of PG-based cement systems on concrete**

27 **Figure 14** summarizes the result from the studies on the concrete compressive strength, with and  
28 without Portland cement. This analysis was performed to find out which study obtained the highest  
29 compressive strength result, and the possible influence of the phosphogypsum mineral phase.  
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**Figure 14 – Compressive strength in concrete with cementitious systems.**

The studies on concrete with PG + GGBFS + Lime cement system reached 80MPa, 75MPa and 70MPa, using 12% anhydrite (A-PG I+ GGBFS + Lime), 17% dihydrate (Di-PG+ GGBFS + Lime) and 6% anhydrite + 6% dihydrate (Di-PG + A-PG I + GGBFS + Lime), respectively.

The concrete manufactured with Supersulphated cement (PG + GGBFS + SS + Clinker) reached 38.6MPa and 45% PG dihydrate. [238]. The lowest compressive strength result was obtained by the cementitious system PG+CSA+GGBFS, 2.96 MPa and 50% PG dihydrate [234]. A higher degree of eco-efficiency may be suggested in this system due to the use of Sulfoaluminate cement (CSA).

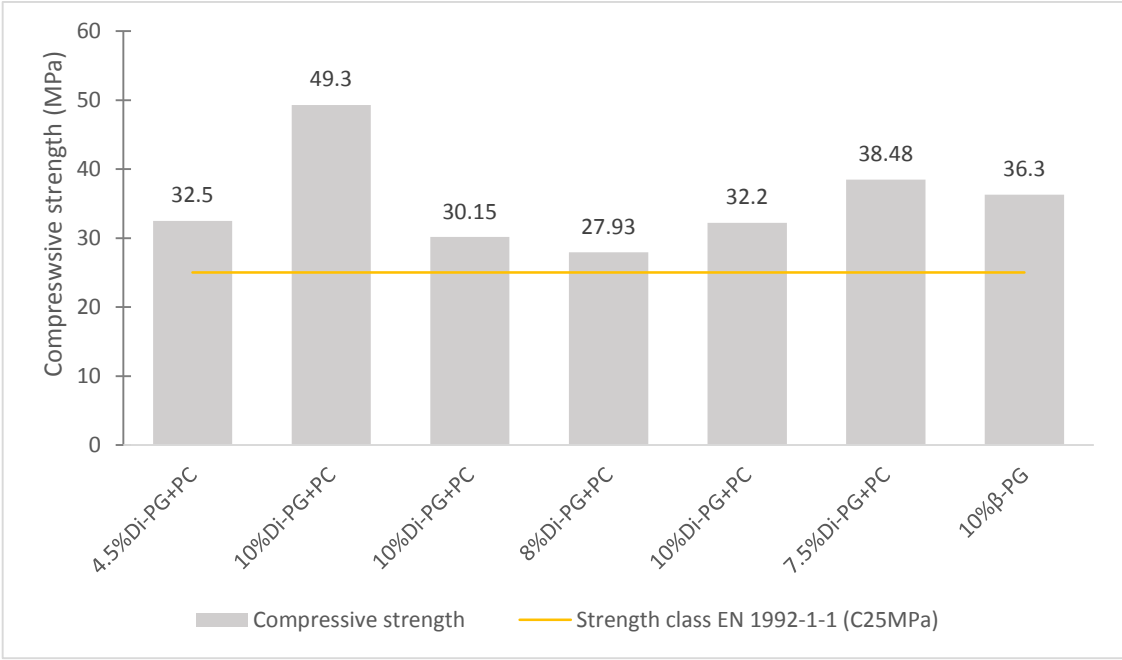
The concrete with quaternary system Di-PC + PG + GGBFS + Lime exhibits the highest result, 48 MPa and 10% PG dihydrate [237]. While concrete produced with Di-PC + PG + FA + Lime cement system gives a value of 20.93 MPa with 35% PG dihydrate [239].

it can be concluded that only concretes produced with cementitious systems Di-PG+CSA+GGBFS and Di-PG + FA + Lime system does not reach the resistance class C25/30 of the European standard EN 1992-1-1 [52]. This resistance class refers to a minimum of 25 MPa for cylindrical specimens and 30 MPa for cubic specimens.

#### **9.4 Effect of the replacement of Portland cement by PG on concrete**

**Figure 15** summarizes the results from the analysed studies on the concrete compressive strength. In those compounds, Portland cement was replaced by phosphogypsum in different ratios. These

studies were performed to compare the compressive strength of concretes with natural additive cement and synthetic gypsum.



**Figure 15 – Compressive strength in concrete with replacement Portland cement by**

Analysing the results, most partial substitutions of Portland cement (PC) by phosphogypsum occurred with PG dihydrate, and in most studies the percentage of PG used is above 5%. However, concrete produced with 4.5% of PG achieves a value of 32.5MPa. [244]. Concrete produced with partial replacement of cement by 10% PG reaches 49.3MPa [242]. However, studies show that the production of concrete with partial replacement of cement in 10% PG, can achieve a compressive strength of 30.5 MPa [245].

The use of 7.5% PG as partial replacement of cement can achieve 38.48 MPa [162]. However, concretes produced with 10% β-hemihydrate (β-PG) and dihydrate (Di-PG) can rise 36.3 MPa and 32.2 MPa, respectively [246]. Nevertheless, the studies showed that concretes produced with 8% PG dihydrate reach only 27.93 MPa. These studies do not inform the cement strength class [247] .

From the literature review it can be concluded that the concretes made with partial replacement of Portland cement by phosphogypsum, accomplish the Eurocode EM 1992-1-1 requirements in terms of compressive strength. The resistance class refers to a minimum of 25 MPa for cylindrical specimens and 30 MPa for cubic specimens [52].



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## 10 Comparison of cement-based studies with and without Portland cement

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2 In order to make easier the analysis of phosphogypsum application in cementitious matrices, Tables  
3 11 and 12 summarize the most significant results on compressive strength of mortars and concrete,  
4 respectively, produced with HCBPG, as well as the production parameters.  
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7 **Table 11-Appendix D** shows that various types of PG mineral phases were used. The Anhydrite  
8 II,  $\beta$ -hemihydrate and dihydrate phases are present in studies without PC mortars. Whereas studies  
9 with PC mortars have most mineral phases ( $\alpha$ -hemihydrate, anhydrite II, anhydrite III,  $\beta$ -  
10 hemihydrate, dihydrate). PG dihydrate is present in most results without PC mortars, while most  
11 PC mortars have PG anhydrite II.  
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16 In addition, it can be observed that mortars with PC had studies with greater variation of the PG  
17 mined phases compared to mortars without PC. In both types of mortar (without and with PC)  
18 results can be observed with PG dihydrate above 44 MPa.  
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22 Among the materials used in the formulation of the mortars, can seen several cementitious systems  
23 with blast furnace slag (GGBFS) and without Portland cement. It is important to mention that fly  
24 ash (FA) was the material most used in mortars with PC. The mortars without PC used only  
25 GGBFS as supplementary cementing materials. Only one study reported the flow between mortars  
26 without PC, while most studies with PC mortars reported the flow, water/cement ratio and  
27 binder/sand ratio.  
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33 **Table 12-Appendix E** provides a comparison of different concretes produced with PG. PG  
34 dihydrate was used as partial replacement in most of the studies, although PG  $\beta$ -hemihydrate was  
35 also used. The compressive strength results accomplish the strength requirements, ranging from  
36 27.93 MPa to 49.3 MPa. In addition, studies on setting times were reported in most studies, as well  
37 as the water/cement ratio and fineness modulus. Only two studies showed the effect of  
38 compressive strength on the ideal PG amount.  
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44 Studies on concrete with cementitious systems have also achieved satisfactory results, being in  
45 accordance with the minimum requirements of compressive strength (25MPa) [53]. Portland  
46 cement, slag (GGBFS) and hydrated lime were the most used materials. The 70 MPa result can be  
47 observed in the formulation PG dihydrate + anhydrite II + GGBFS + lime. Chemical activator  
48 and aluminium powder were also used as raw material. Regarding the analysed parameters, it was  
49 verified that the water/cement ratio was not informed, providing only the fineness module of the  
50 coarse aggregate.  
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## 11 Durability of phosphogypsum-based cement

1 The durability analysis is the study of the agents and mechanisms that have an effect on the material  
2 degradation. Through this study, it is possible to guarantee the performance and integrity of the  
3 material, from its production to the time of use (useful life).  
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6  
7 Phosphogypsum-based cement is prone to undergo moisture, due to the ease of solubilization in  
8 the presence of water. Therefore, its use is restricted to indoor environments. Some studies have  
9 been developed to improve resistance [142], [143], [146], [248]–[250]. As it will be detailed in the  
10 next paragraphs, the solubility of phosphogypsum has been analysed, as well as the water resistance  
11 of cement-based PG, shrinkage by drying, and the expandability of the paste containing PG.  
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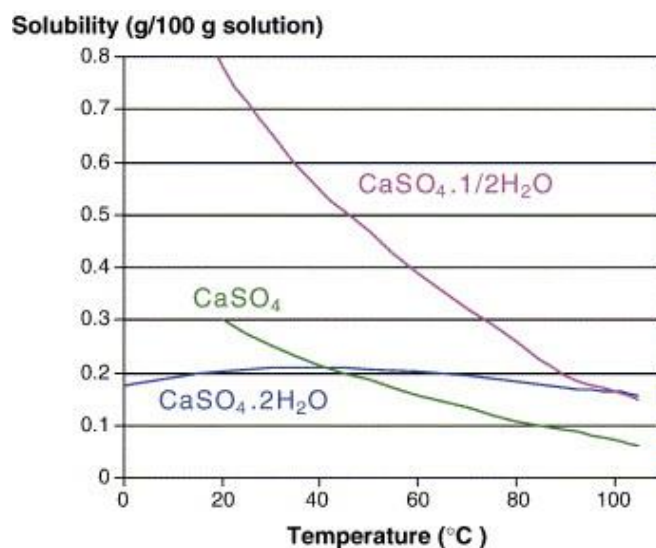
### 16 *11.1 Solubility*

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18 The solubility of calcium sulphate, in all mineral phases, changes if temperature increases. **Figure**  
19 **16** shows the solubility of calcium sulphate in water as a function of temperature  
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22 Calcium sulphate dihydrate solubilizes when the water reaches 20°C. When the water temperature  
23 is above 40 °C the solubilization decreases significantly, resulting in the deposition of the material.  
24 The hemihydrate phase solubilizes from 20°C, and it has greater solubility than both the dihydrate  
25 and the anhydrous phase, due to its lower thermodynamic stability [251]. The calcium sulphate  $\beta$ -  
26 hemihydrate solubilization coefficient (0.92g/100 ml) is lower than that of calcium sulphate  $\alpha$ -  
27 hemihydrate (1.2g/100 ml) [252].  
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33 Anhydrite mineral phase, anhydrous I and II forms are insoluble. However, anhydrite III (soluble  
34 anhydrite) has good solubility and the crystalline structure is an anhydrous state of the hemihydrate.  
35 Furthermore, it can return to the hemihydrate phase very quickly, when in contact with water  
36 (vapor or liquid) at room temperature. For also presenting thermodynamic instability, greater care  
37 is required during preparation [253].  
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43 Summarizing, in cement-based materials, the calcium sulphate solubility differs depending on the  
44 mineral phase, and the importance of this phenomenon is verified. In this way, it is possible to  
45 regulate the setting time during the hydration of the elemental composites. In addition, calcium  
46 sulphate can also improve both mechanical strength and water resistance by adding pozzolan,  
47 cement and hydrated lime.  
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**Figure 16: Solubility of calcium sulphate in water as a function of temperature.**  
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### 11.2 Water-resistant phosphogypsum

Calcium sulphate is a product that has low water resistance, as it has high solubility [126]. In humid environments, crystals tend to lose their adhesion strength. As a result, dissolution occurs under water contact. The environments that are prone to freezing, can also yield to different cracking or collapse patterns, due to the expansion of water that is in the pores [36].

Cement-based materials can be improved through calcination. The phosphogypsum hemihydrate crystals gain water resistance [67]. The water resistance increase in cement-base calcium sulphate can be achieved by the addition of hydrated lime, cement and pozzolana (fly ash, smoke silica, blast furnace slag) [254], [255].

From the performed literature review, the data on the water absorption of phosphogypsum Di-PG,  $\alpha$ -PG,  $\beta$ -PG, A-PG II, A-PG I system are summarized in **Table 13-Appendix F**. However, it is worth noting that it was not possible to find data on the A-PG III. In addition, either data on the compressive strength values or data on water resistance coefficient were not found. Due to the correlation between the compressive strength and the percentage of water absorption, the performance of cement-based PG and plaster is evaluated in this paper.

The Di-PG + PC water absorption was 2% and the compressive strength 32 MPa [156]. The Di-PG + PC + GGBFS system showed a water absorption of 3% and a compressive strength 22 MPa [160]. The water absorption for the Di-PG + PC + FA system was 10% with a compressive strength of 19.6 MPa [160]. The Di-PG + FA + Lime system had a water absorption of 28% and a compressive strength of 12 MPa [256].

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1 The water absorption of  $\alpha$ -PG plaster is 32% [257]. While  $\alpha$ -PG + FA + PC + Lime shows water  
2 absorption and 14% for compressive strength of 27.5MPa [145]. When the  $\alpha$ -PG + FA + Lime  
3 system is subjected to a 50°C curing, the water absorption increases 19.6% and the compressive  
4 strength decreases to 26.4 MPa [141]. However, the PG + GGBFS + Marble dust + chemical  
5 activate system with a curing temperature of 50°C can reach 32.2 MPa, with a water absorption of  
6 only 4% [141].  
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10 From the reviewed data,  $\beta$ -PG plaster can be more soluble compared to  $\alpha$ -PG plaster, with a water  
11 absorption of 30% to leaching. The compression strength values range from 13.20 to 14 MPa, and  
12 the water resistance coefficient is 0.35 [142], [143], [171], [257]–[259]. When analysing the  $\beta$ -PG +  
13 PC + GGBFS system, it is noted that the compressive strength can reach 35 MPa, with a variation  
14 in water absorption between 3-7% % [160], [259]. The use of organic retardant in the  $\beta$ -PG + PC  
15 + GGBFS system maintains the compressive strength at 35 MPa, however the water absorption  
16 can vary between 2.89-6% [143], [160], [257].  
17

18 The study of the  $\beta$ -PG + FA + PC system showed that water absorption can vary between 6.5-  
19 10% and the compressive strength can vary between 25.6-21.3 MPa, respectively. When this system  
20 is cured at 50°C, the water absorption remains at 10% and the compressive strength reduces to 20  
21 MPa, and the use of a retarder causes a compression strength increase of 25 MPa with 8 % water  
22 absorption [146], [160], [171], [250], [260], [261]. The composition 70%  $\beta$ -PG + 30% SAC showed  
23 a resistance coefficient from 0.70 to 22% water absorption [258].  
24

25 The water absorption of the A-PGII + GGBFS system can vary between 9.55 and 10.43% and the  
26 compressive strength follows the variation of 35-56 MPa, respectively [142], [149]. When blast  
27 furnace slag is replaced by Portland cement to form the A-PGII + PC system, water absorption  
28 reduces to 4%, but the compressive strength is slightly reduced to 34.5MPa [156]. The study of the  
29 PG + FA + Red Mud system showed that it is possible to achieve a compressive strength of 40  
30 MPa and a water absorption of 4% [21].  
31

32 In view of the obtained data, it can be concluded that there is a relation between the water content  
33 absorbed by the paste and the compressive strength. That is, the greater the water absorption is,  
34 the lower the resistance to compression is. It appears that the plasters Di-PG,  $\alpha$ -PG and  $\beta$ -PG had  
35 the highest water absorption rates. The Di-PG + FA + Lime,  $\beta$ -PG + Fa + Lime and 70%  $\beta$ -PG  
36 + 30% SAC systems also showed high water absorption and therefore, it can be concluded that  
37 the systems with lime, exhibit high absorption of water compared to systems composed of PG +  
38 GGBFS + PC, PG + FA + PC, PG + PC.  
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It can be seen that most of the data on water absorption of cement-based PG are in agreement with the result of water absorption of cement paste (16%), presented in the study by Selim et al [61]. According to Kovler [255] cementitious systems based on calcium sulphate with water absorption up to 15%, are considered materials with high resistance to water. In the early ages, the absorption water varies between 35-40% on cementitious systems produced with calcium sulphate + silica fume + Portland cement. At 28 days of age the water absorption decreases (30-35%), due to the formation of C-S-H.

### 11.3 Drying shrinkage

Drying shrinkage is a phenomenon that causes the reduction of the volume of cementitious materials, through the decrease of the internal relative humidity of the pores [262]. When there are internal or external restrictions and the volume reduction is impaired, residual tensile stresses may accumulate, and strains and/or micro cracks may occur [262].

The drying shrinkage depends on the following parameters: relative humidity, type of material used (binder, fineness of the binder, addition of pozzolana, volume of cement paste), water / cement ratio, type and size of the aggregate, porosity and fineness and size of the part that will undergo retraction [263].

The drying shrinkage data obtained from the literature review are depicted in **Table 14**. It is worth noting that it was possible to find information only for the mineral phases Di-PG and  $\alpha$ -PG. The performance and technical feasibility of cement systems were compared with the European standards EN 12620 [57], EN 1367-4 [56] and the bibliographic references Hewlett and Liska [54] and; Lamond and Pielert [55].

**Table 14: Drying shrinkage**

Mineral phases	Drying	PG (%)	Binder	Ref.
Di-PG	3	10	HCBPG	[264]
Di-PG	0.06	40	SAC/PC	[265]
Di-PG cured at 80°C	0.06	50	HCBPG	[190]
Di-PG cured at 50°C	0.16	50	HCBPG	[190]
$\alpha$ -PG system +60%FA	0,11	15	HCBPG	[145]
$\alpha$ -PG system +50%FA	0,11	15	HCBPG	[145]
$\alpha$ -PG system +40%FA	0,13	15	HCBPG	[145]
$\beta$ -PG	unknown	unknown	unknown	unknown
A-PG III	unknown	unknown	unknown	unknown
A-PG II	unknown	unknown	unknown	unknown
A-PG I	unknown	unknown	unknown	unknown

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1 Mortars produced with Di-PG + FA + lime with 10% PG resulted in 3% shrinkage when drying  
2 (when curing was carried out in water). For outdoors curing, there was no change in volume [264].  
3 The study of self-levelling mortars with the Di-PG + SAC / PC system, if SAC/PC is in the 6: 4  
4 ratios, showed that the use of 40% PG results in 0.06% shrinkage by drying.  
5

6  
7 The study of mortars produced with PGS-optimal (CM: PG: SG: Lime: SF: USZ: BXF200-L:  
8 Defoaming Agent) with 50% PG resulted in a drying retraction of 0.06% for the curing temperature  
9 80°C, and in a 0.16% retraction by drying, for a curing temperature 50°C. It suggests that the higher  
10 the curing temperature is, the lower the drying shrinkage for mortars produced with PGS-optimal  
11 HCBPG is [190].  
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15 The  $\alpha$ -PG + FA + PC + Lime system, containing 15% PG, was studied in mortars. The  $\alpha$ -PG  
16 system + 60% FA and  $\alpha$ -PG system + 50% FA showed a shrinkage of 0.11%. While the  $\alpha$ -PG  
17 system + 40% FA showed a 0.13% shrinkage on drying.  
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21 In the early ages, the drying shrinkage of Portland cement pastes, with 47% relative humidity varies  
22 between 0.35-0.70%. At the age of 6.5 months, mainly the pastes with the largest amount of pores,  
23 reaches 0.65% retraction, while pastes at the age of 30 months show a 0.45% drying retraction,  
24 when the relative humidity is by 47% [54].  
25  
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27  
28 In concrete, the relative humidity of 50% can influence the shrinkage due to drying between 0.04-  
29 0.08%. In reinforced concrete with normal amount of steel, the retraction can vary between 0.02-  
30 0.03%. In structural lightweight concrete the retraction varies between 0.04-0.15%. It is considered  
31 a great shrinkage effect on drying, in cellular concrete, when the shrinkage varies between 0.3-0.6%  
32 [55].  
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36 According to standard EN 1367-4, when the effect of the aggregate on the concrete shrinkage  
37 exceeds 0.006 mm and 12% the test must be considered unsatisfactory, and another test must be  
38 performed. The EN 12620 standard determines that the shrinkage due to drying in the concrete,  
39 occurs because of the aggregate, and it cannot exceed 0.075%.  
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43 The shrinkage by drying can vary between 0.06-3% for systems with HCBPG. While drying  
44 shrinkage is very low in sulfoaluminate cement-based materials. In view of the data from the  
45 literature review, it can be concluded that the data presented in Table 12 are in accordance with the  
46 standards EN 1367-4, EN 12620, and with the literature on shrinkage by drying [54], [55].  
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#### 11.4 *Expansibility (soundness)*

1 In cementitious materials, in order to guarantee both durability and an adequate useful life, it is  
2 essential to control and minimize the changes in volume [37]. The expandability in cementitious  
3 matrices is directly related with the reaction of tricalcium aluminate (free lime), magnesium sulphate  
4 and the calcium sulphate of the phosphogypsum. During the chemical reaction of these elements,  
5 the formation of ettringite crystals and the subsequent expansion of the cement matrix occur [266].  
6 Therefore, the cement matrix breaks in the hardened state [37].  
7

8 In the literature, the data on expandability was not found only for the mining phase A-PG III. In  
9 **Table 15-Appendix G**, the expansion data for Di-PG,  $\alpha$ -PG,  $\beta$ -PG, A-PG II, A-PG I can be  
10 observed. It is noteworthy that it was not possible to identify, most of the phosphogypsum  
11 percentage used in expandability studies.  
12

13 The expandability study with the Di-PG plaster showed the result of 0.6mm [174]. The  
14 expandability of Portland cement with partial replacement by Di-PG with 12.5-25% PG, can vary  
15 between 6-8 mm [27], [245], [267]. The Di-PG + PC + GGBFS system presented the result of  
16 expandability of 3.6 mm [160], [174]. The expandability of the Di-PG + PC + FA system was  
17 1.6mm [160], [174], while the Di-PG + PC + Red Mud system showed an expandability of 1.6mm  
18 [174]. Among the results on expandability, the Supersulphated cement composed of Di-PG + PC  
19 + GGBFS + SS + LS + NaOH and 45% PG, gave 13.5mm [177].  
20

21 Only the expansion value of  $\alpha$ -PG + PC + FA + Lime system with 10% PG was found. The  
22 expandability value was 2.3mm [145]. The value for the  $\beta$ -PG plaster was 0.6mm [171], [174]. The  
23 study of partial replacement of Portland cement with 70% PG ( $\beta$ -PG + PC) showed an  
24 expansibility of 0.10 mm, while the partial replacement of pozzolanic cement with 70% PG ( $\beta$ -PG  
25 + PCC) presented 0.106 mm expandability [241].  
26

27 The study of the  $\beta$ -PG + PC + GGBFS system with retarder and without organic retarder showed  
28 an expandability of 1.60 mm [160], [174], [249], [257], [259]. The  $\beta$ -PG + GGBFS + tartaric acid  
29 system with 15% PG showed an expandability of 1.2 mm [173]. The  $\beta$ -PG + PC + FA system  
30 showed an expansion range between 0.88-1.00 mm [160], [174]. When a retarding additive is used  
31 for the same system, the expandability value is 0.88 mm [250]. And when the  $\beta$ -PG + PC + FA  
32 system is cured at 50°C, the expandability is 1.8mm [146].  
33

34 The  $\beta$ -PG + FA + PC + Lime + chemical additives system with PG between 70-75% showed an  
35 expandability of 2.0mm. The  $\beta$ -PG + PC + Red Mud system showed an expandability of 1.1 mm.  
36 The highest expansion value found in the literature with the use of  $\beta$ -PG was 12 mm for  
37 Supersulphated cement composed of  $\beta$ -PG + GGBFS + SS + SL with 45% PG [176].  
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1 The expandability of A-PG II plaster is 0.5 mm [142]. The A-PG II + GGBFS system presents a  
2 range of expandability between 0.81-0.90mm, for 70% PG and 15% PG, respectively [149], [173].  
3 While the A-PGII + GGBFS + system chemical activator showed an expandability of 1.5 mm  
4 [142]. The highest expandability value with A-PGII was 3.0 mm for the PG + PC + GGBFS  
5 system, with 10% of PG relative to Supersulphated cement [191]. Only a result of 0.06 mm for A-  
6 PG I + GGBFS + 2% citric acid system is obtained from the literature [21].  
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10 It can be concluded that the plaster has practically no expandability, because the values are very  
11 low. However, when there is a mixture of PG in any mineral phase (with fly ash, blast furnace slag,  
12 Portland cement, setting retarder or chemical activator) the expandability increases. The highest  
13 expansibility values will occur when Portland cement is partially replaced by PG.  
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18 It should be noted that only the PG + GGBFS + SS + SL (Supersulphated cement) system did  
19 not agree with the expandability requirements of the European standards EN15743 [179] and EN  
20 197-1 [268].  
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## 24 **12 Application of phosphogypsum in geotechnical materials**

25 The production of Portland cement-based geotechnical materials require combustion, furthermore,  
26 they consume a lot of energy and pollute the environment [269]. To minimize the environmental  
27 impacts, the construction industry has adopted the use of industrial or agro-industrial by-products  
28 over the years as alternative raw materials, as they provide low cost and lower energy consumption  
29 [270].  
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35 Based on the literature review of works that were done between 1992-2019, **Table 16-Appendix**  
36 **H** shows the studies on phosphogypsum-based geotechnical and building components materials.  
37 In total were found 8 studies on materials to be used as road basement; 5 studies on tile and ceramic;  
38 1 study on lime; 5 studies on self-levelling mortars; 7 studies on non-hydraulic binders; 3 studies  
39 on plasterboard; 13 on blocks and bricks and 7 studies on plaster.  
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45 Recent studies (2017-2019) have not focused on a single theme. Contreras et al. [271] applied PG  
46 as an additive in the production of ceramic bricks. The works by Silva et al. [272] and Dutta [273]  
47 investigated paving and geotechnical materials by using phosphogypsum. In addition, the works  
48 developed by Qiang et al. [274] and Schaefer et al [71] show that it is possible to incorporate PG  
49 in Sulfoaluminate cement and Calcium aluminate cement (CAC) for the production of self-levelling  
50 mortars. In the work by Ding et al. [275], it was found that PG can be used for soil stabilization.  
51 The work by Zhou et al. [276] was focused on the manufacture of ceramics without the combustion  
52 process, and Romero-Hermida [277] investigated the possibility of producing lime from  
53 phosphogypsum as a substitute for commercial lime.  
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1 Regarding the use of PG, it is noted that the most used mineral phases correspond to  
2 phosphogypsum dihydrate (Di-PG) and  $\beta$ -hemihydrate ( $\beta$ -PG). The works by Schaefer et al [71]  
3 and Singh e Garg [21], [165] were performed with anhydrous phase phosphogypsum (A-PG). The  
4 studies of Garg et. al [96]; Garg e Pundir [145] used the phosphogypsum in  $\alpha$ -hemihydrate ( $\alpha$ -PG)  
5 phase.  
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9 Portland cement (PC) is the most used hydraulic binder in all the works, and fly ash (FA) compared  
10 to blast furnace slag was more prominent in the manufacture of building material components. In  
11 addition hydrated lime is present in most of the works due to studies on alternative  
12 phosphogypsum-based hydraulic binders in ternary, quaternary and quaternary systems [160],  
13 [174], [249], [259], [260], [264], [278].  
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18 In view of the discussed issues, it can be concluded that phosphogypsum can also be applied in  
19 geotechnical materials, self-levelling mortars, blocks, tiles, slabs, lime, binders, with the use of PG  
20  $\beta$ -hemihydrate and dihydrate.  
21  
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### 23 **13 Study of radiation in building materials with phosphogypsum**

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25  
26 Natural radionuclides are present in the Earth's geological formation. They are found in rocks,  
27 soils, building materials and industrial by-products. In construction materials, the following  
28 radionuclides appear more frequently: uranium series (U-238), thorium series (Th-232) and  
29 potassium isotopes (K-40) [279]. Building materials manufactured with inputs of terrestrial origin  
30 (sand, granite, clay, marble, gravel, wood) contain less radionuclides than materials produced with  
31 industrial by-products (phosphogypsum, blast furnace slag and fly ash) [280], [281].  
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37 Radiation in building materials occurs in two ways: (i) external radiation - caused by radio isotopes  
38 (Ra) with the emission of gamma radiation (Ra-226 generated by the U238 decay); (ii) internal  
39 radiation - caused by radon isotopes (Rn) with gas exhalation from Rn-222 (generated by the decay  
40 of U238) and exhalation of gas Rn-220 (generated by the decay of Th232) [282]. Radon and its  
41 daughters emit short-lived alpha radiation. Large levels of radiation can be transmitted indoors.  
42 Contact with high concentration can cause respiratory changes and greater chances of developing  
43 cancer when exposed to gas [283], [284].  
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49 The control of radiation in building materials produced with phosphogypsum is essential to  
50 understand the limit amount of incorporation of PG. Depending on the uranium source, the  
51 activity of Ra-226 in PG occurs one or two times stronger, compared to natural gypsum [285].  
52 Because of this, constant research seeks to establish basic safety standards for protection against  
53 the dangerous ones arising from radiation exposure [286].  
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1 The impact of indoor radiation is based on the concentration and exhalation of Rn-222. According  
2 to ICRP 65 [287] the concentration in indoor environments can be classified as follows: (i) normal  
3 (up to 200 Bq/m<sup>3</sup>); (ii) attention (200-400 Bq/m<sup>3</sup>); (iii) monitored (400-600 Bq/m<sup>3</sup>); and (iv)  
4 interventional (600 Bq/m<sup>3</sup>). The Environmental Protection Agency [288] recommends that the  
5 concentration of Rn-222 does not exceed 148 Bq/m<sup>3</sup>.  
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9 The World Health Organization (WHO) [289] recommends that concentrations in indoor  
10 environments be less than 100 Bq/m<sup>3</sup> and if it is not possible to meet, the concentration in the  
11 environment cannot exceed 300 Bq/m<sup>3</sup>. The factors related to the appearance of cancer should  
12 also be noted. Regarding the exhalation rate of Rn-222, the recommendation is not to exceed the  
13 level of 0.074 Bq m<sup>-2</sup> h<sup>-1</sup> or 20 pCi/(m<sup>2</sup>-sec) [290].  
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18 The risks caused by outdoor radiation are verified through the activity concentration index (index  
19 I) and the equivalent radio activity (Ra<sub>eq</sub>). According to RP 122 [280], it is recommended to evaluate  
20 the index I according to the dosage (0.3 and 1 mSv / year) and the characteristics of mass and  
21 surface. The recommended dosage is 1 mSv/year [291]. It is also recommended that Ra<sub>eq</sub> be  
22 evaluated according to the annual exposure to gamma rays, considering residential locations  
23 [292]. The maximum limit for Ra<sub>eq</sub> is 370 Bq/kg [293], [294].  
24  
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28 Indoor and outdoor radiation data found on phosphogypsum building materials are shown in  
29 **Table 17-Appendix I** The following types of building materials have been identified with PG:  
30 autoclaved plaster (α-PG), Portland cement paste, binary system based-PG, lime mortar, Portland  
31 cement concrete, mortar, sulfur polymer concrete, bricks, board, plate and ceramic tiles.  
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36 Most data regarding Rn-222 concentrations agree with the EPA [288] and ICRP 65 [287]  
37 recommendations and can be classified as normal. However, the data from studies by Gijbels et al.  
38 [295] on the binary cement system are classified between attention (200-400 Bq m<sup>3</sup>) and monitored  
39 (400-600 Bq/m<sup>3</sup>). Fournier's study [296] on bricks shows radon concentrations between normal  
40 classification (less than 200Bq/m<sup>3</sup>) and the attention classification (200-400 Bq/m<sup>3</sup>).  
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45 Based on WHO recommendations on Rn-222 concentration [289], it is noted that the study by  
46 Gijbels et al [295] meets the limit of 300Bq/m<sup>3</sup>. While Fournier's study [296] meets the  
47 recommendations within the recommended concentration (100Bq / m<sup>3</sup>) and within the limit of  
48 300Bq/m<sup>3</sup>.  
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53 The studies about plaster, paste, mortar and concrete materials agree RP 112 [280] and within the  
54 of dosage criteria classified as “mass” (I is smaller equal 1). The studies about brick, board and  
55 plate materials can be classified as “surface” (I is smaller equal 6). All materials were analysed  
56 considering the dosage of 1 mSv/year.  
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The data on radio equivalent radioactivity ( $R_{eq}$ ), show that materials produced with igneous phosphogypsum can exceed the recommended limit (370Bq/kg). Although the sedimentary phosphogypsum contains a higher level of radioactivity, it is possible to produce materials that meet the limits of  $R_{eq}$ . Among all the studies found, only Campos et al [31] and Nisti et al [297] have samples that exceed the recommended radioactivity limit.

The amount of PG incorporated in the materials was also investigated. The study by López et al [298] show that there is a correlation of  $R^2 = 0.99$  between the percentage of PG and the equivalent activity of radio-226. In addition, it show that the equivalent activity of radio-226 reaches approximately 300 Bq/kg with the use of 50% PG. The concentration of radon-222 was 196 Bq/m<sup>3</sup> and the exhalation rate was 11.7 Bq m<sup>-2</sup> h<sup>-1</sup> when 50% PG was used.

It was not possible to find data about the exhalation rate of radon in paste, mortar and concrete, and the percentage of PG used. Only the studies by Campos et al [31] and Nisti et al [297] show that plates (0.16 - 0.41 Bq m<sup>-2</sup> h<sup>-1</sup>) and bricks (0.11 - 0.41 Bq m<sup>-2</sup> h<sup>-1</sup>) meet the requirements of the exhalation rate (0.074 Bq m<sup>-2</sup> h<sup>-1</sup>).

The studies by Gijbels et al [101] and García-Díaz et al [299] show that the incorporation of 30% of PG meets the RP 112 (I is smaller or equal than 1). While the study by Contreras et al (Contreras et al., 2018) shows that the use of 10% PG in ceramic tiles and ceramic tiles with 100% PG are in accordance RP 112 (I is smaller or equal than 6) and  $R_{eq}$  (370 Bq/m<sup>3</sup>).

It can be concluded that the radiation levels in materials produced with phosphogypsum, depend on the type of material that will be produced. It also depends on its physical characteristics (thickness and density) and chemical properties (quantity of radionuclides). Although sedimentary phosphogypsum contains a higher level of radiation compared to igneous phosphogypsum, the extraction site will strongly influence the radiation level of the material.

#### **14 Other ways of valorizing phosphogypsum**

The environmental problems caused by phosphogypsum have been studied over the years, in order to find a more adequate purification treatment, which contributes to enhance the use of phosphogypsum, mainly in the manufacture of construction materials [29], [30]. There is also an incentive for the industry to mitigate phosphogypsum in different ways, such as fertilizers, soil conditioning, landfill for road construction and others [300].

Mitigation of dihydrate phosphogypsum can also occur, through conversion to portlandite (calcium hydroxide) and sodium sulphate, with a high carbon dioxide capture efficiency. In addition, the impurities present in the phosphogypsum were transferred to the final product [301].

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1 The calcium present in phosphogypsum, in 96% of the cases, is capable of sequestering CO<sub>2</sub> from  
2 the atmosphere, through the process of alkaline dissolution and aqueous carbonation under  
3 pressure and room temperature. In addition, a CO<sub>2</sub> sequestration process, based on the recycling  
4 of phosphogypsum, could help to mitigate greenhouse gas emissions. The estimated production of  
5 phosphogypsum worldwide would be able to sequester 70 Mt of carbon dioxide per year, and as  
6 an example, in SW Spain the phosphogypsum cells could capture an estimated value of 30 Mt CO<sub>2</sub>  
7 [302].  
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12 With the production of portlandite that is generated from the dissolution of phosphogypsum,  
13 radionuclides <sup>226</sup>Ra, <sup>238</sup>U, <sup>230</sup>Th and <sup>210</sup>Pb-<sup>210</sup>Po are almost completely transferred. The  
14 calcium carbonate generated through the sequestration of CO<sub>2</sub> also receives the radionuclides  
15 present in the PG. EU regulations consider portlandite and calcite generated from PG as NORM  
16 materials , and therefore they must be controlled [302].  
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22 The treatment of bauxite with phosphogypsum can promote the significant transformation of  
23 alkalinity cations by neutralization, precipitation and substitution reactions. The alkalinity of the  
24 bauxite residue can be reduced by adding phosphogypsum by 92.2%. Studies show that the  
25 transformation of bauxite into an aggregate structure can facilitate the treatment of the deposition  
26 areas of the bauxite residue (BRDA). In addition, it achieves the objective of “using waste and  
27 treating waste”, reducing the existing risks in the BRDA and promoting the process of forming  
28 bauxite waste soils [303].  
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35 Currently, waste management is considered a challenge. Waste such as steel slag, phosphogypsum  
36 and fly ash can play a key role in mitigating methane and nitrous oxide emissions from rice paddies.  
37 Studies show that these residues have nutrients that can provide additional benefits to paddy rice  
38 soils. Although these residues show promising potential, comprehensive analyses on several  
39 features (e.g. the mechanical performance, the recommended doses, the application time and the  
40 hydrological processes in the short, medium and long term) are still required [22].  
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## 46 **15 Research Gaps**

47 This section describes the issues that have not yet been studied or are not yet fully resolved within  
48 the framework of a solid and structured scientific knowledge. Thus, identifying research gaps could  
49 contribute to future scientific research.  
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53 The SWOT analysis (Strengths, Weaknesses, Opportunities, and Threats), **Figure 17**, is a tool that  
54 is used in any type of scenario or analysis. It tries to find the strengths, weaknesses, opportunities  
55 and threats within a given subject or field of work. Due to its methodological scope, the SWOT  
56 analysis is used to find the research gaps in this work.  
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During the performed research, the main strength points were the availability of scientific papers on phosphogypsum, the available results on pastes, mortars and concrete, and the standardization of binders with a high amount of calcium sulphate (Supersulphated cement).

Focusing on the weaknesses, it is observed that there are few scientific papers that synthesize the application of phosphogypsum in building materials. Most scientific papers deal with investigating a specific material or the environmental impact caused by phosphogypsum. It is important to comment that among all the existing data on phosphogypsum, only 6 scientific papers ([24], [29], [32], [130], [304], [305]) on synthesized content were found. Besides, is possible noted the absence of studies about Life Cycle Analysis and Global Warming in PG-based material building and; the absence of studies about sulphate resistance and attack by chloride, carbonation and chloride ingress in PG-based concrete.

The issues considered as opportunities show that it is possible to use phosphogypsum in the mineral dihydrate phase in cementitious matrices, contributing to the mitigation of phosphogypsum and its commercial valorization, without causing risks to human health [306]. The presence of impurities in the chemical composition, as well as heavy metals, were considered threatening points in the use of phosphogypsum. The setting time increase in cementitious matrices, the effects on the quality of using PG without treatment, as well as the high degree of radiation of phosphogypsum with sedimentary origin, are also threats.

**Figure 17: SWOT analysis on phosphogypsum in cement-based materials**

		Helpful	Harmful
Internal	<b>Strength:</b>	<ul style="list-style-type: none"> <li>Scientific papers on the issue.</li> <li>Satisfactory results with various PG mineral phases.</li> <li>Standards on calcium sulphate binder (Supersulphated cement /EN15743).</li> <li>Standards on calcium sulphate screed material and floor screeds (EN 13813)</li> </ul>	<b>Weaknesses:</b> <ul style="list-style-type: none"> <li>Absence of documents with systematic review on the influence of PG in cement-based</li> <li>Absence of Life Cycle Analysis and Global Warming studies on PG to be used as building material.</li> <li>Absence of studies on sulphate resistance and attack by chloride, carbonation and chloride ingress in concrete produced with PG</li> </ul>
	<b>Opportunities:</b>	<ul style="list-style-type: none"> <li>PG dihydrate application in cementitious matrices. valorization and commercial enhancement of PG without risks to human health</li> </ul>	<b>Threats:</b> <ul style="list-style-type: none"> <li>Presence of impurities in the phosphogypsum, (setting time increase and decrease compressive strength).</li> <li>Non-treatment of PG influences on the quality.</li> <li>High radiation degree in the phosphogypsum of sedimentary origin.</li> </ul>
External			

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## 16 Conclusions

The feasibility of using phosphogypsum as raw material in different applications is an open and active research field. For instance, several research works focus on its use in cementitious matrices transforming it into a commercial by-product. From a comprehensive review of the literature, this paper highlights the following conclusions:

- 1) Phosphogypsum dihydrate changes the crystalline structure when it undergoes dehydration. Through autoclave or calcination process, it can reach the hemihydrate, anhydrite III, anhydrite II and anhydrite I mineral phases.
- 2) The study of the fineness and distribution of PG particles helps to determine the degree of reactivity, hydration kinetics, the development of the microstructure and the development of the properties of the PG-based binder. PG particles can reach a maximum size of 0.5 mm. Most of the particles (50-75%) are smaller than 0.075 mm and the specific area varies from 2.5 to 17.5 m<sup>2</sup>/g.
- 3) The chemical composition of phosphogypsum depends on the geological origin (sedimentary or igneous), the type of process for obtaining phosphoric acid, the efficiency of the industrial process, the storage time, and on the presence of contaminants. Heat treatments such as calcination or autoclaving, in addition to increasing the mechanical strength of PG, contribute to the reduction of impurities.
- 4) The impurities of phosphate and fluoride influence setting time increase and the reduction of resistance to compression in the early ages. Although phosphogypsum is a NORM waste, if it is of igneous origin, it is possible to use it without compromising human health as raw component of building materials.
- 5) The Di-PG and  $\beta$ -PG particles absorb more water, and therefore the normal consistency of the Di-PG (66-80%) and  $\beta$ -PG (60-64%) plaster is greater than the normal consistency of the  $\alpha$ -PG (23.7-58%), HBBPG (27,42-44%) and Portland cement plaster with partial PG replacement (23-26,5%). There is a correlation between compressive strength and consistency. Higher compressive strength values are related to lower normal consistency.
- 6) Most of the analysed data have a fluid spread flow greater than 220 mm and, therefore, mortars with this type of spread flow can be used as self-leveling mortars. The spread flow of mortars with  $\beta$ -PG + GGBFS + PC,  $\beta$ -PG + FA + PC and  $\beta$ -PG + FA + Clay + lime cementitious systems only accomplish the ASTM C270 requirements. While the mortar

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with the cementitious system 50% Di-PG + PC presented a light density (greater than 1200 kg/m<sup>3</sup>) fulfilling the EN 1015-2 requirements.

- 7) Most of the data found on slump are in accordance with the standard EN 206 and, furthermore, match the S2 consistency class (50-90 mm) of the standardized type, to be used in slabs, foundations and structural elements in general. Only the concrete produced with the Di-PG + GGBFS + Lime cement system satisfy the S4 consistency class (160-210 mm) requirements, with high workability and application in concrete foundations, slabs, pumping and piling.
- 8) By using scanning electron microscopy, it is possible to identify the presence or absence of impurities. It is suggested that the visualization of changes in the crystalline structure occurs when the phosphogypsum is hydrated.
- 9) The setting times of the Di-PG,  $\alpha$ -PG and  $\beta$ -PG plasters are shorter than that of A-PG I. The use of succinate and potassium citrate setting time retarders increases the initial setting time. The results of the initial and final setting time of the HCBPG are greater when the phosphogypsum is being used in the A-PG II phase. HCBPGs with  $\alpha$ -PG showed a fast initial setting time. While the HCBPGs produced with Di-PG and  $\beta$ -PG showed a medium setting time. The HCBPGs produced with A-PG II and cementitious systems as Supersulphated cement showed a slow setting time. In the setting time of Portland cement with partial replacement of PG, it was observed that all data of initial setting time meet the requirement of EN 197-1 (45 min). It is also noted that the final time was below that of required by the ASTM C150 standard (375 min). The final setting time was longer for replacements with Di-PG.
- 10) The studies on phosphogypsum applied in paste were mostly carried out in ternary, quaternary and Supersulphated cement systems. PG dihydrate and PG  $\beta$ -hemihydrate were the most used mineral phases. Studies on mortars used mainly PG dihydrate, and the most commonly found cementitious systems are: (I) without Portland cement: PG-FA-lime; PG-GGBFS-file; PG-GGBFS-FA-lime, (II) with Portland cement: PC-PG-FA-lime; PC-PG-GGBFS-file; PC-PG-GGBFS-FA-lime. The other systems found were binary (PC-PG) or systems developed for Supersulphated cements (GGBFS-PG-PC). It was found that the studies related to concrete follow two research lines: (i) the partial replacement Portland cement by phosphogypsum, and (ii) the production of concrete with HCBPG binder.
- 11) Focusing on the mortar mechanical strength, most studies without Portland cement were carried out with Di-PG, whereas those of with PC used phosphogypsum A-PG II. In addition, mortars without PC have compressive strengths ranging from 6.3-70 MPa. The

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compressive strength of mortars with PC varied between 10-70 MPa. Besides, most mortars produced with cementitious systems without and with Portland cement accomplish the compressive strength requirements the standards ASTM C 270 standard, EN 998-1, EN 998-2 and EN 13813. The data on phosphogypsum mortars point out the feasibility of using them in plastering mortars for indoor and outdoor environments, structural mortar (masonry foundation and masonry below grade), in addition to self-levelling mortars.

- 12) As far as the concrete compressive strength is concerned, the compounds that are produced with cementitious systems ranged between 38.6-80 MPa, accomplishing the Eurocode 1992-1-1 requirements. The cementitious systems Di-PG + CSA + GGBFS and Di-PG + FA + Lime do not meet the minimum requirements because they have a compressive strength below 25 MPa. All the analysed mixtures, with partial replacement of cement by PG, accomplish the Euro Code 1992-1-1 prescriptions.
- 13) Regarding durability, the data show that the plaster, regardless of the mineral phase, has high water absorption and low resistance to compression. However, cementitious systems and partial substitutions of PC for PG, have less water absorption and greater compressive strength. The drying shrinkage of the SAC/PC binary cement system is lower compared to HCBPG. The expandability of the Plaster is low compared to the binary system PG + PC and HCBPG, in all mineral phases.
- 14) Phosphogypsum can also be applied in geotechnical materials, self-levelling mortars, blocks, tiles, slabs, lime, binders. Most of the studies were developed with Di-PG and  $\beta$ -PG.
- 15) Other forms of PG valorization occur with the production of portlandite and sodium sulphate. CO<sub>2</sub> capture contributes to the mitigation of PG, as well as the treatment of bauxite and use in agriculture.
- 16) The health risks, due to radiation exposure of phosphogypsum building materials, depends on the origin of the PG, as well as the density and thickness of the material. The literature showed that up to 50% phosphogypsum can be used without causing contamination by radon-222 and up to 30% of PG can be used without causing contamination by radio-226.

The data mentioned in this paper show an overview on the feasibility of using phosphogypsum in cementitious materials accomplishing relevant requirements of the current Standards. The use of PG of sedimentary origin is still a challenge, and therefore ways of controlling or minimizing the effect of radiation should be studied, as well as the application of raw state PG (igneous and sedimentary). In addition, it is also necessary to research on methods of removing impurities in an



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1 economical way. In future research the studies on high performance concrete, hydration of  
2 cementitious systems, stabilized mortar, high workability concrete, (by using PG-based  
3 cementitious systems), could contribute to foster both the research and the use of more to eco-  
4 efficient building materials. Moreover, circular economy would be promoted within the  
5 construction industry by valorizing the phosphogypsum: now it is waste but a it could be a raw  
6 material in the future. This could contribute in reducing the negative environmental impacts  
7 construction sector produces.  
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12 **17 Appendix: supplementary data**  
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**Table 8 - Appendix A: Studies on phosphogypsum pastes**

25	26 Author	27 Year	28 Phosphogypsum (PG) mineral phase				29 Supplementary cementitious materials							30 Cement types					31 Alkali-activated materials		32 Cementitious systems					33 % Weight the raw materials															
			Di-PG	A-PG	α-PG	β-PG	GGBFS	FA	SS	EAFS	BA	CSR	SF	PC	PS	PL	CAC	CSA	Lime	Solution	SSC	B	T	QA	QI	PG	GGBFS	FA	SS	EAFS	BA	CSR	SF	PC	PS	PL	CAC	CSA	Lime		
28	Bijen and Niel [206]	1981	x				x					x							x	x				14-15	83-79	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
29	Abeni et al. [212]	1988	x					x										x					20	-	60	-	-	-	-	-	-	-	-	-	-	-	-	-	-	40	
30	Singh and Garg [142]	1990		x			x												x				-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-		
31	Singh and Garg [142]	1990			x		x											x					-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-		
32	Singh and Garg [160]	1992			x		x					x								x	x		-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-		
33	Singh and Garg [160]	1992			x		x					x								x			-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-		
34	Demir and Ölmöz [191]	1993		x															x	x			10-18	77-85	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
35	Singh and Garg	1993			x		x					x									x		-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-		
36	Beiz et al. [189]	1994			x		x	x				x						x			x		18,5-28,5	61,5-69	43-50	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	1-28,5
37	Singh and Garg [149]	1995		x			x												x				-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-		
38	Sherman et al. [307]	1995	x				x	x					x							x			-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
39	Singh, Garg, kumar [213]	1996			x		x											x					-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
40	Singh and Garg [143]	1996			x		x					x							x	x			40	-	40	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	20
41	Singh and Garg [146]	1997			x		x					x						x					-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-		
42	Singh and Garg [250]	2000			x		x					x								x			-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
43	Singh and Garg [150]	2000	x				x					x									x		-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
44	Singh and Garg [173]	2002			x		x					x									x		15	75	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
45	Singh and Garg [173]	2002			x		x					x									x		15	75	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
46	Singh and Garg [173]	2002		x			x					x									x		15	75	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
47	Mishra et al. [214]	2008			x			x										x			x		15	-	68-85	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	15-35
48	Delgado et al. [192]	2011		x			x					x			x								15,5	85	-	-	-	-	-	-	-	-	-	-	-	-	-	-	73,5	-	
49	Lucas et al. [200]	2011			x					x	x										x		60-85	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
50	Sharma et al. [308]	2012	x				x					x								x	x		-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
51	Mishra et al. [201]	2012	x				x	x				x							x				-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
52	Bovorkin et al. [309]	2013	x				x					x							x	x			5-10	80-85	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	5-10
53	Chen et al. [310]	2014	x				x			x		x							x				45	48	-	2	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
54	Kuczyk et al. [202]	2013	x						x	x													12,5-30	-	-	-	19-38,85	13,4-22	-	-	-	-	-	-	-	-	-	-	-	-	-
55	Kashad [215]	2015			x			x													x		5-15	-	85-100	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
56		2016	x				x				x										x		50	30	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	2
57	Wang, Yao [190]	2016	x				x					x											50	30	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	2-6
58		2016	x				x					x											50	30	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
59	Gracioli et al. [311]	2017	x				x													x	x		10-20	89-90	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
60	Suljanis et al. [312]	2018			x																x		-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-		
61	Liu and Wang [203]	2018	x									x	x										-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-		
62	Lan [313]	2018	x				x					x									x		30-60	30-60	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	5-10
63	Kramar et al. [210]	2019					x														x		20	5-10	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
64	Wang, and Yu [209]	2019	x				x					x									x		15	81	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
65	Cheels et al. [178]	2019	x				x					x									x		10	90	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-

Abbreviations: α-PG: α-hemihydrate; A-PG: Anhydrite; A-PG-II: Anhydrite-II; A-PG-III: Anhydrite-III; β-PG: B-hemihydrate; B: Binary; BA: Bottom ash; CAC: Calcium aluminate cement; CRS: Carbonate silica rock; CSA: Sulfaluminate cement; Di-PG: Dihydrate; EAFS: Electric arc furnace fteel slags; FA: Fly ash; FGD: Flue-gas desulfurization gypsum  
GGBFS: Ground granulated blast-furnace slag; H-CABPG: Hydraulic Cement Based on Phosphogypsum; LS: Limestone; PC: Portland Cement; PG: Phosphogypsum; PL: Portland cement limestone; PS: Portland cement slag; QA: Quaternary; QI: Quinary; RM: Red mud; SF: Silica fume; SS: Steel slag; SSC: Supersulphated cement; T: Ternary

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**Table 9-Appendix B: Studies on phosphogypsum mortars**

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Author	Year	Phosphogypsum mineral phase						Supplementary cementitious and Alkali-activated							Cement types					Cementitious systems			% Weigh the raw materials							Studies parameter			
		Di-P-G	A-P-G	A II-PG	A III-PG	α-PG	β-PG	GGBFS	SS	FA	RM	LS	Clay	Lime	PC	PS	PL	CAC	CSA	B	T	QA	PG	GGBFS	FA	PC	Lime	clay	SS	Water/binder	Binder/Sand	Flow (mm)	
Singh and Garg [160]	1992					x	x							x					x			-	-	-	-	-	-	-	-	-	-	1:2-1:5	105
Singh and Garg	1992					x								x					x			-	-	-	-	-	-	-	-	-	-	1:2-1:5	105
Singh and Garg	1999								x				x									10		45		20	25		0,5	1:3-1:5	100-105		
Singh and Garg [174]	2001					x	x							x					x			65-										1:2-1:5	-
Singh and Garg [174]	2001					x								x					x			65-										1:2-1:5	-
Singh and Garg [174]	2001					x								x					x			65-										1:2-1:5	-
Singh and Garg [174]	2007			x			x						x						x			8-14	91-85			1			0,5	2,45		-	
Mun et al. [175]	2007	x		x			x						x						x		x	2-10	87			1			0,5	2,45		-	
Mun et al. [175]	2007			x			x						x						x			12-	87-			1			0,5	2,45		-	
Mun et al. [175]	2007			x			x						x						x			11	87-79			2-10			0,5	2,45		-	
Mun et al. [175]	2008	x		x		x							-	x					x			5-10			87-			0,45	2		-		
Mun and So [222]	2008	x		x		x							-	x					x			7,5		2,5	90			0,45	2		-		
Mun and So [222]	2008	x		x		x		x					-	x					x			7,5	2,5		90			0,45	2		-		
Mun and So [222]	2008	x		x		x		x					-	x					x		x	7,5	1	1,5	90			0,45	2		-		
Huang and Li A	2010	x					x		x				-								x	25-	22,2-								5-13,6	-	
Huang and Li B	2010	x					x		x				-									45	40								5	-	
Garg and Pundir	2012					x							x	x								8-10		56-	15-	5-20			1:6	1:3-1:6		-	
Garg and Pundir	2012	x											x	x							x	33		42	20	3			2:1	1:3	342-375		
Ren et al. [158]	2012					x							x	x							x	33		42	20	3			2:1	1:3	342-375		
Ren et al. [158]	2012			x									x	x							x	33		42	20	3			2:1	1:3	342-375		
Hyung et al. [221]	2015	x					x						x						x			7-15	92-84			1-1,5			-	-	-	-	
Hyung et al. [221]	2015			x			x						x						x			7-15	92-84			1-1,5			-	-	-	-	
Huang et al. [314]	2016	x											x	x					x			45			3-	30-50			1:2	1:3		-	
Ren et al. [159]	2017					x							x	x							x	52		33	10	3			2:1	1:3	190-270		
Li et al. [315]	2018						x						x								x	90-		7,5-		2,5-			0,2-1,0	1:1-1:4		-	
Wang et al. [220]	2019	x					x						x								x	-	-	-	-	-		0,5	0,25		-		
Wang et al. [220]	2020			x									-	x					x			10-			50-			0,5	0,5	175-325		-	
Gong et al. A [156]	2020	x												x					x			10-			50-			0,5	0,5	175-325		-	
Gong et al. A [156]	2020													x					x			10-			50-			0,5	0,5	175-325		-	
Gong et al. B [157]	2020												x	x					x			-	-	-	-	-		-	-	-	274-320		

**Abbreviations:**

α-PG: α-hemihydrate; A-PG: Anhydrite; A-PG-II: Anhydrite-II; A-PG-III: Anhydrite-III; β-PG: B-hemihydrate; B: Binary; BA: Bottom ash; CAC: Calcium aluminate cement; CRS: Carbonate silica rock; CSA: Sulfoaluminate cement; Di-PG: Dihydrate; EAFS: Electric arc furnace steel slags; FA: Fly ash; FGD: Flue-gas desulfurization gypsum; GGBFS: Ground granulated blast-furnance slag; HABPG: Hydraulic alternative binder phosphogypsum-based; LS: Limestone; PC: Portland Cement; PG: Phosphogypsum; PL: Portland cement limestone; PS: Portland cement slag; QA: Quaternary; QI: Quinary; RM: Red mud; SF: Silica fume; SS: Steel slag; SSC: Supersulphated cement; T: Ternary

**Table 10-Appendix C: Studies on phosphogypsum**

Ref.	Year	Phosphogypsum mineral phases				Cement types		Supplementary Cementitious and Alkali-activated Materials					Cementitious systems				Proportion of raw materials (W%)								Parameter studied						
		PG-Di	α-PG	β-PG	PG-A	PC	CSA	GGBFS	FA	SF	SS	Lime Hydrated	Alkaline Activating Solutions	T	QA	QIA	Replacement partial	PG	PC	CSA	GGBFS	FA	Lime	SS	SF	Water/binder	Optimus water/binder	Optimus % PG	Fineness modulus (mm)		Effect strength the rep pho
																										Fine aggregate		Coarse aggregate			
Smaddi et al. [241]	1999			x		x										X	10-90	10-90	-	-	-	-	-	-	-	-	0,6	10	-	-	
	1999				x	x										X	10-90	10-90	-	-	-	-	-	-	-	-	0,6	10	-	-	
Diop et al. [244]	2005	x				x									x	1-10	20-80	-	-	-	-	-	-	-	-	-	-	4,5	-	-	
Reddy et al. [242]	2010	x				x									x	10-40	60-90	-	-	-	-	-	-	-	0,4-0,65	0,4	10	2,9	6,9		
Bagade and Satone [245]	2012	x				x									x	10-20	80-90	-	-	-	-	-	-	-	0,4	0,4	10	2,9	6,9		
Yang et al. [237]	2013	x				x	x				x				x	0-55	15	-	0-35	-	2-16	-	-	-	0,43-0,45	0,45	55	-	-		
Ding et al. [238]	2014	x				x	x			x					x	45	48	-	5	-	-	2	-	-	-	-	45	2,73	-		
Dhinakaran and Shanthi [247]	2015	x				x									x	2-10	90-98	-	-	-	-	-	-	-	-	0,5	8	-	-		
Nigade and Bagade [267]	2015	x				x									x	5-25	75-95	-	-	-	-	-	-	-	0,45 - 0,50	-	10	-	-		
	2015				x		x				x				x	12	-	-	87	-	1	-	-	-	-	-	12	-	6,68		
	2015	x					x				x				x	6	-	-	87	-	1	-	-	-	-	-	6	-	6,68		
Yoo et al. [163]	2015	x			x		x				x				x	17	-	-	82	-	1	-	-	-	-	-	17	-	6,68		
	2015				x		x				x				x	11	-	-	85	-	4	-	-	-	-	-	11	-	-		
	2015				x		x				x				x	11	-	-	81	-	8	-	-	-	-	-	11	-	-		
Tian et al. [234]	2016	x				x		x							x	40-70	-	25	-	20-50	0-12	-	-	-	-	-	-	45-55	-	-	
	2016	x				x	x								x	40-70	-	25	20-50	-	0-12	-	-	-	-	-	-	45-55	-	-	
Buhari and Raju [161]	2016	x				x			x						x	5-15	75-90	-	-	-	-	-	-	10	-	0,45	10	3,71	3,87		
Islam et al. [246]	2017	x				x									x	5-15	85-95	-	-	-	-	-	-	-	-	0,45	10	2,7	-		
Srinivasulu and Raghava [162]	2017	x				x									x	2,5-10	90-97,5	-	-	-	-	-	-	-	-	-	7,5	2,8	7,2		
Sukmana et al. [239]	2019	x				x				x		x			x	35-45	26-34	-	-	-	8-12	-	-	-	-	-	35	-	-		

**Abbreviations:**

α- PG: α-hemihydrate; A-PG: Anhydrite; A-PG-II: Anhydrite-II; A-PG-III: Anhydrite-III; β- PG: B-hemihydrate; B: Binary; BA: Bottom ash; CAC: Calcium aluminate cement; CRS: Carbonate silica rock; CSA: Sulfoaluminat cement; Di-PG: Dihydrate; EAFS: Eletric arc furnace feel slag; FGD: Flue-gas desulfurization gypsum; GGBFS: Ground granulated blast-furnance slag; HABPG: Hydraulic alternative binder phosphogypsum-based; LS: Limestone; PC: Portland Cement; PG: Phosphogypsum; PL: Portland cement limestone; PS: Portland cement slag; QA: Quaternary; Red mud; SF: Silica fume; SS: Steel slag; SSC: Supersulphated cement; T: Ternary

**Table 11- Appendix D: Comparison of cement-based studies with and without Portland cement - mortars**

<b>Mortar without cement Portland - EN 998-1, EN 998-2, EN 1015-2, EN 13454-2 and ASTM C270</b>																
PG phases	Ref.	Compressive strength (MPa)	Flow (mm)	Water/ binder	Binder/ sand	Setting time				%Formulation						
						Initial	Final	PG	FA	GGBFS	Lime	PC	SS	LS	Chemical activator	
Anhydrite II	[175]	70	-	0,5	2,45	05:15	09:30	12		91	1	-	-			
	[221]	42	-	-	-	-	-	7		92	1	-	-			
β-hemihydrate Dihydrate	[148]	6,3	100-105	0,5	1:3	02:21	05:10	-	-	-	-	-	-			
	[175]	60	-	0,5	2,45	06:05	09:40	17		82	1	-	-			
	[176]	47	-	-	1:3	08:05	11:57	33		41,4			11,8	11,8		
	[177]	44,7	-	-	-	02:19	04:12	45		40			-	-	14	1
	[221]	40	-	-	-	-	-	12		87	1,5	-	-	-	-	-
[220]	20.5	-	-	0,5	0,25	-	-	-	-	-	-	-	-	-	-	-
Dihydrate + Anhydritell	[175]	65	-	0,5	2,45	05:30	09:15	12 (6+6)		87	1	-	-			
<b>Mortar with cement Portland EN 998-1, EN 998-2, EN 1015-2, EN 13454-2 and ASTM C270</b>																
PG phases	Ref.	Compressive strength (MPa)	Flow mm	Water/ binder	Binder/ sand	Setting time (min)				%Formulation						
						Initial	Final	PG	FA	GGBFS	Lime	PC	SS	LS	Chemical activator	
α-hemihydrate	[158]	20	342-375	2:1	1:3	1404	2020	33	42	-	3	20	-	-		
	[145]	10	-	1:6	1:3	22	38	15	60	-	5	20	-	-		
Anhydrite II	[159]	31,8	190-270	2:1	1:3	-	-	52	33		3	10	-	-		2
	[222]	72	-	0,4	2	-	-	7,5	2,5			90	-	-		
	[158]	30	342-375	2:1	1:3	517	705	33	42		3	20	-	-		
	[156]	40	275	0,5	0,5	-	-	30				70	-	-		
Anhydrite III	[222]	70	-	0,45	2	-	-	7,5		2,5		90	-	-		
	[156]	30	325	0,5	0,5	-	-	-				-	-	-		
β-hemihydrate	[160]	13,31	105	-	-	70	145	-	-	-	-	-	-	-	-	-
	[222]	60	-	0,45	2	-	-	7,5	1,5	1		9	-	-		
	[157]	36,71	283	-	-	311	493	-	-	-	-	-	-	-	-	-
β-hemi (PC+PG)	[222]	70	-	0,45	2	-	-	7,5				92,5	-	-		
Dihydrate	[158]	15	342-375	2:1	1:3	2934	3625	33	42	-	3	20	-	-		
	[243]	45	-	1:2	1:3	-	-	45		48		7	-	-		
	[156]	34,5	225	0,5	0,5	-	-	30				70	-	-		1

**Table 12- Appendix E: Comparison of cement-based studies with and without Portland cement - Concrete**

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**Replacement Portland cement by phosphogypsum – EN 206:2013 and Eurocode 1992**

Phosphogypsum phases	Ref.	Compressive strength (MPa)	Strength class (MPa)	Setting time		%Formulation							Fineness modulus		Effect on compressive strength in respect to the optimum replacement of PG			
				Initial	Final	PC	PG	GGBFS	Lime	SS	Chemical activator	aluminium powder	Water/binder	Fine aggregate		coarse aggregate		
β-hemihydrate  Dihydrate	[246]	36,33		175	285	90	10	-	-	-	-	-	-	0,45	2,7	-	-	
	[244]	32,5	32,5	-	-	90	4,5	-	-	-	-	-	-	-	-	-	-	
	[242]	49,3	53	35	190	90	10	-	-	-	-	-	-	0,4	2,9	6,9	25 % increased	
	[245]	30,15	53	185	870	90	10	-	-	-	-	-	-	0,4	2,9	6,9	-	
	[247]	27,93	-	-	-	-	92	8	-	-	-	-	-	-	0,5	-	-	20 % increased
	[246]	32,2	-	-	72	159	90	10	-	-	-	-	-	-	0,45	2,7	-	-
	[162]	38,78	-	-	-	92,5	7,5	-	-	-	-	-	-	-	2,8	7,2	-	

**Concrete with cementitious systems - EN 206:2013 and Eurocode 1992**

Phosphogypsum phases	Ref.	Compressive strength (MPa)	Strength class (MPa)	Setting time		%Formulation							Fineness modulus		Effect on compressive strength in respect to the optimum replacement of PG		
				Initial	Final	PC	PG	GGBFS	Lime	SS	Chemical activator	aluminium powder	Water/binder	Fine aggregate		coarse aggregate	
Dihydrate +Anhydrite II Anhydrite II Dihydrate	[163]	70	-	-	-	-	12 (6+6)	87	1					-	-	6,8	-
	[237]	48	-	-	-	15	55	30	7	1,6	0,074			-	-	6,8	-
	[238]	38,6	-	-	-	5	45	48	-	2				-	2,73	-	-
	[163]	75	-	-	-	-	17	82	1					-	-	6,8	-
	[316]	2,92	-	-	-	-	25	50	25					-	-	-	-
	[239]	20,93	-	-	-	-	34	35		10				-	-	-	-

**Table 13- Appendix F: Water-resistant phosphogypsum- Kovler (2001); Piasta and Zarzycki (2017); Selim et al (2020)**

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Mineral phase	Water Absorption (%)	Compressive Strength (MPa)	Composite binder	Water resistance coefficient	Ref.
Di-PG	2	32	PG+PC	Unknown	[156]
	3	22	PG+PC+GGBFS	Unknown	[160]
	10	19.6	PG+PC+FA	Unknown	[160]
α-PG	28	12	PG+FA+Lime	Unknown	[256]
	32	Unknown	PG	Unknown	[257]
	14	27.5	PG+FA+PC+Lime	Unknown	[145]
	19,6	26.4	PG+FA+Lime 50°C	Unknown	[141]
	4	32.2	PG+ GGBFS+ Marble dust+ chemical	Unknown	[141]
	leaching	13.20	PG activator 500C	Unknown	[142]
β-PG	Leaching	14	PG	Unknown	[171]
	30	Unknown	PG	Unknown	[257]
	30.5	Unknown	PG	0.35	[258]
	32.09	Unknown	PG	Unknown	[143]
	42	Unknown	PG	Unknown	[259]
	7	35	PG+PC+GGBFS	Unknown	[259]
	3	35	PG+PC+GGBFS	Unknown	[160]
	2,89	Unknown	PG+PC+GGBFS+	Unknown	[143]
	6	35	PG+PC+GGBFS+	Unknown	[249],
	10	21.3	PG+FA+PC	Unknown	[160]
	6.5	25.6	PG+FA+PC	Unknown	[171]
	10	20	PG+FA+PC 50°C	Unknown	[146]
	8	25	PG+FA+PC+ retarder	Unknown	[250]
	21.6	4.11	PG + FA + Lime	Unknown	[260]
	10	12	PG+FA+Lime	Unknown	[256]
22	Unknown	70%PG+30%SAC	0.70	[258]	
A-PG III	Unknown	Unknown	Unknown	Unknown	Unknown
A-PGII	10.43	56	PG+GGBFS	Unknown	[142]
	9.55	35	PG+GGBFS	Unknown	[149]
A-PG I	4	34.5	PG+PC	Unknown	[156]
	4	40	PG+FA+Red Mud	Unknown	[21]

Table 15-

G: Expansibility cold (soundness) – EN 15743/ EN 197-1

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Mineral phases	Soundness Cold expansion (mm)	%PG	System	binder	Ref.
<b>Di-PG</b>	0.6	100	PG	Plaster	[174]
	6	12.5	PG +PC	Parcial replacement	[27]
	8	20	PG +PC	Parcial replacement	[245]
	9	25	PG +PC	Parcial replacement	[267]
	3.6	Unknown	PG+PC+GGBFS	HABPG	[160], [174]
	1.10	Unknown	PG+PC+FA	HABPG	[160], [174]
	1.6	Unknown	PG+PC+Red Mud	HABPG	[174]
	13.5	45	PG+PC+GGBFS+SS+LS+NaOH	Supersulphated	[177]
<b>α-PG</b>	<b>2.3</b>	<b>10</b>	<b>PG+PC+FA+Lime</b>	<b>HABPG</b>	[145]
<b>β-PG</b>	0.6	100	PG	Plaster	[174], [171]
	0.1	70	PG+PC	Parcial replacement	[241]
	0.106	70	PG+PPC POZOLANIC	Parcial replacement	[241]
	1.60	Unknown	PG+PC+GGBFS	HABPG	[160], [174], [259]
	1.60	Unknown	PG+PC+GGBFS+ organic retarder	HABPG	[249], [257]
	1.2	15	PG + GGBFS + tartaric acid	HABPG	[173]
	0.88	Unknown	PG+PC+FA	HABPG	[160]
	0.88	Unknown	PG+FA+PC+ retarder	HABPG	[250]
	1.00	Unknown	PG+PC+FA	HABPG	[174]
	1.8	Unknown	PG+PC+FA 50°C	HABPG	[146]
	2.0	70-75	PG+FA+PC+Lime+chemical additives	HABPG	[171]
	1.1	Unknown	PG+PC+Red Mud	HABPG	[174]
	12	45	PG+GGBFS+SS+SL	Supersulphated	[176]
<b>A-PG III</b>	<b>Unknown</b>	<b>Unknown</b>	<b>Unknown</b>	<b>Unknown</b>	<b>Unknown</b>
<b>A-PG II</b>	0.5	100	PG	Plaster	[142]
	0.81	70	PG+GGBFS	HABPG	[149]
	0.90	15	PG+GGBFS	HABPG	[173]
	1.5	Unknown	PG+ GGBFS+ chemical activator	HABPG	[142]
	3.0	10	PG+PC+GGBFS	Supersulphated	[191]
<b>A-PG I</b>	0.06		PG+ GGBFS+2% citric acid	HABPG	[21]



**Table 16-Appendix H: Application of phosphogypsum in geotechnical materials in construction and components**

Ref	Year	Phosphogypsum mineral phase				Raw materials									Cement types			Cement based Phosphogypsum				Application in other materials building studies							Studies parameter		
		Di-PG	A-PG	α-PG	β-PG	GGBFS	FA	SS	Clay	Adobe	Fibre	RM	Fluoro	LH	PC	CSA	CAC	B	T	QA	QI	Road base	Brick and Blocks	Tile and Ceramic	Binder	Plaster	Self-leveling	Board	%PG	Optimum % replacement of phosphogypsum	Study objective
Singh and Garg [249]	1992				x					x															x				-	-	Binder
Singh and Garg [160]	1992				x	x								x				x				x		x					-	-	Binder
Singh and Garg [259]	1993				x		x							x				x					x						-	-	Binder
Singh and Garg [257]	1994				x					x														x					-	-	Plaster board
Gutti et al. [317]	1996	x												x			x					x						8	-	Soil stabilization	
Singh and Garg [165]	2000		x																				x					-	-	Anhydrite cement	
Kumar [318]	2000				x						x						x					x						10-40	-	Binder	
Singh and Garg [174]	2001				x																			x				-	-	Plastering	
Kumar [260]	2003				x									x														10-40	-	Binder	
Pérez and Ambroise [319]	2004	x												x	x		x									x		25	-	Topping mortar	
Singh and Garg [144]	2005		x								x	x					x											-	-	Plastering	
Degimercı et al. [278]	2007	x									x			x			x				x							2,5-5	-	Soil stabilization	
She et al. [320]	2007	x												x			x				x							0-92	18-23	Binder	
Degimercı et al A [261]	2008	x												x			x					x						10-50	-	Binder	
Degimercı et al B [321]	2008	x												x			x					x						10-50	-	Binder	
Ajam et al. [322]	2009	x									x											x						-	25	adobe stabilization	
W. Shen et al. [323]	2009	x									x						x				x							5-40	30	Fired clay	
Yang et al. [324]	2009	x									x						x					x						0-5	2,5	solidified material	
Yang et al. [324]	2009				x						x						x					x						35	-	Wall	
Garg, Mirrocha and Jain [96]	2011				x						x						x							x				30-50	30	Wall plaster	
					x						x													x				-	100	plaster	
					x						x														x			-	-	Multiphase	
					x						x																	-	-	Binder	
					x						x																	-	-	boards	
					x						x																	-	-	flooring	
Garg and Pudir [145]	2012				x																				x				-	100	Plaster
					x																			x				8-15	15	Binder	
Zhou et al. [325]	2014				x																								8-15	15	Binder
Yang, Zhang and Yan [265]	2016	x																										75	65-85	Non-fired mortar	
Zhou et al. [326]	2016				x																							40-55	45	Green tiles	
Dutta et al. [273]	2017	x																										-	100	Geotechnical underlayments	
Schaefer et al. [116]	2017		x																									2-10	2	geotechnical underlayments	
	2017				x																							-	19,8	underlayments	
Contreras et al. [327]	2018	x																										-	19,8	underlayments	
Ding, Garlwen et al. [275]	2019	x																										5-10	7,5	Additive	
Qiang et al [274]	2019	x																										-	26	Stabilized soil	
Romero-Hermida et al. [328]	2019	x																										-	-	mortar	
	2019				x																							-	-	Rheological characteriza lime	
Silva et al. [329]	2019				x																							-	11	Pavements	
Zhou et al. [276]	2019				x																							-	41	Pavements	
	2019				x																							-	100	non-fired ceramic	

**Abbreviations:**  
 α-PG: α-hemihydrate; A-PG: Anhydrite; A-PG-II: Anhydrite-II; A-PG-III: Anhydrite-III; β-PG: β-hemihydrate; B: Binary; BA: Bottom ash; CAC: Calcium aluminate cement; CRS: Carbonate silica rock; CSA: Sulfoaluminate cement; Di-PG: Dihydrate; EAFS: Electric arc furnace steel slags; FA: Fly ash; FGD: Flue-gas desulfurization gypsum; GGBFS: Ground granulated blast-furnace slag; HABPG: Hydraulic alternative binder phosphogypsum-based; LS: Limestone; PC: Portland Cement; PG: Phosphogypsum; PL: Portland cement limestone; PS: Portland cement slag; QA: Quaternary; QI: Quinary; RM: Red mud; SF: Silica fume; SS: Steel slag; SSC: Supersulfated cement; T: Ternary

Table 17-Appendix I: Study of radiation in building materials with phosphogypsum

Building materials	% PG	Indoor (Rn-222)		Outdoor (Ra-226)		Geological origin		Geographical location of the extraction site	Ref.
		Activity concentration (Bq/m <sup>3</sup> )	Exhalation rate (Bq m <sup>-2</sup> h <sup>-1</sup> )	Activity concentration Index (I)	Radium equivalent activity (Ra <sub>eq</sub> ) (Bq/kg)	Sedimentary	Igneous		
Plaster α-PG	100	-	-	0,36-0,51	-	x		Russia	[330]
Portland cement Paste	3-5	28,27-36,49	<b>a</b>	-	-	x		Syria	[331]
Binary system cement paste	10	281-464	-	0,8	190		x	Russia	[295]
Ettringite-based paste	10-30	-	-	0,2-1,00	50-300		x	Russia and Finlandia	[101]
Lime mortar	-	-	-	0,8	250	x		Morocco	[332]
Ettringite-based mortar	10-30	-	<b>b</b>	0,1-0,2	50-100		x	Russia and Finlandia	[101]
Mortar	-	-	-	0,227	68,2 ±0,9		x	Brazil	[333]
Portland cement concrete	3-5	63-81	<b>c</b>	-	-	x		Syria	[331]
Sulfur Polymer cemente/concrete	10-30	-	-	0,43-0,75	117-253	x		Morocco	[299]
Sulfur Polymer cemente/ concrete	8,1-8,4	-	-	0,36-0,42	-	x		Morocco	[334]
Sulfur Polymer cemente/ concrete	<b>d</b>	-	<b>d</b>	-	61,5-74,1	x		<b>e</b>	[335]
Sulfur Polymer concrete	-	10-50	-	-	-	x		Moroco	[298]
Bricks	-	-	0,11-1,2	-	25-400		x	Brazil	[336]
Bricks	-	100-300	6-10,2	-	-		x	Brazil	[296]
Bricks	25	15-125	-	-	-	x		Tunisia	[337]
Brick	10-50	-	1-1,5	-	-	x		Morroco	[298]
Brick	-	-	0,41-5,67	-	84-780		x	Brazil	[297]
Board	100	78,07-136,16	-	-	259-289	x		South of korea	[338]
Plate	100	-	0,19-1,3	-	25-400		x	Brazil	[336]
Plate	100	47-99	-	0,2-2,6	-		x	Brazil	[339]
Plate	-	67-196	4-11,7	-	-	x		Morocco	[298]
Plate	-	-	0,16-4,30	-	55-755		x	Brazil	[297]
Ceramic tiles	5-10	-	-	0,76-4,45	15-106	x		Morocco	[271]

**Note:**

**a:** 8,89-10,83 JmBq m<sup>-2</sup> h<sup>-1</sup>; **b:** 30-98,3 mBq/(kg\*h); **c:** 75,8-92,5 JmBq m<sup>-2</sup> h<sup>-1</sup>; **d:** 232-438 mBq m<sup>-2</sup> h<sup>-1</sup> and PG of 0,091 (w/w)

**e:** The author does not inform the country that the phosphogypsum was generated, only that it was generated on the African continent

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**No conflict of interest**