Chapter 10

By-product Phosphogypsum Valorisation Possibilities in the Context of Circular Economy of Building Materials

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Abstract Phosphogypsum (PG) is the most significant solid waste in the world. However, only 15% of it is recycled, and the rest is stored in useless and environmentally-damaging stockpiles, which are continually growing. Therefore circular economy of PG through its utilisation in useful applications, is relevant and critical from an ecological perspective. In this context, its utilisation in building materials is among the most promising and attractive recycling possibilities, since PG typically exhibits excellent binding properties. However the applicability of PG is often restrained by its radiological risks and economic non-competitivity in comparison to traditional building materials, such as natural gypsum. In some cases, these obstacles refer to cultural prejudices and misinformation rather than real issues. Therefore, through a comprehensive literature review, this article considers the real potential of PG in building materials, providing useful information to interested stakeholders.

Keywords Phosphogypsum · Waste valorisation · Building materials · Circular economy

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10.1 Introduction

In the context of global warming and sustainable use of energy and natural resources, the need to develop real circular economies (CE) is becoming of critical importance. The main goals of CE are: '(1) eliminate waste and pollution; (2) circulate products and materials; (3) and regenerate nature' [1]. In this sense, the recycling of phosphogypsum (PG), the "world's most significant solid waste [2], is crucial for CE on a global scale since it directly relates to the first and second CE goals and, indirectly, to the third one".

PG is a by-product of the industry of mineral fertilisers, which, nowadays, are irreplaceable in agricultural activities. The main nutrients needed for plant growth are nitrogen, potassium, and phosphorus [3]. However, the production of these fertilisers carries notorious secondary effects, some of which are hazardous to the environment. The case of phosphorus-based fertilisers deserves special attention, as they are responsible for the production of the leading solid residue in the world. In fact, every year the PG global production increases and currently it is about $1.8–2.8 \times 10^8$ t [2]. However, until now, most of the produced by-product, approximately 85%, has been stored in open dumps or discharged in water bodies, and only with 15% of it being recycled.

The most problematic issue related to long-term storage is not only the fact that humungous stockpiles occupy extensive area but also cause other kinds of ecological damage: radionuclides, waste-soluble phosphates, fluorides, sulphates, and many heavy metals leach into underground waters and end up being absorbed by plants, thus seriously harming the entire ecological environment [4, 5]. Heavy metals migrate along the food chain and enter the human body [6].

Considering the global relevance of this problem, it is critical for CE to find effective ways to utilise PG. Abundant research is carried out in this area, but the obtained results do not necessarily reach lead to implementation due to economic, regulatory, or even cultural reasons [7]. Thus, the current publication aims to review the PG utilisation possibilities in the field of building materials, thus providing PG use cases which might be of interest to stakeholders of both the public and private sector stakeholders.

To address this objective, a comprehensive literature review has been carried out, within the *Google Scholar* database, using keywords "phosphogypsum utilisation", "building materials", "binding materials", "press-forming", "agriculture", "radioactivity", and pertinent synonyms.

10.2 Characterisation of Industrial By-product Phosphogypsum

PG is an undesirable by-product resulting from the production process of phosphate fertilisers. The raw material used during the process consists mainly of phosphate ores of igneous or sedimentary nature [8]. According to the mineral composition, impurities and microstructure, the main minerals of these rocks belong to the apatite or phosphorite group, for which the general chemical formula is $Ca_{10}(PO_4,\pm CO_3,\pm OH)_6(OH,F,Cl)_2$ [7]. To produce fertilisers, phosphate ores are processed into orthophosphoric acid (H_3PO_4), through the so-called "wet process", during which the phosphate rock is digested by concentrated sulfuric acid [7]. Furthermore, it can be clarified that H_3PO_4 is not the final product but an intermediate feedstock to manufacture the final phosphate fertilisers [8]. The general reaction is provided by the International Atomic Energy Agency (IAEA) [7] in Eq. (10.1). This reaction occurs in two stages: first, the phosphate raw material reacts with a mixture of sulfuric and phosphoric acids (the latter formed during the process); then, subsequently, calcium sulphate compounds crystallise.

$$Ca_5(PO_4)_3F + 5H_2SO_4 + 5nH_2O \rightarrow 5CaSO_4 \times nH_2O + HF + 3H_3PO_4 \quad (10.1)$$

Equation (10.1) reveals that in addition to H_3PO_4, a by-product based on calcium sulphate crystals ($CaSO_4$) and known as PG is produced. For each tonne of orthophosphoric acid, up to 4.5–5.5 t of PG by-product are generated [9, 10]. Depending on the process conditions (sulfuric acid concentration, temperature, phosphate raw material composition, and impurities), the solid phase of calcium sulphate can present three alternative forms: dihydrate (DH): $CaSO_4{\cdot}2H_2O$; hemihydrate (HH): $CaSO_4{\cdot}0.5H_2O$; anhydrous: $CaSO_4$.

According to the mineral composition, PG belongs to the group of gypsum raw materials, as it contains 80% to 98% calcium sulphate [4]. Furthermore, PG is typically characterised by a certain content of undesirable impurities, such as unreacted apatite or phosphorite and H_3PO_4, soluble and insoluble phosphate and fluoride compounds, salts of iron, aluminium and other heavy metals, REE and quartz [6, 11, 12]. PG usually contains an elevated radionuclide content [7, 13, 14], which significantly hinders its applicability.

10.3 A Sustainable Approach to the Utilisation of PG

Currently, the main applications of PG, according to IAEA [7], are soil stabilisation and road base construction [15–18], agriculture and building materials. There are other promising PG valorisation opportunities and fields of activity, such as is the biochemical approach presented by Chernysh et al. [5], consisting in the exploitation of various bioprocesses, with PG valorisation from existing stockpiles

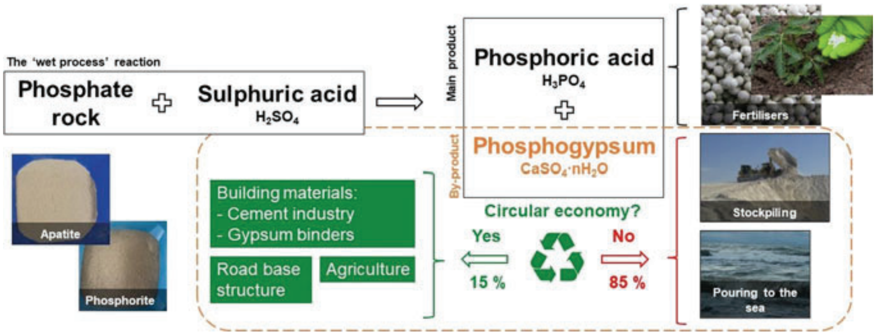

Fig. 10.2 Scheme of PG production, disposal, and main ways of utilisation

and implementing innovative biotechnological solutions for phosphorus raw materials processing. A schematic view of the PG production and main options for its utilisation is presented in Fig. 10.2. Although the current article focuses on the field of building materials, the utilisation of PG in agriculture is also commented on due to its relevance.

10.3.1 PG in Agriculture: Acidic and Saline Soils Improvement and Other Uses

Agricultural activity is one of the main ways to use PG on large scales, as it has shown good performance in soil, improving it in various ways.

First, PG can be used to improve acidic soils. As explained by Qi et al. [2], PG is an excellent alternative to more expensive inorganic improvements for acidic soils, such as natural gypsum (which is scarcely available) or lime, which exhibits poor solubility, so its nutrients within the soil profile are limited to a thin layer [19]. Acidic soils appear due to long-term fertilisation. The problems of acidic soils are related to toxic metals, especially exchangeable aluminium ions (Al^{3+}), and the lack of calcium [20], which is necessary for plants to grow. The addition of PG to the soil determines the reduction of the exchangeable aluminium ions of $Al(OH)_3$, binding them to $Al(OH)SO_4$ compounds. This process was named the "self-limiting effect" by Reeve and Sumner [21]. Moreover, PG is a suitable amendment for saline soils, effectively neutralising Na^+ ions typical of their composition [2].

PG treatment can also improve agricultural soils by enhancing their resistance to crusting [22] and rainwater runoff-induced erosion [23] or immobilising toxic heavy metals in the soil by binding them in complex and non-soluble metal-fluorine compounds [2]. In addition, PG can be used as a source material for calcium and sulphur, which are necessary elements for the growth of crops [24, 25]. In summary, PG can be used effectively to improve agricultural activities.

10.3.2 PG in Building Materials

According to the content of the primary substance, calcium sulphate, PG could be exploited as a raw material for cement and gypsum binders. However, compared to natural gypsum, the quality of PG is usually lower because of the acid-soluble impurities (phosphate and fluoride), which limit its use. Soluble phosphates prolong gypsum hydration and reduce gypsum specimens' compressive strength (*CS*) [26, 27]. It is also the main cause of a long setting time and a low early cement strength [26]. Therefore, extensive research has been conducted on neutralising or removing soluble phosphate and fluoride compounds from the PG. The principal methods are the following: washing of PG with water; neutralisation of impurities in citric, nitric, or sulphuric acid solutions with subsequent washing with water [26–28]; application of various neutralising, mineralising and crystallisation-regulating additives before dehydration of PG [29, 30]; thermal treatment and neutralisation in lime suspension [31, 32].

10.3.2.1 The Problem of Radioactivity

Radioactivity is the key factor that hinders the application of PG in building products. PG generally contains a relatively high amount of ^{226}Ra radionuclide, which decays to ^{222}Rn [33, 34], a toxic gas to which exposure in indoor environments causes between 3 and 14% of global lung cancer cases, according to the World Health Organisation [35]. This issue restricts the use of PG in building materials, as the competent authorities limit the radioactivity levels of building products. For example, the European Commission established a limit gamma emission rate of 1 mSv/year [36]. Usually, this limit is not depicted directly but is evaluated through the radioactivity concentration index I, calculated from the activity concentration of radionuclides ^{226}Ra, ^{232}Th, and ^{40}K. A material is considered safe from a radiological perspective when $I \leq 1.0$, if employed in bulk amounts (slabs, wall blocks, etc.) or $I \leq 6.0$, if used in superficial applications (wall coatings, ceilings, etc.) [37]. If this condition is not met, the material could be hazardous to human health, so additional investigations must be performed.

According to Kovacs et al. [38], the PG produced in most countries presented I value between 0.7 and 2.0, although in some cases (UK and Morocco), the values were much higher. This variety depends on the nature of the phosphate rock from which PG is processed [39–41]. For instance, the PG produced from igneous phosphate ores tends to exhibit a lower radioactivity level than those manufactured from sedimentary rocks. It must be considered that the imposed limits apply to the final building material, not to each ingredient. This is the main reason why PG is mainly utilised in small amounts as a supplementary material of OPC or asphalt (the final I index will not exceed the limit) and not so frequently as the main binding material since, in this case, the I limit could easily be surpassed. In the latter case, employing a PG with $\mathrm{I} \leq 1.0$ index is crucial.

10.3.2.2 PG as a Cement Retarder

One of the most important uses of natural gypsum is the manufacture of ordinary Portland cement (OPC), which is included in amounts of 2.5–5%, acting as an OPC setting time retarder [42, 43]. If gypsum was not present during mixing OPC with water, tricalcium aluminate compounds (C3A) would immediately hydrate into calcium hydro aluminate compounds (CAH), which would not allow further mixing. When there is gypsum, a layer of ettringite and calcium hydro silicates (CSH) precipitate on the surfaces of the cement particles, and so the start of hydration is postponed (by about 2 h), allowing workability for a longer time [42]. Therefore, a retarder such as gypsum is crucial for suitable OPC hydration. In addition, gypsum is a retarder of the setting time and acts as an accelerator of the early strength of OPC mixtures.

In the same way, PG could be utilised as an OPC retarder. The retarding effect could be attributed not only to the ettringite coating but also to the soluble content of phosphorus and fluorine in PG, which, during OPC mixing with water, precipitates in the form of phosphate and fluoride compounds ($Ca_3(PO_4)_2$ and CaF_2), forming a layer that hinders further cement hydration and determines a later setting time than OPC with gypsum [26]. However, this additional retarding effect caused by the fluoride and phosphate impurities is usually too long. Therefore, the purification of the PG from the soluble phosphate and fluoride compounds is necessary to obtain a good performance as an OPC retarder.

10.3.2.3 PG as a Binding Material

Most of the research on using PG in building materials attempts to explore the potential of PG as a secondary material that acts as an additive or modifier of the main binding material, such as OPC or asphalt, in building products or roads. The problem with this approach is self-evident: PG is used in rather small amounts. In contrast, it must be considered that PG, in essence, is the same material ($CaSO_4$) as natural gypsum, which is one of the most widely used binding materials in the world. Therefore, in the same way, PG can be used as the primary binding material for building products. Its utilisation rate would be much higher, allowing it to solve the environmental problems derived from PG stockpiling more efficiently. It must be observed that PG (and natural gypsum) exhibit binding properties only in its HH form. When mixed with water, it hydrates and hardens, achieving its DH phase [44].

Researchers are exploring ways to increase the applicability of PG as a binding material by improving its main properties: mechanical strength, degree of hydration, water resistance, thermal and acoustic insulation performance, etc. The main focus is on improving mechanical properties. The mechanical performance of PG mostly depends on its microstructure, chemical composition, and processing method through which the final products are obtained.

First, abundant research has been conducted on the influence of microstructure and chemical composition. Valančius et al. [45] created a laboratory imitation of the “wet process” to determine the influence of reaction conditions on the microstructure and properties of the produced PG. Applying wet process conditions of 8 h duration, 97 °C temperature, and 1.03 apatite/sulfuric acid ratio, the α-type HH PG crystals were regularly orientated and presented needle shapes. Later, the HH PG powder was ground and mixed with water. The suitable microstructure of the HH crystals determined the fast hydration of the pastes and the excellent mechanical properties of the hardened specimens, even reaching a *CS* of 27 MPa. In a similar way, Leškevičienė et al. [46] studied the influence of the mineralogical composition of HH-PG on the physical and mechanical properties of hardened specimens.

Moreover, the mechanical properties of DH PG can also be improved by processing the specimens in certain ways, such as adding fibre, press-forming, autoclaving, or treating with a constant magnetic field. Press-forming has proven to be a very effective method for improving the mechanical properties of specimens. Zhou et al. [47] suggested a processing method called ‘the two-step hydration process’, including press-forming and subsequent immersion in water, with which high-strength non-fired PG mortar bricks were manufactured. During the investigation, it was found that the higher the press-forming pressure determined higher *CS* values, even reaching a 7-day *CS* value of 32 MPa when the pressure was 20 MPa. However, for economic reasons, the author recommends using a lower press-forming pressure of 10 MPa, with which the specimens still present a satisfactory *CS* value of 29 MPa. Similarly, Villalon Fornes et al. [48] explored this method using plain PG (instead of mortar) and obtained high *CS* values. In addition, Zhou et al. employed intermittent press-forming hydration to produce load-bearing tiles [49] and plasterboards [50]. Therefore, strong PG products, such as tiles, plasterboards, bricks or blocks, could be created by applying a suitable processing method and a pertinent type of PG.

10.4 Discussion

10.4.1 Insights and Risks of PG Utilisation in Agriculture

As described in Sect. 10.3.1, PG exhibits suitable properties to be utilised to amend and improve certain soil, render the types to be suitable for agricultural purposes. However, certain aspects must be considered. PG contains noticeable amounts of P, F, and heavy metals, so it can contaminate the soil or leach water, creating even more serious problems than those it is intended to solve. Phosphorus dissolved in leaching water can lead to the eutrophication of rivers and lakes [51], and plants with large amounts of accumulated fluorine can led to dental or skeletal fluorosis if plants containing high amounts of it are digested [52]. Similarly, heavy metals (especially Cd) and radionuclides in the PG composition can also cause health problems when ingested. Furthermore, an excessive amount of PG in the soil can reduce microbial activity in the soil, reducing agricultural yield [2]. Hence, the chemical composition of PG and the extent of its utilisation in the soil must be evaluated and monitored.

10.4.2 Possibilities in the Field of Building Materials

The potential of PG to be used in building materials as a substitute for natural gypsum is great. However, there are some objective constraints for PG usage in building materials: aside from the mentioned radioactivity, the economic factor is also relevant, since the additional costs derived from PG processing (purification, drying, and slightly different processing methods) may make PG non-competitive when compared to natural gypsum. However, in places where natural gypsum is scarcely available (for example, Japan, the Netherlands, and France), PG could be a competitive solution [40].

Some representative examples of PG applications in building materials are given in Table 10.1.

10.5 Conclusions

Valorisation of by-product PG is a non-trivial subject for circular economy. The real constraints to effective PG application in building materials are high radioactivity and economic non competitively (regarding natural gypsum). However, in some cases these risks do not apply. If that is the case, stakeholders must be open to the incorporation of PG to their products (OPC retarder, tiles, blocks, bricks, ceiling, partition boards, etc.), not behaving according to prejudices, cultural obstacles or simply, inertia, but making a step forward towards a more real concrete circular economy.

Table 10.1 PG processing methods to make it suitable for building materials

Processing method	Final product	References
Cement retarder was prepared from PG. It was dried and ground	Belite-ferroaluminate cement	Yang et al. [53]
Calcined at 500 °C and neutralisation with 4.00% of lime	Super sulphated cement	Liu et al. [12]
Addition of aqueous citric acid solution (1–5%)	Gypsum plaster, Portland and Portland/slag cements	Singh et al. [26]
Bitumen modified with 10% phosphogypsum	Asphaltic bitumen	Cuadri et al. [10]
Use of lime modified PG as a partial filler of white paints	White textured paints	Valančius et al. [54]
Neutralisation in 1% lime suspension at 60 and 80 °C temperature	PG binding material	Nizevičienė [4]
Addition of waste fluid catalytic cracking (0.5–10%), with and without sonication	PG binding material	Vaičiukynienė et al. [55]
Addition of carbide slag (1–5%)	PG binding material	Wu et al. [6]
Addition of $Ca(OH)_2$ (0.5–1.5%)	PG binding material	Villalón Fornés et al. [41]
Calcination and further addition of fly ash and lime	PG plasterboards	Kumar [56]
Granulation with water, press-forming and hydration with intermittent pressing	Paper-free and fiber-free plasterboards	Zhou et al. [50]
Intermittent press-forming hydration	PG load-bearing tiles	Zhou et al. [49]
Press-forming with two-step hydration process	PG load bearing non-fired bricks and blocks	Zhou et al. [47] Villalon Fornes et al. [48]
Partial substitution 10–30% of natural gypsum in the gypsum binder	Gypsum-based biocomposite	Bumanis et al. [57]

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