

# Characterization of a more sustainable cement produced with recycled drywall and plasterboards as set retarders



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## HIGHLIGHTS

- RCS can be used as set retarders in cement.
- Porosity of cements with RCS has been studied through image analysis.
- Cement samples with RCS treated at temperatures over 150 °C comply cement standards.
- The porosity of cement is reduced when RCS are preheated.
- The management of RCS facilitates the manufacture of more sustainable cements.

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## ABSTRACT

This paper deals with the study of a process that allows the use of waste sulphates from drywalls and plasterboards in the cement chemistry without affecting the resistance of cement, since cement industry is an important sulphate consumer. The role of the recycling temperature to obtain recycled calcium sulphates (RCS) and their SO<sub>3</sub> contents, when RCS are used as set retarders in cements, is also studied. Gypsum from drywalls, and plaster of Paris from plasterboards, were heated at three recycling temperatures (110 °C – 150 °C – 170 °C) and partially dehydrated to obtain RCS. Chemical composition and SO<sub>3</sub> purity of these RCS and of natural gypsums were determined and compared via XRD and DSC/TG. Setting time, the compressive strengths and the microstructure of cements with RCS used as set retarder were studied. The main products formed after cement hydration were determined by XRD and their microstructure observed by SEM/EDX. Internal porosity of different blends, area per mm<sup>2</sup> and diameter distribution, was determined through image processing with computer-aided design (CAD) software in SEM images. Cements with RCS from plasterboards as set retarder, with the higher SO<sub>3</sub> content and higher amount of hemihydrates, preheated at 150–170 °C, have mechanical strengths according to cement standards. They show a denser microstructure, lower porosity and more homogeneous matrix after final setting than cements with RCS from drywalls.

The selection here suggested of waste sulphates and the preheating process will allow using recycled sulphates from debris in the cement industry, according to EU standards.

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## 1. Introduction

Developing more sustainable or ecofriendly cement is necessary, since cement is the most consumed building material [1]. Additions from industrial waste have been researched for reducing the amount of clinker in the cement blends. Nowadays artificial

pozzolans such as blast furnace slag, silica fume, and fly ashes coming from industrial processes or natural pozzolans volcanic ash glasses, calcined clays and shale are commonly used as addition in cements. Najim et al. studied the effect of Cement kill dust (CKD) additions in mortars and cements [2]. Nguyen et al. so designed an eco-friendly cement with waste additions in ternary mixtures [3]. Moreover, hardazous waste additions have been incorporated in cement with the purpose of their encapsulation in mortar matrixes [4,5].

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On the other hand, sulphate compounds from recycling, commonly known as waste gypsum (WG), are also valorized as material in the construction sector. Recycled sulphates (RS) may be obtained from many and diverse recycling sources and therefore their chemical composition is variable.

RS may come from the industrial by-product derived from flue gas desulfurization process called FGD gypsum [6,7] and from other industrial synthetic gypsums such as fluorogypsum [8], phosphogypsum [9], jarosite/alunite precipitate [10,11] and borogypsum [12]. RS may also come from slip casting moulds of ceramic factories [13], from the TiO<sub>2</sub> production (red gypsum) [14] or from construction debris such as drywalls, plasterboards, and wallboards [15].

RS has been studied as set retarder in Portland cements [7,16–18], as anhydrite cement [19], or as calcium sulphoaluminate cements [20]. RS has been improved with low additions of granulated blast furnace slag and pozzolans like silica fume or fly ash to be used as binder [6,21,19]. However, many of these studies have presented a reduction of resistances, limiting the possibilities of its real use in the cement industry.

Sulphates have been used as set retarders in Portland cement because they avoid the flash setting of Portland cement, caused by the quick calcium aluminate hydration, through the formation of ettringite in the first hours. At a second stage of the reaction, when the supply of sulphate ions runs out, there is an increase in C<sub>3</sub>A dissolution and the remaining C<sub>3</sub>A reacts with ettringite and water to form monosulfoaluminate and hydroxy-AFm [22]. Therefore, for any particular clinker and source of SO<sub>3</sub> there is an optimum content of SO<sub>3</sub> at early ages, but usually in practice a 0.5–0.9 SO<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> ratio is commonly observed [23].

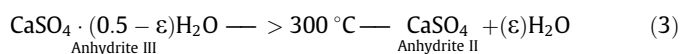
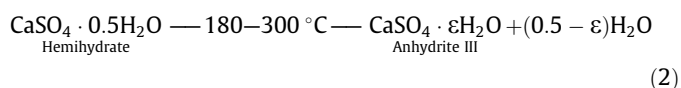
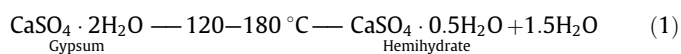
Development of sulphate compounds in the cement paste varies depending on whether the original sulphate is dihydrated (gypsum), hemihydrated or anhydrite. Some authors have pointed out that formation of AFm phases occurs when CA is hydrated with gypsum [22,24,25]. The use of gypsum, dihydrates and hemihydrates mixtures, has been suggested as set retarder [23]. The crystals of ettringite play a role in the cement strengths in the early hours and monosulphates in the first days because they fill the voids of the cement hydrated matrix [26].

On the other hand, a thorough study of the influence of RCS in cements depending on their temperature of recycling is presented in this work. As it has been shown, the amount of hemihydrates and dihydrates modifies the properties of cement especially in the first days (1–3 days) [27]. This is caused by the different solubility of both compounds, since the hydration reactions can be modified. The slower the dissolution of the hemihydrate used as set retarder, the lower compressive strength of cement in the first day [28]. This difference in strength development is reduced after 7 days from the setting, when the strength provided by ettringite

and monosulphate is less important than the strength provided by the Hydrated Calcium Silicates. Cement pastes with hemihydrates also present a higher amount of hydrated sulfoaluminate phases, mainly monosulfoaluminate formations, than those with dihydrates [27]. The use of natural anhydrite as set retarder has been also studied, and the reduction of strengths in the first three days was the main difference found [7].

Although some studies have pointed out the possibilities of the use RS different to RCS, – Fluorogypsum, phosphogypsum, or flue gas desulfurization (FGD) gypsum –, as set retarders [7,16–18], a modification of setting time, early strength and final strength of cements has been observed [29]. This fact has not encouraged the valorization of RS in industrial cement.

In this paper, properties of cements with calcium sulphates from the recycling of construction debris, and with natural calcium sulphates (NCS) have been compared when they are used as set retarders. Moreover, to modify the different phases in the gypsum, the samples have been preheated at 110–150 and 170 °C. It is known that at low temperatures, partial dehydration of gypsum may be obtained, even though in recycled gypsum. Reactions (1)–(3) show these dehydration phases.



ε not exact percent obtained, partially dehydrated less than 0.5 molecule of H<sub>2</sub>O.

As it has been preheated hydrated gypsum from 110 °C up to 170 °C it has been gotten only dihydrates and hemihydrates in the RCS (Reaction (1)). An analysis of the RCS with XRD and DSC/TG has been done and their microstructure observed by SEM/EDX. Setting time, the compressive strengths and the microstructure of cements with RCS used as set retarder were tested.

## 2. Materials and test procedure

As cement hydration is a holistic process, relevant factors such as clinker source, w/c ratio, temperature, amount of set retarder and grinding fineness have been kept constant.

The clinker (CL) used was obtained from the CEISA S.A. cement factory and was grounded in laboratory with a steel ball mill. The reference set retarder was natural gypsum (NG) used also in the

**Table 1**

Chemical analysis of the clinker used (according to UNE-EN 197:2000) and chemical analysis of natural and recycled calcium sulphates at different recycling temperatures (in accordance with UNE 102032). (NTM: Not thermally modified).

Chemical analysis	RC	NG	E-0	E-1	E-2	E-3	Y-0	Y-1	Y-2	Y-3
TEMP (°C)	NTM	NTM	NTM	110	140	170	TA	110	140	170
Hum (%)	0.08	0.1	0.2	0.02	0.79	0.28	0.2	0.34	0.71	0.02
SiO <sub>2</sub> (%)	21.32	2.01	1.58	1.58	1.98	1.64	4.22	4.49	4.61	4.37
Al <sub>2</sub> O <sub>3</sub> (%)	5.07	0.05	0.22	0.21	0.26	–	0.48	–	0.43	0.4
Fe <sub>2</sub> O <sub>3</sub> (%)	4.26	0.13	0.08	0.14	0.19	0.26	0.3	0.3	0.35	0.32
CaO (%)	64.96	32.9	33.88	37.1	40.6	38.92	33.32	36.4	38.08	38.08
MgO (%)	1.8	0.8	3.2	2.8	2.9	2.7	2.8	3.2	2	2.1
SO <sub>3</sub> (%)	1.71	43.57	40.14	43.37	46.75	46.88	38.62	41.61	43.79	44.52
LOI (%)	0.16	21.64	23.21	17.46	11.53	11.56	22.62	17.35	12.03	12.14
RI (%)	0.87	1.84	0.79	1.49	1.82	1.07	3.32	4.03	4.85	2.66
H <sub>2</sub> O COMB (%)	–	19.16	17.49	11.92	5.74	5.61	17.09	11.46	6.17	6.42
I. PURITY SO <sub>3</sub> (%)	–	–	85.73	85.65	85.22	85.31	82.74	82.2	80.61	82.1

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