Artificial weathering pools of calcium-rich industrial waste for CO$_2$ sequestration

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ABSTRACT

Processes of carbonation of calcium-rich aqueous industrial wastes from acetylene production were performed mimicking rock weathering, using the atmospheric carbon dioxide as reactant. This residue was carbonated exposing it to the air in artificial pools with controlled solid-to-liquid and surface-to-volume ratios, and the efficiency of this simple mineral carbonation process was maximized. Considering realistic values of just one acetylene production plant, the intelligent handling of the calcium-rich waste would make it possible to counteract the emission of around 800 t of carbon dioxide per year, so the CO$_2$ emissions of the acetylene production could be completely compensated and its carbon footprint significantly reduced.

X-ray diffraction patterns and thermogravimetric analyses reported the conversion, up to 88%, of the calcium hydroxide into calcium carbonate under atmospheric conditions. So, considering a realistic industrial scale-up, 476 kg of CO$_2$ could be captured with 1 t of dry waste. The morphology of the grains is resolved by electron microscopy, and can be described as needles 15 nm wide and 200 nm long arranged in grains smaller than 1 micron. We exploit these nanometric textural parameters (nanometric pores and particles having a specific surface area $\sim$50 m^2/g) to design an efficient carbon fixation procedure. The aim of this work is to propose this simple carbonation technology, based on aqueous alkaline industrial waste, as a contribution to reducing global CO$_2$ emissions.

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1. Introduction

The increase in the concentration of CO$_2$ in the atmosphere from the pre-industrial level of 280 to 380 ppm [1] has led to alarm about the likely effects on the earth's climate. The seriousness of the damage caused by climate change to the way of life of the developed countries has forced them to assign enormous resources to mitigate this situation. Nowadays, the challenge is to develop and improve technologies for energy production and to reduce the concentration of greenhouse gases in the atmosphere. In this work, a feasible technology for alkaline industrial residues weathering is proposed. The process mimics the actual geological rock weathering because it only considers the interaction between industrial wastes with greenhouse gases present in the atmosphere, as carbon dioxide. Therefore, the proposed weathering management considers only the industrial waste and the greenhouse gases, and no extra devices, reactants, additives, or activation heat or pressure are needed. This technology is possible thanks to the properties of the industrial waste, as the composition or the microstructure of the solid phase, namely, grain size, specific surface area and porosity. These structural parameters promote and enhance the reaction rate.

Currently, most of the works about carbon capture and storage (CCS) technologies comprise geological storage and ex situ mineral sequestration, and they work once the CO$_2$ is separated from the flue gas, mainly to reduce CO$_2$ emissions from localized industrial sources (large industries). The first one has been considered since decades ago when CO$_2$ injection-based enhanced oil recovery systems were implemented in mature oil fields [2,3]. However, it presents several problems such as the reaction of carbon dioxide with the cementitius seals of the injection wells [4] which can affect the isolation of the reservoirs, or the diffusion of the CO$_2$ via rock cracks over centuries. The second technology considered is a mineral carbonation process [5]. This process consists in chemically binding the carbon dioxide molecule to a mineral to form carbonates, similar to the weathering process that has regulated the CO$_2$ concentration of the earth's atmosphere for millions of years. Mineral sequestration allows the removal of CO$_2$ coming from not only power plants or other localized industrial sources but also diffuse sources (typically transport means) and past CO$_2$ emissions. In this case, the atmosphere carries the carbon

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dioxide produced elsewhere above cities or countries, and it can be sequestered thousands of miles away.

The strongest advantage of mineral sequestration [5–8] is that the hazardous greenhouse gas is converted into innocuous, stable and environmental benign carbonate mineral, fixing the carbon dioxide definitively. Its weakest points are the low speed of the reaction, the carbonate by-product that is produced and should be managed, and the costs of the process [9,10]. Therefore, the carbonation reaction ought to be sped up to become a realistic solution for carbon sequestration, but without penalizing its efficiency due to a high energy consumption or negative net carbon balance. Besides, the by-product has to be innocuous and easy to handle so that hundreds of tons can be managed without complex technologies or extra risks. Moreover, one by-product with an added value will be the most desirable option to support this technology.

The use of industrial alkaline wastes will contribute to reducing the costs of carbon sequestration enormously, besides avoiding the ecological impacts of mineral extraction on a massive scale. Some prototypes based on the absorption of the atmospheric CO_2 have already been presented, and the costs of one sequestered ton of carbon dioxide are estimated to be between 10\$ [11] and 127\$ [12]. In the last decade, several industrial products have been considered for sequestration of carbon dioxide. Cementitius materials were initially considered [13] followed by municipal solid waste incinerator bottom ashes [14], steel slag [15,16], paper mill waste [17], coal combustion fly ash [18], air pollution control residues [19] or oil-shale wastes [20]. In summary, the current research focused on portlandite or calcium oxide for mineral sequestration is quite wide, but very few raised and studied the relationship between kinetics and microstructure [21].

In this work, we have considered the calcium rich waste of the industry of the production of acetylene as a new material for carbon dioxide sequestration. Acetylene produced via calcium carbide generates an aqueous suspension of portlandite (calcium hydroxide, $Ca(OH)_2$). The experimental conditions to obtain the maximum carbonation efficiency are researched. The carbonation process is carried out under atmospheric conditions, paying special attention to the texture of the samples and to strategies to enhance the reaction speed as well [22–24]. The experimental results allowed us to design the weathering pools, a very simple atmospheric carbon dioxide sequestration process. The reaction by-product obtained is calcium carbonate that is easy to handle and could be disposed of or even commercialized.

2. Materials and methods

The sample is an industrial waste discarded by the acetylene industry, and it was supplied by the AIR LIQUIDE España S.A. acetylene factory located in Seville (Spain). The waste is an inhomogeneous greyish water slurry.

2.1. Characterization of the samples

Chemical analyses performed on the original sample, prior to any carbonation experiment, were done by X-ray fluorescence (AXIOS, Panalytical, with an Rh tube) using the semi-quantitative method. Crystalline phases of the samples were identified by X-ray diffraction (XRD) in a Philips X'Pert diffractometer with Cu K_α radiation, from 5.00° to 70.00° with a step of 0.05° and counting time of 80 s. The morphology of the waste was characterized by nitrogen physisorption experiments and scanning electron microscopy (SEM). Physisorption experiments were performed in a Micromeritics device model ASAP2010 at a constant temperature of 77.35 K. Samples were milled and degasified at 150 °C under vacuum for 2 h prior to the experiment. In order to keep the original

structure before any possible carbonation due to atmospheric CO_2, the "original sample" was taken from the wet industrial waste container, dried, and degasified inside the physisorption experimental device. Specific surface area and pore size distribution (PSD) were obtained by analysing the physisorption isotherm using the BET and BJH methods, respectively. SEM was performed in a SEM-FEG Hitachi S480 using an acceleration voltage of 2 kV.

Finally, the carbonation degree of the samples was estimated by thermogravimetric analyses (TGA) in a STD Q600 experimental device. The experiments were carried out under a nitrogen flux of 100.0 ml/min, starting from ambient temperature and increasing by 10 °C/min up to 1000 °C. As a reference, we have born in mind that for a pure calcite sample, the stoichiometric weight loss due to decarbonation is 44% in the range between 550 °C and 900 °C. To compare the experimental results with this theoretical maximum, mass of the samples from TGA experiments was normalized to the weight of the dehydrated sample, that is, the mass at $T = 200$ °C.

2.2. Carbonation process

Calcium hydroxide reacts with CO_2 to form calcium carbonate $CaCO_3$ and water:

$$Ca(OH)_2 + CO_2 \leftrightarrow CaCO_3 + H_2O \tag{1}$$

The carbonation of this industrial waste was studied using the atmospheric CO_2. The weathering pools consist in very shallow flat ponds where the waste is disposed as a film. The carbonation of the waste occurred during drying under atmospheric conditions, typically taking from 3 days to one week. With the uncontrolled disposal of this portlandite-based waste, some carbonate is precipitated on the outer face of the grains due to the drying process, but the core of the sample remains as portlandite. In this research, two parameters were analysed to explore their influence on the efficiency of the carbonation process. The first was the solid-to-liquid ratio of the aqueous system, defined as the ratio between the mass of the solid phase and the mass of the liquid in which the solid is in suspension. To control and to measure this ratio, an amount of wet paste is weighed and its volume measured. Then a certain volume of supernatant is added up to a defined volume, and then the system is weighed again.

The second studied parameter is the height of the volume that occupies the aqueous system. This parameter is critical as it defines the surface-to-volume ratio of the system, that is, the quotient of the surface in contact with the air by the volume of the system. In other words, it defines the surface in contact with the atmosphere, which controls the kinetics of the drying as well as the dissolution of the CO_2 from the atmosphere into the system. Systems giving two different heights were used: a glass where the system was set to a height of 2.3 cm, and spreading the aqueous sample on a flat surface, giving this system the theoretical height of 0. In this work, the containers will be referred to as "weathering pools".

Finally, the influence of rewetting daily or stirring was also tested. Rewetting the sample consisted in adding 3 ml of distilled water to 10 g of dry sample on each rewetting. Sampling of each configuration was done until the maximum degree of carbonation was achieved.

3. Results and discussion

3.1. Characterization of the original sample

The original waste was characterized prior to any carbonation process. The results of the chemical analysis by XRF (Table 1) showed a calcination weight loss of 22.45%, a calcium content of 52.41 wt.%, an oxygen content of 23.10 wt.%, and a silicon content of 1.04%. Some traces of Al, S, Fe, Cl, Mg, and Sr were also found.

Table 1
Chemical composition (XRF) and structural features of the original sample.

Chemical composition (%)	
Calcination weight loss	22.45%
Major elements	Ca: 52.41%; O: 23.09%; Si: 1.04%
	Normalized to the calcined mass:
	CaO: 94.56%; SiO$_2$: 2.88%
Traces (<0.6%)	Al; S; Fe; Cl; Mg; Sr
Structural data	
Specific surface area	47.5 m^2/g
Pore mean size	3.7 nm and 16.8 nm
Particle shape and size	Nanometric needles of 15 nm × 200 nm arranged in clusters of 500 nm

This result matches a mineralogical content of 97.0 wt.% of portlandite and silica content of 2.2 wt.%, where the calcination weight loss matches the release of water due to the conversion of the portlandite into lime (CaO), supporting that the waste is composed of portlandite. This result was confirmed by XRD analysis (Fig. 1). The sound sample was found in the core of large dried pieces of the waste. The core of these grains remained uncarbonated despite the drying process. In the outer face of the grains, calcite and portlandite instead of pure portlandite could be observed by XRD (not shown). Note that spontaneous weathering occurs but only the controlled management and conscious weathering of the waste will mean significant amounts of captured carbon dioxide.

The morphology of the portlandite grains suspended in the aqueous waste was studied by nitrogen physisorption and electron microscopy. Measured values of the structural parameters are listed in Table 1. The PSD showed two typical pore sizes of 3.7 nm and 16.8 nm and the specific surface area is 47.5 m^2/g. The SEM images showed that portlandite precipitates in the form of nanometric needles of around 15 nm wide and 200 nm long (Fig. 2). These needles form aggregates of around 500 nm and these aggregates are agglomerated in larger grains. But the specific surface area is smaller than that corresponding to one single needle of portlandite and larger than that of one grain of portlandite of 500 nm size. In addition, the pore sizes measured correspond to the spaces among the nanometric portlandite needles. Consequently, it can be concluded that the nanometric structural features are still present in the large agglomerates.

Up to now, the main drawback of some of the carbon mineral sequestration technologies was the cost of milling to obtain a small grain size (and large reactive area) from bulk mineral, which is necessary to obtain significant values of carbonation efficiency. In the samples presented in this work, large reactive surface areas are present due to the particular morphology of the calcium hydroxide
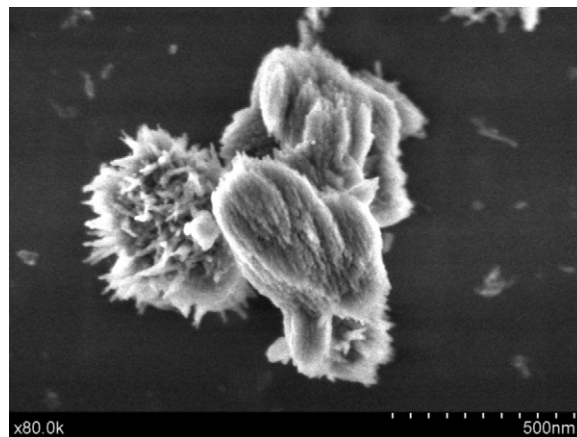
**Fig. 2.** SEM image of the original sample.

rich waste, enhancing the carbonation reaction as the carbonation efficiency is very sensitive to particle size [25] or the porosity of the sorbents [21]. So, the nanostructure of the portlandite allows the CO$_2$ dissolved into the aqueous system to attack the whole sample before the passivating layer [26] hinders the carbonation of the grains, yielding large carbonation efficiencies.

To avoid the precipitation of some carbonate while handling the sample for thermogravimetry, and to verify the absence of calcite in the original waste, a sample of the original wet paste was taken and quickly submitted to the TGA. The drying process was carried out in an inert atmosphere of nitrogen inside the experimental device. The curve obtained (not shown) presented a first large weight loss at around 100 °C due to the dehydration process. The second weight loss at around 400 °C is associated with the dehydroxylation[1] of the portlandite [27,28]. Reported values of dehydroxylation of portlandite are slight larger than the 400 °C found for these samples (450 °C and 467 °C in the referred papers, respectively). It is well known that experimental conditions as well as sample size and morphology can affect greatly the rate of thermal dehydroxylation [28], so this decrease in the dehydroxilation temperature can be obtained due to the high reactive area of the sample.

3.2. Ambient carbonation

The carbonation of the samples due to the atmospheric carbon dioxide, the so-called weathering, was studied by measuring the degree of carbonation for different experimental configurations. First of all, the carbonation reaction was verified by the appearance of different calcium carbonate polymorphs (calcite, vaterite, and aragonite) in the samples, as revealed by XRD. In Fig. 1, the XRD pattern of a carbonated sample (P0, regarding to labels in Table 2) is shown. Total carbonation is not present as portlandite is still observed. Once the samples were completely dry, no more carbonation was observed, as expected. The influence of the solid-to-liquid ratios and the height of the aqueous system were studied to control and to maximize the weathering process. Both parameters are directly related to the kinetics of the drying process of the sample, and both were revealed to be critical to the degree of carbonation.

Firstly, the influence of the surface-to-volume ratio was studied. Two different samples were considered. Details of their configuration and carbonation efficiency are listed in Table 2 and the TGA
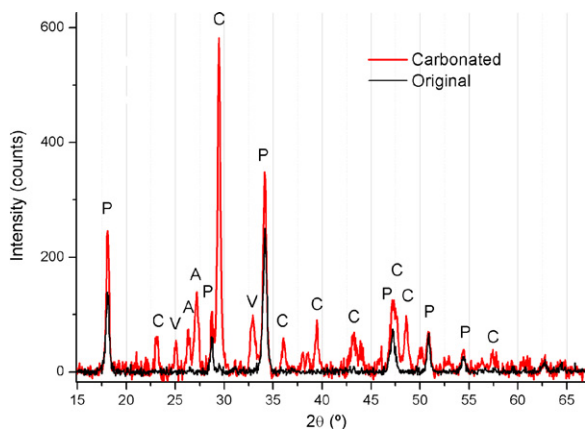
Fig. 1. XRD pattern of the original and carbonated samples. The letters "P", "C", "V", and "A" indicate portlandite, calcite, vaterite, and aragonite peaks, respectively.

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[1] Note that in this work, the terms "dehydration" and "dehydroxylation" are used to refer to the two different processes where water molecules are released from the sample. The term "dehydration" refers to drying of the wet paste, whereas the term "dehydroxylation" refers to the release of the water from the portlandite.

Table 2
Different configurations considered for ambient carbonation and their carbonation efficiencies.

		Carbonation efficiency[a] (%)
Surface-to-volume ratio (height)		
P4	Sol/liq = 0.24; height = 2.3 cm	15
P5	Sol/liq = 0.24; height = 0	65
Solido-to-liquid ratio (sol/liq)		
P3	Sol/liq = 0.07; height = 2.3 cm	10
P4	Sol/liq = 0.24; height = 2.3 cm	15
P6	Sol/liq = 0.33; height = 2.3 cm	38
Stirring		
P3_S	Stirred, sol/liq = 0.07; height = 2.3 cm	15
P4_S	Stirred, sol/liq = 0.24; height = 2.3 cm	18
Rewetted samples		
P4_w	P4, rewetted once	41
P4_w2	P4, rewetted (after 4 days)	58
P5_w	P5, rewetted once	81
P0	Height = 0	88
	Rewetted and stirred for 7 days	

[a] Carbonation efficiency is the ratio between the sample weight loss between 550 °C and 750 °C measured in the TGA analyses and the theoretical maximum for calcium carbonate of 44%.

curves of samples P4 and P5 are plotted in Fig. 3. It can be seen how decreasing the height of the system from 2.23 cm (P4) to 0 cm (P5) increases the efficiency of the carbonation from 15% to 65%. If the aqueous system is not shallow enough, one layer of floating calcite will precipitate in the interphase liquid/air slowing down the carbonation. The high carbonation efficiency obtained in such a simple system as this shallow weathering pool P5 makes this waste a very promising carbon dioxide sequester for industrial implementation. In Fig. 4, the morphology of the precipitated calcite floating layer is shown. Nanometric spheres of calcite (confirmed by SEM and XRD, not shown) precipitated and clumped together to form large arrays and clusters (branched bottom of the precipitated phase) where the calcite rhombohedrals appeared later.

Secondly, the influence of the solid-to-liquid ratio of the wet paste was researched. The considered values of the ratio and the carbonation results are listed in Table 2 (P3–P4–P6 series). These results are shown in Fig. 5. Again, it can be stated that complete carbonation was not achieved in any case. Nevertheless, the higher the solid-to-liquid ratio is, the higher is the carbonation efficiency. Then, this ratio is found to be critical given that, for a similar geometry, it controls the appearance of the calcite floating layer which hinders the CO_2 flux to the solution and eventually the carbonation of the sample. It is worth mentioning that the original waste has a
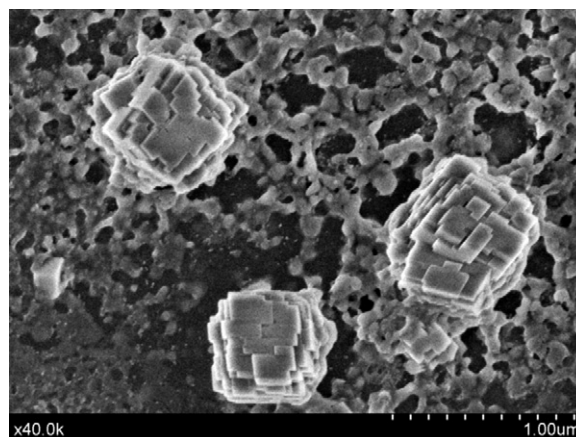

Fig. 4. SEM image of the floating calcite layer. Submicrometric spheres of precipitated calcium carbonate formed branched film and turned into calcite crystals.

solid-to-liquid ratio close to 0.33, the maximum value considered in this work. So, the addition of water to the system will result in a delay in the drying process and impede the carbonation process.
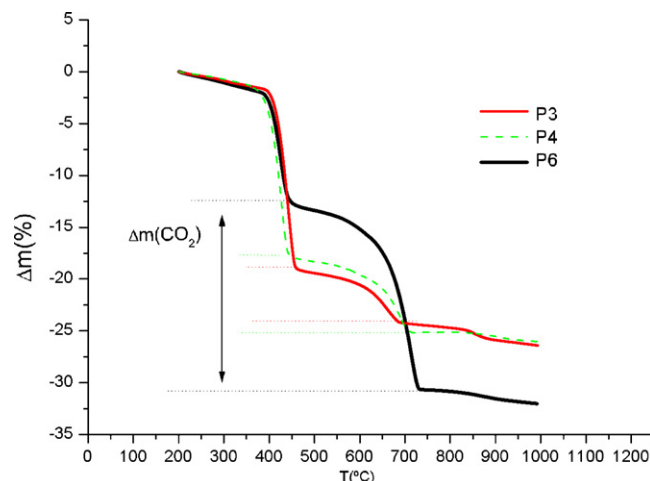
3.3. Assisted ambient carbonation

Some additional technical measures were considered to improve the efficiency of carbonation. Firstly, stirring the sample was tested to avoid the appearance of the calcite floating layer. It was observed that the floating layer appeared even under vigorous stirring. Stirring yields small increases in the carbonation efficiency from 10% up to 15% for the pool P3_S and from 15% to 18% in the case of pool P4_S. Details of the pools and carbonation efficiencies are listed in Table 2.
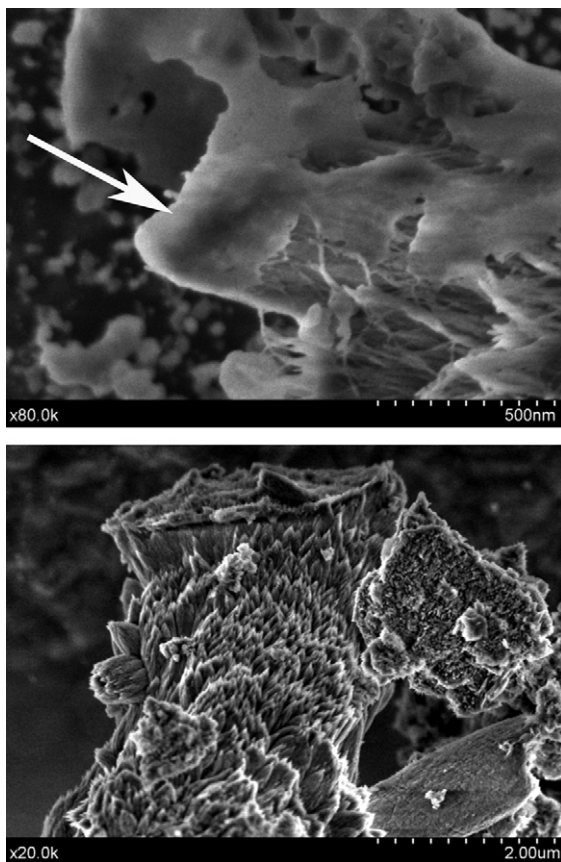
Rewetting the sample was also considered to achieve higher carbonation efficiency. In Fig. 3, TGA curves of the rewetted samples in pools P4_w2 and P5_w are also plotted and their carbonation efficiencies are compared in Table 2. Results reveal the enhancement of the carbonation efficiency with the rewetting of the pools. Thus, pool P4_w rewetted once with distilled water increases its carbonation efficiency up to 41%, almost three times the efficiency of the non-assisted pool P4. Rewetting the pool again (sample P4_w2) reveals that the carbonation reaction can continue up to 58%. But again, the geometrical configuration was essential to obtain carbonation efficiencies close to 100%. Rewetting the sample in pool P5_w, with a height equal to zero (spread on a flat surface), gave an
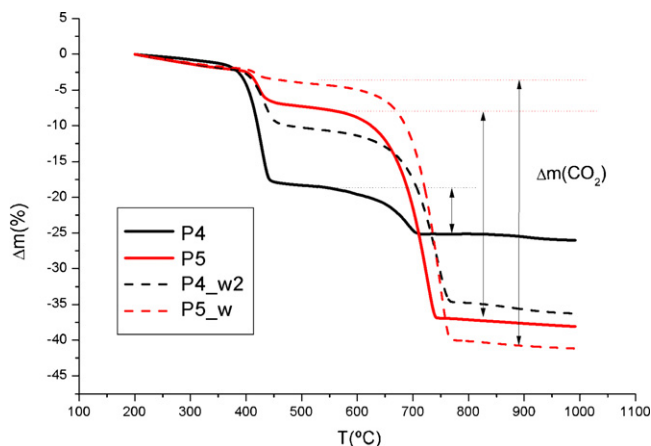
Fig. 3. Comparison of the geometrical effect on the TGA of carbonated samples. The weight loss $\Delta m(CO_2)$ represents the weight loss due to the release of CO_2 from the calcium carbonate.

Fig. 5. Comparison of the solid-to-liquid ratio in the TGA of carbonated samples.

Fig. 6. SEM image of the calcium carbonate layer precipitating on the portlandite clusters, indicated by the arrow (top) and bunches of calcium carbonate grains piled up (bottom).

Table 3
Daily production of one acetylene plant, alkaline waste (portlandite) and different elements of the acetylene carbon footprint. The mitigation power of this technology is evaluated for 1 ha weathering pool.

Acetylene production	1.67 t
Portlandite production	4.74 t
Maximum stoichiometric theoretical CO_2 absorption	2.82 t
Indirect CO_2 emissions (energy consumption)	0.96 t
CO_2 sequestered (80% efficiency considered)	2.28 t
CO_2 balance	−1.94 t (total mitigation)
Calcium carbide consumption	4.10 t
Indirect CO_2 emissions	6.35 t
CO_2 balance	5.03 t (31% reduction)

could be established at 7%, or 47.6% considering the dry waste (476 kg of captured CO_2 per ton of $Ca(OH)_2$) much higher than that from municipal solid waste bottom-ash (2.3%), carbon fly ash (2.6%) or even paper mill waste (21.8%) [17]. Although acetylene synthesis does not produce carbon dioxide directly, indirect CO_2 emissions produced by the energy consumed (estimated at 288 t CO_2/year) could be completely mitigated by a weathering pool of 3000 m². So, the carbon footprint of the acetylene plant could be reduced.

But, considering the 1 ha weathering pool set-up, it would be possible to capture not only the carbon dioxide indirectly emitted by the acetylene production but also some of the CO_2 emitted during the synthesis of the calcium carbide (CaC_2), the precursor of the acetylene. The calcium carbide is obtained by burning coke and lime in electric furnaces, with high energy consumption. The energetic requirement to obtain 1 t of CaC_2 at 80% purity is around 3300 kWh [32]. Thus, 1.55 t of CO_2 is indirectly emitted to the atmosphere for each ton of calcium carbide. Consequently, 1 day's production of acetylene yields total indirect carbon dioxide emissions of 7.3 t of CO_2, whereas one weathering pool will capture 2.3 t (Table 3). So, 30% of total indirect emissions of CO_2 could be mitigated. Finally, it must be remarked that the carbon sequestration by-product is calcite, a valuable product that can be simply disposed of, used in different industries or commercialized as precipitated calcium carbonate [33].

Secondly, regarding to the assisted carbonation procedures, the experimental set-up with maximum carbonation efficiency was considered, that is, rewetting the weathering pool with minimum height. Water consumption was estimated by Dunne and Leopold [34] and Meyer [35] empirical equations and the costs of the rewetting process have been evaluated. Using the local mean weather and solar radiation data of the area of Seville (where the acetylene plant is located) for a 1 ha weathering pool, 41.15 m³/day will be consumed. Rewetting will cost 45.68 €/day so it will not be a major drawback of this technology. But, given that carbonation efficiency will be increased by only 15%, the rewetting process, water pipes, and logistics could not be interesting at an industrial scale. Consequently, this should be considered and evaluated for each particular case.

increase in the efficiency from 65% to 81%. This improvement of the efficiency was found to saturate in pool P0, which was rewetted and slightly stirred daily for 7 days. This sample showed an efficiency of 88%, but then it was not possible to improve it further. So, by rewetting these samples, carbonation efficiency close to 90% can be obtained.

SEM images have revealed some steps of the structural evolution of the carbonation of the fibrous clusters of portlandite to calcium carbonate crystals. In Fig. 6, top, it can be seen how the calcium carbonate precipitated on the surface of the clusters of portlandite fibrous grains as a layer (indicated by the arrow), but this sort of passivating layer [26] does not prevent high carbonation efficiencies thanks to the small size of the portlandite grains. In Fig. 6, bottom, wheat-sheaf-like bundles of calcium carbonate can be seen. It has been reported that the precipitation of the calcium carbonate in these morphologies is caused by the presence of impurities of Mg [29,30]. The existing impurities of Mg among others in our system (Table 1) cause the precipitation of the calcite into these morphologies of bundles. SEM imaging also confirms the precipitation of the calcium carbonate into rhombohedral morphologies (not shown), together the wheat-sheaf-like bundles.

3.4. Industrial scale-up

Firstly, considering an acetylene industrial plant of 80 kW with a potential production of 50 t/month of acetylene [31], 1707 t/year of $Ca(OH)_2$ are produced (6800 t/year of aqueous waste, considering a solid-to-liquid ratio close to 0.33), so thanks to this technology, around 800 t of CO_2 could be captured each year directly from the atmosphere. Thus, the fixation power of the carbonation pools

4. Conclusions

This research work has proven that the management of aqueous industrial alkaline wastes can be an effective strategy for carbon dioxide mineral sequestration. Carbonation efficiencies up to 88% were achieved using just carbonation pools where the aqueous residue is disposed and let to dry, under atmospheric conditions. Actually, this work is proposing a change in the waste management pointing to CO_2 sequestration. In the studied case, the calcium-rich acetylene industry wastes have been shown to be a promising material for atmospheric CO_2 sequestration. It is possible to sequester up to 800 t per year of carbon dioxide just

considering the wastes of one simple acetylene plant, reducing the carbon footprint of the acetylene production. Knowing that this amount is not a huge CO_2 removal, the most important point to remark is the change of the philosophy on the waste management. For any other industry producing alkaline wastes, the amount of carbon dioxide that could be captured will depend on the amount of waste and its composition. Up to now, several solid industrial residues were considered for CO_2 sequestration, but in this work it has been shown that it is technologically simpler and more efficient to capture CO_2 using the aqueous alkaline industrial wastes, as those coming from the acetylene industry.

Acknowledgements

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