Immediate Aqueous Carbonated of Gypsum Used in Flue Gas Desulfurization, Calcium Carbonate Precipitates

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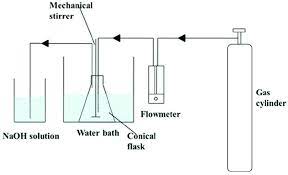
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**Abstract:** It was examined to see if the carbonate of calcium precipitated after the immediate water carbonation of desulfurization of flue gas (FGD) gypsum, was an unneeded byproduct from industry. Two-step fermentation was additionally tried as an alternative. In the experimental two-step carbonation process, calcite predominated, but in the straight carbonation, the vaterite and calcareous components were mixed under all circumstances. By contrasting the highest intensity for every stage's X-ray diffraction spectrum, the corresponding quantities of every phase have been determined. For a constant CO2 flow rate, it was discovered that the quantity of vitriol phases in the mixture increased with acidification duration. XPS (X-ray photoelectron spectroscopy) was used to figure out if there was an initiation phase prior to the deposition of tangible carbonate of calcium particles. At the 1 L/min CO2 consumption rate, a calcium carbonate C1s spike was clearly visible after more than 15 minutes of carbonation. The brine collected throughout the inducement time resulted in the precipitation of single-phase, essentially impurity-free limestone stones.

**Keywords:** Aqueous; Gypsum; Calcium carbonate; carbonation reactor; Polymorphs.

# Introduction

The main byproduct generated by the decomposition technique employed for eliminating SOx from coal combustion gases is the desulfurization of flue gases (FGD) travertine. The creation of the fertilizer sodium sulfate has been a long-standing use of leftover plasterboard. In addition, the mineral calcium carbonate is created as a byproduct, and based on its innocence, shape, etc., it may be valuable commercially. Its applications include traditional sectors like paper or cosmetics as well as the pharmaceutical sector. The two subsequent sequential responses that make up the procedure typically are: The high yield as well as the purity (up to 99%) of the sulfuric acid generated by the aforementioned processes are its distinguishing features. Burns et al [1]. convincingly showed that the majority of FGD travertine impurities stayed in the solid residue, guaranteeing the purity of the ammonium sulfate that was produced. Filtering is a simple method for removing contaminants from ammonia sulfate solutions when they are present in a solid state. According to the concentrations of the two compounds and a solution of ammonia, the majority of the somewhat soluble calcium dioxide will precipitate, while the latter, the one with great solubility, should be present in liquid. Therefore, following filtering out the insoluble contaminants, crystalline sulfates of ammonium crystals may be extracted [2]. The latter, on the other hand, has limited dissolution and is unable to be processed in a comparable way. In recent times, we have assessed the viability of complete oxygenation of FGD travertine in just one stage. Figure 1 shows the Straight water acidification reactors arrangement.



**Fig. 1** shows the Straight water acidification reactors arrangement.

Since process is exothermic in nature, nitrogen must typically be refrigerated below 20 C to prevent nitrogen evaporation. As a result, a one-step direct carbonation of FGD limestone is undoubtedly superior to a two-step traditional procedure. By routinely checking the slurry's temperature, pH, and carbonated rate of conversion, the impacts of the ammonium material, carbon dioxide rate of flow, solid-to-solution proportion, and CO2/N2 mixture of gases ratios were investigated. As anticipated, somewhat pure sodium sulfate might be produced as a result. Under all circumstances, the thermally unsteady vaterite as well as the stable calcareous phase were constantly mixed together in the calcium carbonate solution that precipitated during the initial carbonated reaction [3]. The formation of precipitates of carbonate of soda and its variability are the main topics of this essay. Also highlighted are our efforts to create extremely pure sodium carbonate from FGD lime in a way comparable to that of ammonium sulfate manufacturing. Several optical as well as imaging methods were used to investigate the innocence, stage, and other tangible characteristics of the generated calcium carbonate. Additionally, a comparison investigation involving carbonate of soda made using the traditional two-step procedure is provided [4].

# Experimental works

## Material

To eliminate the top layer of fluid, FGD limestone was bought from thermal power stations in Tamil Nadu, India, and evaporated at 45 °C 24/7. After digestion with acids (HNO3/HClO4), the primary elements were determined using wet chemical testing, and the minor elements were determined using inductively controlled plasma-optical fluorescence detection. The laser-bouncing particle size analyzer was used to determine the range of particle sizes. A charged probing microanalyzer was employed to do the chemical analysis.

## Methods

As illustrated in Fig. 1, indirect aquatic FGD calcium carbonation was performed by infusing carbon dioxide (CO2) (99.99%) into an FGD calcium nitrogen solution. The solid-to-solution proportion within a spectrum of 25 to 60% did not significantly affect the degree of carbonation or effectiveness, according to our preliminary investigation. Here, the mass-based solid-to-solution proportion (%) was determined. The solid-to-solution ration was determined at 25% according to this outcome. Therefore, 200 milligrams of FGD limestone and 3.9% (v/v) ammonia from water were used. A store-bought 25-weight percent solution of ammonia (as well as an average density of 0.95 g/mL) was used to modify the nitrogen content. The volume of nitrogen utilized (Eq. 3)) was slightly over the solubility ratio 2 (120%) by volume. While injecting CO2 gas (1-3 L/min), the ammonium chloride mixture, including FGD-gypsum, had been stirred with a motorized agitator for five minutes at 500 rpm. At predefined periods, the solution was gathered (in 20 mL parts). The collected samples were passed through filters to measure the amount of calcium ions and soluble sulfate particles. The filtrated sample was subsequently refiltered after letting it settle for 10 hours to figure out the amount of calcium in the dissolved atoms.

The distinction among the aggregate and soluble levels of calcium was used to determine the CaCO3 level. Therma Scientific's Horizon 410A was used to measure both pH and temperature, while all tests were carried out at ambient temperature and the pressure of the atmosphere. A Sartorius acetate-cellulose membrane filter measuring 0.25 lm was employed for the filtering process. ICP-OES was used to identify anions, particularly calcium ions. This was performed after the specimen was acidified to pH 2 using equipment grade HNO3. Following purification of the specimens, soluble sulfate ions were measured using the method of ion chromatography. Filled specimens of solids had been dried for a whole evening at 70 °C after being rinsed in the deionized water that had been filtrated with a Milli-Q 18 MX cm device. These specimens were subsequently investigated using energy dispersion X-rays.

For contrast, a two-stage FGD limestone effervescent process was used, and the ensuing reactions took place in that order: Ammonia (bi)carbonate is created, and then FGD plasterboard reacts with the resulting urea (bi)carbonate. The identical methane level of 3.9% (v/v) was used in the initial step to create ammonium bicarbonate, in addition to an injection rate of one liter per minute of carbon dioxide (CO2). Following the signal of pH stabilization, this process was stopped. When the sodium bicarbonate liquid cooled to ambient humidity, the subsequent reactions between the resulting ammonia bicarbonate and FGD plasterboard began. Additional analytical procedures followed the same guidelines formerly used in indirect freshwater oxygenation.

# Results

## Characterization of the FGD-gypsum

The FGD gypsum's XRD spectrum reveals that it is a sodium sulfate dihydrate with small amounts of Russian and limestone. According to the wet chemical information, the weight formula of FGD plasterboard has been found to be CaSO41.9H2O. Based on the chemical makeup data, it was calculated that the minimum quality of the calcium sulfate dihydrate was around 97.2 percent [5]. The results showed that the minor contaminants were 0.8 percent silicon dioxide, 0.6% Al2O3, 0.3% iron oxide, and 0.1 percent potassium hydroxide. Nevertheless, lead, mercury, zinc, magnesium, and Cadmium heavy metal contamination was not found. The volume that was the average diameter of the fragments, which varied in size between 20 and 50 lm, was 32 lm; however, the majority of the fragments are under 75 lm. Because these co-occur, trace anions silicon Al, which is while Ka is likely related to the Muscovite, according to The EPMA technique mappings [6]. Figure 2 shows the XRD pattern of gypsum[13-19].

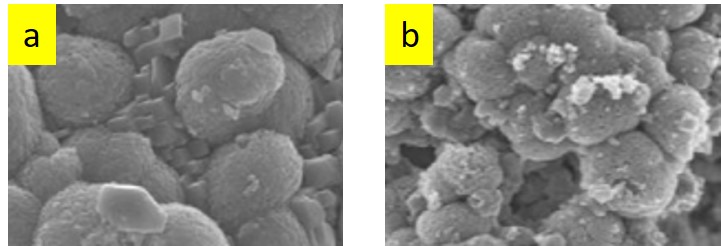


**Fig. 2** shows the XRD pattern of gypsum

## Carbonation of FGD gypsum

At the outside temperature and pressure in the atmosphere, FGD plasterboard exhibited a strong carbonated reaction, generating carbonate of calcium and sulfate of ammonium. Despite all circumstances, calcium and vaterite were present in the magnesium carbonate [20-22]. Since the majority of the intractable contaminants were able to be removed by simple filtering, the sulfate of ammonium was incredibly pure. Typical metal contaminants such as Si, K, Na, Al, which is, and Mgcl were found in the filtration mixture at quantities lower than 20 mg/L, while Fe is below the level of detection threshold. This finding shows that mostly insoluble stones like Russian and limestone were the source of the majority of the pollutants. The purity of the compound was therefore often above 97% natural, making it sufficient for usage in business [7].

Upon being charged with FGD studies, the pH level of the water-ammonia mixture was approximately 12.0 at first. The starting levels of calcium ions and sulfate were discovered to be 555 mg/L (15.0 mM) as well as 1250 mg/L (14.0 mM), accordingly, at this juncture, prior to the infusion of carbon dioxide (CO2). These amounts make up just 1.5 percent of the overall calcium supply. On the other hand, at this elevated pH, carbon dioxide can dissolve readily as CO2O3. The pH level and sulfate content of the solution's contents were measured to track the development of the method of carbonation. The sulfate ion level and pH both reached consistent levels of around 900 mM and 6.0 once the carbonating process was finished. Additionally, the FGD gypsum's speed of dissolution improved with the circulation rate of carbon dioxide, as has been shown. This shows that the synthesis of carbonates in the solution, which combine with calcium ions to promote carbonation, is made easier by a boost in CO2 flow rate [23-28]. Additionally, it's probable that the additional agitation caused by a higher CO2 flow will speed up the dissolving of FGD plasterboard. The hard residues created following acidification are depicted in Fig. 5 in FE-SEM photographs, which demonstrate the morphological alterations of the resulting product over time. According to the less magnified micrographs, after ten to fifteen minutes, FGD travertine saw a substantial size decrease. As was previously seen in the pure water carbonated manipulation, the formation of carbon is more likely to happen via the scratching of FGD limestone particles. More powerful images demonstrate that the carbonate of calcium did not coat the outermost layer of the FGD plasterboard but rather developed independently of it. The EDX projected pictures of the material that were acquired after 15 minutes of direct acidification (Fig. 6) provide additional support for this assertion. Imaging using C-Ka (CaCO3) and S-Ka (CaSO4) differs, and this shows that calcium carbonate is not produced on the outermost layer of FGD plasterboard. Additionally, following 45 minutes of carbonation, it was seen that a spongy layer around the vaterite phase grew [8]. The two-step fermentation procedure was also used to create magnesium carbonate for comparison's sake. The first stage of the process, which results in the creation of sodium bicarbonate, is endothermic, and Fig. 7 shows how the temperature changes. The perfect pH, which was about 8, once more showed that the processes were finished. This pH level causes nitrogen hydroxide (NH4- HCO3) to be produced. Increased CO2 circulation equally aided two-step carbonation as it did straight fermentation. Additionally, Bonenfant et al. showed that increasing the CO2 flow velocity causes a boost in the pace of absorbing carbon dioxide in amide systems [29-33]. After carbonating for five minutes, the dissolved sulfate concentration was 523 mM (55%) in the two-step carbonation, but it was only 27 mM (3.5%) in the initial carbon dioxide. This indicates that throughout the initial phase of the subsequent step, the measure of dispersed sulfates and, consequently, the content of magnesium have become significantly higher compared to what happened in the project bubbling [10]. According to the XRD analysis, calcite was the predominant solid formed during the whole two-step bubbling process. Figure 3 shows the SEM and TEM images of the gypsum [34-39]



**Fig. 3** (a) (b) shows the SEM and TEM images of the gypsum

## Polymorphs of calcium carbonate

Amongst all three polymorphism types of carbonates of calcium, Vaterite is the most thermally stable stage; however, given certain circumstances, that metastable in nature form is regularly encountered [11, 50-52]. The carbonating cycle, carbonated duration, etc. were discovered to affect the variants of carbonate of calcium in our research. At a carbon dioxide circulation rate of 1 L/min, the phase shift that occurred throughout the acidification period was observed [12, 46-49]. The XRD structures for goods following indirect carbonation for different time frames are shown in Fig. 9. When the carbonation duration was less than 15 minutes, the calcium carbonate wasn’t visible by Diffraction. The quantity of vaterite at that stage varies with time for both indirect carbonated and two-step carbon dioxide, as seen [13]. The vaterite at that stage developed further during the course of the indirect carbonating, as has been shown by FE-SEM (Fig. 5). It can be observed that the vaterite stage grew quickly and plateaued after 45 minutes. On the other hand, calcite predominated over the whole reaction time of the two-step acidification. According to Dickinson et al., a lack of calcium ions in the air causes the formation of thermally stable calcite [40-45]. They assert that pCO2 affects crystalline as well as vaterite production, which is dynamically encouraged by elevated pCO2 levels whenever the amount of calcium ion is more than 90 mM. Due to rising CO2 quantities, Han et al. likewise noticed a similar trend of growing percentages of vitriol as they increased CO2 velocity. The results presented are in line with our findings. The amount of sulfate ions in the air is not very high, as mentioned in Subsection [15].

# Conclusion

Investigations were made into the formation of calcium carbonate crystals that formed after the immediate aqueous carbonation of plasterboard used in the desulfurization of flue gas (FGD). Significant carbonated sensitivity was found in FGD limestone at normal pressure and temperature conditions. Increasing CO2 flow boosted carbonation since the synthesis of ammonia bicarbonate served as the speed-limiting stage in the straight water carbonation of FGD limestone. Rhombic limestone and round vaterite are calcium bicarbonate variations formed by mechanical fermentation; agate was not found. For a constant Carbon Dioxide circulation rate, it was discovered that the quantity of vaterite, a component of the combination, increased with acidification duration. On the other hand, during all effervescent periods, two-step carbonation primarily created calcium particles. The product that was taken out throughout the initial stage involving direct carbonation solidified with almost no impurities.

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