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Title: Experimental tests for acid flue gas emissions treatment in industrial applications.

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Abstract

Removal of acid gases from exhaust emissions of industrial applications (SO_x, HCl, HF) still represents a challenge for many sectors, in particular for waste-to-energy plants. New and existing installations must comply with even tighter emission requirements as new regulations are implemented. Typically, these industrial facilities employ wet or dry scrubbing technologies for compliance. In dry absorption processes, the solid reagent is injected counter-current to the gas in a reaction chamber that allows for optimal contact between the phases. Efficiency is influenced by the specific surface area of the solid (m² kg⁻¹ reagent), by the degree to which it is mixed with the gas, the concentration of the gas to be adsorbed, the temperature and humidity of the flow and the concentration of the reagent, usually expressed in terms of stoichiometric ratio. In the case of carbonaceous sorbents (lime-based), the optimum operating temperature is around 140 - 150°C. As an alternative to using lime, sodium bicarbonate (NaHCO₃) can be used as an adsorbent agent. The optimum performance of this reagent is observed at temperatures around 170 - 180°C. Sodium bicarbonate has higher collection efficiency than lime, but it is more expensive.

After reacting, solid sorbents can be separated from the flue gas together with the dust particles via a fabric filter. The formation of a filter cake is necessary for the neutralization process, as it influences the retention time of the additive and, hence, the contact time of additive and pollutant in the flue gas. Recently, new formulation of calcium and sodium-based sorbents have been investigated. Hence, the design and development of accurate systems for the experimental characterization of these material is needed.

The object of this study was the characterization of solid sorbents for hydrochloric acid neutralization. The characterisation consisted in experimental tests for 1) the characterization of the sorbents, and 2) the calculation of adsorption capacity. Four different samples were considered, two of hydrated lime (samples C1 and C2) and two of sodium bicarbonate (samples C3 and C4). All samples were provided by industrial partners, except for C4 that was commercial sodium bicarbonate.

Adsorption tests were carried out using an experimental test bench, designed in the laboratory, with the aim of simulating the temperature, pressure and flue gas velocity conditions found in a Duct Sorbent Injection (DSI) configuration (Figure 1) [1]. Sorbent samples were put in contact with a mixture of nitrogen and hydrochloric acid (500 ppm), and measuring the concentration of chlorides dissolved in a downstream absorber using an ion chromatograph. The amount of chlorine retained by the sample, i.e. the adsorption capacity, was calculated assuming a plug-flow system model. Adsorption capacities were tested at 150°C. These tests showed that sodium-based sorbents showed up to 15% higher sorption capacity than lime-based sorbents. Important differences were also found among samples of the same type (e.g. C1 vs. C2).

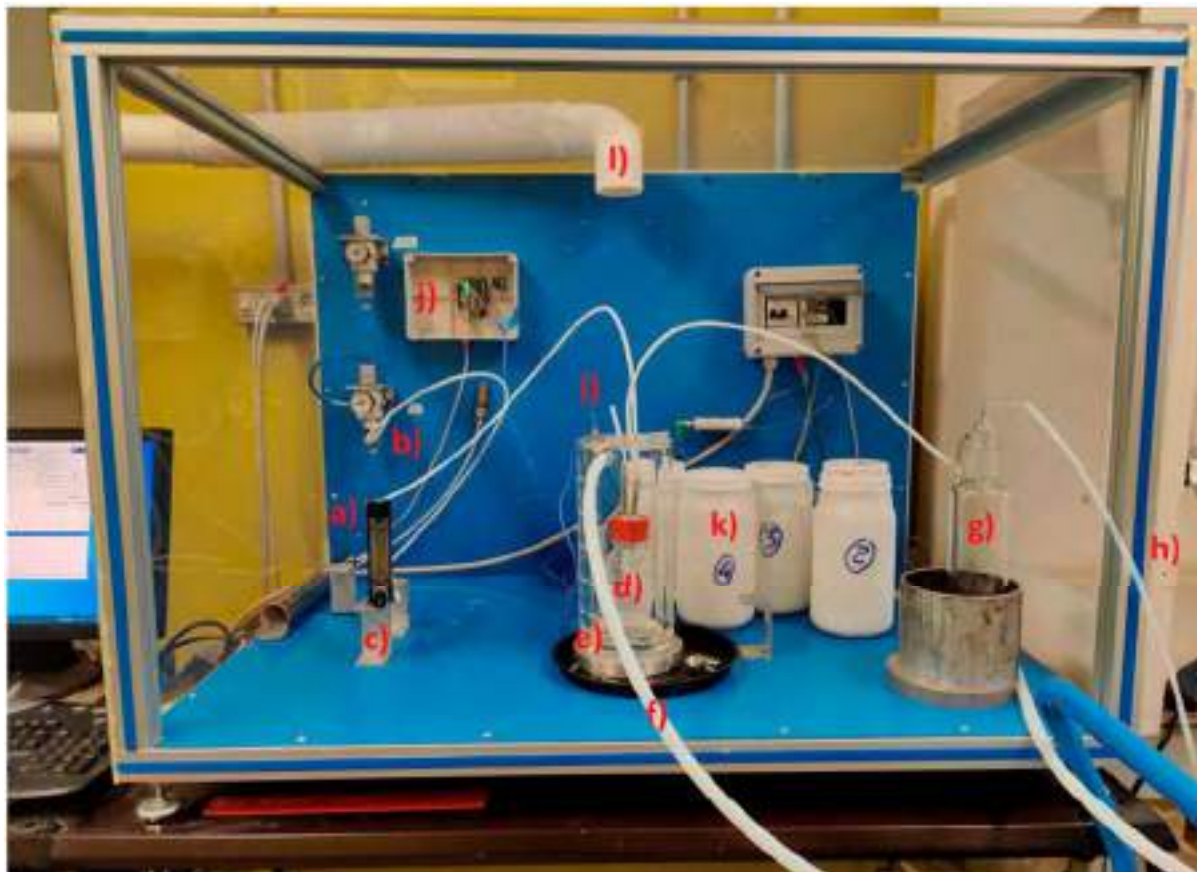


Figure 1. Photo of the experimental system: (a) HCl input line; (b) N₂ input line; (c) Flowmeter; (d) Reactor, (e) jacketed beaker; (f) Thermo-conditioning circuit; (g) Water absorber; (h) vent line; (i) thermocouple; (j) Arduino device; (k) sorbent samples; (l) air extraction to ventilated hood [1].

Sorbent materials were also subjected to additional tests for their chemical-physical characterisation, i.e. analyses with scanning electron microscope (SEM, Figure 2), X-ray diffraction (XRD), inductively coupled plasma mass spectrometry (ICP-MS) and BET analyses with a porosimeter. SEM analyses identified hydrated lime samples composed of coarse elements, an intermediate fraction and a fine fraction, which were distributed homogeneously in space. The bicarbonate samples visually revealed the presence of coarse and intermediate elements (no fine fraction), which appeared more heterogeneously arranged in space.

X-ray diffraction analyses enabled a quantitative or semi-quantitative analysis of the composition of the samples (Rietveld method). XRD analyses showed that both lime samples consisted mainly of calcium hydroxide (Ca(OH)₂). In addition, calcium carbonate (CaCO₃) was found in C1 and C2, to an amount of 10% and 18% w/w respectively. This component probably represents the portion not converted during the calcination process. In sample C2, the presence of dolomite (CaCO₃·MgCO₃) and magnesium oxide (MgO) was found. The presence of magnesium in the form of magnesium oxide or magnesium hydroxide (Mg(OH)₂) depends on the different hydration modes. C3 and C4 were found to be entirely composed of sodium bicarbonate (NaHCO₃).

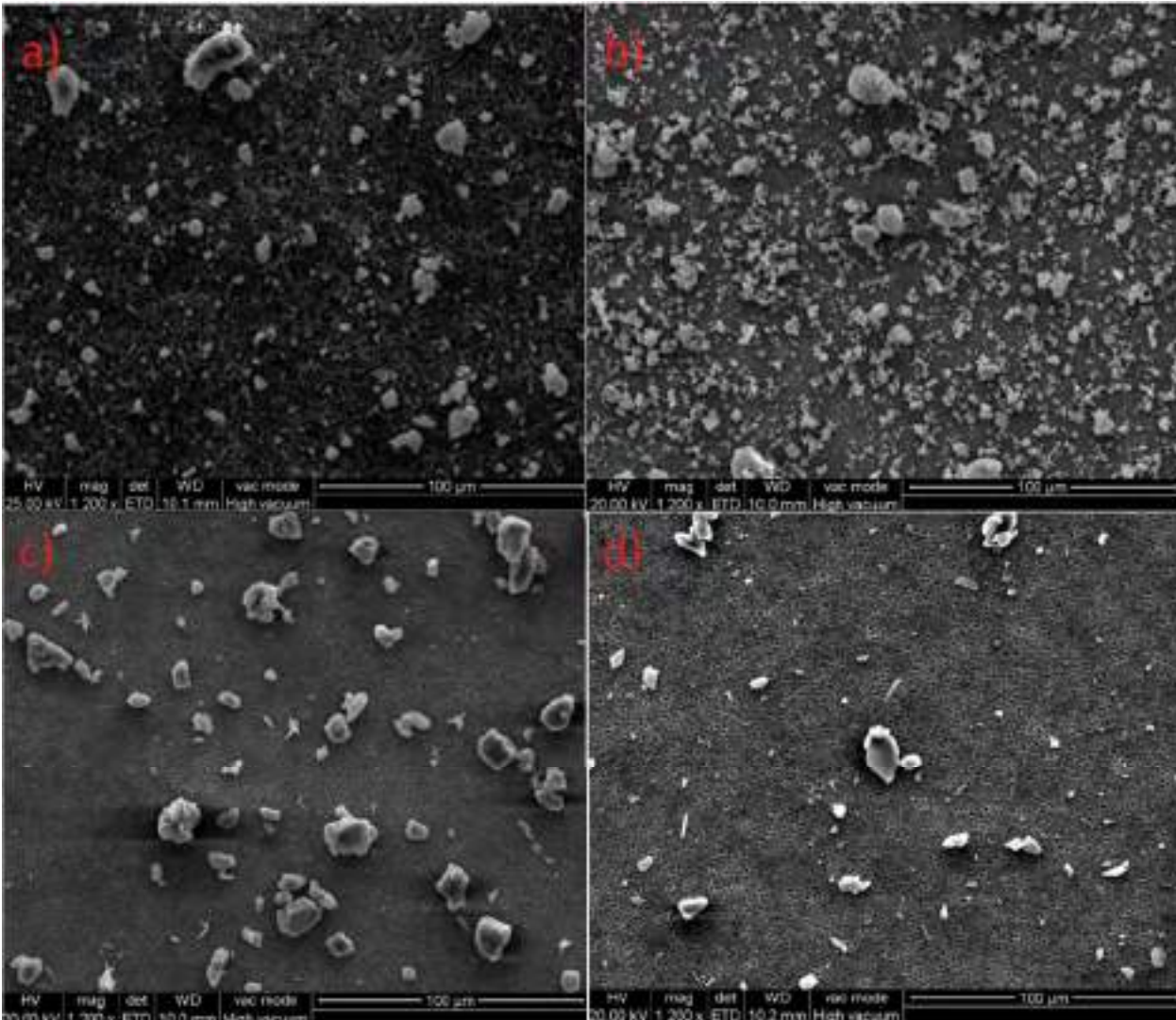


Figure 4. Scanning electron microscopy (SEM) pictures of Sample C1 (a), Sample C2 (b), Sample C3 (c), and Sample C4 (d).

ICP-MS analyses provided a quantitative composition of single elements contained in the samples. Lime-based samples showed dominating percentages of calcium. Sample C2 also contained around 8% of magnesium. The composition of samples C3 and C4 was confirmed to be mostly sodium.

BET analysis was conducted with a porosimeter and allowed determining the specific surface area and porosity values of each sample. Sample C1 showed specific surface area and porosity values approximately four times higher than other samples.

Finally, tests were conducted on the sorbent samples after the reaction, to investigate the reaction products. An amount of 1 g of reacted sample was dissolved in ultrapure water during prolonged stirring, then the concentration of chlorides was measured. These tests showed that, except for C4, the measured chlorine mass values were lower than the expected. This is probably due to an incomplete dissolution of the reacted chlorine.

The results of the present study showed that a combination of properties, i.e. specific surface area, reactivity, favourable thermodynamic conditions, particle size, affect the adsorption capacity of solid



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sorbents with respect to acid components in the flue gas. The good adsorption capacity of C1 is probably due to its high specific surface area, whereas that of C3 is more related to the higher reactivity. In order to achieve better abatement efficiencies, it would be necessary to ensure more uniform grain sizes and avoid undesired components (e.g. the presence of unreacted carbonates). Conversely, agglomeration of fine particles may have adversely affected the performance of sorbents.

References

[1] Ravina, M.; Marotta, E.; Cerutti, A.; Zanetti, G.; Ruffino, B.; Panepinto, D.; Zanetti, M. Evaluation of Ca-Based Sorbents for Gaseous HCl Emissions Adsorption. *Sustainability* 2023, 15, 10882, doi:10.3390/su151410882.