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Comparing the Performance of Recirculating Cyclones Applied to the Dry Scrubbing of Gaseous HCl with Hydrated Lime

Vania G. Chibante,† Ana M. Fonseca,*,‡ and Romualdo R. Salcedo†

DEQ/LEPAE, Faculdade de Engenharia da Universidade do Porto, Rua Dr. Roberto Frias s/n, 4200-465 Porto, Portugal, and CIAGEB, Universidade Fernando Pessoa, Praça 9 Abril 349, 4249-004 Porto, Portugal

Recirculating cyclone systems, combining a numerically optimized reverse flow gas cyclone (RS_VHE geometry) with a straight-through cyclone concentrator, were employed as reaction chambers for the dry scrubbing of gaseous hydrogen chloride with solid hydrated lime particles. The performance of this technology was tested at laboratory scale with two differently sized RS_VHE cyclones (0.020 and 0.026 m internal diameter) coupled to the same recirculator of 0.020 m internal diameter. The experimental conditions were the following: reaction temperature ≈ 326 K, gas flow rate ≈ 2.9×10^{-4} $m^3 \cdot s^{-1}$ at STP, and relative humidity of the gas ≈ 8.5%. The experimental variables were the solids feed rate [$(1.0-9.2) \times 10^{-7}$ $kg \cdot s^{-1}$] and HCl concentration in the inlet gas [$(0.35-2.8) \times 10^{-2}$ $mol \cdot m^{-3}$], giving different values for the ratio between the amount of fresh hydrated lime and HCl feed at the inlet of the recirculating cyclone systems (R) and that corresponding to the stoichiometric quantity (SR). The acid removal efficiencies ranged from 10 to 96%, and the best performances were obtained for high values of the ratio R/SR. Increasing the cyclone diameter while maintaining cyclone geometry improved the performance of the acid gas removal as well as the solid reactant conversion, but the average particle collection efficiency was lowered from (98.0 ± 0.7)% to (89.3 ± 1.7)%.

1. Introduction

Environmental quality is a key issue of human society. Due to the important role of industry in economic development, industrial plants should operate while minimizing the aggression to the environment. In order to guarantee the human welfare in the future, governments are establishing increasingly rigorous regulations, and industry has to make severe economic efforts to comply. Therefore new technological alternatives have to be developed to ensure industry's ability in complying with new standards in environmental performance.

The incineration of solid wastes (municipal, hazardous, industrial,...) and the combustion of certain solid fuels (coal, biomass,...) are environmental menaces since these processes may produce large amounts of air pollutants (HCl, SO_x, HF, CO, NO_x, VOCs, heavy metals, fine particulate matter), which are important sources of local and global environmental pollution.[1−4]

Among these pollutants, acid gases such as HCl, SO_x, and HF are causes of acid rain and fog, which are harmful to human life, modifying the lungs' defenses and aggravating cardiovascular or chronic lung diseases.[4,5]

The processes used for acid gas removal in gaseous emissions are classified as "wet", "semidry", or "dry". Although wet scrubbing technologies have proven to be more efficient for acid gas cleaning purposes, this technology implies the generation of a wastewater problem and has economical drawbacks when compared to dry or semidry processes. Acid gas cleaning with dry or semidry technologies are becoming more widespread since they are easy to use, efficient, and of simple implementation.[2,4,6] Dry sorbent injection can be performed at low (150 °C) or moderate (200−315 °C) temperature in the so-called in-duct injection or directly in the combustion chamber at higher temperatures (750−1100 °C).[1]

* To whom correspondence should be addressed. E-mail: afonseca@ufp.pt.
† Universidade do Porto.
‡ Universidade Fernando Pessoa.

Calcium compounds such as $Ca(OH)_2$, CaO, and $CaCO_3$ are sorbents frequently used in dry scrubbing due to their low cost and high efficiency.[4,6−9] Nevertheless, the dry scrubbing process needs to be improved since the very short contact time available for the sorbent and acid gases to react results in low sorbent conversion efficiencies (<25%) and generates a large amount of solid particles.[6] Moreover, in order to improve the sorbent conversion and to achieve the increasing stringent emission limits set by regulations, the partially spent sorbent should be recirculated[10] or new sorbents with higher removal efficiencies should be used.[1,8,11]

In the dry scrubbing process, the gases are removed through adsorption and chemical reaction at the solid's surface and a dry powdery product results. This product is usually separated downstream the reactor chamber from the cleaned flue gas by bag filters or electrostatic precipitators. The promotion of the dry scrubbing and the dust removal processes in the same equipment would have considerable practical and economical implications, and in the authors' opinion this can be made possible using a cyclone as reaction chamber.

Cyclones are well-known gas−solid separators, associated with low investment and maintenance costs, which are robust when operating at heavy solids' loads and at high temperature and pressure. Some studies have proven that cyclones can be powerful gas−solid reactors.[12−15]

The main design of industrial gas cyclones is the tangential reverse-flow type,[16,17] and recent studies have confirmed that it is possible to design significantly improved reverse-flow gas cyclones by solving adequate numerical optimization problems.[17−19] On the basis of this approach, a new cyclone geometry (RS_VHE) was identified.[17]

The RS_VHE geometry was designed to avoid saltation, a nonideal phenomenon that reentrains large particles (>5 μm) in the exit gas from the cyclone, reducing collection below design values. This new geometry revealed at laboratory, pilot, and industrial scales to have much lower emissions at comparable pressure losses, with typical reductions of 50% over other

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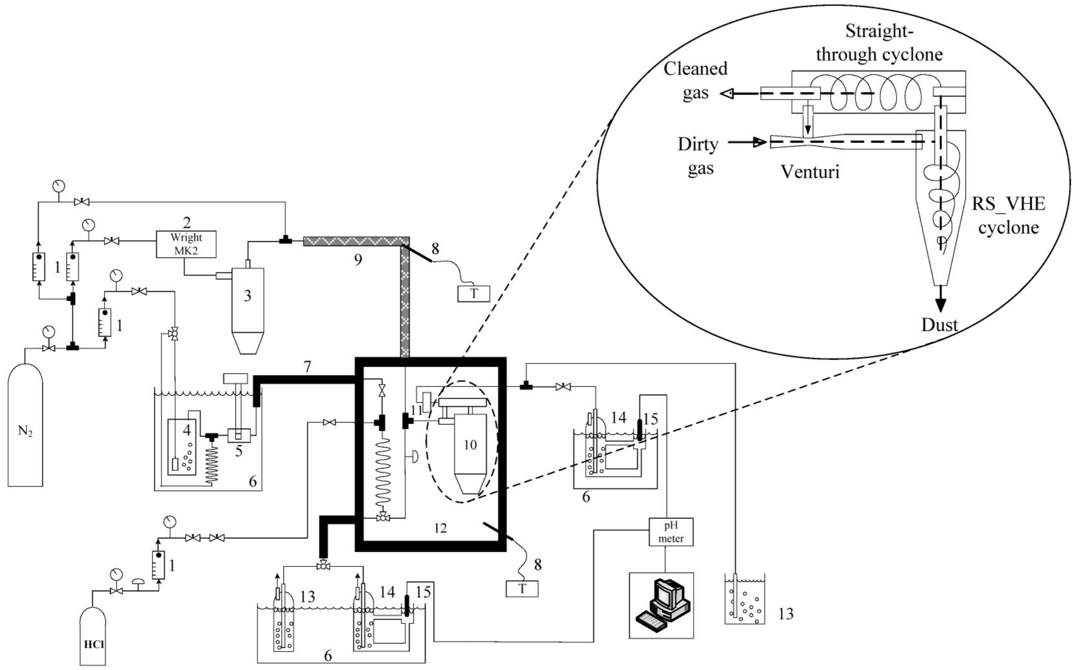

( ) manometer; ( ) pressure regulator; ( ) on/off valve; ( ) needle valve; ( ) three-way valve; ( ) check valve. 1: rotameter; 2: solid particles feeder; 3: deagglomerating cyclone; 4: humidification column; 5: hygrometer; 6: temperature controlled bath; 7: thermal insulation; 8: thermocouple; 9: electrical heating tape; 10: cyclone reactor; 11: filter; 12: oven; 13: absorber; 14: absorber with lateral chamber; 15: pH electrode.

Figure 1. Experimental setup.

reverse-flow cyclone designs like, for example, the Stairmand HE geometry.[17,19,20]

Recirculating cyclone systems increase the performance over cyclones and are used in this work by employing a RS_VHE reverse-flow cyclone (collector) upstream from a straight-through cyclone (concentrator). This concentrator promotes the partial recirculation of the gas and solids by a venturi, blower, or ejector. These recirculating systems have been tested at laboratory, pilot, and industrial scales, achieving, under some circumstances, particle removal efficiencies comparable to those of a pulse-jet bag filter.[19−21]

The high capacity to purify a gaseous stream contaminated with HCl in a dry scrubbing process using hydrated lime was already proven in previous studies using reverse flow cyclones[14,15] or recirculating cyclone systems.[15] The results obtained in the latest study suggest that the contact time of the gas with the particles in the cyclone reactor is an important parameter for this process, and this should be further exploited. Increasing the cyclone's volume will increase the contact time between the gas and the particles through the combined effect of increased gas mean residence time and lowered solid particles' capture. This is expected to improve the HCl removal efficiency as well as the conversion of the solid particles, which could lower the fresh reactant's consumption.

The aim of the present study is to verify, at laboratory scale, the extent of the effect of the gas and particle contact time in the process of dry scrubbing of HCl with $Ca(OH)_2$ in recirculating cyclone systems. For this purpose, two different sized RS_VHE cyclone reactors (0.02 and 0.026 m internal diameter) were built and used in the recirculating systems.

2. Experimental

The experimental laboratory-scale setup is schematically shown in Figure 1. A detailed description of the equipment can be found elsewhere.[14,15] The experimental setup was designed to promote the continuous contact in the recirculating cyclone system between the solid lime particles injected in a gaseous stream containing HCl. The manipulated operation variables were the following: gas flow rate, solids feed rate, HCl concentration, temperature, humidity level in the gas, and reaction time.

The solid particles of hydrated lime dust (Riedel-de-Haën, pro-analysis, minimum $Ca(OH)_2$ of 96% and maximum $CaCO_3$ of 3%) have a median volume diameter of 2.3 μm and a specific gravity of 2350 $kg \cdot m^{-3}$. The particles were injected in a carrier gas (N_2), which contacts a gaseous stream of N_2 with a controlled concentration of HCl and humidity level, immediately before the inlet to the recirculating cyclone system. The HCl concentration in the effluent gas is measured by bubbling a known fraction of this stream into distilled water by means of an external circulation airlift absorption vessel.[14,15,22] The HCl is completely absorbed in the water, and the H^+ concentration in the resulting solution is monitored online with the aid of a computerized data acquisition system (Labview routine).

The solid particles are collected in the cyclone reactor, and those escaping the system are captured in a GFA filter located at the exit of the recirculator. This backfilter was employed to avoid particles exiting the systems to reach the pH measuring absorption vessel. For a cross check, at the end of the experimental run, the solids captured are separately collected and chemically analyzed by potentiometric titration. The amount of chloride contained in a sample was evaluated by titrating the chloride ion with a silver nitrate solution, using a combined Ag electrode. The conversions of the solid reactant in the cyclone reactor and in the filter are then obtained through material balances, considering $CaCl_2 \cdot 2H_2O$ as the reaction product.

Table 1. Experimental Conditions for the Dry Scrubbing Tests

	RS_VHE 2 system[15]	RS_VHE 2.6 system
cyclone reactor internal diameter (m)	0.020	0.026
relative humidity[a] (%)	9.0 ± 0.3	8.3 ± 0.1
HCl concentration[a] × 10^2 (mol·m^{-3})	0.35−2.8	0.69−1.8
fresh solids feed rate[a] × 10^7 (kg·s^{-1})	1.1−9.2	1.0−6.7
total experimental time (min)	10.6 ± 0.9	11.6 ± 0.8
total gas flow rate[a] × 10^4 (m^3·s^{-1} at STP)	2.9 ± 0.1	
reaction temperature (K)	326 ± 0.4	

[a] At the inlet to the recirculating cyclone system (before recirculation loop).

Two sets of experiments were performed, varying the internal diameter of the RS_VHE cyclone in the recirculating cyclone system. Table 1 shows the experimental conditions for both systems at a 5% significance level.

The mean residence time of the gas was calculated[23] to be 0.026 and 0.057 s, respectively for the RS_VHE cyclone reactors 2 and 2.6, and for the recirculating cyclone systems 2 and 2.6 (collector and concentrator),[24] respectively of 0.091 and 0.12 s.

The ratio R/SR is calculated through the expression

$$R/\mathrm{SR} = \frac{\dfrac{W_{\mathrm{Ca(OH)_2}}}{QC_{\mathrm{HCl}}}}{\dfrac{M_{\mathrm{Ca(OH)_2}}}{2}} \quad (1)$$

where $W_{Ca(OH)_2}$ is the fresh solids feed rate at the inlet of the recirculating cyclone system (before the recirculation loop), C_{HCl} and Q are respectively the HCl concentration and the gas flow rate at the same location, and $M_{Ca(OH)_2}$ is the molar mass of $Ca(OH)_2$. The experimental variable R/SR can be manipulated either by changing the solids feed rate or by changing the HCl concentration, or both.

3. Results and Discussion

In all the experiments performed, it was verified that when the feeding of the solid reactant starts, the HCl concentration in the effluent gas, given by the slope of H^+ versus time, reaches a constant value, corresponding to a steady state removal efficiency.

The results presented in Figure 2 show that, for similar experimental conditions, the RS_VHE 2.6 system (Figure 2b) has a higher acid removal capacity than the RS_VHE 2 system (Figure 2a).

The slope of the line H^+ versus time, once the steady-state has been reached, corresponds to the HCl constant concentration in the outlet gas and was obtained through a statistical test:[25] starting from the last five end points and going backward in time sequentially one data point at the time, for a cumulative increasing number of experimental points (n_p), a linear fit $\hat{y} = a + bx$ is performed on the experimental data, after rescaling the time scale to the start of the hydrated lime injection. Then the corresponding F^* parameter is calculated through the expression

$$F^* = \frac{1}{n_p - 1} \times \sqrt{\frac{\sum (\hat{y} - y)^2}{\sum y^2}} \quad (2)$$

According to this statistical test, the slope of the line to be considered is the one corresponding to the smaller value of F^*: as can be observed in eq 2, the number of points and the accuracy of the fitting (given by the deviation of the fitted points from the experimental data) are simultaneously taken into account. Therefore, a large data set lowers F^*, and for a small data set to be as good, it must have a higher accuracy. This compromise means that there is some optimum number of data points to be considered in the regression.

The experimental values obtained for the HCl removal efficiency (η_{HCl}) varied from ≈10% (C_{HCl} = 1.3 × 10^{-2} mol·m^{-3}, R/SR = 0.6) to ≈96% (C_{HCl} = 0.67 × 10^{-2} mol·m^{-3}, R/SR = 6.5) in the RS_VHE 2 system,[15] and between ≈17% and ≈94% (C_{HCl} = 1.2 × 10^{-2} mol·m^{-3} and R/SR = 0.7 or R/SR = 4.2, respectively) in the RS_VHE 2.6 system.

Figure 3a shows the experimental acid removal efficiencies (η_{HCl}) and Figure 3b shows the solid reactant's conversion obtained in the cyclone reactor (X_C^*) and in the filter (X_F^*) relative to the fresh feed at the inlet of the recirculating cyclone system, for different R/SR values and for both recirculating cyclone systems. The choice for this basis was dictated by the practical application of this technology, since it is the conversion relative to the fresh feed that would mostly interest industrial practice. However, to provide further information on the currently accepted meaning of conversion, Figures 3c and d show the solid reactant's conversion in the filter (X_F) as compared to that in the cyclone (X_C), relative to the captured samples, for both cyclone systems. These figures show, as expected, that the filter conversion is always larger than the cyclone conversion.

As can be observed in these figures, the RS_VHE 2.6 system has a better performance than the RS_VHE 2 system, both for the acid gas removal efficiencies and for the solid reactant's conversions. These results confirm the expectation that increasing the contact time between gas and particles in the cyclone reactor would improve the acid removal efficiency and the solid conversions. Nevertheless, the usage of the solid reactant is still low: the average solid conversions (X_C^*) are lower than 25% and apparently not affected by R/SR. As already indicated in previous studies,[15,26] the reaction is most probably controlled by diffusional limitations.

Given the high efficiency of the cyclone system for particle collection, the mass of particles exiting the recirculating cyclone systems and collected in the filter was very low. In spite of having experienced the longest contact time with the gas, and therefore having higher solid conversions (X_F) than those obtained in the cyclone reactor (X_C), their contribution to the HCl removal was not relevant: the conversion level of these particles, relative to the total amount of fresh reactant fed to the recirculating cyclone system (X_F^*), was on the average less than 3%. The HCl removal efficiencies experimentally obtained are thus essentially due to the RS_VHE cyclone by itself, as already found in previous works.[14,15] Figures 3c (RS_VHE 2 system) shows that the solid conversion occurring in the back filter, based in the captured sample (X_F) is larger than that of Figure 3d (RS_VHE 2.6 system). This is due both to the higher particles' collection efficiency and the lower acid removal of the smaller cyclone (RS_VHE 2).

As shown in Figure 3a, the HCl removal efficiency increases with R/SR, but since this parameter is based on a ratio between the solid reactant's load and the HCl concentration, it is important to separately evaluate the influence of these experimental variables. Therefore, two sets of experimental results were selected: Figure 4 illustrates the influence of the fresh solid's reactant feed rate on the experimental acid removal

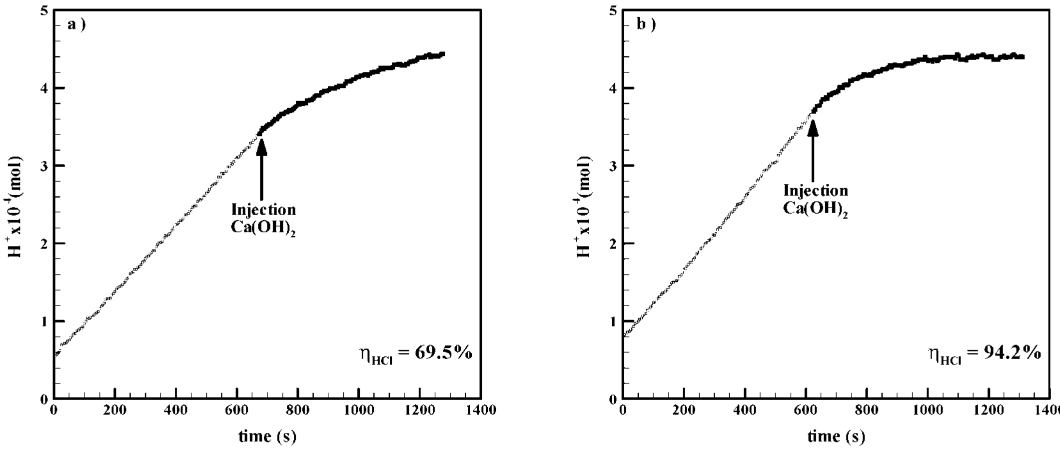

Figure 2. Experimental variation of H^+ vs time (R/SR = 4.2). (a) RS_VHE 2 system, $C_{HCl} = 1.10 \times 10^{-2}$ mol·m^{-3}. (b) RS_VHE 2.6 system, $C_{HCl} = 1.20 \times 10^{-2}$ mol·m^{-3}.

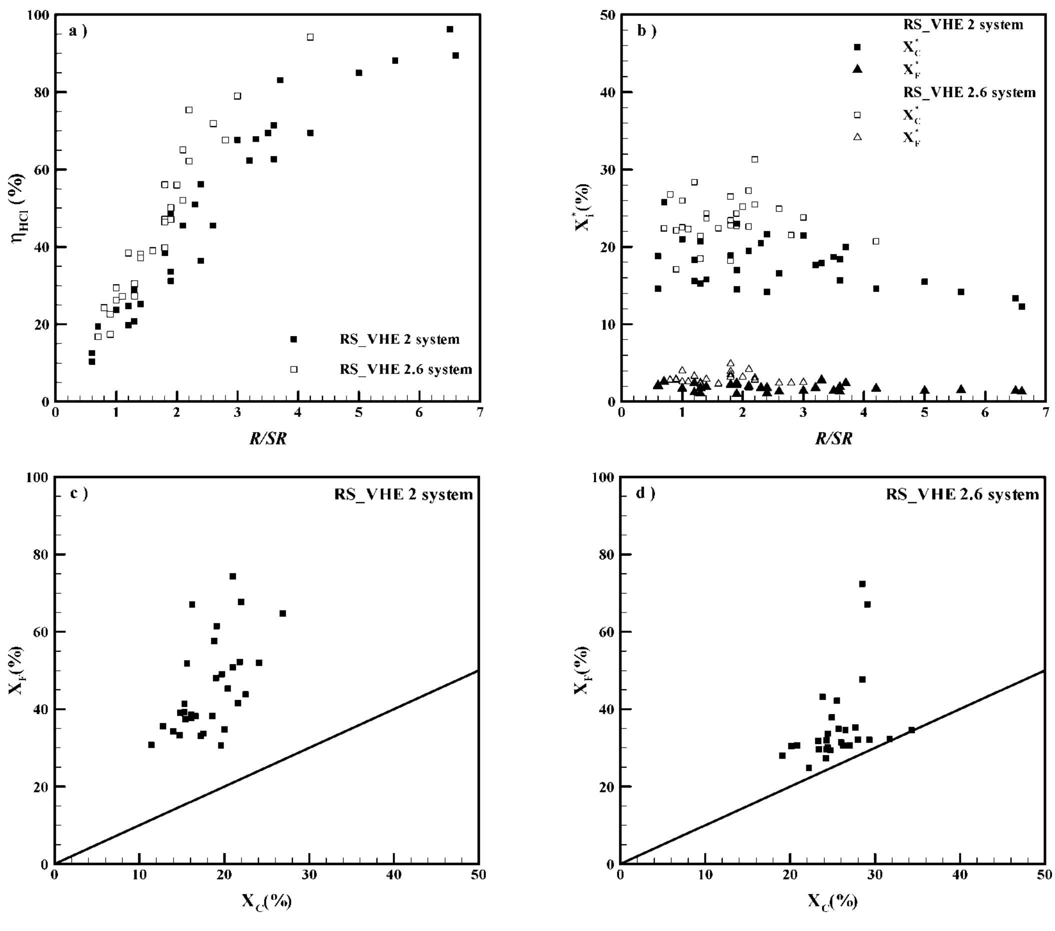

Figure 3. Experimental results for the acid gas removal efficiency (a), for the solid reactant's conversion relative to the fresh feed (b) and for the solid reactant's conversion relative to the captured sample (c and d) for the two recirculating cyclone systems tested. The solid line is filter conversion if equal to cyclone conversion (X_C).

efficiencies, for a fixed HCl concentration (C_{HCl}); and Figure 5 shows the influence of the acid gas concentration for a fixed feed rate of fresh hydrated lime ($W_{Ca(OH)_2}$). Both the reactant feed rate and the initial gas concentration were measured at the inlet of the recirculating cyclone system, i.e., prior to the recirculation loop.

In these figures, the selected interval (±10%) reflects the range of approximately similar conditions that could be chosen from the experimental data, since it was virtually impossible to run different experiments at exactly the same acid gas inlet concentration and solid feed rate values.

These results show that, under the present experimental conditions, and for both systems tested, the dry-scrubbing process is more efficient for high values of the ratio *R*/*SR*.

4. Particles' Collection Efficiency. One of the potential advantages of these recirculating cyclone systems is that, when at pilot or industrial scales, they are supposed to operate without any electrostatic precipitator or bagfilter downstream. Therefore a set of experiments was carried out to evaluate the particles' collection efficiency and the grade efficiency curves of the recirculating cyclone systems in the absence of reaction. Fine

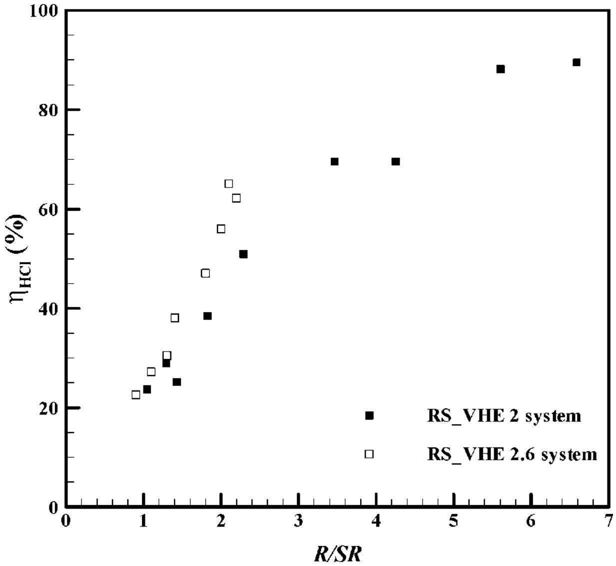

Figure 4. Experimental results for acid gas removal varying the fresh solid reactant's feed rate and $C_{HCl} = (1.0 \pm 0.1) \times 10^{-2}$ mol·m^{-3}.

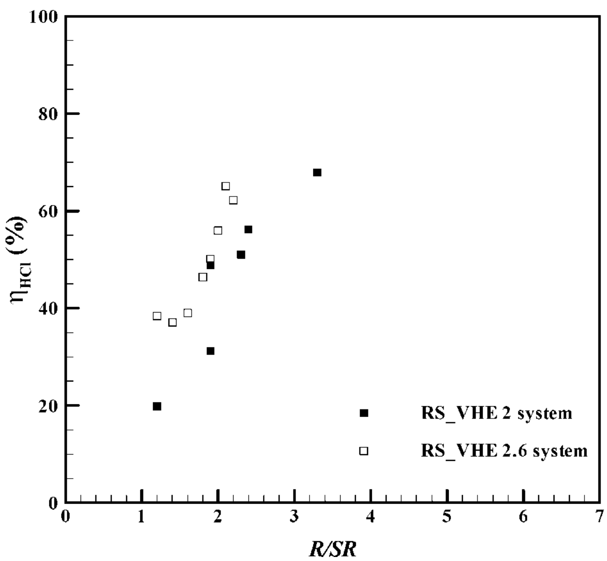

Figure 5. Experimental results for acid gas removal varying the acid gas concentration and $W_{Ca(OH)_2} = (3.1 \pm 0.3) \times 10^{-7}$ kg·s^{-1}.

hydrated lime particles were injected in a carrier gas (N_2) using a Wright dust feeder at ambient temperature. Grade efficiency curves were obtained sampling the total dust entering and exiting the recirculation cyclone system on GFA filters, and by weighting the filters on an analytical balance after proper drying and conditioning. Particle size distributions were analyzed off-line down to 0.04 μm using a Coulter LS 230 laser sizer, after ultrasonic redispersion in ethanol. For the RS_VHE 2.6 system, the experimental gas and solid flow rates were those of the dry-scrubbing tests, while for the experiments with the RS_VHE 2 system, extensively described elsewhere,[15,21] a wider range of experimental conditions, shown in Table 2, were tested.

The average particle size distributions of the $Ca(OH)_2$ at inlet and outlet of the recirculating cyclone systems are illustrated in Figure 6.

Figure 6 shows that the feed is weakly bimodal and that all particles are below 10 μm in diameter. This figure also shows that particles characterized by diameters above about 4 μm and below 0.2 μm are, for practical purposes, completely removed by the recirculating cyclone systems.

Table 2. Experimental Conditions for the Particles' Collection Tests

	RS_VHE 2 system[15,21]	RS_VHE 2.6 system
total gas flow rate[a] × 10^4 (m^3·s^{-1} at STP)	2.5−4.8	2.9
airflow recirculating[b]	0.10−0.14	0.14
fresh solids concentration[a] (mg·m^{-3} at STP)	226−961	221−854
inlet velocities to the cyclone reactor[c] (m·s^{-1})	8.8−17.9	6.5

[a] At the inlet to the recirculating cyclone system (before recirculation loop). [b] Ratio of gas flow rate in the recirculation loop over the total gas flow rate. [c] After recirculation loop.

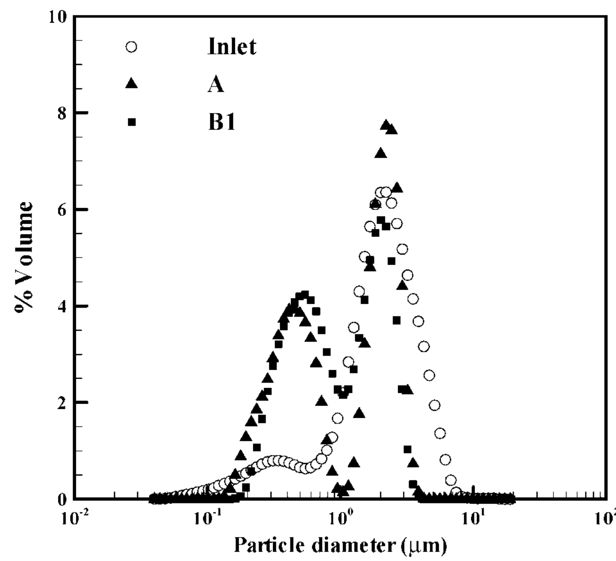

Figure 6. Particle size distributions at the inlet (average) and at the outlet for the two recirculating cyclone systems (**A**: RS_VHE 2 system, 226 mg·m^{-3} at STP, 4.0 × 10^{-4} m^3·s^{-1} at STP; **B1**: RS_VHE 2.6 system, 221 mg·m^{-3} at STP, 2.9 × 10^{-4} m^3·s^{-1} at STP).

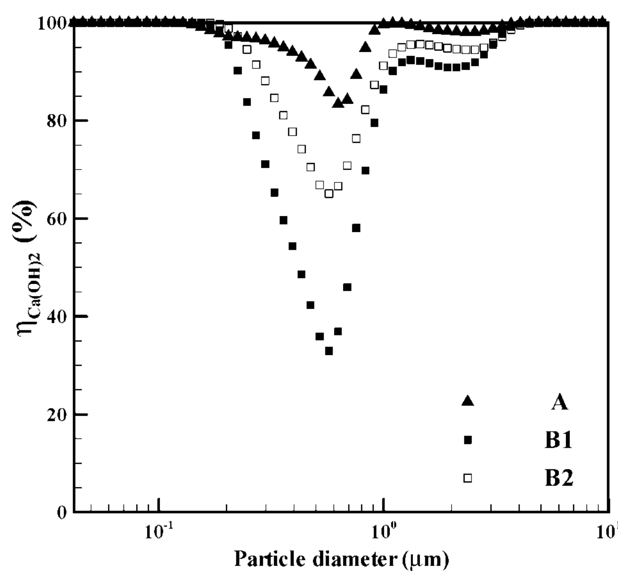

Figure 7. Grade efficiency curves for the capture of $Ca(OH)_2$ particles (**A**: RS_VHE 2 system, 226 mg·m^{-3} at STP; **B1**: RS_VHE 2.6 system, 221 mg·m^{-3} at STP; **B2**: RS_VHE 2.6 system, 683 mg·m^{-3} at STP).

The grade efficiency curves obtained are shown in Figure 7. Two different inlet solids' concentrations are presented for the RS_VHE 2.6 system, showing, as expected, that for the higher solids' load the capture efficiency is the best. Comparing, for the same solids' concentration, the performance of both systems, shows that the smallest cyclone has the higher efficiency. This is not only due to the smaller cyclone size, which by itself gives rise to a larger inlet mean velocity for the same gas flow rate, but also to the fact that the gas flow rate was actually higher for the smaller cyclone (see Figure 6).

The average particle collection efficiency in the absence of reaction, at a total gas flow rate of 2.9×10^{-4} $m^3 \cdot s^{-1}$ at STP was (89.3 ± 1.7)% for the RS_VHE 2.6 system and (98.0 ± 0.7)% for the RS_VHE 2 system,[15,21] both at a 5% significance level. These results, obtained at laboratory scale, are hardly representative of industrial applications, but pilot and industrial tests using recirculating cyclone systems in biomass boiler applications and in fine chemicals recovery confirmed their high performance, obtaining total particle removal efficiencies in the range of 90−99.6%.[20,21]

The abnormal collection of very fine particles, illustrated in Figure 7, had been observed before.[15,17,19−21] It is attributed to capture of small particles by larger ones in the turbulent cyclone flow field,[21] much as it happens in recirculating fluidized beds,[27] and work is currently being carried out to model this phenomenon.

Conclusions

In the experimental conditions tested, the RS_VHE 2.6 system has better performance than the RS_VHE 2 system both for the acid removal and for the solid reactant's usage. Since the mean residence time of the gas in the RS_VHE 2.6 cyclone is larger than in the RS_VHE 2 cyclone, these results confirm the conclusions of previous studies that the reactor vessel is essentially the RS_VHE cyclone and not the recirculation loop.

Increasing the cyclone's volume while keeping its geometry increases the contact time between the gas and the particles through the combined effect of increased mean gas residence time and lowered particle capture. This improves the HCl removal efficiency as well as the conversion of the solid particles.

The experimental results are highly dependent on the experimental conditions, but higher acid removals were obtained for high values of the ratio *R*/SR.

The solids' reactant conversions obtained relative to the fresh feed were on the average lower than 25%, revealing poor solids' usage. These results indicate that the reaction is controlled by diffusion limitations. This is supported by preliminary modeling.

The dry scrubbing of acid gases in recirculating cyclone systems is a technology with a high potential, since it is simple, efficient, and may have the advantage of not requiring a postreaction deduster. However, this depends on the particulate emission legal limits, since mechanical recirculating systems may be inadequate at industrial scale[19−21] below about 50 $mg \cdot m^{-3}$ at STP.

Testing this technology at pilot or industrial scales is important, since the longer contact times between HCl and the solid particles due to the larger volume of the RS_VHE cyclones at these scales is expected to result in both an increase in HCl removal and in solid conversion. Also, since recirculation can then be controlled by a blower rather than by a venturi, the recirculating capacities will be much improved, which could lead to lower fresh solids' loads.

It is also expected that the adoption of electrostatic recirculation[28] may allow these systems to operate where stringent emission limits for acid and particles are enforced. Here, a high voltage is imposed on a discharged wire located axially in the concentrator, thus providing an ionized electric field, with the objective of electrically charging the fine particles that escape the cyclone. Since charging operates best at low gas velocities, electrostatic recirculation may substantially improve fine particle capture, solids recirculation, and contact times.

The theoretical modeling of the experimental results, currently under development, is an important step in the understanding and scale-up of this new technology.

Acknowledgment

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Nomenclature

C_{HCl} = HCl concentration at the inlet of the recirculating cyclone systems ($mol \cdot m^{-3}$)
F^* = statistical parameter
$M_{Ca(OH)_2}$ = molar mass of the $Ca(OH)_2$ ($kg \cdot mol^{-1}$)
n_p = number of experimental points
Q = total gas flow rate at the inlet of the recirculating cyclone systems ($m^3 \cdot s^{-1}$)
R/SR = ratio between the amount of fresh hydrated lime and HCl feed at the inlet of the recirculating cyclone systems (R) and that corresponding to the stoichiometric quantity (SR)
$W_{Ca(OH)_2}$ = fresh solids feed rate at the inlet of the recirculating cyclone systems ($kg \cdot s^{-1}$)
X_C^* = solid reactant's conversion obtained in the cyclone reactor, relative to the fresh feed at the inlet of the recirculating cyclone system (%)
X_C = solid reactant's conversion obtained in the cyclone reactor, relative to the captured sample (%)
X_F^* = solid reactant's conversion obtained in the back filter, relative to the fresh feed at the inlet of the recirculating cyclone system (%)
X_F = solid reactant's conversion obtained in the back filter, relative to the captured sample (%)
y = experimental data for the quantity of H^+ present in the bubbling solution
$\hat{y}$ = fitted data for the quantity of H^+ present in the bubbling solution
Greek Symbols
η_{HCl} = HCl removal efficiency (%)
$\eta_{Ca(OH)_2}$ = $Ca(OH)_2$ removal efficiency (%)

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