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Sorbent behavior in urban waste incineration: acid gas removal and thermogravimetric characterization

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Abstract

A thermogravimetric procedure has been developed for the characterization of a partially reacted sorbent from a dry cleaning process (TURBOSORP®) which was operated in an urban waste incineration plant. The amounts of calcitic compounds in the sorbent: $Ca(OH)_2$, $CaCO_3$, $CaCl_2$ and sulfur containing products were determined by means of TGA, after validation with standard chemical analysis techniques. Different desulfurization products were obtained in a laboratory fixed bed reactor working under conditions of the large scale plant, in the range of relative humidity 20–45%. The results obtained show the relevance of $CaCl_2$ in the sorbent allowing a higher calcium utilization, in terms of mole of sulfur per mole of calcium, when working at 40–45% of relative humidity. The values achieved were significantly higher than those reported in previous studies working with hydrated lime as sorbent.

Keywords: TGA characterization; Calcium chloride; Acid gas removal; Urban waste incineration; Calcium utilization

1. Introduction

Different technologies have been developed for acid gas removal in emissions from incineration. The post-combustion acid gas removal technologies have been widely incorporated in the operation of large coal power plants in order to achieve the regulations of SO_2 and NO_x mainly. These technologies are based on the use of sorbents, usually calcitic sorbents, such as $CaCO_3$, CaO in desulfurization processes at high and medium temperatures, and hydrated lime in low temperature processes [1,2].

In acid gas removal processes, a great deal of work is performed in order to improve the sorbent utilization, studying in particular the alternatives of: (i) additives, such as NaOH, $CaCl_2$, other sodium-based salts and highly hygroscopic compounds, (ii) new silica-enhanced lime sorbents, on the basis of the reuse of fly ash and its reaction with $Ca(OH)_2$, (iii) activation with steam or liquid water, and (iv) recycling of partially reacted sorbents in the system [3–8].

When hydrogen chloride is generated during combustion, as in waste incineration plants and coal-fired power plants when chlorine-rich coals are used, the presence of calcium chloride is reported to have an important influence on the desulfurization efficiency [9,10].

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The thermogravimetric technique has been used for the analysis of calcium-based sorbents and products, as well as for desulfurization through the direct recording of the weight increase during the reaction [11–15]. The results obtained with the TG technique are in good agreement with other fixed bed reactor studies, indicating that the TG technique is also a satisfactory method for screening sorbents [16].

A new process called TURBOSORP® has been developed for the reduction of acid gases, SO_2 and HCl mainly, from a municipal waste incineration plant based on the application of sorbent injection at low temperatures. The process has been tested on a pilot scale by using a fraction of the flue gas stream proceeding from an incineration unit. The process consists of a circulating fast fluidized reactor (turbo-reactor), a multi-cyclon and a bagfilter [17,18]. The pilot plant treats a flue gas flow of 2900 $m^3 h^{-1}$ (STP), using as sorbent, a mixture of $Ca(OH)_2$, fly ash and partially reacted solids.

The analysis of the recycled sorbent and its behavior in the desulfurization reaction are the main objectives of this work. A laboratory fixed bed reaction system is used for the evaluation of the SO_2 sorption ability and the calcium utilization, working within a similar range of relative humidity as that which operates in the pilot plant.

The study focuses on: (i) the thermogravimetric characterization of the partially reacted sorbent collected from the pilot plant, and (ii) the behavior of the partially reacted sorbent in a fixed bed desulfurization system at laboratory scale in order to evaluate the utilization of the calcium contained in the sorbent compared with the utilization when fresh $Ca(OH)_2$ is used as sorbent. The relevance of the sorbent recycling in the increase of the calcium utilization in the desulfurization process is then evaluated.

2. Experimental

2.1. Thermoanalytical equipment

The analysis of solid samples was carried out in a Perkin-Elmer TGA-7 apparatus provided with a high temperature furnace, up to 1500 °C; a microprocessor PE 7500 and a thermal analysis controller TAC-7. The carrier gas was synthetic air, 30 ml min^{-1} flow, and the analysis started with sample amounts in the range of 7–16 mg.

Table 1
Heating rate program TG analysis of desulfurization products, fresh $Ca(OH)_2$ as sorbent

Temperature (°C)	Holding time (min)	Heating rate (°C min^{-1})
40	0	20
550	20	20
1300	80–140[a]	5
1350	20–50[a]	0

[a] Required time depending on the solid conversion (content of sulfate).

Desulfurization products based on $Ca(OH)_2$ were studied by means of TGA with the heating rates given in Table 1 [13,14]. Since the reacted sorbent analyzed in this study also contained $CaCl_2$ from the acid gases treatment, some modifications were necessary in the heating rate program. The thermoanalytical procedure and the experimental results are shown in the following section.

2.2. Chemical analysis

The chemical analysis of the sorbent was performed for calculating the content of the calcitic compounds by following the standard chemical procedures:

- $Ca(OH)_2$: neutralization titration with HCl (EN 451-1);
- $CaSO_3$: iodometric titration with $Na_2S_2O_3$;
- $CaSO_4$: oxidation with H_2O_2 and dissolution in HCl/HNO_3 acid (DIN 38414), and determination from the total sulfur (DIN 38405) by precipitation with $BaCl_2$;
- $CaCl_2$: titration with $AgNO_3$ (DIN 38405);
- $CaCO_3$: dissolution and measurement of CO_2 by ortho-phosphoric acid and solid NaOH impregnation.

The chemical analysis methods pointed out were devised especially for the analyses of mixed Ca compounds from sorbents of flue gas cleaning by the German Institute for Standardization and they fulfil the respective DIN norms.

2.3. Structural properties

A Micromeritics ASAP 2000 apparatus was used for the determination of the structural properties of the sorbent from N_2 adsorption/desorption. The sample was degassified prior to the analysis. The specific surface area, calculated from the Brunauer–Emmett–Teller (BET) method, is $16.9 \pm 0.2\,m^2\,g^{-1}$. The void space for pores between 20 and 2000 Å diameter is $0.079\,cm^3\,g^{-1}$, assuming cylindrical pores (Barret–Joyner–Halenda method). Approximately 65% of the pore volume corresponds to the contribution of the mesopores, which belong to the diameter range 20–500 Å (IUPAC classification).

2.4. Laboratory fixed bed reactor

A partially reacted sorbent obtained from the acid gas treatment process, namely TURBOSORP® process [17,18] applied to the urban waste incineration plant of Spittelau (Wien), was the reactant for the desulfurization tests in the laboratory scale fixed bed reactor. The experimental set-up is shown in Fig. 1, and the details are given elsewhere [19]. The sorbent samples located in a glass- made jacketed reactor were mixed with inert sand and exposed to a humidified flue gas stream generated by mixing commercial gases in compositions 230–1000 ppmv SO_2, 12% CO_2, 5% O_2 and N_2 balance, with a total flow of $900\,ml\,min^{-1}$.

The water vapor content in the flue gas was adjusted in the range of 20–45% of relative humidity in the reaction conditions, as it operated in the large plant. The amount of sorbent in the fixed bed was 250 mg, dispersed into inert silica sand with a ratio of 1/90 in weight terms of sorbent/silica sand.

During the desulfurization reactions, the SO_2 concentration in the reactor outlet was continuously monitored by means of a Non-dispersive Infrared Analyser. The data were recorded on-line, obtaining the corresponding breakthrough curves of SO_2 in the gas phase.

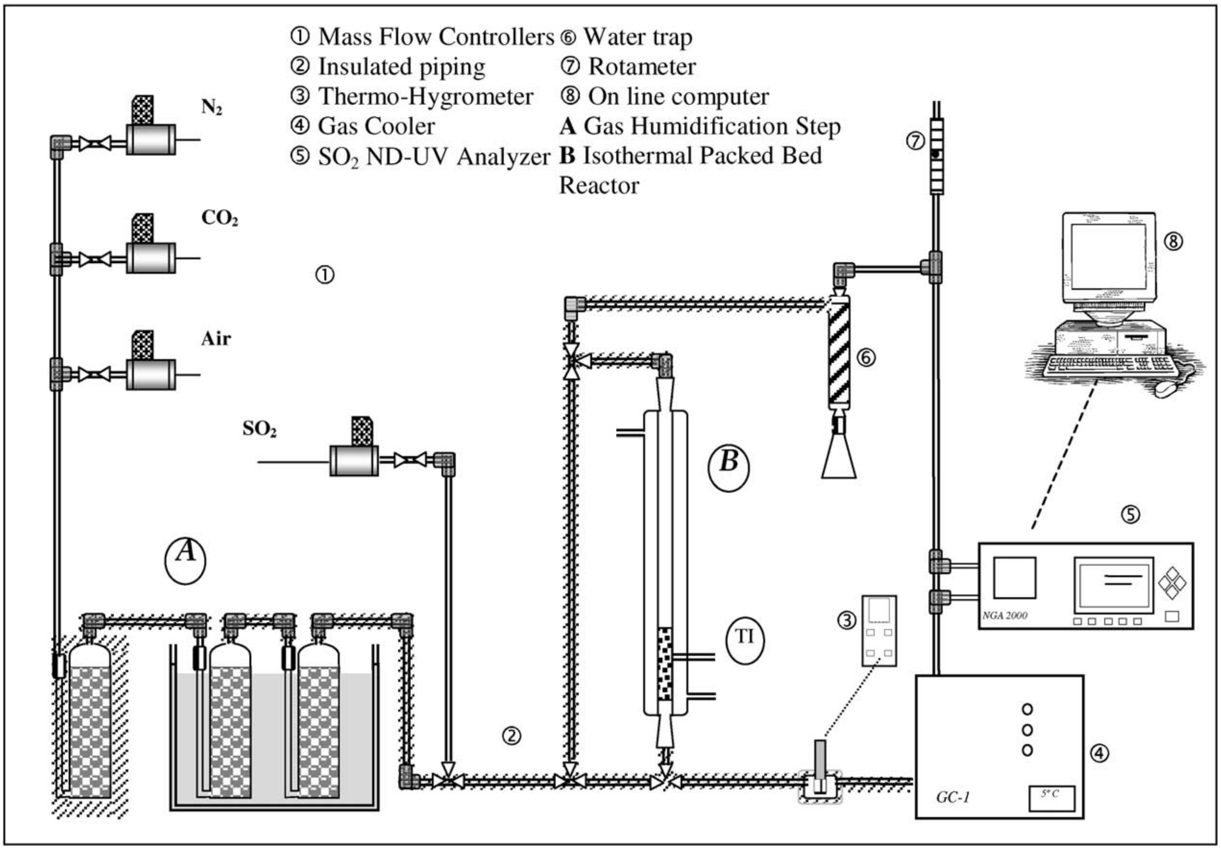

Fig. 1. Experimental set-up of the inert sand fixed bed reactor at laboratory scale.

At the end of the reactions, the solids were removed from the reactor and the silica sand was separated by sieving. The products were analyzed by the thermoanalytical procedure.

3. Results and discussion

3.1. Thermoanalytical characterization of the partially reacted sorbent

A first approach to the characterization of the partially reacted sorbent was carried out with the heating rate program shown in Table 1, which is usually applied to desulfurization products in order to determine the content of $Ca(OH)_2$, $CaCO_3$ and sulfur compounds ($CaSO_3$, $CaSO_4$) from the associated reactions to the weight changes in the temperature ranges [13,14]:

$$Ca(OH)_{2(s)} \rightarrow H_2O_{(g)} + CaO_{(s)} \qquad 350\text{–}450\,^{\circ}C$$

$$CaSO_{3(s)} + \tfrac{1}{2}O_{2(g)} \rightarrow CaSO_{4(s)} \qquad 450\text{–}550\,^{\circ}C$$

$$CaCO_{3(s)} \rightarrow CO_{2(g)} + CaO_{(s)} \qquad 600\text{–}850\,^{\circ}C$$

$$CaSO_{4(s)} \rightarrow SO_{2(g)} + \tfrac{1}{2}O_{2(g)} + CaO_{(s)} \qquad 1200\text{–}1350\,^{\circ}C$$

It should be noted that the content of $CaCl_2$ was not considered in the TGA program described in Table 1, though this program was useful for the determination of $Ca(OH)_2$, $CaCO_3$ and sulfur compounds in the desulfurization products when commercial $Ca(OH)_2$ was the sorbent and no calcium chloride was present.

Hydrogen chloride in the flue gas stream coming from the incineration plant produced the $CaCl_2$ content in the partially reacted sorbent of this study. In order to evaluate $CaCl_2$ in the thermoanalytical procedure, an additional TG analysis of calcium chloride, analysis reagent quality, was performed corresponding to the temperature range of 800–1250 °C for the total weight loss of the sample.

The thermoanalytical TG and DTG curves of a sorbent sample are shown in Fig. 2, applying the heating rate program shown in Table 1. It can be

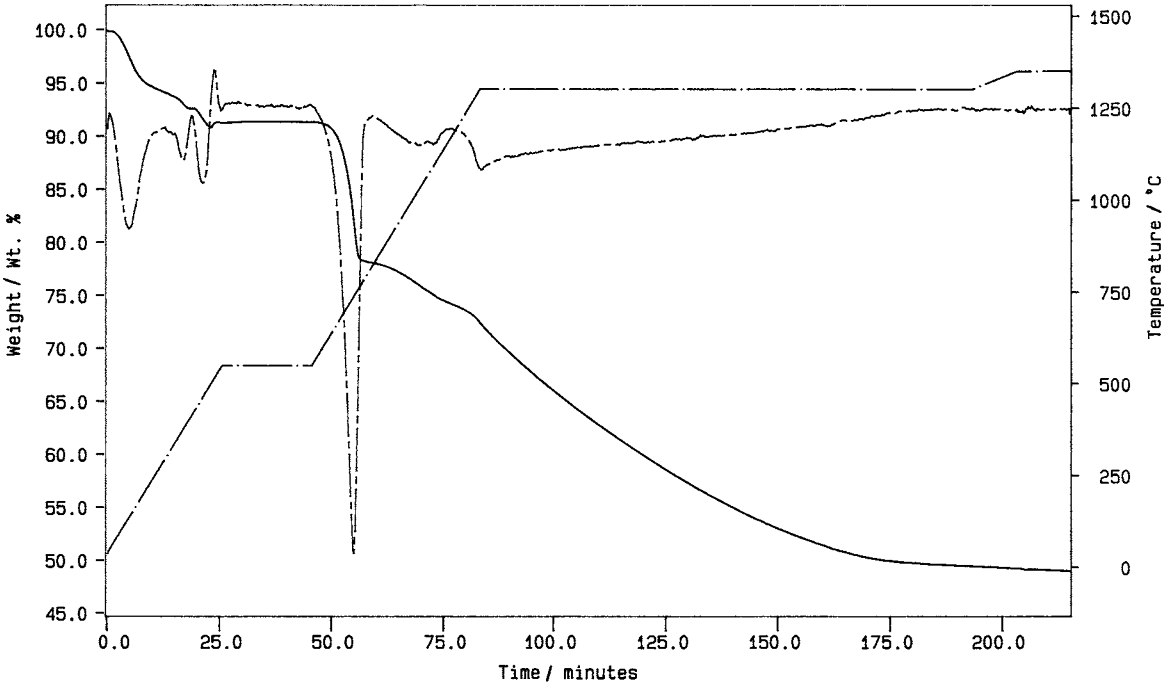

Fig. 2. Thermoanalytical TG (—) and DTG (- - -) curves of a reacted sorbent from urban waste incineration. Heating rate program (–·–) of Table 1.

observed from the baseline of the DTG curve that there is no clear difference between the decomposition of $CaCl_2$ and $CaSO_3 + CaSO_4$ in the temperature range of 1150–1250 °C.

The content of the calcitic compounds $Ca(OH)_2$, $CaSO_3$, $CaCO_3$, $CaCl_2$ and $CaSO_4$ in the partially reacted sorbent was calculated from the corresponding weight changes observed in the TG analysis. The amount of each compound, denoted by *i* in the sorbent can be calculated in terms of mole per mass of sorbent (100 g) as follows:

$$\frac{\text{mole}_i}{100\ \text{g}_{\text{solid}}} = \frac{\Delta W_i(\%)}{\nu \text{PM}_{\text{gas},i}} \tag{1}$$

ΔW_i (%) being the corresponding weight change for the compound *i*, in percentage related to the initial load of sample in the thermobalance; $\text{PM}_{\text{gas},i}$ the molecular weight of the gas component involved in the reaction corresponding to the compound *i*, and ν the stoichiometric coefficient of the gas component in this reaction. As an example, the total content of sulfur compounds in the sorbent was calculated from the weight loss corresponding to the $CaSO_4$

$$\frac{\text{mol}_{CaSO_4}}{100\ \text{g}_{\text{solid}}} = \frac{\Delta W_{CaSO_4}(\%)}{1\text{PM}_{SO_2} + (1/2)\text{PM}_{O_2}} \tag{2}$$

The results expressed in mole per 100 g of sorbent are shown in Table 2. The values obtained from chemical analysis were also included for checking.

As regards the content of $CaCl_2$ (Table 2), the estimation of calcium chloride in the solid calculated by TA was 0.036 mole per 100 g, while the evaluation from the chemical analysis was 0.073 mole per 100 g. Taking into account this difference, the heating rate program in the TA method (Table 1) was modified in order to give a longer time of analysis for this decomposition up to the temperature of sulfate. The following heating rates were added to the first program in the temperature range of 900–1300 °C:

$$550\,^{\circ}\text{C} \xrightarrow{20\,^{\circ}\text{C/min}} 900\,^{\circ}\text{C} \xrightarrow{5\,^{\circ}\text{C/min}} 1150\,^{\circ}\text{C}\ (100\ \text{min}) \xrightarrow{5\,^{\circ}\text{C/min}} 1300\,^{\circ}\text{C}$$

Table 3
TG modified heating rate program for analysis of the partially reacted sorbent

Temperature (°C)	Holding time (min)	Heating rate (°C min^{-1})
40	0	20
250	0	5
375	30	5
550	0	20
900	0	5
1150	100	5
1300	130	5
1350	0	0

The complete heating rate program is given in Table 3. Fig. 3 shows the results of the thermoanalytical TG and DTG curves of the reacted sorbent, showing that the weight loss corresponding to $CaCl_2$ in the modified program, with a longer period of time before sulfate decomposition, was clearly differentiated as is shown in the DTG baseline.

The content of the calcitic compounds calculated from the weight changes were also included in Table 2 for comparison. The contents of $CaCl_2$ and $CaSO_3 + CaSO_4$ calculated from the TGA were in good agreement with the chemical analysis: the values from TG are 0.061 mole $CaCl_2$ per 100 g, and 0.183 mole $CaSO_3 + CaSO_4$ per 100 g, the chemical analysis results being 0.073 and 0.194 mole per 100 g, respectively.

On analyzing the experimental results it was decided that a modification in the heating rate program

Table 2
TG results of the reacted sorbent with different heating rates and chemical analysis (mole per 100 g of reacted sorbent)

Compound	Basic program	Modified program	Chemical analysis
$Ca(OH)_2$	0.099 ± 0.002	0.053 ± 0.014	0.143
$CaSO_3$	0.036 ± 0.012	0.044 ± 0.003	0.125
$CaCO_3$	0.295 ± 0.005	0.320 ± 0.022	0.323
$CaCl_2$	0.036 ± 0.002	0.061 ± 0.001	0.073
$CaSO_3 + CaSO_4$	0.284 ± 0.047	0.183 ± 0.012	0.194
Total mole of Ca	0.714 ± 0.021	0.626 ± 0.010	0.733

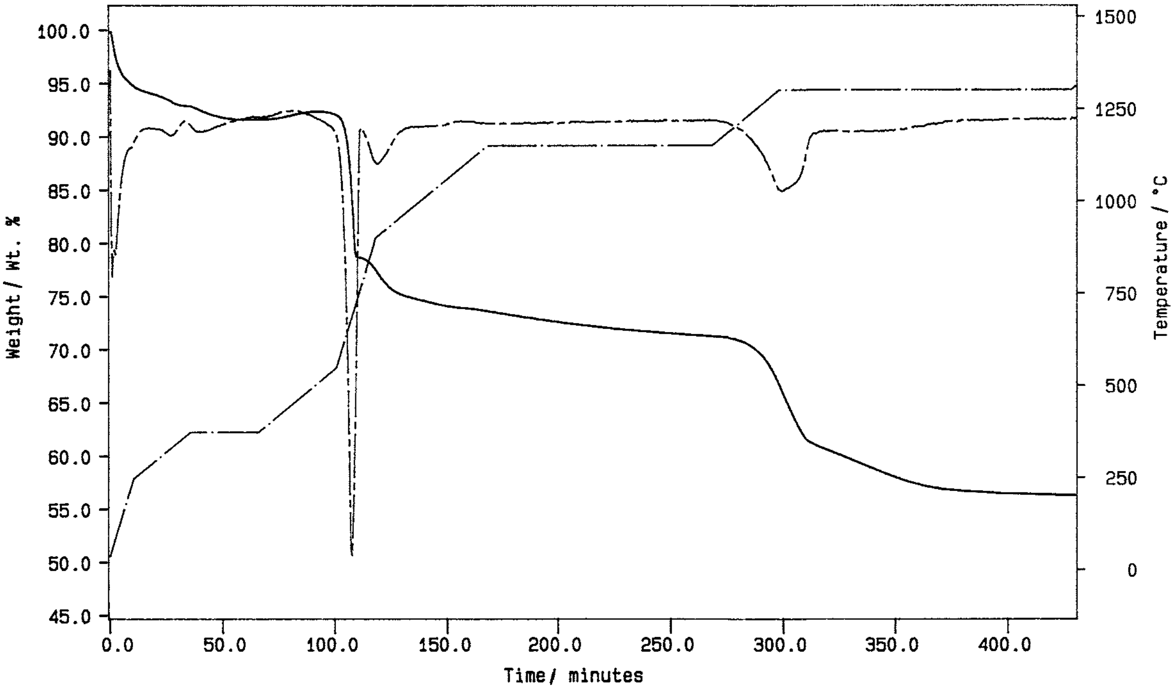

Fig. 3. Thermoanalytical TG (—) and DTG (– – –) curves of the reacted sorbent. Modified heating rate program (– · –) described in Table 3.

was required for the determination of $CaCl_2$ in the reacted sorbent containing sulfation products (Table 3).

The TA results of $CaSO_3$ content lead to significantly lower values than those corresponding to the chemical analysis (Table 2). They were calculated under the assumption of the complete oxidation to $CaSO_4$ in the thermobalance, within the temperature range of 450–550 °C. This result was considered in the modified program by using the following heating rate below 550 °C,

$$250\,^{\circ}\mathrm{C} \xrightarrow{5\,^{\circ}\mathrm{C/min}} 375\,^{\circ}\mathrm{C}\ (30\,\mathrm{min}) \xrightarrow{5\,^{\circ}\mathrm{C/min}} 550\,^{\circ}\mathrm{C}$$

but there was no significant weight increase in comparison with the results of the basic program: values of 0.036 (basic) and 0.044 (modified), while the chemical analysis led to 0.125 mole $CaSO_3$ per 100 g. The complex behavior of $CaSO_3$ could not be explained from the TA results.

It is reported in the literature that the determination of the $CaSO_3$ content in a solid sample by means of the thermoanalytical technique is not easy, due to the complex disproportionation of $CaSO_3$ observed in nitrogen atmosphere and also in air atmosphere that proceeds according to the following scheme of reactions [20]:

$$CaSO_3 + \tfrac{1}{2}O_2 \rightarrow CaSO_4$$

$$4CaSO_3 \rightarrow 3CaSO_4 + CaS \qquad \text{(no change in mass)}$$

$$CaS + 2O_2 \rightarrow CaSO_4$$

CaS cannot be decomposed in the temperature window and consequently both calcium and sulfur remain undetected. The presence of sulfur was also revealed by a yellowish color in some desulfurization products after thermal treatment [13,14].

The complex behavior of $CaSO_3$ could also affect the TGA determination of calcium hydroxide due to mutual interferences. There may be a possible overlapping zone between the weight loss associated to the $Ca(OH)_2$ in the temperature range of 350–450 °C and the weight increase corresponding to the $CaSO_3$ oxidation (450–550 °C), with the consequence of lower contents than the corresponding chemical analysis.

The total amount of calcium in the reacted sorbent, calculated from the sum of the contributions of the

Table 4
TG results of the reacted sorbents (mole per mole of calcium, 0.733 mole Ca)

Compound	Basic program	Modified program	Chemical analysis
$Ca(OH)_2$	0.135 ± 0.003	0.073 ± 0.020	0.195
$CaSO_3$	0.049 ± 0.016	0.060 ± 0.004	0.170
$CaCO_3$	0.402 ± 0.008	0.437 ± 0.030	0.440
$CaCl_2$	0.049 ± 0.003	0.084 ± 0.001	0.099
$CaSO_3 + CaSO_4$	0.387 ± 0.065	0.250 ± 0.016	0.265

calcitic compounds determined in the TGA: $Ca(OH)_2$, $CaCO_3$, $CaCl_2$ and $CaSO_3 + CaSO_4$, was affected by the selected heating rate program (Table 2). The effect may be explained by the formation of stable compounds when the analysis time was extended due to the complex $CaSO_3$ decomposition involving a reduction–oxidation mechanism, which is still unresolved.

Taking the chemical analysis for calcium content, 0.733 mole of calcium per 100 g, the amounts of the calcitic compounds from the TG results were also expressed as a molar fraction in terms of mole per mole of calcium (Table 4).

3.2. Laboratory fixed bed SO_2 sorption experiments

The desulfurization experiments in the sand bed fixed bed reactor were carried out under conditions prevailing in the large scale operation. It was studied with a synthetic flue gas stream of 230 ppmv SO_2, 12% CO_2, 5% O_2 and N_2 balance. The temperature of reaction was 80 °C, the relative humidity varying within the range of 20–45%.

The SO_2 capture of the reacted sorbent in the fixed bed was calculated from the integration of the breakthrough curves describing the SO_2 outlet concentration during the period of reaction. As examples, the breakthrough curves corresponding to the experiments at 20, 40 and 45% relative humidity were included in Fig. 4, these being the mean curves of three repeated experiments.

At the end of the reactions the spent sorbents were also analyzed by the thermogravimetric technique following the heating rate program shown in Table 5, the weight changes corresponding to $CaCl_2$ and $CaSO_3 + CaSO_4$ being clearly different. The thermo-analytical TG curves of the products from reactions at 20 and 40% of relative humidity are shown in Fig. 5,

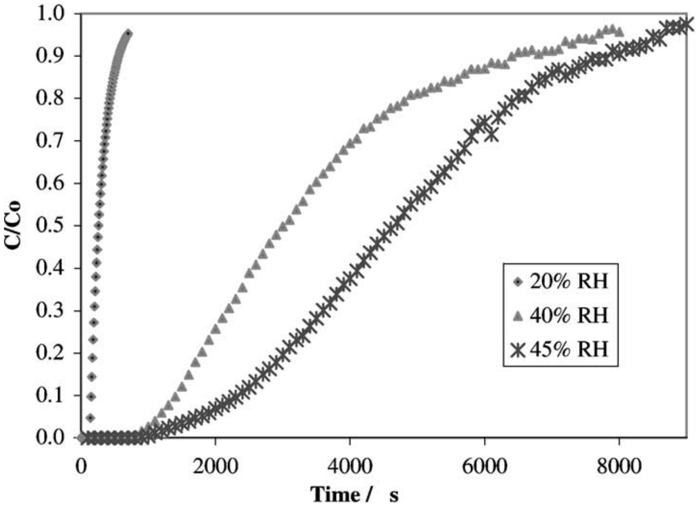

Fig. 4. Experimental breakthrough curves, reactions in the fixed bed at 20, 40 and 45% relative humidity. C/C_0: dimensionless SO_2 concentration at the reactor outlet.

Table 5
TG heating rate program for analysis of desulfurization products of the fixed bed using the partially reacted sorbent

Temperature (°C)	Holding time (min)	Heating rate (°C min^{-1})
40	0	20
550	20	20
900	20	20
1300	80–140[a]	5
1350	20–50[a]	0

[a] Required time depending on the solid conversion (content of sulfate).

which also includes the DTG curve corresponding to 20% as representative.

The results of $Ca(OH)_2$, $CaCO_3$, $CaCl_2$ and $CaSO_3 + CaSO_4$ are shown in Table 6.

The verification of the SO_2 captured in the solid phase and the SO_2 removed from the gas phase was performed after comparison of the following values (Table 7):

- the total $CaSO_3 + CaSO_4$ calculated in the TGA;
- the SO_2 removed from gas phase (breakthrough curves) plus the initial content of $CaSO_3 + CaSO_4$

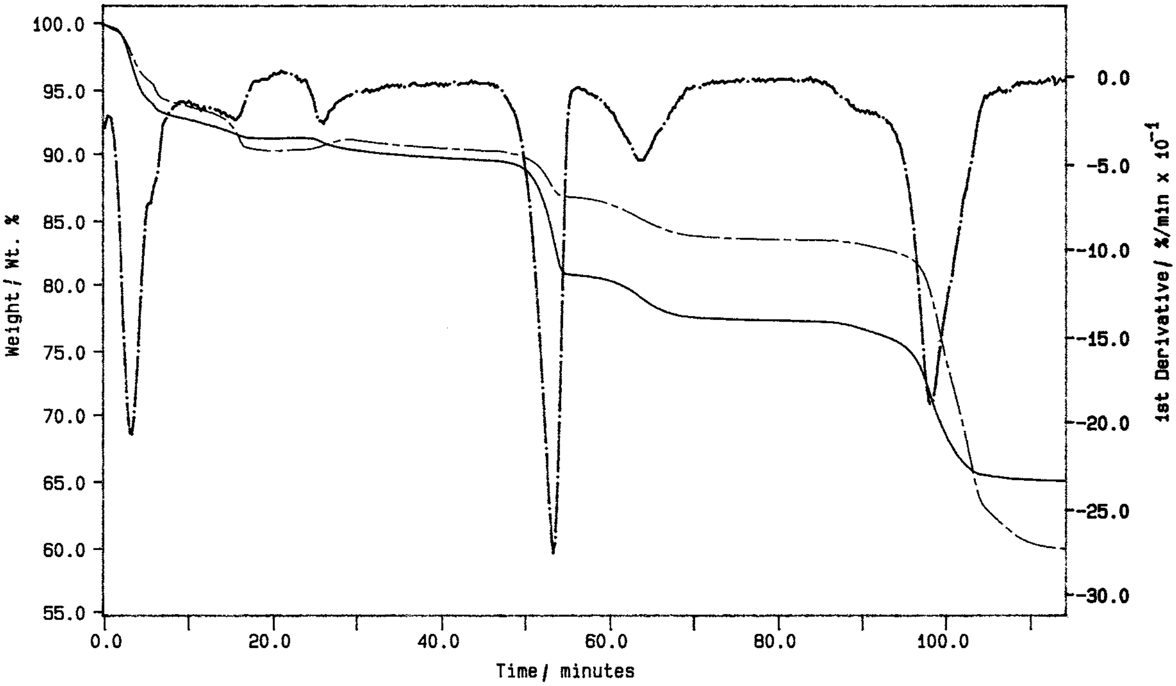

Fig. 5. Thermoanalytical TG curves of the reaction products at relative humidity 20% (—) and 40% (– – –). DTG curve (– · –) corresponding to 20%.

Table 6
TG results of the desulfurization products at different relative humidities (mole per mole of calcium)

Compound	20% RH	40% RH	45% RH
$Ca(OH)_2$	0.048 ± 0.030	0.051 ± 0.027	0.034 ± 0.051
$CaCO_3$	0.554 ± 0.027	0.272 ± 0.025	0.187 ± 0.072
$CaCl_2$	0.071 ± 0.006	0.071 ± 0.003	0.048 ± 0.008
$CaSO_3 + CaSO_4$	0.309 ± 0.022	0.607 ± 0.051	0.731 ± 0.111

Table 7
Comparison of the desulfurization results calculated from the TG analysis of products and from the breakthrough curves (mole per mole of calcium)

RH (%)	From TG analysis	From gas phase[a]
20	0.309	0.275
40	0.607	0.565
45	0.731	0.667

[a] The values are the sum of the SO_2 removal from the gas phase + the initial content in the solid.

in the reacted sorbent before reaction in the fixed bed.

An acceptable level of agreement was observed between the calculations of the desulfurization in the fixed bed by using the gas and the solid phases, which also validated the TG determination of the sulfation products.

The contents of calcitic compounds determined in the reaction products revealed: (i) the negligible SO_2 captured by the solid at 20% of relative humidity, where only the content of $CaCO_3$ increases after reaction in the fixed bed, and (ii) the consumption of $Ca(OH)_2$ and $CaCO_3$ in the reactions at 40 and 45% relative humidity, the solid utilization increasing in sulfation.

It should also be noted from the results obtained that the calcium utilization in the desulfurization reaction using a partially reacted sorbent (Table 7), up to 0.731 mole per Ca mole at 45% relative humidity, is much higher than that achieved when working with $Ca(OH)_2$ as fresh sorbent, up to 0.10 mole per Ca mole at similar relative humidity [14].

Calcium chloride, a hygroscopic salt, may work as an activation agent or additive to increase the reactivity of $CaCO_3$ in the desulfurization reaction, which was reported in previous studies as very low at these temperatures [3]. From comparison of the $CaCl_2$ contents after the fixed bed experiments (Table 6), it became evident that the amount of $CaCl_2$ reduced and that $CaSO_3/SO_4$ was formed out of it, but only at the relative humidity 45%. This phenomenon was also reported by Chisholm and Rochelle [10].

The strong dependence of the solid utilization on the relative humidity is related to the appearance of a film of adsorbed water on the surface of the sorbent. The use of deliquescent sorbent additives is expected to promote the appearance of moisture, providing higher calcium utilization in the desulfurization reaction. At a higher relative humidity the reaction product can build up differently, which results in a better accessibility of the still unreacted material.

The influence of the relative humidity in the desulfurization reaction working with $Ca(OH)_2$ and $Ca(OH)_2$/fly ash based sorbents was described in previous kinetic studies by an exponential function related to the sorbent surface heterogeneity in non-ideal adsorption modeling approaches [15,19,21]. Liu et al. [21] indicated that the thicker water layer provides a wider range for the product molecules to deposit. Thus, at lower relative humidity, the reaction product covers the sorbent surface more uniformly, whereas at higher relative humidity, the product builds up more cluster-like and covers less surface and the reaction reaches a higher solid conversion.

The proposed mechanism for the increased lime conversion at high water vapor concentration has also been proposed for other gas–solid reactions under similar conditions, e.g. in hydrogen chloride kinetics with lime at low temperatures, it was related to the formation of a saturated aqueous phase that is able to break up the crystal lattice of the lime, thus, exposing a larger fraction of the reactant for reaction [9].

From the analysis of the structural properties of the sorbent used in this study, the contribution of the mesopores to the pore volume of the solid is evident. It was measured that 65% of the pore volume corresponds to the mesopore range (20–500 Å diameter). Previous studies concluded that the region of mesopores is the most effective pore range for the desulfurization reaction from the characterization of the structural changes in the solid sorbent [22].

Taking into account that the flue gas desulfurization reaction with $Ca(OH)_2$ under practical conditions of operation is limited due to the low utilization as reported in several studies [4,6,15,19,23], a great effort has been made in order to optimize the utilization of the sorbent. From the results of this study, it can be pointed out that the reuse of the sorbent in the system may be relevant in the calcium utilization.

4. Conclusions

Thermoanalysis was used for the characterization of a partially reacted sorbent with recycling in a desulfurization process (TURBOSORP® process) applied in an urban waste incineration plant.

Some tests were carried out in order to modify the heating rate program for a better evaluation of $CaCl_2$, contained in the partially reacted sorbent due to the flue gas procedence. A temperature step at 900 °C was introduced, prior to that corresponding to the sulfate, for a better separation of $CaCl_2$ and $CaSO_4$.

The possibility of increasing the utilization in desulfurization was studied in a laboratory fixed bed reactor operating in the range of relative humidity of 20–45%, this being the humidity used in the large scale plant.

The TG determination of the sulfate content in the reaction products was validated with the SO_2 removed from the gas phase. The desulfurization results show that the SO_2 removal ability of the partially reacted sorbent was relevant in the range of relative humidity 40–45%, achieving the values of 0.61 and 0.73 mole of total sulfur per mole of calcium, respectively (with 0.26 mole per mole as the initial content before the fixed bed reaction).

The conversion level of the reused sorbent is significantly higher than that obtained when the sorbent is fresh hydrated lime in the range of 0.05–0.10 mole per mole working with the same relative humidity levels.

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