COMPARISON OF THE REACTIVITY OF DIFFERENT SODIUM COMPOUNDS AND Ca(OH)$_2$ TOWARDS SO$_2$ AND MIXTURES OF SO$_2$ AND NO$_x$

Karel Mocek, Kvetoslava Stejskalova[1], Pavel Bach, Erich Lippert, Zdenek Bastl[2], Ilona Spirovova and Emerich Erdos

The J. Heyrovsky Institute of Physical Chemistry, Academy of Sciences of the Czech Republic, 182 23 Prague 8, Czech Republic; e-mail: [1] qetastej@jh-inst.cas.cz, [2] bastl@jh-inst.cas.cz

The reactivity of solid substances towards acid gases (SO$_2$, NO$_x$) was studied with a view to finding the best conditions for an efficient removal of the gases. The solids included various sodium compounds (sodium bicarbonate, active soda, sodium carbonate monohydrate, dense soda ash) and Ca(OH)$_2$, and the reaction with sulfur dioxide was examined over a broad range of experimental variables (temperature, gas composition). The reactivity of selected samples was also investigated in reactions with gaseous mixtures containing SO$_2$ and NO$_x$ in the dependence on temperature, the SO$_2$/NO$_x$ and NO/NO$_2$ ratios and the hydrodynamic conditions of the fixed bed flow reactor. The ESCA and SEM techniques were employed to identify the solid reaction products and to study their evolution as a function of reaction parameters. It was found that with the NaHCO$_3$ precursor, a simultaneous removal of 90% SO$_2$ and nearly 50% NO$_x$ is feasible. The results indicate that NaHCO$_3$ shows promise particularly for the purification of waste gases from incinerators and power plants.
Key words: Sulfur dioxide; Nitrogen oxides; Waste gases purification; Dry processes.

The abatement of gaseous noxious pollutants formed during the combustion of various materials including wastes is an integral part of every modern combustion technology. Economically advantageous and efficient is the elimination of such pollutants by using solid substances in dry processes. The dry methods usually possess additional advantages in comparison with the wet and semi-wet ones:

1. no water is required

2. no or minimum corrosion and handling problems

3. simplicity of operation

4. capital costs are significantly lower

5. flue gas reheating is not necessary.

A sufficient reactivity of the proposed solid material and its reasonable use capacity are prerequisites for a successful application of any dry method. The present study is concerned with the reactivity of various solid substances with respect to gaseous acid pollutants over a broad range of experimental variables.

EXPERIMENTAL

Desulfurization Experiments

Kinetic measurements. The kinetics of reaction of the sodium samples including active soda[1] with gaseous SO_2 was measured in an integral flow fixed bed reactor[2] at five temperatures (130, 150, 180, 300 and 350 °C) and two SO_2 concentrations (approximately 1 600 and 400 ppm). The concentrations of oxygen (6 vol.%), carbon dioxide (11 vol.%) and water vapour (2 or 5 vol.%) were kept constant throughout all experimental runs. The particle size of the solid lay mostly within the range of 0.25–0.33 mm.

The gas mixture was prepared from two single gas streams. The first stream contained a low concentration of SO_2 in nitrogen saturated with water vapour at 18 °C which was prepared by a saturation method[3]. The second gas stream contained 33 vol.% CO_2, 18 vol.% O_2, and 49 vol.% N_2, and was also saturated with water vapour at 18 °C. The two gas streams were mixed in a ratio of approximately 2 : 1. The required concentration of SO_2 was obtained by choosing a suitable concentration of the saturation solution by respecting the mixing ratio of the two gas streams.

Analytical procedures. During the kinetic experiments, the SO_2 concentration in the feed was determined by passing a part of the gaseous mixture through a blank line identical with the reactor line without the reacting sample. In the reactor line the unreacted SO_2 was absorbed in a 1.5% H_2O_2 solution and determined conductometrically. After completing the experiment, the unreacted amount of solid Na_2CO_3 (or $Ca(OH)_2$) was determined acidimetrically. The gas mixture volume that had passed the bed during the experiment was measured eudiometrically[4]. The experiment was completed after the stoichiometric amount of SO_2 had passed through the bed. The stoichiometric excess, expressed as the SO_2/Na_2CO_3 (or $Ca(OH)_2$) molar ratio, lay mostly within the interval of 1–1.7.

Simultaneous Removal of SO_2 and NO_x

A new kinetic apparatus was designed for studying the heterogeneous reactions of selected solid compounds with the gaseous mixture containing nitrogen oxides and sulfur dioxide (Fig. 1). The reacting gaseous mixtures of the required composition were prepared by mixing two gas streams. The composition of stream I was determined by the concentration and temperature of an acidified solution of sodium sulfite and by the original content of NO in nitrogen (source I). Source I containing the $(NO + N_2)$ mixture and source II containing NO_2, O_2, CO_2 and N_2 (stream II) were prepared by mixing the components from commercially available sources (steel pressure vessels). The first and second gas streams were mixed in an approximate ratio of 3 : 1 before entering the reactor. The concentrations of SO_2 and NO_x in the feed were quantitated immediately before starting and after terminating the experiment, based on the gas absorption in a dilute solution of H_2O_2 followed by conductivity measurement. NO and nonabsorbed NO_2 were determined by using a chemiluminescent NO_x analyzer. The absorbed fraction of NO_2 (in H_2O_2) was determined with an ion selective electrode (ISE), and the conductivity contribution of HNO_3 was subtracted from the total conductivity (the sum of $H_2SO_4 + HNO_3$).

During the whole experiment the total amount of unreacted NO and of NO_2 which had not been absorbed in H_2O_2 were recorded by the NO_x analyzer connected to a line recorder. The unreacted SO_2 and NO_2 were absorbed in a H_2O_2 solution and determined as described above.

The residual alkalinity in the solid after the experiment was determined titrimetrically; the NO_2^- and NO_3^- contents were measured with ion selective electrodes.

PRESENTATION OF REACTIVITY EXPERIMENTS

Desulfurization

All kinetic experiments were performed in approximately constant hydrodynamic conditions characterized by the M/k ratio[2], which is defined as

$$M/k = P \, n_s/F = p_0 \, n_s/F_g \; , \tag{1}$$

where M is a dimensionless parameter describing the working regime of the reactor in isobaric–isothermal conditions, k (s^{-1}, kPa^{-1}) is the effective rate constant, P (kPa) is the total gas pressure, n_s (mol) is the initial amount of reacting solid in the reactor, F (mol s^{-1}) is the overall feed rate, p_0 (kPa) is the partial pressure of SO_2 at the reactor inlet, and F_g (mol s^{-1}) is the feed rate of the reacting gaseous component SO_2.

Additional dimensionless quantities suitable for the description of the results include the relative outlet SO_2 concentration, denoted as Y, and the degree of stoichiometric
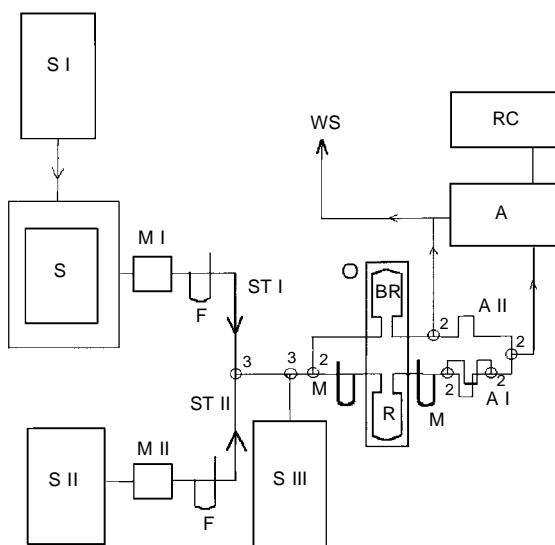

Fig. 1

Experimental set up of the apparatus for SO_2 and NO_x removal. A analyzer; A I, A II absorber; BR blank reactor; F flowmeter; M manometer; M I, M II manostat; O oven; R reactor; RC recorder; S saturators (SO_2 + H_2O); S I source (NO + N_2); S II source (NO_2 + CO_2 + O_2 + N_2); S III source (N_2); ST I, ST II stream; WS waste stream

loading of the bed W (which also has the meaning of the dimensionless time). These quantities are defined as

$$Y = y_{out}/y_{in} \tag{2}$$

and

$$W = y_{in} F t/n_s = F_g t/n_s \quad , \tag{3}$$

where y_{out} and y_{in} are the mole fractions of SO_2 at the reactor outlet and inlet, respectively, and t (s) is time.

At $W = 1$ the stoichiometric amount of SO_2 has just passed through the bed. The complementary quantity to Y is $E = (1 - Y) . 100$ expressing the percent degree of gas purification. The contact time of the reacting gas with the solid was roughly the same in all kinetic experiments (0.02–0.03 s) even though the duration of the entire experiment differed about three times (approximately 60 min for 1 600 ppm SO_2 and 180 min for 400 ppm). The gas flow rate through the bed was 90–100 ml min^{-1} for higher SO_2 concentrations and 35–40 ml min^{-1} for lower SO_2 concentrations.

Relatively smooth Y vs W kinetic curves were obtained and employed to calculate the conversion degree of the solid substance at a unit stoichiometric loading ($W = 1$). The final degree of conversion of the solid (usually at $W > 1$) gives information on the limiting utilization degree of the solid substance.

Simultaneous Removal of SO_2 and NO_x

The kinetic experiments were evaluated as in the desulfurization experiments by introducing the following quantities: Y_{SO_2}, Y_{NO_x}, Y_{NO}, Y_{NO_2}, Y_t and F_{gt}. For NO and NO_2 the stoichiometry of the reactions was taken into account: thus the reciprocal value of W indicates directly the stoichiometric excess.

The primary experimental data were transformed into breakthrough curves describing the dependences of the relative outlet concentrations of SO_2 and NO_x, Y_{SO_2}, Y_{NO}, Y_{NO_2}, Y_{NO_x}, Y_t on the total stoichiometric loading of the bed W. For a potential practical application, the Y versus W dependences were transformed into dependences of the gas purification degree E (with respect to the individual component) on the stoichiometric ratio of the reacting solid (normalized stoichiometric ratio, NSR). The average gas purification degree with respect to a given component (SO_2, NO_x) was evaluated for values of NSR = 1.0 and 1.5, which can be considered as limiting with respect to a practical application of sodium-based sorbents.

RESULTS

Desulfurization

Typical breakthrough curves of SO_2 are shown in Figs 2 and 3, representing the temperature dependence of reactivity for the $NaHCO_3$ sample and the dependence on the solid nature, respectively. The results of reactivity experiments within the temperature range of 130–350 °C at a water vapour content of 2 vol.% are summarized in Table I. The influence of the water vapour content in the gas phase can be inferred from Table II, where the degrees of conversion of selected solid samples in dependence on temperature are compared for two water vapour levels. The following conclusions can be drawn from the desulfurization experiments:

1. $NaHCO_3$ exhibits the absolutely highest reactivity with respect to SO_2 within the temperature region of 130–180 °C (Table I, Fig. 3). The reactivity of active soda within the temperature region of 300–350 °C is comparable to that of $NaHCO_3$ and is even higher at lower SO_2 concentrations (Table I). Sodium carbonate monohydrate and dense soda ash displayed the lowest reactivity in the same conditions.

2. The $Ca(OH)_2$ sample is almost unreactive in all circumstances. Only at lower SO_2 concentrations at temperatures of 300–350 °C the degree of conversion at the unit stoichiometric ratio is higher than 10% (Table I).

3. Higher water vapour concentrations in the gas phase have a highly stimulating effect on the reactivity of $NaHCO_3$ and active soda at 150 °C, whereas a very distinct
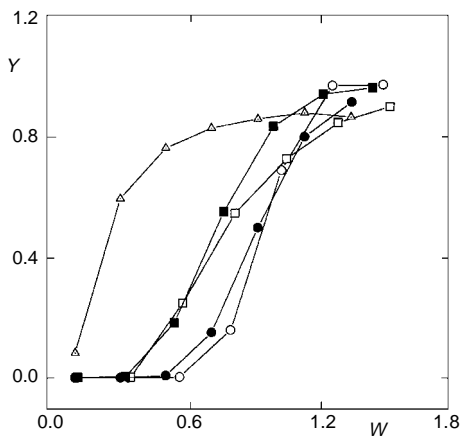
FIG. 2

Temperature dependence of reactivity of the $NaHCO_3$ sample with respect to SO_2; c = 1 370 ppm, v = 2%. Temperature (°C): ○ 130, □ 150, △ 180, ● 300, ■ 350
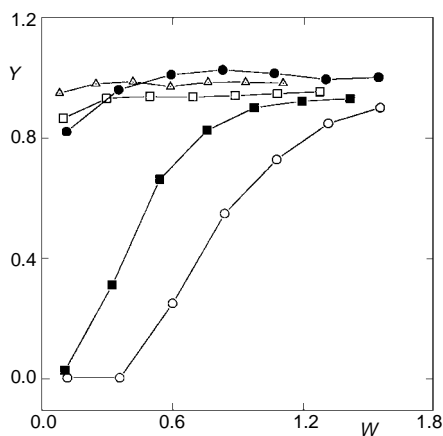
FIG. 3

Comparison of the reactivity of different samples with respect to SO_2 at 150 °C; c = 1 400 ppm, v = 2%. Samples: ○ $NaHCO_3$, □ monohydrate of sodium carbonate, △ dense soda ash, ● $Ca(OH)_2$, ■ active soda

adverse effect of the higher water vapour content is observed at 300 °C (Table II). Work on an explanation of this effect is in progress.

4. The morphological microstructure of the received solid samples was examined by scanning electron microscopy (SEM), which revealed a significant porous structure for active soda and $Ca(OH)_2$; the $NaHCO_3$ sample exhibited a significant porous structure after thermal treatment at 130 °C. A similar microstructure was also found for the $NaHCO_3$ sample after partial conversion with SO_2 at 130 °C ($x = 0.255$).

5. The surface composition of selected samples was determined by electron spectroscopy for chemical analysis (ESCA) (i) in the state as obtained; (ii) after activation by heating; (iii) after adsorption of SO_2; and (iv) after reaction with gas mixtures containing SO_2. In the case of reaction with gas mixtures containing SO_2, H_2O and N_2 only, sulfite was the main reaction product. In the presence of oxygen, the conversion of tetravalent to hexavalent sulfur occurs through incorporation of oxygen supplied from

TABLE I
Degree of conversion of the solids x at a unit stoichiometric loading ($W = 1$) in dependence on temperature and SO_2 concentration ($c_1 = 1\ 400$ ppm, $c_2 = 350$ ppm) at a water vapour content of 2 vol.%

Sample	130 °C		150 °C		180 °C		300 °C		350 °C	
	c_1	c_2	c_1	c_2	c_1	c_2	c_1	c_2	c_1	c_2
$NaHCO_3$	0.91	0.91	0.77	0.73	0.38	0.70	0.89	0.73	0.75	0.74
Active soda	0.64	0.29	0.50	0.33	0.32	0.52	0.86	0.92	0.73	0.85
$Na_2CO_3 . H_2O$	0.06	0.04	0.03	0.08	0.02	0.06	0.07	0.08	0.07	0.11
$Ca(OH)_2$	0.02	0.02	0.01	0.02	0.02	0.03	0.05	0.12	0.07	0.15

TABLE II
Degrees of conversion of selected solids at two different water vapour contents in treated gas ($v_1 = 2$ vol.%, $v_2 = 5$ vol.%) as a function of temperature; $W = 1$, $c_1 = 1\ 400$ ppm

Sample	150 °C		300 °C		350 °C	
	v_1	v_2	v_1	v_2	v_1	v_2
$NaHCO_3$	0.77	0.92	0.89	0.69	0.75	0.71
Active soda	0.50	0.93	0.86	0.44	0.73	0.61
$Ca(OH)_2$	0.01	0.01	0.05	0.05	0.07	0.07

the gas phase. The ESCA measurements with sodium carbonate before and after activation (by heating in the reactor or in a vacuum in the ESCA device or by sputtering with Ar^+ ions) revealed a sodium overstoichiometry in the superficial layers. This overstoichiometry, which decreases with time and on exposure to ambient air, may be due to the decomposition of some of the carbonate groups at the sample surface during the activation process (e.g. by heating). It should be noted that the information depth of the ESCA technique used is about 5 nm.

Simultaneous Removal of SO_2 and NO_x

Based on the results obtained from the isolated desulfurization and denoxification experiments, the $NaHCO_3$ sample was selected for a detailed experimental study of the simultaneous reactions with the $SO_2 + NO_x$ mixture. A systematic reactivity study was performed at SO_2/NO_x ratios within the range of 0.5–2.0; a few experiments were also conducted at SO_2/NO_x ratios higher than 4, within the temperature range of 130–180 °C, at two different NO/NO_2 ratios (NO/NO_2 < 5 and NO/NO_2 = 7–10), in two different hydrodynamic regimes of the reactor M/k, which is proportional to the contact time of the gas with the solid, and different partial pressures of SO_2 and nitrogen oxides. Most of the experiments were performed with 5 vol.% water vapour.

Typical breakthrough curves of SO_2 and NO_x are shown in Figs 4, 5 and 6. The dependences of the gas purification degree $E_{SO_2}(E_{NO_x})$ on temperature and the hydrodynamic conditions of the reactor for two values of NSR = 1.0 (or 1.5) are shown in Figs 7
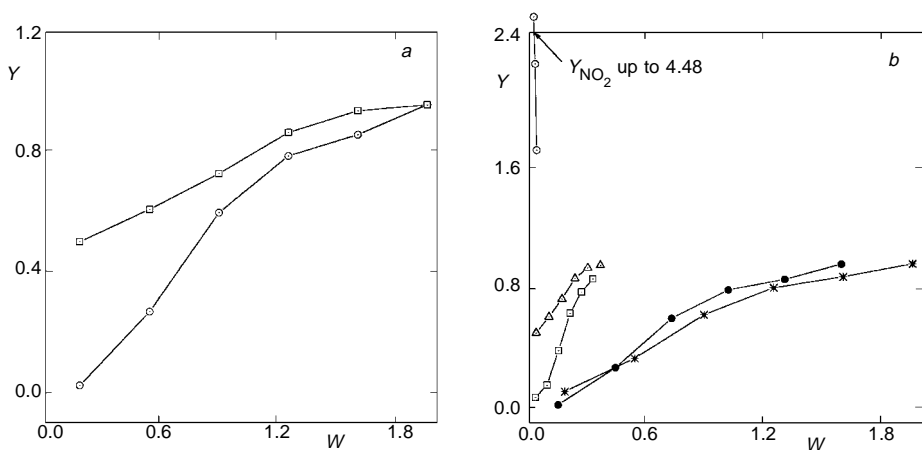

FIG. 4

SO_2 and NO_x breakthrough curves for the $NaHCO_3$ sample at 150 °C; SO_2/NO_x = 2.2, NO/NO_2 = 8.5, v = 5.2%, τ_g = 0.03 s. *a* W is calculated with respect to the loading by the sum of all components: ◯ SO_2, ☐ NO_x. *b* W is calculated with respect to the loading by individual components or by their sums (NO_x, total): ◯ NO_2, ☐ NO, △ NO_x, ● SO_2, ✳ total

and 8. The results of the combined SO_2/NO_x reactivity experiments with the $NaHCO_3$ sample over the temperature range of 130–180 °C are summarized in the Tables III and IV. The following conclusions can be drawn from the reactivity experiments.

SO_2 Removal

1. The reactivity of the $NaHCO_3$ sample with respect to SO_2 in terms of E_{SO_2} does not depend on the NO/NO_2 ratio at 130–180 °C but it depends appreciably on the SO_2/NO_x ratio.

2. At the constant ratio of $SO_2/NO_x = 2$, a change in the partial pressures of SO_2 and NO_x by a factor of 2 has no significant effect on the degree of SO_2 removal, E_{SO_2}.

3. Higher degrees of SO_2 removal can be achieved by extending the time τ_g of contact of the gas with the solid; the degree of NO_x removal, E_{NO_x}, remains virtually constant.

4. Increase in the water vapour content from 2 to 5 vol.% has no significant effect on E_{SO_2}.

5. Increase in the NSR value from 1.0 to 1.5 brings the increase in the average SO_2 removal E_{SO_2} by about 15%.

NO_x Removal

1. The degree of NO_x removal at 130–180 °C depends significantly on the SO_2/NO_x ratio and to a lesser extent on the NO/NO_2 ratio; the degree of NO_x removal, E_{NO_x}, increases with increasing SO_2/NO_x ratio, while the NO/NO_2 ratio has a minor effect only.
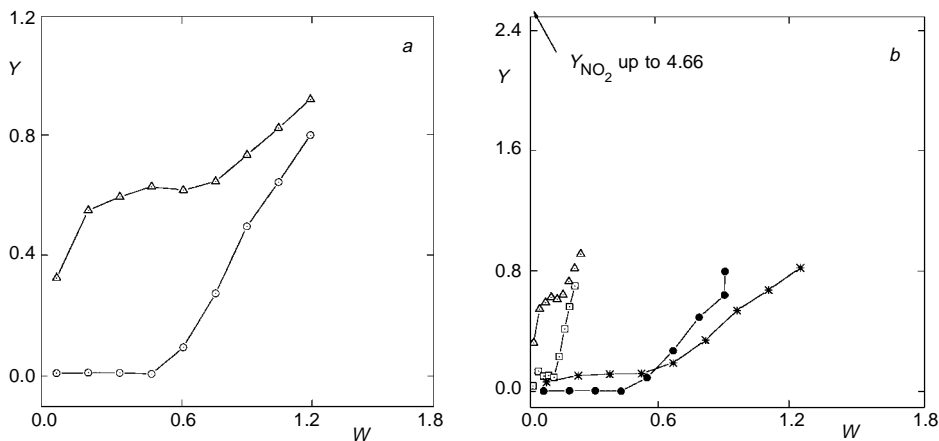

Fig. 5

SO_2 and NO_x breakthrough curves for the $NaHCO_3$ sample at 151 °C; $SO_2/NO_x = 2.1$, $NO/NO_2 = 7.8$, $v = 5.2\%$, $\tau_g = 0.07$ s. *a* W is calculated with respect to the loading by the sum of all components: ○ SO_2, △ NO_x. *b* W is calculated with respect to the loading by individual components or by their sums (NO_x, total): □ NO, △ NO_x, ● SO_2, * total

2. Increase in the water vapour content from 2 to 5 vol.% brings about a roughly 9% increase in the average NO_x removal.

3. Increase in the NSR value from 1.0 to 1.5 increases the average NO_x removal, E_{NO_x}, by 6–11% in dependence on temperature and the SO_2/NO_x ratio.

DISCUSSION

Let us recall some other experimental findings which are worth mentioning. The total degree of conversion, x, of the $NaHCO_3$ sample (or more accurately, Na_2CO_3 formed by

TABLE III
Average degrees of gas removal E_{SO_2} and E_{NO_x} in dependence on temperature and on the NSR value (1.0 and/or 1.5) for $NaHCO_3$

Ratios	130 °C		150 °C		180 °C	
	1	1.5	1	1.5	1	1.5
E_{SO_2}						
$SO_2/NO_x = 0.5$						
$NO/NO_2 < 5$	0.79	0.91	0.81	0.96	0.84	0.95
< 10	0.80	0.93	0.86	0.95	0.67	0.77
$SO_2/NO_x = 1.0$						
$NO/NO_2 < 5$	0.52	0.61	0.66	0.81	0.77	0.88
< 10	0.56	0.73	0.66	0.82	0.77	0.89
$SO_2/NO_x = 2.0$						
$NO/NO_2 < 5$	0.58	0.74	0.56	0.72	0.72	0.84
< 10	0.52	0.63	0.73	0.85	0.77	0.92
E_{NO_x}						
$SO_2/NO_x = 0.5$						
$NO/NO_2 < 5$	0.16	0.24	0.14	0.20	0.12	0.18
< 10	0.11	0.18	0.12	0.16	0.14	0.19
$SO_2/NO_x = 1.0$						
$NO/NO_2 < 5$	0.19	0.26	0.08	0.14	0.07	0.13
< 10	0.16	0.23	0.15	0.23	0.22	0.28
$SO_2/NO_x = 2.0$						
$NO/NO_2 < 5$	0.33	0.43	0.30	0.37	0.33	0.39
< 10	0.35	0.46	0.44	0.52	0.23	0.32

its decomposition) during the simultaneous SO_2/NO_x experiments varied over a wide range in dependence on the experimental conditions. The highest values, typically $x = 0.75$–0.80 with a maximum of $x = 0.85$, were achieved at 150 °C. In the reaction with SO_2 alone in roughly identical conditions, the conversion degree of the solid was as high as $x = 0.97$. The observed decrease in the maximum total conversion (by about 20%) due to the presence of NO_x can be plausibly explained by the difference in the volumes of the reacting and produced solid substances. This difference can be crudely calculated from the known molar volumes of the solids involved and from the stoichiometry of the reactions. The molar volume increased about 14% during the formation of Na_2SO_3 from SO_2 and Na_2CO_3 and about 50–80% during the reaction of Na_2CO_3 with NO_x (NO, NO_2) leading to $NaNO_2$ and/or $NaNO_3$, in dependence on the nitrite-to-nitrate ratio. A high increase in volume can lead to a blocking and/or hindrance of the access of the gas to the reaction interface, thus decreasing its effective surface area. As a result, a lower useful capacity of the solid and, consequently, a lower degree of conversion is achieved.

During the initial stage of the simultaneous SO_2/NO_x experiments an increase in NO_2 concentration occurs, increasing the Y_{NO_2} value to above 1. This NO_2 formation takes place at the expense of NO (Figs 4, 5 and 6). This formation of NO_2 by oxidation of NO is a result of the combined effect of the presence of SO_2 as an oxygen transferring agent and of the simultaneous influence of the surface of the active form of Na_2CO_3. In the reaction of the nitrogen oxides with the $NaHCO_3$ sample in the absence of SO_2 in otherwise identical conditions, NO_2 formation did not take place.
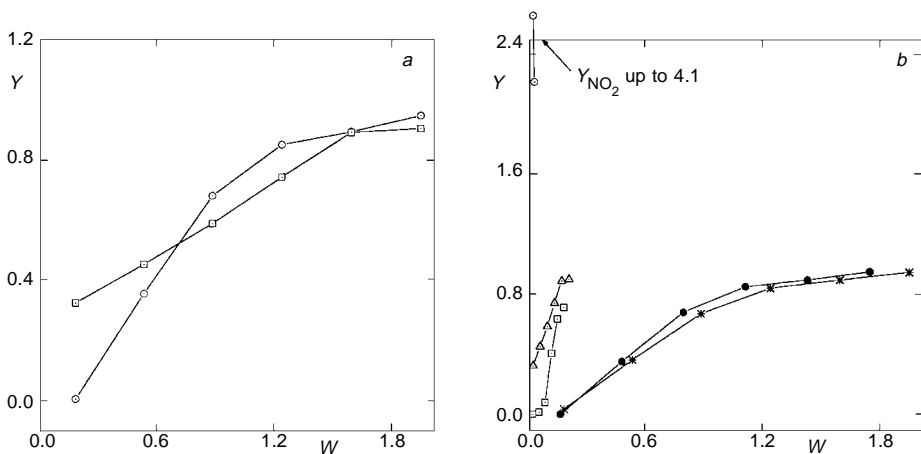

Fig. 6

SO_2 and NO_x breakthrough curves for the $NaHCO_3$ sample at 152 °C; $SO_2/NO_x = 4.3$, $NO/NO_2 = 7.0$, $v = 5.2\%$, $\tau_g = 0.06$ s. *a* W is calculated with respect to the loading by the sum of all components: ◯ SO_2, ▭ NO_x. *b* W is calculated with respect to the loading by individual components or by their sums (NO_x, total): ◯ NO_2, ▭ NO, △ NO_x, ● SO_2, * total

Comparison of the desulfuration and denoxification reaction rates (in terms of E_{SO_2} and E_{NO_x}) for the simultaneous ($SO_2 + NO_x$) and isolated (SO_2, NO_x) cases showed that, at 150 °C and in otherwise identical conditions, the following changes in reaction rate take place:

TABLE IV
Effect of temperature, NSR value (1 and/or 1.5) and the residence time of gas in bed τ_g on the average degree of gas removal E_{SO_2} and E_{NO_x} for NaHCO$_3$; SO$_2$/NO$_x$ = 2, NO/NO$_2$ < 10

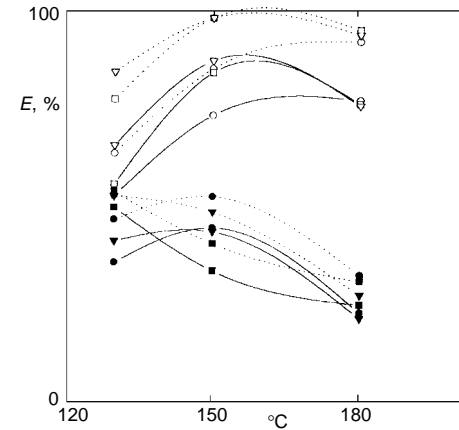| τ_g	130 °C		150 °C		180 °C	
	1	1.5	1	1.5	1	1.5
			E_{SO_2}			
0.03	0.52	0.63	0.73	0.85	0.77	0.92
0.07	0.65	0.84	0.87	0.98	0.76	0.94
			E_{NO_x}			
0.03	0.35	0.46	0.44	0.52	0.23	0.32
0.07	0.41	0.52	0.43	0.48	0.21	0.27

Fig. 7
Dependence of the average degree of gas removal E_{SO_2} and E_{NO_x} on temperature, the NSR value and residence time of gas in bed for the NaHCO$_3$ sample at SO$_2$/NO$_x$ = 2 and NO/NO$_2$ < 10. NSR = 1 (full lines), NSR = 1.5 (dotted lines), E_{SO_2} (empty points), E_{NO_x} (full points), ○ ● τ_g = 0.03 s, ▽ ▼ τ_g = 0.07 s, □ ■ τ_g = 0.07 s (at half values of SO$_2$ and NO$_x$ concentrations)
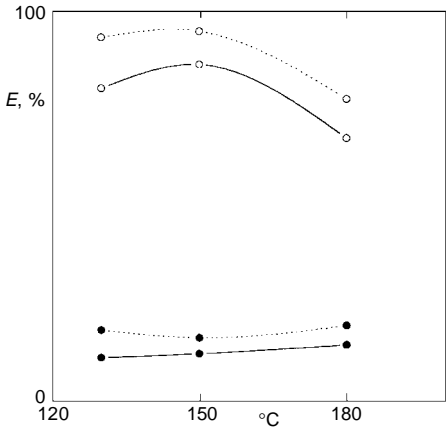

Fig. 8
Dependence of the average degree of gas removal E_{SO_2} and E_{NO_x} on temperature and the NSR value for the NaHCO$_3$ sample at SO$_2$/NO$_x$ = 0.5 and NO/NO$_2$ < 10. NSR = 1 (full lines), NSR = 1.5 (dotted lines), ○ E_{SO_2}, ● E_{NO_x}

1. decrease in the desulfurization reaction rate with the $SO_2 + NO_x$ mixture on average by 35% in comparison with the isolated desulfurization reaction

2. increase in the rate of NO_x removal from the $SO_2 + NO_x$ mixture by a factor of *three* in comparison with the isolated denoxification reaction.

Sulfur dioxide in the mixture with nitrogen oxides acts as a promoter of the NO_x removal during the reaction with the activated $NaHCO_3$ sample. The course of the NO_x removal from the mixture $SO_2 + NO_x$ is also influenced by the catalytic effect of water vapour.

Experiments applying an SO_2/NO_x ratio higher than 4 confirmed that the NO_x removal is significantly affected by this ratio. The NO_x removal was about 60% ($E_{NO_x} = 0.60$) and more in dependence on the NSR value whereas the SO_2 removal decreased slightly (see Fig. 6). This feature is characteristic of competitive reactions.

Sulfate and nitrate were found to be the main reaction products in the solid phase after the reaction of $NaHCO_3$ with the $SO_2 + NO_x$ gaseous mixture. On the basis of ESCA measurements and in agreement with the complete analysis of the solid and gaseous phases during and after the reaction, the formation of an additional chemical species of nitrogen in the solid phase besides nitrate and nitrite is conceivable. Research in this direction is in progress.

CONCLUSIONS

It has been shown that activated sodium hydrogencarbonate, which is one of the most efficient materials for SO_2 removal in gas–solid systems, is also very efficient for simultaneous SO_2 and NO_x removal. A simultaneous removal of 90% SO_2 on average and 50% NO_x at least is feasible. The results obtained are very promising with respect to the utilization of $NaHCO_3$ particularly for the purification of waste gases from incinerators.

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