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Precipitation of Sulfur Dioxide from Gaseous Feed by Oxidative Chemisorption

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ABSTRACT

Several sorption processes based on precipitation of sulfur dioxide by absorption with chemical reaction have been investigated. The target of this study was the evaluation of process boundaries and boundaries of application for the following processes:

- Absorption of sulfur dioxide with caustic soda.
- Catalytically supported oxidation of sulfur dioxide in the liquid phase.
- Absorption of sulfur dioxide with dichromate.
- Absorption of sulfur dioxide with sulfate acidic hydrogen peroxide.

The processes were investigated in a lab-scale falling-film absorption column. Mass transfer area of the film was 0.22 m^2. In absorption of sulfur

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dioxide with aqueous solution of dichromate below pH values of 2.5, the rate of reaction of dichromate with sulfur dioxide is very fast. As a consequence, absorption of sulfur dioxide is mainly controlled by mass transfer in the gaseous phase. Absorption of sulfur dioxide with caustic additives from an oxygen-containing gaseous phase is followed by oxidation in the liquid phase. Oxidation is accelerated by heavy metal catalysis. The role of iron(II) and manganese(II) in oxidation of sulfur dioxide has been investigated. Parallel to the oxidation of sulfur dioxide, the catalyst iron(II) is subject of irreversible deactivation by oxidation. Manganese(II) accelerates oxidation. Hydrogen peroxide, sometimes suggested for application in technical scale absorption of sulfur dioxide, causes instantaneous formation of sulfuric acid in the aqueous bulk phase. As long as the concentration of sulfuric acid can be kept below 20% by weight, absorption of sulfur dioxide is again controlled by the gas phase mass transfer resistance.

Key Words: Absorption; Sulfur dioxide; Oxidation; Chemisorption.

INTRODUCTION

Wet flue gas desulfurization (FGD) processes are based on the absorption of sulfur dioxide in the absorbent phase and subsequent neutralization to keep the concentration of protonated sulfur dioxide low and thus enhance the mass transfer of sulfur dioxide.

In the aqueous phase several species of sulfur dioxide, which we will refer to as S(IV), are formed according to the dissociation properties of S(IV)-oxides in aqueous solution: hydrated sulfur dioxide, bisulfite, and sulfite. The distribution among the protonated and nonprotonated species is determined by the pH value.[1]

$$SO_2 \cdot H_2O \xleftrightarrow{K_1} H^+ + HSO_3^- \quad K_1 = 1.7 \times 10^{-2} \tag{1}$$

$$HSO_3^- \xleftrightarrow{K_2} H^+ + SO_3^{2-} \quad K_2 = 6.0 \times 10^{-8} \tag{2}$$

To obtain high absorption efficiency, the concentration of protonated S(IV), $SO_2 \cdot H_2O$, has to be kept low. This can be achieved by:

- Neutralization of the dissolved sulfur dioxide by a caustic additive, commonly lime or limestone,[2] which results in a shift of the S(IV)-equilibrium to predominant nonprotonated species (bisulfite, sulfite), which are then oxidized with oxygen when it is also absorbed from the gaseous phase in parallel with SO_2, to form sulfate. This oxidation reaction is known to be supported catalytically by transition metal ions.[1,3–6]

$$\underset{\substack{\mathrm{HSO_3^-} \\ \mathrm{SO_3^{2-}}}}{\mathrm{S(IV)}} + \frac{1}{2}\mathrm{O_2} \xrightarrow{\text{Catalyst}} \underset{\mathrm{SO_4^{2-}+H^+}}{\mathrm{S(VI)}} \tag{3}$$

- Oxidation of the dissolved sulfur dioxide with an additive, i.e., dichromate or hydrogen peroxide, both in acidic media. The first process combines both off-gas desulfurization and wastewater detoxication.[7,8] Sulfur dioxide is absorbed from gaseous phase with an acidic solution of Cr(VI) and oxidized to sulfate, while Cr(VI) is reduced to Cr(III), which can be precipitated as chromium(III)-hydroxide in a consecutive step.

  $$\mathrm{Cr_2O_7^{2-} + 3SO_2 \cdot H_2O + 2H^+ \longleftrightarrow 2Cr^{3+} + 3SO_4^{2-} + 4H_2O} \tag{4}$$

  The second process should yield a marketable product of high purity.[9–11] Sulfur dioxide is absorbed from the gaseous phase with sulfate acidic hydrogen peroxide, forming sulfuric acid.

  $$\mathrm{SO_2 \cdot H_2O + H_2O_2 \longleftrightarrow H_2SO_4 + H_2O} \tag{5}$$

EXPERIMENTAL PROCEDURE

The experiments were performed in a falling-film absorption column. The column and its calibration has been described in detail elsewhere.[12] Falling-film reactors have proven applicable in testing gas–liquid mass transfer (Roberts and Danckwerts[13] and Sherwood et al.[14]).

The falling-film absorber used for investigation is designed as a concentric tube apparatus with a mass transfer area of 0.22 $\mathrm{m^2}$. The equipment was calibrated with the test system $\mathrm{SO_2}$/air/aqueous sodium hydroxide, as suggested by Danckwerts.[15] The results of the calibration tests confirmed the applicability of Eq. (6) to describe the gas phase mass transfer resistance.[14,16]

$$Sh_{\mathrm{G}} = 0.023 Re_{\mathrm{G}}^{0.83} \cdot Sc_{\mathrm{G}}^{0.44} \tag{6}$$

The liquid phase mass transfer resistance was derived from dimensional analysis according to Eq. (7).[17]

$$Sh_{\mathrm{L}} = 0.126 Re_{\mathrm{L}}^{0.55} \cdot Sc_{\mathrm{L}}^{0.4} \tag{7}$$

The experimental procedure, process conditions, and analytical aspects of the processes under investigation are described in detail elsewhere.[7,18,19]

RESULTS AND DISCUSSION

Absorption of Sulfur Dioxide with Sulfate Acidic Solution of Chromate

Opposite to the absorption of sulfur dioxide in caustic soda, the absorption of sulfur dioxide in acidic solution of chromate is controlled by the rate of the redox reaction of sulfur dioxide and chromate in the liquid film. The results of the mass transfer experiments point out that absorption of dilute sulfur dioxide from a carrier gas is enhanced by pseudo first-order reaction when the concentration of dichromate is kept above a level of $c_{\mathrm{Cr(VI)}} > 0.8\,\mathrm{g/L}$.[7] For a lower concentration of dichromate, the absorption of sulfur dioxide is enhanced by fast second-order reaction.

Catalytically Supported Oxidation of Sulfur Dioxide in the Liquid Phase

The enhancement of the oxygen mass transfer by iron in oxidation state II, which is the thermodynamically preferred iron species in aqueous solution in the presence of S(IV), was investigated by varying the Fe(II) content (1.2×10^{-5}–$4.36 \times 10^{-2}\,\mathrm{mol/L}$) and the pH value ($4.5$–$6 \pm 0.1$ at the bottom of the column). The results are shown in Fig. 1.

Process control of the mass transfer experiments was based on fixed pH value at the bottom of the column. Elevated oxygen consumption required adjustment of the feed pH to a higher value due to proton formation during oxidation of HSO_3^-.

The results shown in Fig. 1 indicate an increasing rate of S(IV) oxidation at fixed pH value with an increasing amount of Fe(II) in the absorption liquor. The effect of Fe(II) on oxidation of S(IV) passes a maximum, which is pH-dependent.

The increase in rate of oxidation with increasing pH value can be explained with sulfite being the predominating S(IV) species at elevated pH value (pH > 5), the oxidation of which is thermodynamically favored over the oxidation of bisulfite. The increase in rate of oxidation with increasing Fe(II) concentration is a result of catalyst content and the pH value.

Providing Fe(II) in excess would not increase the rate of oxidation further but would yield a constant maximum rate of oxidation. Thus, for Fe(II) content below the maximum concentration, oxidation of S(IV) is limited by the mass transfer of oxygen, above a pH-dependent maximum concentration of Fe(II), and oxidation of S(IV) would be limited by reaction control of the

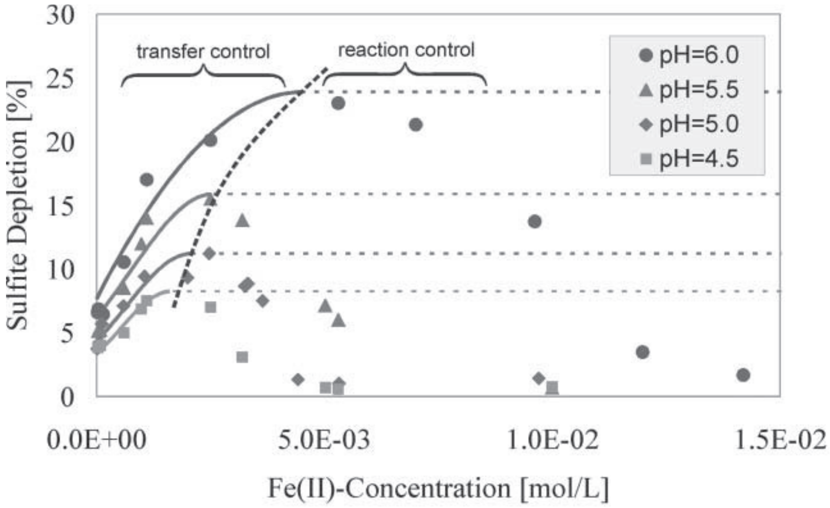

Figure 1. S(IV) depletion as a function of the pH value and Fe(II) content; $T = 20°\mathrm{C}$; pH value of the outlet stream, as indicated.

oxidation reaction. Actually, using Fe(II) in excess will even cause a rapid breakdown of S(IV) oxidation, as shown in Fig. 1.

The breakdown of S(IV) oxidation is reported to be caused by formation of (colloidal) Fe(III) hydroxide precipitate.[6] The higher the rate of oxygen mass transfer the more S(IV) is converted in the liquid film, until finally oxidation of the catalyst Fe(II) and therefore deactivation of the catalyst itself by oxidation and precipitation takes place.[4,6,20] Due to the experimentally observed fact that the maximum content of Fe(II) can be increased with increasing pH value, the rate of oxidation of S(IV) with oxygen must increase when shifting the predominating species of S(IV) from HSO_3^- to SO_3^{2-}.

Investigation of the role of the catalyst mixture Mn(II) plus Fe(II) qualitatively shows a similar trend. The results are shown in Fig. 2. When adding the oxidation catalyst Mn(II) to Fe(II)-doped absorption liquor, oxidation of S(IV) is significantly enhanced. The maximum of mass transfer controlled S(IV) oxidation can nearly be doubled by acceleration of the rate of S(IV) oxidation, and the maximum amount of Fe(II) can be increased by about 50%. These results indicate that the role of Fe^{2+} is rather limited to an enhancement of the oxygen mass transfer, while acceleration of S(IV) oxidation is based on the catalytic support of Mn(II).

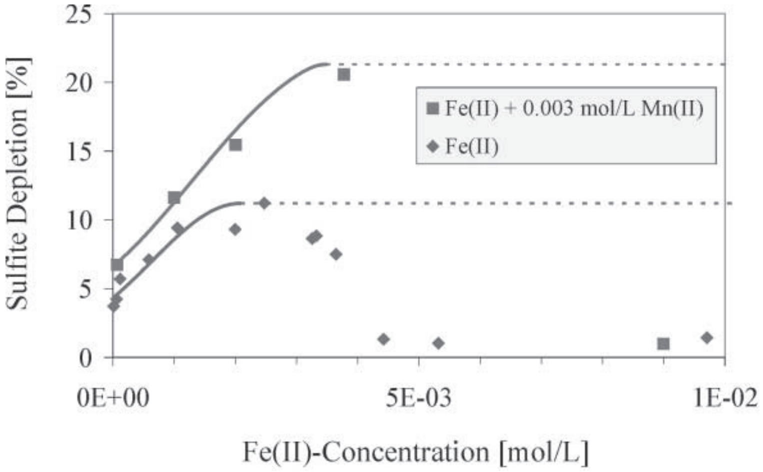

Figure 2. S(IV) depletion, observed for Fe(II)-doped absorption liquor, and varying Fe(II) content but fixed amount of Mn(II) (3×10^{-3} mol/L); operating conditions: $T = 20°C$, pH = 5 ± 0.1 in the outlet stream

Absorption of Sulfur Dioxide with Sulfate Acidic Hydrogen Peroxide

Oxidation of sulfur dioxide with hydrogen peroxide is comparable to the oxidation of sulfur dioxide with sulfate acidic solution of dichromate. Opposite to the latter process, oxidation with hydrogen peroxide does not consume protons due to the mechanism of reaction.

For elevated concentrations of hydrogen peroxide, the absorption efficiency of sulfur dioxide absorption in aqueous solution of sulfuric acid plus hydrogen peroxide approaches the efficiency of absorption of sulfur dioxide in aqueous solution of sodium hydroxide, as shown in Fig. 3. Due to the low solubility of sulfur dioxide in sulfuric acid, the rate of absorption decreases when exceeding a concentration level of sulfuric acid of 20 wt%. Absorption efficiency of sulfur dioxide finally approximates an amount as low as physical absorption. The small rate of sulfur dioxide absorption at elevated concentration of sulfuric acid is the major reason for failing technical application of this absorption process.

The results clearly indicate that with hydrogen peroxide in excess, mass transfer of sulfur dioxide is controlled by the mass transfer resistance of the gaseous phase. Further, the oxidation reaction Eq. (5) is very fast compared with mass transfer kinetics ("instantaneous reaction" according to Levenspiel[21]), and the reaction takes place at the gas–liquid interphase.

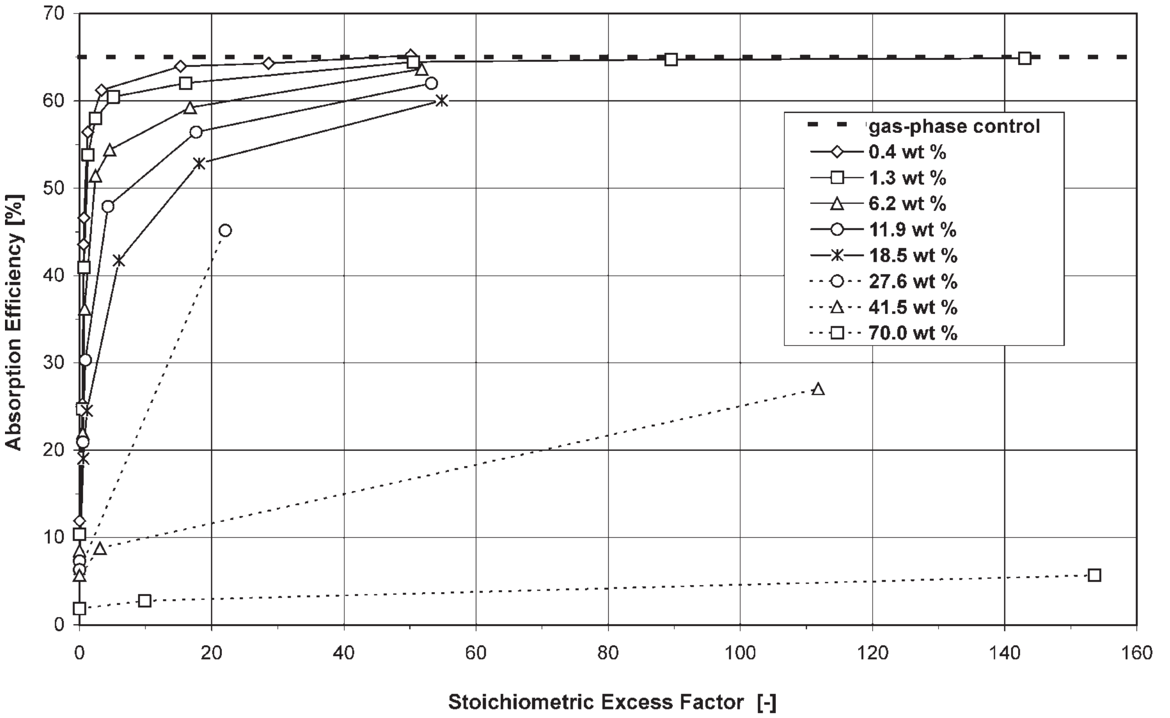

Figure 3. Absorption efficiency of sulfur dioxide absorption with sulfate acidic hydrogen peroxide compared with mass transfer control of sulfur dioxide in the gaseous phase. Operation conditions: $G = 7\,\mathrm{Nm}^3/\mathrm{hr}$, $L = 20\,\mathrm{L/hr}$, $c_{\mathrm{SO}_{2,\mathrm{feed}}} = 910$ mg/m^3, $c_{\mathrm{H_2SO4}} = 0.4\text{–}70\,\mathrm{wt\%}$, $c_{\mathrm{H_2O2}} = 0\text{–}16\,\mathrm{g/L}$; stoichiometric excess factor: molar ratio of H_2O_2 to $SO_{2,\mathrm{feed}}$.

The liquid-phase mass transfer resistance can be neglected. Reaction at the interphase is rate-determining when the boundary condition of Eq. (8) is given.

$$k_{G,SO_2} \cdot p_{SO_2} \leq k_{L,H_2O_2} \cdot c_{H_2O_2} \tag{8}$$

The rate of absorption can be expressed by Eq. (9):

$$-r''''_{SO_2} = k_{G,SO_2} \cdot a \cdot p_{SO_2} \tag{9}$$

For hydrogen peroxide concentration below the limiting value shown in Eq. (8), the reaction plane shifts toward the liquid bulk. Then the liquid-phase mass transfer resistance has to be considered as well. The rate of absorption is then described by Eq. (10):

$$-r''''_{SO_2} = \frac{(D_{L,H_2O_2}/D_{L,SO_2})c_{H_2O_2} \cdot H_{SO_2} + p_{SO_2}}{(1/k_{G,SO_2} \cdot a) + (H_{SO_2}/k_{L,SO_2} \cdot a)} \tag{10}$$

The height of an absorption column is calculated according to Eq. (11), assuming plug flow for both phases:

$$H = \frac{\dot{G}}{S}\frac{1}{RT}\int_{p_{SO_2,\mathrm{out}}}^{p_{SO_2,\mathrm{in}}} \frac{dp_{SO_2}}{(-r''''_{SO_2})} \tag{11}$$

The rate of absorption ($-r_{SO_2}''''$) is expressed by Eqs. (9) or (10), depending on the comparison of the operating conditions and the boundary condition of Eq. (8). When the rate-controlling regime shifts within the column, both regimes have to be considered in column design. The location of the shift within the column can be calculated according to the boundary condition of Eq. (8).

Based on the data of Fig. 3, the column height necessary to obtain the same outlet sulfur dioxide concentration as derived from experimental data was calculated with Eqs. (9) or (10). The calculated height was compared with the actual height of the pilot column.

The prediction error:

$$\frac{H_{\mathrm{calc}} - H_{\mathrm{eff};}}{H_{\mathrm{eff};}} 100\% \tag{12}$$

is a measure for the quality of the applied model, shown in Fig. 4.

As shown in Fig. 4, the height of the column can be predicted very precisely for negligible liquid-phase mass transfer resistance (large excess of hydrogen peroxide). For both straight physical absorption (no hydrogen peroxide added) and small excess of hydrogen peroxide, however, the quality of the models is rather poor. This can be explained by the shift of the rate-controlling regime within the absorption column, which is not modeled properly.

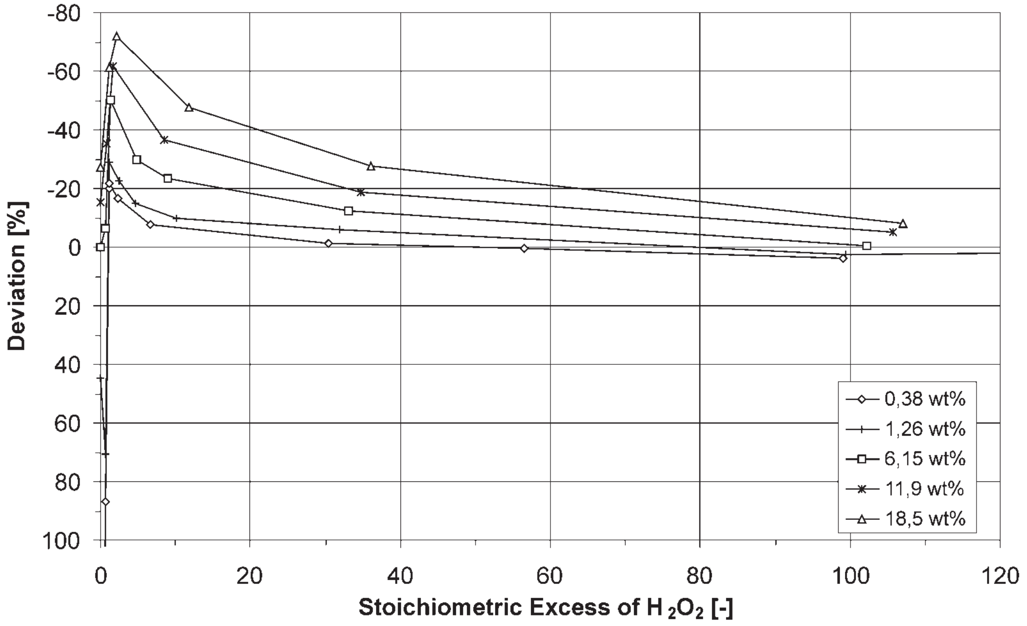

Figure 4. Prediction error derived from comparing the calculated column height according to Eq. (11) and the actual column height. Operating conditions: $G = 7\,\mathrm{Nm^3/hr}$, $L = 40\,\mathrm{L/hr}$, $c_{\mathrm{SO_{2,feed}}} = 910\,\mathrm{mg/m^3}$, $c_{\mathrm{H_2SO4}} = 6.2\,\mathrm{wt\%}$.

SUMMARY

Several processes of sulfur dioxide absorption with chemical reaction have been investigated in a falling-film absorption column. Investigation considered absorption of S(IV) with oxidizing scrubbing liquor and catalytically supported oxidation of S(IV) with oxygen.

The target of this investigation was the evaluation of process boundaries and boundaries of application.

Oxidation of sulfur dioxide in liquid phase with oxygen absorbed from gaseous phase is accelerated by heavy metal catalysis. The role of the catalysts iron (II) and manganese(II) in oxidation of sulfur dioxide was investigated. The results point out the significance of Fe(II)-containing caustic additives in flue gas absorption with preferred sulfate formation and the role of the pH value. Enhancement of the mass transfer of oxygen by Fe(II) and the catalytic acceleration of the oxidation process by Mn(II) was analyzed. As shown by the results, the Fe(II) content must not exceed a pH-dependent maximum concentration because, parallel to the oxidation of sulfur dioxide, the catalyst Fe(II) is subject of irreversible oxidative deactivation. Oxidative deactivation of Fe(II) can be suppressed by excess S(IV) even at high oxygen content and by catalytic acceleration of S(IV) oxidation.

Precipitation of sulfur dioxide by absorption with dilute sulfate acidic hydrogen peroxide is controlled by the gas-phase resistance. Absorption efficiency is comparable with neutralizing absorption of sulfur dioxide with aqueous sodium hydroxide and oxidative absorption of sulfur dioxide with sulfate acidic Cr(VI)-containing absorption liquor.

The results indicate that oxidation of sulfur dioxide with hydrogen peroxide in the liquid phase is very fast. Oxidation does not need catalytic support. Due to the low solubility of sulfur dioxide in sulfuric acid, the rate of SO_2 absorption decreases when concentration of sulfuric acid exceeds an amount of 20 wt %. For elevated concentration of sulfuric acid, the rate of SO_2 absorption finally approximates physical absorption of SO_2 in sulfuric acid.

For high sulfuric acid concentration and low hydrogen peroxide concentration, absorption rate is poor. Thus, the technical applicability of sulfur dioxide absorption with hydrogen peroxide has to be checked carefully.

ABBREVIATIONS AND SYMBOLS

a	Specific mass transfer area (m^2/m^3).
c	Concentration (mol/L, g/L).
D	Diffusion coefficient (m^2/sec).
E	Enhancement factor (−).

G	Gaseous-phase flow rate (m^3/hr).
H	Henry's constant ($bar\,m^3/mol$).
H	Height of absorption column (m).
k_G	Gas-phase mass transfer coefficient (m/sec, mol/m^2 sec bar).
k_L	Liquid-phase mass transfer coefficient (m/sec, mol/m^2 sec bar).
k	Reaction rate constant ($m^3/mol\,sec$).
L	Liquid phase flow rate (L/hr).
p	Partial pressure (bar).
r''''	Mass transfer rate ($mol/sec\,m^3$).
Re	Reynolds number (–).
Sc	Schmidt number (–).
Sh	Sherwood number (–).

Subscripts

calc	Calculated.
eff	Effective.
G	Gaseous phase.
H_2O_2	Hydrogen peroxide.
I	Interface.
feed	Entering the absorption column.
L	Liquid phase.
OG	Overall gas.
out	Leaving the absorption column.
SO_2	Sulfur dioxide.

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