H$_2$S REACTIONS WITH LIMESTONE AND CALCINED LIMESTONE

SHIYING LIN, ALI AL-SHAWABKEH, HITOKI MATSUDA, MASANOBU HASATANI
Department of Energy Engineering & Science/Chemical Engineering, Nagoya University, Chikusa-ku, Nagoya 464-01
MASAYUKI Horio
Department of Chemical Engineering, Tokyo University of Agriculture and Technology

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Sulfur capture reactions of CaCO$_3$-H$_2$S and CaO-H$_2$S have been investigated under various gaseous mixtures conditions using limestone and calcined limestone of 1 mm diameter in a thermogravimetric analyzer.

Sulfidation of limestone (CaCO$_3$-H$_2$S) had a very low initial reaction rate and final conversion than the sulfidation of calcined limestone (CaO-H$_2$S) due to its product layer having less porosity than those produced by calcined limestone. The products (CaS) produced by sulfidation of limestone have larger grain size and lower specific surface area than those produced by calcined limestone. Both sulfidation reactions of limestone and calcined limestone were first order with respect to H$_2$S partial pressure. The presence of CO$_2$ inhibited both the initial reaction rate and final conversion of sulfidation of calcined limestone. This was explained by the fact that CO$_2$ accelerated sintering of CaO, and thus reduced the specific surface area of CaO. The initial sulfidation rate of limestone decreased linearly with increasing CO$_2$ partial pressure. The initial reaction rates of both limestone and calcined limestone decreased with increasing steam partial pressure. The effect of steam on final conversion of sulfidation of calcined limestone was found to be insignificant. Only with the presence of CO$_2$ was the sulfidation rate of calcined limestone appreciably reduced by H$_2$ addition.

Introduction

Recently, attention has been focused on pressurized coal gasification technology to produce fuel gas for gas turbines in combined cycle generations such as the Topping Cycle (the second generation PFBC). However, during coal gasification, sulfur is produced in the form of harmful hydrogen sulfide, H$_2$S. Since the removal of H$_2$S by traditional gas absorption techniques requires cooling the resulting gases to lower temperatures, high-temperature H$_2$S removal is economically favorable.

Most recent works on H$_2$S removal from coal gas is now under high temperature and pressure are concerned with injection of solid sorbents such as limestone into the gasifier with coal simultaneously.

In the pressurized gasifier, as the temperature and the CO$_2$ concentration are not uniform, the CO$_2$ partial pressure may be higher or lower than the equilibrium pressure $P^*_{CO_2}$ of limestone decomposition. Decomposition of limestone in the gasifier can be represented by the following reaction:

$$\text{CaCO}_3 \rightleftharpoons \text{CaO} + \text{CO}_2 \quad (1)$$

Both calcined limestone (CaO) and limestone (CaCO$_3$) can react with H$_2$S according to reactions (2) and (3), respectively.

$$\text{CaO} + \text{H}_2\text{S} \rightleftharpoons \text{CaS} + \text{H}_2\text{O} \quad (2)$$

$$\text{CaCO}_3 + \text{H}_2\text{S} \rightleftharpoons \text{CaS} + \text{H}_2\text{O} + \text{CO}_2 \quad (3)$$

In previous studies on H$_2$S removal with limestone, most efforts have been concentrated on the kinetics of calcined limestone (CaO) under atmospheric conditions where the CO$_2$ partial pressure was always less than the equilibrium pressure $P^*_{CO_2}$. Freund (1984) studied CaO-H$_2$S reaction in the temperature range of 1400-1700 K and reported that the reaction was strongly pore diffusion limited. Kamath and Petrie (1981) studied the CaO-H$_2$S reaction with a fully calcined dolomite and showed that the apparent reaction rate was first order with respect to CaO. Nguyen and Watkinson (1993) studied the effects of sorbent surface area and percentage of calcination on H$_2$S capture using calcined limestone or dolomite during coke gasification. They found that, under atmospheric conditions and 1203 K, after an initial stage of chemical control the reaction appeared to be controlled by diffusion through the product layer. Ruth et al. (1972) studied the reaction of a half-calcined dolomite with H$_2$S. Simons and Rawlins (1980) discussed the mass transport which occurs when either SO$_2$ or H$_2$S reacts with calcined limestone and indicated that the reaction of H$_2$S with CaO proceeded almost as fast as that of SO$_2$ with CaO. However, there are only a few studies on the sulfidation of limestone under high CO$_2$ partial pressures. Borgwardt and Roache (1984) studied the reaction of limestone with H$_2$S using 1.6 μm-100 μm particles. They reported that the activation energy

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was 175 kJ·mol⁻¹ between 873-973 K by using the reaction rate at 0.2 conversion. Fenouil et al. (1994) reported that sintering of uncalcined limestone was insignificant between 1023-1173 K, but sintering of the CaS product layer influenced the reaction rate. Some experimental studies (Fujioka et al. (1994) and Goyal et al. (1990)) on H₂S capture at high pressures have also been done in a real gasifier. However, kinetic data on H₂S capture under pressurized gasification conditions are still lacking. Moreover, the effects of gases which usually exist in a coal gasifier, such as CO₂, H₂O and H₂, on both sulfidation rate of limestone and calcined limestone still have not been clearly verified.

Accordingly, the objective of this study is to clarify the characteristics of H₂S capture reactions with both limestone and calcined limestone under various conditions. To do this, experimental studies were performed at different temperatures and different CO₂, H₂O and H₂ partial pressures using a thermogravimetric analyzer with approximately 1 mm diameter limestone and calcined limestone particles.

1. Thermodynamic Equilibrium

**Figure 1** shows the equilibrium curves of CaCO₃ calcination (a), CaO sulfidation (b) and CaCO₃ sulfidation (c). Thermodynamic data were obtained from Barin (1989).

The calcination equilibrium constant shows a profound increase with increasing temperature. For instance, the equilibrium CO₂ pressure is 8.4 kPa at 1023 K and 140.4 kPa at 1223 K. As CaO sulfidation is an exothermic reaction, the equilibrium constant decreases with increasing temperature and sulfidation is thermodynamically favorable in the temperature range up to 1273 K at constant pressure. The equilibrium constant of the endothermic CaCO₃ sulfidation reaction increases with increasing temperature and is favorable above 800 K at constant pressure.

The system of reactions of CaCO₃ calcination, CaO sulfidation and CaCO₃ sulfidation contains six components CaCO₃, CaO, CaS, CO₂, H₂O, and H₂S. **Fig. 2** shows the phase diagram for this system at 1073 K, 1123 K and 1173 K. It can be seen that an increase in temperature reduces the stability of CaCO₃ and CaS in favor of CaO. With increasing temperature, the equilibrium points of three phases of CaCO₃, CaO and CaS move toward increasing the CO₂ partial pressure and decreasing the ratio of [H₂O]/[H₂S].

The above trends identify optimum conditions for H₂S adsorption, but the real reaction occurring in the gasifier is always affected by some other factors, such as gas composition and gas diffusion into solid particles.

2. Experimental

2.1 Material

A limestone of 98 wt% CaCO₃ (from Chichibu, Japan) was used in this work. It was crushed and sieved to produce particle sizes of 0.84 - 1.0 × 10⁻³ m range. The chemical composition of this limestone is shown in **Table 1**.

2.2 Set-up

**Fig. 3** shows a schematic diagram of the thermobalance used to measure the sulfidation rates throughout
all our experiments. The alumina reaction tube (18 × 10^{-2} m long, 2.4 × 10^{-2} m i.d.) is divided into two parts, the lower part (15 × 10^{-2} m long) is the preheating zone and the upper part (3 × 10^{-2} m long) is the reaction zone. H_2S and steam were individually introduced into the preheating zone, while CO_2, H_2 and N_2 gases were purged through the TGA microbalance to contact with H_2S and steam in the preheating zone. Then, the gas mixture flows upward to the reaction zone. A quartz glass evaporator of 1 × 10^{-2} m i.d. and 4.5 × 10^{-2} m length was employed to generate steam. It was fed with a 8.33 × 10^{-12} 8.33 × 10^{-10} m^3 s^{-1} water flow rate through a stainless steel tube (1 × 10^{-3} m i.d.) by a microwater pump. A schematic drawing of the evaporator is shown in Fig. 4.

In the presence of CO_2 gas, H_2S may react with CO_2 according to the reaction:

\[ \text{CO}_2 + \text{H}_2\text{S} \rightleftharpoons \text{COS} + \text{H}_2\text{O} \]  \hspace{1cm} (4)

The variation of the thermodynamic equilibrium concentrations of the various species was calculated by Krishnan and Sotirchos (1994). Their results showed that nearly 85% of H_2S would be converted to other sulfur-containing species at 1023 K. Norgwardt and Roache (1984) let a gas mixture of 70 vol.% CO_2 and 5000 ppm H_2S pass through a long reactor tube (the residence time of gases was longer than 7 s) then measured the exit gas composition with gas chromatography. They reported that up to 1223 K, only 18% of the H_2S was converted to COS and 24% of the H_2S to S. Krishnan and Sotirchos (1994) also reported that only less than 3% of the H_2S was converted in their TGA system at 1023 K. Achieving equilibrium concentration was found to take a long time. In our TGA equipment, the residence time of the gases in the reactor tube was less than 1 s, H_2S converted to COS and S must be much less than the value reported by Borgwardt and Roache, and probably close to that of Krishnan and Sotirchos (1994).

2.3 Procedure

Limestone weighing about 2 × 10^{-5} kg was spread evenly in a sample pan (10 × 10 × 2 × 10^{-3} m) made from a ceramic foam (Bridge Stone #30) and heated at a constant heating rate of 3.33 K s^{-1}. The total gas flow rate was set at 1.67 × 10^{-5} m^3 s^{-1}. The fact that the reaction rate was only slightly changed with altering the total gas flow rate from 1.67 × 10^{-5} to 2.5 × 10^{-5} m^3 s^{-1} confirmed the absence of the gas film resistance. For the sulfidation of limestone, the sample was heated above the CO_2 equilibrium pressure P_0^* to avoid decomposition of limestone. For the sulfidation of calcined limestone, the sample was calcined at a predetermined temperature under a certain CO_2/N_2 atmosphere (0 MPa CO_2, 0.101 MPa N_2). When no further weight loss was noticed, the complete calcination was reached which was also confirmed from stoichiometry. Sulfidation reaction was initiated by a change in the gas stream to the CO_2, H_2, H_2S and N_2 gas mixture, and thereafter the weight gain (or loss) upon sulfidation was continuously recorded.

2.4 Sample characteristics

The specific surface areas of the sample before and after sulfidation were measured by an N_2 adsorption technique (SHIMADZU ASAP 2000). The products in the sample taken after sulfidation were confirmed by XRD analysis (RIGAKU-XRD). The morphology of the particle surface of the sample taken after sulfidation was examined by a scanning electron microscope (SEM).

3. Results and Discussion

3.1 Reaction product analysis

Fig. 5 shows the results of XRD analysis of the sulfided samples. The detected species after the sulfidation of limestone and calcined limestone were CaS and unreacted CaCO_3, and CaS and unreacted CaO, respectively. The properties of the samples and the produced CaS were examined in terms of specific surface area and SEM.
Table 2: Specific surface area of sample before and after sulfidation (1123 K)

<table>
<thead>
<tr>
<th></th>
<th>S_{CaS} [m^2 kg^{-1} \times 10^3]</th>
<th>S_{CaS}^{'} [m^2 kg^{-1}]</th>
<th>X</th>
<th>t [s \times 10^3]</th>
<th>S_{CaS} [m^2 kg^{-1} \times 10^3]</th>
</tr>
</thead>
<tbody>
<tr>
<td>limestone</td>
<td>0.19</td>
<td>2.40</td>
<td>0.12</td>
<td>0.60</td>
<td>25.4</td>
</tr>
<tr>
<td></td>
<td>2.48</td>
<td>0.75</td>
<td>0.60</td>
<td>22.0</td>
<td>3.63</td>
</tr>
<tr>
<td>calcined</td>
<td>1.20</td>
<td>1.85</td>
<td>0.48</td>
<td>0.60</td>
<td>27.5</td>
</tr>
<tr>
<td>limestone</td>
<td>6.40</td>
<td>0.91</td>
<td>3.80</td>
<td>6.84</td>
<td></td>
</tr>
</tbody>
</table>

photographs. The specific surface area of the samples before and after sulfidation are shown in Table 2. From the specific surface area and the sulfidation conversion, the specific surface area of the product CaS can be correlated by equation (5).

\[
S_{CaS^{'}}, f = \frac{S_{CaS} - W \cdot S_{CaS^{'}}, - A}{W_{CaS^{'}},} 
\]

(5)

\[
S_{CaS^{'}}, = S_{CaS}, f
\]

(6)

where, \( f \) is the factor of surface area reduction during sintering, \( k \) is a sintering constant and \( \gamma \) is a parameter for different transport mechanisms. Since the sintering of limestone was found to be insignificant in the temperature range 1023 - 1173 K by Fenouil et al (1994), the \( k \) for limestone is zero. However, for sintering of calcined limestone, Borgwardt (1989b) reported that \( k \) was \( 8.15 \times 10^9 \exp (\frac{-26862}{T}) \) and \( \gamma = 2.7 \).

The \( S_{CaS^{'}}, \) calculated by using the data from Fig. 7 are shown in Table 2. It can be seen that, at 600 sec., the \( S_{CaS} \) produced by sulfidation of limestone \( (25.4 \times 10^3 \text{ m}^2 \text{ g}^{-1}) \) is about the same as that produced by sulfidation of calcined limestone \( (27.5 \times 10^3 \text{ m}^2 \text{ g}^{-1}) \). However, \( S_{CaS} \) is affected by longer reaction time, such as: the \( S_{CaS} \) produced by sulfidation of limestone drops to a value of \( 3.60 \times 10^3 \text{ m}^2 \text{ g}^{-1} \) at 2.2 \times 10^4 sec., and \( S_{CaS} \) produced by calcined limestone drops to \( 6.84 \times 10^3 \text{ m}^2 \text{ g}^{-1} \), even at \( 3.80 \times 10^3 \text{ sec.} \) sulfidation time.

Fig. 6 shows SEM photos of the sample taken after sulfidation. It may be noticed that the CaS grains produced by sulfidation of limestone at \( 2.2 \times 10^4 \text{ sec.} \), Fig. 6a, seem to be larger in size and less porous than those produced by sulfidation of calcined limestone at \( 3.80 \times 10^3 \text{ sec.} \), Fig. 6b. These larger grains resulted in a lower specific surface area than those produced by sulfidation of calcined limestone.

3.2 Reactivities of H₂S with limestone and calcined limestone

According to the products, reactants and the stoichiometry of reactions (2) and (3), the conversions of sulfidation can be calculated with the weight change of solid in the thermogravimetric experiments.

The conversion of the sulfidation of limestone was calculated by equation (7):

\[
X = \frac{M_{CaCO}_3 \cdot W_{CaS}^{'} \cdot (M_{CaS} - M_{CaO})}{W_{CaCO}_3 \cdot (M_{CaS} - M_{CaO})} 
\]

(7)

The conversion of the sulfidation of calcined limestone was calculated by equation (8):

\[
X = \frac{M_{CaS} \cdot W_{CaS} \cdot (M_{CaS} - M_{CaO})}{W_{CaS} \cdot (M_{CaS} - M_{CaO})} 
\]

(8)

Fig. 7 shows the conversion - time curves of the sulfidations for both limestone and calcined limestone at
1123 K. It is seen that both the initial reaction rate and final conversion of limestone were lower than those of calcined limestone. Since the rate of sulfidation of limestone was lower than that of calcined limestone, it is concluded that CaS produced from limestone will be exposed to sintering conditions much longer than that of CaS produced from calcined limestone, which causes the sulfidation of limestone to have lower final conversion than calcined limestone.

The effect of H$_2$S partial pressure on the initial reaction rate for both sulfidations of limestone and calcined limestone are shown in Fig. 8. The fitted lines for both reactions showed a first order reaction with respect to H$_2$S partial pressure.

3.3 Effect of CO$_2$ partial pressure

Fig. 9 shows the effects of CO$_2$ partial pressure on sulfidations of limestone and calcined limestone. Different patterns are found, depending on whether CO$_2$ partial pressure is lower or higher than its equilibrium value of about 20 kPa at 1073 K. Although CO$_2$ has no influence on the equilibrium of reaction (2), it was reported (Borgwardt (1989a,b) and German and Munir (1976)) that CO$_2$ accelerates the sintering of CaO. The specific surface area of CaO calcined in different CO$_2$/N$_2$ atmospheres is shown in Table 3. It is noticed that the specific surface area of CaO calcined in 18 kPa CO$_2$ has a lower value than those calcined in a N$_2$ atmosphere. Thus, the effect of CO$_2$ on sulfidation of calcined limestone may be explained by the fact that the specific surface area of CaO particles is reduced by the sintering effect accelerated by CO$_2$ presence.

When CO$_2$ partial pressure was higher than $P_{CO_2}$, sulfidation proceeded via reaction (3) (sulfidation of limestone). The sulfidation rate was significantly decreased by increasing CO$_2$ partial pressure. It may be due to the fact that the rate of the reverse reaction of equation (3) is enhanced by higher CO$_2$ partial pressure. Fig. 10 shows that the initial sulfidation rate decreases linearly with increasing CO$_2$ partial pressure.

However, from Fig. 9, it can also be seen that the final conversion also decreased with increasing CO$_2$ partial pressure. Thus it is concluded that the higher the partial pressure of CO$_2$ gas outside the particle, the more difficult for the CO$_2$ gas that has been produced by reaction (3) to diffuse through the product layer, which in turn suppresses formation of more products by reaction (3).

3.4 Effect of steam

Fig. 11 shows the effect of steam on sulfidation of
calcined limestone. Increasing steam partial pressure (from 0 to 5.2 kPa) resulted in a decrease in the initial reaction rate. Although the initial reaction rate was almost halved at 5.2 kPa steam partial pressure, no influence of steam on the final conversion after 90 minutes was noticed.

Both the initial reaction rate and final conversion of limestone were significantly affected by the presence of steam as Fig. 12 shows. At 2.9 kPa steam partial pressure, the initial reaction rate value dropped to about 15% of its initial value without steam. The final conversion after 200 minutes was also found to drop to lower than 10%. The effect of H₂O pressure on limestone sulfidation can also be explained in a similar way to the CO₂ effect described above. That is increasing H₂O partial pressure enhanced the reverse reaction of equation (3).

### 3.5 Effect of H₂

At the temperature range prevailing in a typical gasifier, H₂S usually undergoes decomposition according to Eq. (9):

\[
\text{H}_2\text{S} \rightleftharpoons \frac{1}{2}\text{S}_2 + \text{H}_2 \tag{9}
\]

Many previous researchers (Borgwardt and Roache (1984) and Ruth et al. (1972)) tried to investigate H₂ or H₂O addition effects on H₂S decomposition and, accordingly, on the sulfidation rates. However, these effects still have not been clearly explained. For instance, Borgwardt and Roache (1984) found an inhibiting effect of H₂ on sulfidation, while no significant effect was shown by Ruth et al. (1972).

Figs. 13 and 14 show the effects of H₂ addition on sulfidation of calcined limestone at 1073 K and different H₂/CO₂/N₂ partial pressures. As seen in Fig. 13, in the range between 0 and 2.6 kPa, H₂ had no significant effect on H₂S capture which agrees well with the results reported by Ruth et al. (1972). However, at 9 kPa CO₂ in different H₂ partial pressures 0, 2.4 and 8.9 kPa, the initial sulfidation rates as well as final conversions were appreciably reduced with H₂ partial pressure increase. In Fig. 14, the initial rates of sulfidation of calcined limestone (including data of Fig. 13) are plotted against H₂ partial pressure with and without CO₂. The fact that the sulfidation rates remained unchanged with H₂ addition (without CO₂) ensures no effect of H₂S decomposition on sulfidation.

The effect of H₂ partial pressure of 0-2.9 kPa on the sulfidation of limestone at 1123 K in 60 kPa CO₂ and 1 kPa H₂S atmosphere is shown in Fig. 15. The CO₂ gas was presented in all tests. The inhibiting effect of H₂ resulted in a remarkable reduction in the initial sulfidation rates as well as in final conversions. The results in Fig. 15 agree with the results reported by Borgwardt and Roache (1984).

The decreasing sulfidation rate with increasing H₂ at a constant CO₂ partial pressure is most probably caused by the effect of H₂O formed via the reaction of CO₂ with H₂ according to Eq. (10):
\[ \text{CO}_2 + \text{H}_2 \leftrightarrow \text{H}_2\text{O} + \text{CO} \quad (10) \]

Within the equilibrium limitations of this reaction, \( \text{H}_2\text{O} \) content increases with increasing \( \text{H}_2 \) partial pressure.

Conclusions

The findings drawn from this experimental study can be itemized as follows:

Sulfidation of limestone had a much lower initial reaction rate and final conversion than that of the sulfidation of calcined limestone due to its less porous product layer.

Both sulfidation reactions of limestone and calcined limestone are first order with respect to \( \text{H}_2\text{S} \) partial pressure.

The presence of \( \text{CO}_2 \) inhibited both the initial reaction rate and the final conversion of the sulfidation of calcined limestone. This may be due to the fact that \( \text{CO}_2 \) accelerates sintering of \( \text{CaO} \), which results in reducing its specific surface area of \( \text{CaO} \). The initial sulfidation rate of limestone decreases linearly with increasing \( \text{CO}_2 \) partial pressure.

Only the initial reaction rate of sulfidation of calcined limestone was decreased with increasing steam concentration. Both the initial reaction rate as well as the ultimate conversion of sulfidation of limestone decreased with increasing steam concentration.

For efficient \( \text{H}_2\text{S} \) capture in a gasifier, it is necessary to operate the gasifier under conditions where CO\(_2\) partial pressures are below \( P^{*}_{\text{CO}_2} \).

Nomenclature

- \( A' \): surface area of impurity in limestone \([\text{m}^2]\)
- \( l_d \): inside diameter \([\text{m}]\)
- \( f \): factor of surface area reducing during sintering \([-]\)
- \( F_G\text{CO}_3 \): fraction of \( \text{CaCO}_3 \) in limestone \([-]\)
- \( k \): sintering constant \([\text{s}^{-1}]\)
- \( K_{C_1} \): equilibrium constant of reaction (1) \([-]\)
- \( K_{C_2} \): equilibrium constant of reaction (2) \([-]\)
- \( K_{C_3} \): equilibrium constant of reaction (3) \([-]\)
- \( M \): molecular weight of \( \text{CaO} \) or \( \text{CaCO}_3 \) \([\text{kgmol}^{-1}]\)
- \( M_{\text{GCO}_3} \): molecular weight of \( \text{CaCO}_3 \) \([\text{kgmol}^{-1}]\)
- \( M_{\text{Gao}} \): molecular weight of \( \text{CaO} \) \([\text{kgmol}^{-1}]\)
- \( M_{\text{Gas}} \): molecular weight of \( \text{CaS} \) \([\text{kgmol}^{-1}]\)
- \( P_{\text{H}_2S} \): partial pressure of \( \text{H}_2\text{S} \) \([\text{Pa}]\)
- \( P^{*}_{\text{CO}_2} \): equilibrium partial pressure of \( \text{CO}_2 \) \([\text{Pa}]\)
- \( P_{\text{CO}_2} \): partial pressure of \( \text{CO}_2 \) \([\text{Pa}]\)
- \( P_{\text{FBC}} \): pressurized fluidized-bed combustor \([-]\)
- \( S_g \): initial specific surface area of sample \([\text{m}^2\text{g}^{-1}]\)
- \( S_{C_1} \): specific surface area of unreacted \( \text{CaCO}_3 \) or \( \text{CaO} \) at time \( t \) \([\text{m}^2\text{g}^{-1}]\)
- \( S_{\text{CaS}} \): specific surface area of \( \text{CaS} \) \([\text{m}^2\text{g}^{-1}]\)
- \( S_{\text{CaS},t} \): specific surface area of product \( \text{CaS} \) at time \( t \) \([\text{m}^2\text{g}^{-1}]\)
- \( S \): specific surface area of sample at time \( t \) \([\text{m}^2\text{g}^{-1}]\)
- \( t \): sulfidation reaction time \([\text{s}]\)
- \( W_{C,1} \): weight of unreacted \( \text{CaCO}_3 \) or \( \text{CaO} \) at time \( t \) \([\text{kg}]\)
- \( W_{\text{GCO}_3} \): initial weight of \( \text{CaCO}_3 \) \([\text{kg}]\)
- \( W_{\text{Gao}} \): initial weight of calcined limestone \([\text{kg}]\)
- \( W_{\text{Gas}} \): weight gain upon sulfidation of \( \text{CaO} \) \([\text{kg}]\)
- \( W_{\text{Gas}} \): weight loss upon sulfidation of \( \text{CaCO}_3 \) \([\text{kg}]\)
- \( X \): fractional conversion of \( \text{CaO} \) or \( \text{CaCO}_3 \) into \( \text{CaS} \) \([-]\)

Literature cited


