

# The effects of limestone type on the sulphur capture of slaked lime

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## Abstract

This study examines the effect of the chemical composition and origin on the performance of two calcitic and two dolomitic limestones from different sources in South Africa. The experiments were carried out in a fixed bed reactor maintained at 80 °C. The raw sorbent materials were calcined at 900 °C and the resulting quicklime hydrated to produce the relevant hydrates which were used in the tests. Results obtained show that the maximum temperature rise during the hydration of the samples varied from 5 to 65 °C depending on the chemical composition of the sorbent. Sorbents with higher temperature rise resulted in products with a more porous structure and a better performance in the sulphur capture. The maximum sorbent conversion in terms of mol of SO<sub>2</sub> per mol of sorbent varied from 0.0274 for dolomitic limestones to 0.1823 for the calcitic limestones. The presence of Fe<sub>2</sub>O<sub>3</sub> in small quantities was observed to have a positive effect on the performance of the sorbent.

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## 1. Introduction

South Africa is an energy intensive country, with a high reliance on electricity and primary energy resources such as coal and imported oil. Coal presently provides about 75% of South Africa's primary energy needs, and over 90% of electricity in the country is coal-derived. This is likely to remain the scenario until alternatives such as nuclear energy, natural gas, hydro or other renewables start to play a more significant role. In 2001, South Africa's domestic consumption of coal was 160.2 million metric tons. Coal reserves will last several hundred years at current consumption rates, making it the country's most abundant indigenous primary energy resource. Presently, South African power plants have particulate control but no gaseous emission control requirements, mainly due to the relatively low sulphur content of local coals (i.e. low expected SO<sub>2</sub> emissions). However, future plants will have to contend with far

stricter scrutiny from the local and international communities. The concept of flue gas desulphurisation (FGD) is thus one which needs to be seriously explored in the country.

Almost all commercial FGD processes are based on the fact that SO<sub>2</sub> is acidic in nature, and remove the SO<sub>2</sub> from the flue gases by reaction with a suitable alkaline substance. The most commonly used alkaline materials are limestone (calcium carbonate), quicklime (calcium oxide) and hydrated lime (calcium hydroxide). Limestone is an abundant and therefore relatively cheap material and both quicklime and hydrated lime are produced from limestone by calcination. The induct sorbent injection is one of the dry flue gas desulphurisation (FGD) processes. In this process the sorbent particles are normally introduced into humidified and temperature controlled flue gas in the duct between the air pre-heater and particulate control device. The total SO<sub>2</sub> removal achieved is the sum of the removal in the duct section and in the particulate control device. The sorbent used widely in the humidified duct injection processes is hydrated lime (Ca(OH)<sub>2</sub>) [1]. In the preparation of hydrated lime from raw limestone, two main processes are necessary; calcination and hydration.

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Table 1  
Some properties of CaO prepared from calcining limestone

Limestone	% CaCO <sub>3</sub>	Temp. (°C)	Specific surface area (m <sup>2</sup> g <sup>-1</sup> )		Porosity		Reference
			Before	After	Before	After	
Rummu L	92.26	850	0.7	5	0.0716	0.141	[4]
Hellamaa D	52.36	850	0.34	22.18	–	0.159	[4]
Maria	99.1	850	0.424	17.66	0.014	0.43	[5]
Andorra	55.9	850	0.777	24.81	0.066	0.547	[5]
Blanka	97.1	900	0.3	19	0.03	0.56	[6]
Sierra de Arcos	52.5	900	9.57 <sup>a</sup>	30.4	0.35 <sup>a</sup>	0.57	[6]

<sup>a</sup> Half-calcined samples.

During limestone calcination some changes will occur in the structural properties of the final product depending on the calcination temperature and the limestone type. During their investigation of Estonian Oil Shale carbonates, Trikel and Kuusik [2] observed that the specific surface area and the porosity of the samples studied mainly increased. Paolo Davini [3] obtained a threefold increase in the BET surface area and more than doubling of the porosity of raw limestone samples calcined at 900 °C. Other researchers have reported similar changes (Table 1).

Since the relative molar volumes are 36.9 cm<sup>3</sup> mol<sup>-1</sup> for CaCO<sub>3</sub> and 16.9 cm<sup>3</sup> mol<sup>-1</sup> for CaO [7], if there is negligible particle shrinkage during calcination, the porosity of the product from a pure non-porous carbonate will increase to a theoretical value of 0.55. Hence a lime may have a porosity greater than 0.6. For dolomite, the porosity of a completely calcined, non shrinking particle of dolomite is as large as 0.552 [8]. For maximum adsorption efficiency, care must be taken on the one hand to ensure that calcination is complete, and on the other, that the CaO grains formed do not sinter after formation. An optimum temperature of 900 °C for the calcination reaction has been suggested by Irfan and Gulsen [9], which is the temperature used in traditional kilns. Thermogravimetric Analysis (TGA) results by Hartman et al. [8] demonstrate that for dolomite, the decomposition starts at about 600 °C and end at about 760 °C. At temperatures less than 350 °C, quicklime (CaO) rapidly reacts with water, exothermically, to produce hydrated lime. During the hydration of quicklime, two simultaneous phenomena are usually observed; the temperature increases (exothermic reaction) and the volume of the paste is expanded.

In the present work, the calcination, hydration, and sulphation of several calcium-based materials has been carried out and the results reported. It is the aim of this work to relate the physical and chemical changes that take place during the calcination and hydration processes to the eventual performance of the materials in the removal of sulphur dioxide under fixed bed conditions.

## 2. Experiments

### 2.1. Materials

Four samples of commercial calcareous material (quarried in South Africa and herein named A, B, C, and D)

were used as sorbents. The samples have varying amounts of calcium and magnesium carbonates. Table 2 shows their composition as obtained by a chemical analysis. For comparison, a 99.8% pure commercial Ca(OH)<sub>2</sub> sample (LC) was used. The raw samples were heated at 900 °C for 3 h to obtain the relevant calcines. The weight of the samples before and after calcination were recorded to determine the percentage weight loss. Hydration was achieved by weighing 10 g of the calcinated sample and mixing, while stirring, with 20 ml of water. A thermometer immersed in the hydrating mixture recorded the maximum temperature attained for each sample. The resulting slurry was dried off in an oven maintained at 105 °C to produce a dry solid. All samples were then milled and sieved to a particle size below 200 µm before being used in the fixed bed.

### 2.2. Procedure

The experiments for the SO<sub>2</sub> absorption of the samples were carried out in a laboratory – scale fixed bed reactor (Fig. 1). The reaction zone is contained in a 0.008 m inner diameter stainless steel tube fitted in a furnace for isothermal operation. 0.1 g of the sorbent material is dispersed in 3 g of inert silica sand, packed in the centre of the reactor and is supported by glass wool. The inert silica sand helps to ensure isothermal operation, improve gas solid contact and avoid channeling of the flue gas. A N<sub>2</sub> stream is passed through a humidification system consisting of two 250 ml conical flasks immersed in a water bath maintained at constant temperature. The temperature in the water bath can be varied to obtain the desired relative humidity in the reactor. After humidification, the N<sub>2</sub> stream is mixed with a gas stream consisting of SO<sub>2</sub>, NO, and O<sub>2</sub>. The simulated flue gas then flows through the reactor at a flow rate of 150 ml/min. The SO<sub>2</sub> concentration of the effluent gas is continuously monitored by an IMR 2800 P flue gas

Table 2  
Chemical analysis of raw sorbent materials

Compound	A	B	C	D	LC
SiO <sub>2</sub> (%)	5.95	13.33	–	4.48	–
Al <sub>2</sub> O <sub>3</sub> (%)	1.53	1.67	–	0.36	–
Fe <sub>2</sub> O <sub>3</sub> (%)	1.83	0.67	–	1.61	≤0.05
CaO (%)	88.39	81.67	44	53.54	≥99.8
MgO (%)	1.26	1.67	39.0	36.52	–

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