

Recovery of Sulphur from Waste Gypsum

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Introduction

- **Gypsum is produced as a waste product by various industries (the fertilizer industry and the mining industry) as well as by power stations**
- **Gypsum waste disposal sites are responsible for leachates of saline water into surface and underground water.**
- **Gypsum waste is accountable for environmental pollution**

Sulphur from gypsum

- **Gypsum is a good source for the recovery of Sulphur**
- **Sulphur is an essential raw material for many manufacturing industries like fertilizers, acids, etc.**
- **The need for finding alternative sources to produce Sulphur is therefore essential.**
- **South Africa can effectively supply other African countries with Sulphur, as these countries face shortages and are presently importing from other countries.**

Sulphur recovery reactions

Reduction roasting of gypsum to form CaS



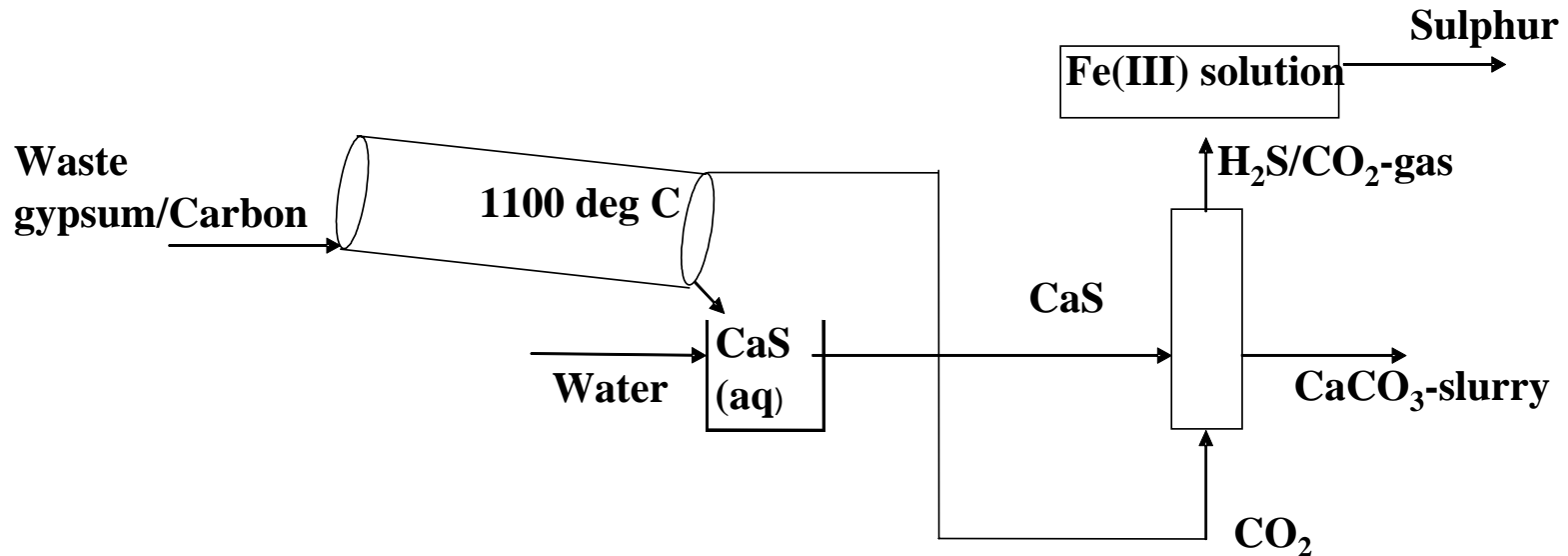
Dissolution and stripping of CaS to form H₂S and CaCO₃



H₂S converted to elemental sulphur via the iron (III) route



S^0 recovery flow diagram



Aim

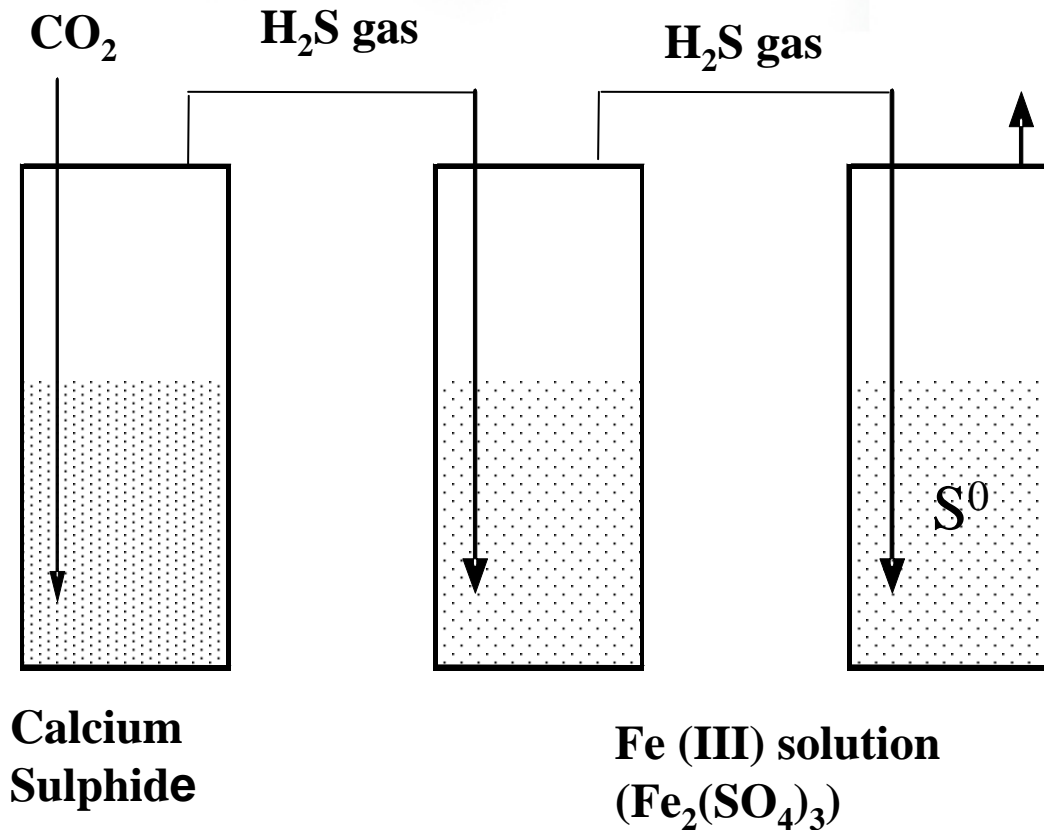
- **Develop the following stages of the sulphur recovery process to the stage prior to full-scale implementation**
 - **Production of calcium sulphide through thermal reduction of gypsum**
 - **H₂S-stripping with CO₂**
 - **Crystallization of CaCO₃ with CO₂**
 - **Sulphur production**

Experimental conditions

- *Thermal batch studies:* A tube furnace, silica boat and silica tube were used for the reduction of gypsum with carbon



H₂S batch stripping: CO₂ gas and Ferric sulphate were used for H₂S stripping and absorption of the stripped H₂S-gas and sulphur formation



Experimental programme

Thermal studies

Temperature	900 to 1150°C
Reaction time	5 to 60 min
Carbon to gypsum mole ratio	0 to 3 moles
Particle sizes of gypsum	38, 63 and 125 μm

H₂S stripping studies

- **The following parameters were investigated as a function of time:**
 - **CO₂ dosed**
 - **% Sulphide stripped**
 - **% Sulphur produced**

RESULTS

THERMAL STUDIES

Effect of Time

Parameter	Value	% Gypsum converted to CaS
Time (min)	2	24
	5	48
	10	60
	20	96
	60	90

Constant parameter: temperature = 1100 °C and mole ratio (carbon:gypsum) = 3: 1

Effect of Temperature

Parameter	Value	% Gypsum converted to CaS
Temperature (°C)	900	35
	950	39
	1000	45
	1050	76
	1100	95
	1150	94

Constant parameter: time = 20 min, mole ratio (carbon:gypsum) = 3: 1

Effect of carbon to gypsum ratio

Parameter	Value	% Gypsum converted to CaS
Gypsum: Carbon mole ratio	1:0	0
	1:2	97
	1:3	94

Constant parameter: temperature = 1100 °C, time = 20 min

Effect of gypsum particle size

Parameter	Value	% Gypsum converted to CaS
Gypsum particle sizes (μm)	125	19
	63	61
	38	90

Constant parameter: temperature = 1100 °C, time = 20 min, mole ratio (carbon:gypsum) = 3: 1

H₂S STRIPPING

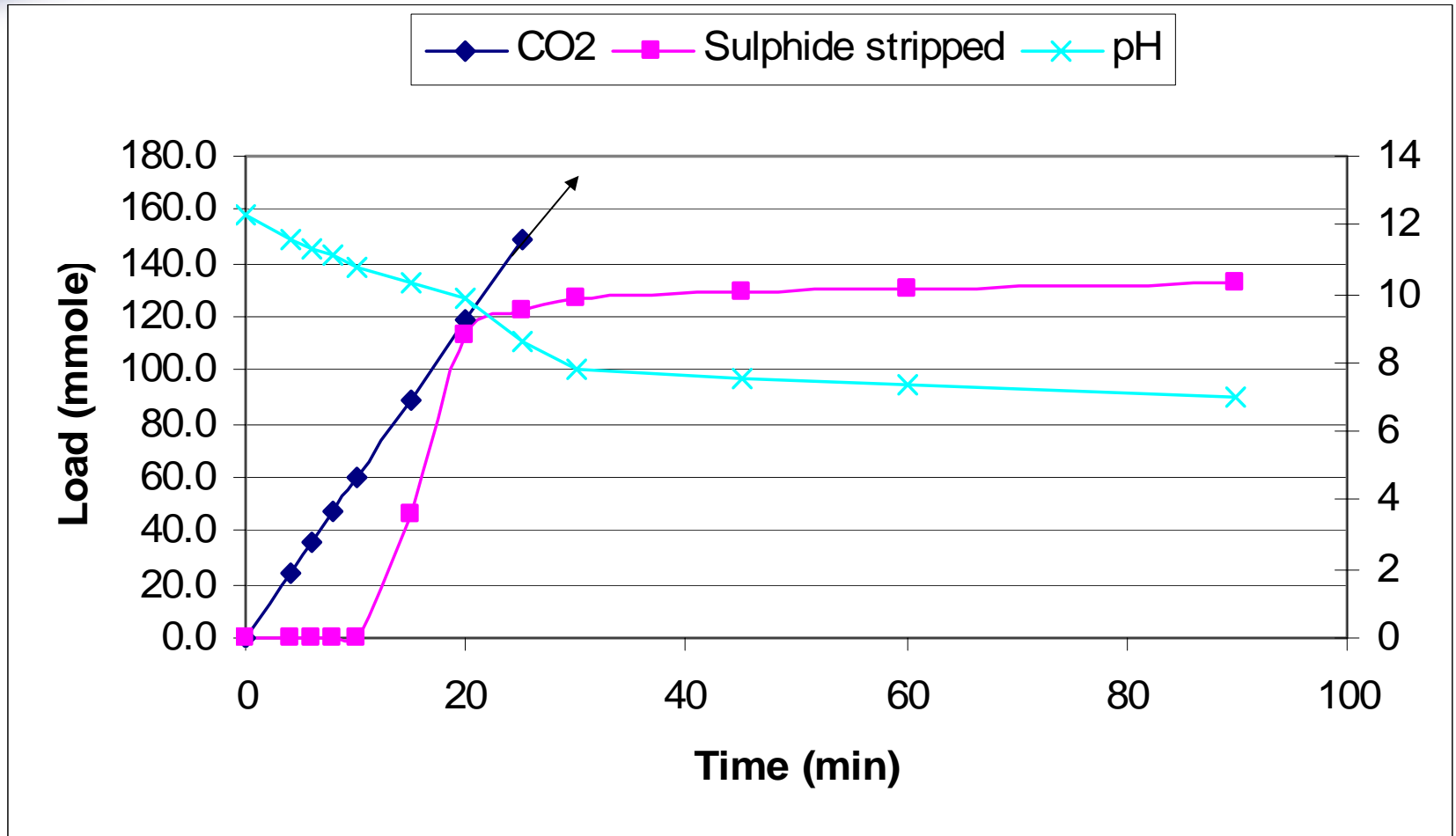
Reaction Conditions

Parameters	Value
CO₂ flow rate (ml/min)	105
Period of run (min)	90
Sulphide at start (mmole/l)	300
Sulphide at end (mmole/l)	0.5
Fe²⁺ at start (mg/l)	28
Fe²⁺ at end (mg/l)	3000

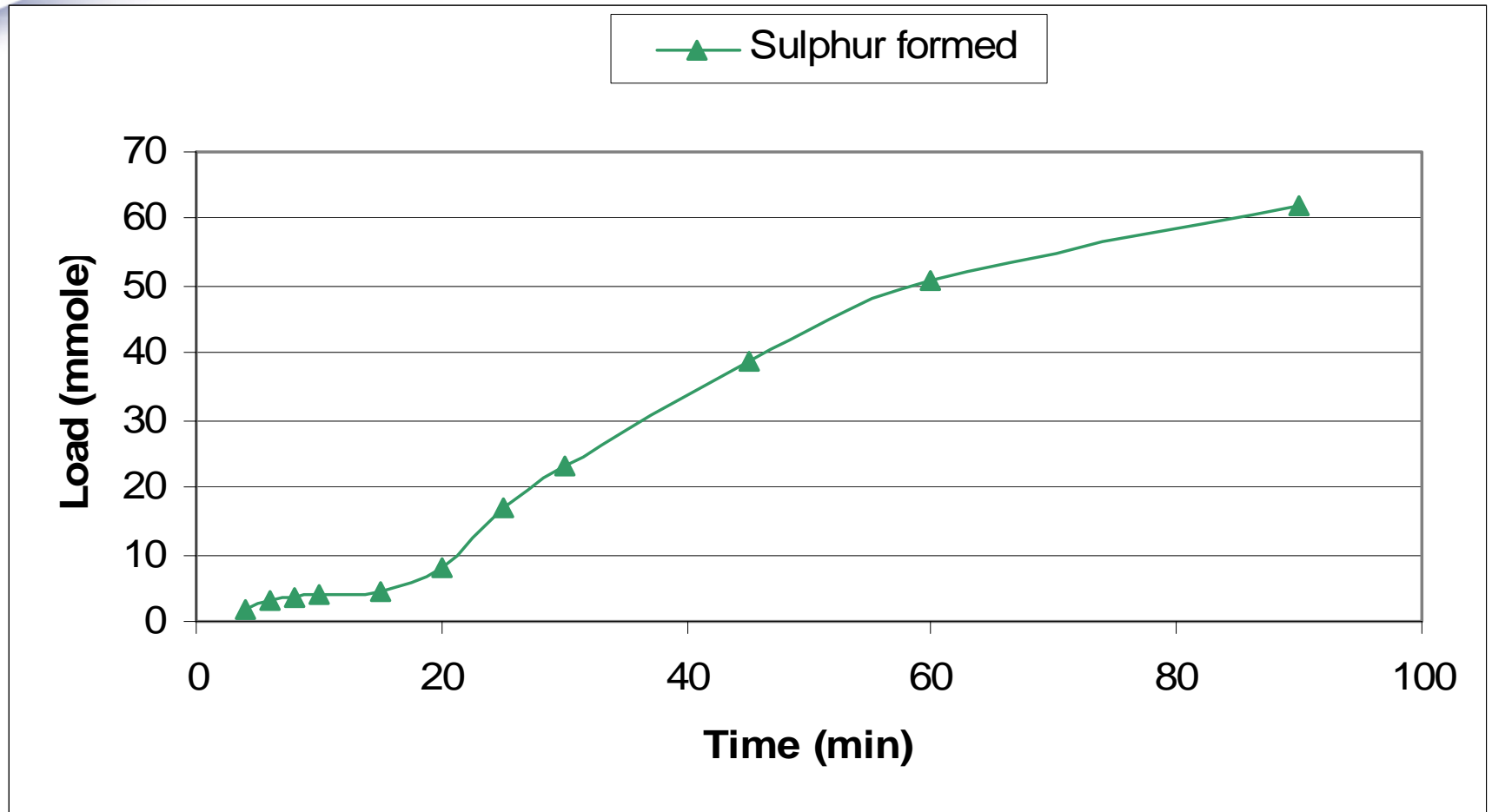
Efficiencies

Parameter	Value (%)
Sulphide stripped	96
Sulphur recovered from sulphide stripped	50.5

Relationship between CO₂, sulphide stripped



Sulphur production



CONCLUSIONS

Thermal studies

Optimum thermal reduction of gypsum to CaS is obtained when:

- furnace temperature is 1100 °C,
- activated carbon is used as reducing agent
- mole ratio of gypsum to carbon is 1:2
- a small particle size of gypsum is used
- reaction time is 20 minutes.

H₂S stripping studies

- ✓ H₂S gas can be stripped with CO₂ gas from solutions of CaS
- ✓ Sulphur can be recovered from gypsum