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Removal of sulphur from coal by air oxidation at 350-450°C

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The extent of sulphur removal from seven U.S. coals has been studied by low-temperature oxidation. The total sulphur in these coals varied between 3 and 7% by weight. The pyritic sulphur constituted approximately 50% of the total sulphur. A major part of the study has been carried out at oxidation temperatures of 350, 400 and 450°C. Temperature, up to 450°C, is a prime factor determining the extent of desulphurization. The efficiency of removal is controlled by the diffusion of oxygen into coal and its pyrite. More than 90% of the pyritic sulphur can be removed at 450°C in 10 min in some cases. The rate of removal is unaffected by the build-up of sulphur dioxide concentration in the air stream to at least 10%. That is, the level of sulphur dioxide concentration in the effluent gases can be increased for sulphuric acid manufacture by successive recycling of the effluent gases over fresh charges of coal. The calorific value and the VM decrease during this desulphurization treatment. The importance of the loss can be minimized if the desulphurized coal is fed directly to the boiler while at about 450°C. The process can be made self-sustaining by proper control of the coal/air ratio.

A volume of work has been carried out on the decomposition of pyrite in inert atmospheres in the temperature range of 500–1300°C. In most cases the results on the equilibrium constants, the kinetics of decomposition, and the mechanism of decomposition are at variance. This is mainly because of (1) impurities in the pyrite used and (2) a complicated sequence of decomposition reactions. However, it is generally agreed that the decomposition occurs in steps and that the composition of the residue depends upon the decomposition temperature^{1,2}.

The reaction of pyrite with air is much more complicated than its decomposition in an inert atmosphere^{3–13}. FeO, FeS, Fe₃O₄, Fe₂O₃, Fe₂(SO₄)₃, S₂, SO₂, and SO₃ are frequently reported reaction products. The nature and relative proportions of the products of any particular experiment in a system as complex as this will depend largely on the conditions at which it is carried out, particularly since, as in other reactions involving solid components, the rate of reaction depends very much on the grain size of the reactants and on the rates of diffusion in and between the particles. Added to this is the effect of adventitious impurities in the pyrite. Sulphur dioxide and sulphur trioxide are reported as gaseous products of the reaction.

Most of the studies on the reactions of pyrite upon the heating of coal, coke or carbon have been prompted mainly from the standpoint of producing low-sulphur metallurgical cokes. Numerous chemicals have been added to coal prior to its carbonization^{14,15} in order to have a coke with very low sulphur content. Technical success has been limited, and the cost of such desulphurization processes is relatively expensive.

Blum and Cindea¹⁶ studied the desulphurization of coals in an 85:15 steam/air mixture and found that by heating for 1 h at 380°C, 90–95% of the sulphur could be removed from a coal containing not more than 2.5% sulphur with a carbon loss of only 4–5%. Muntean¹⁷ studied the kinetics of this reaction at 380°C and concluded from his results that the topochemical oxidation of pyrite in coal is similar to the oxidation of pure pyrite; the rate of oxidation of pyrite in coal is not influenced by the coal substance.

With increasingly strict air pollution laws being invoked, there is a clear necessity for reducing the emission of sulphur dioxide from power plant stack gases. Much attention is being paid to the removal of sulphur dioxide produced by combustion by using sorption techniques. The other obvious approach is to reduce the sulphur content of the fuel prior to combustion. Successful commercial plants are in operation to reduce the sulphur content of liquid fuels. Since it is clear that we will continue to rely on coal as a prime fuel source for years to come, studies should be accelerated on the removal of sulphur from coal prior to its combustion. In this study, the extent of desulphurization of seven U.S. coals by oxidation with air at 350, 400 and 450°C has been investigated.

EXPERIMENTAL

A 2 g sample of coal dried at 110°C for 1 h of a particular sieve fraction was thinly distributed in a silica boat. The

Table 1 Proximate analyses of coal samples

PSOC Sample No.	Seam No. and location	Rank	Moisture (%)	VM (%)	Ash (%)	Dry basis			
						Sulphur (%)			Calorific value (Btu/lb*)
						Total	Pyrite	Sulphate	
113	Lower Kittaning, Tirehill, Pa.	LV	0.8	18.1	10.6	3.0	2.0	0.16	13 745
116	Freeport Ehrenfeld, Pa.	MV	1.1	20.6	12.9	4.1	2.6	0.20	13 233
185	Illinois No.6 Sullivan, Ind.	HVC	9.9	38.0	14.9	3.3	1.6	0.18	12 126
24	No.2 Colchester, Vermont, Ill.	HVB	4.8	36.9	6.0	3.8	1.9	0.03	12 827
26	Illinois No.6, Carrier Mill, Ill.	HVC	12.1	40.7	10.8	7.0	4.2	0.35	12 773
184	Upper & Lower Bloc, Carbon, Ind.	HVC	10.7	34.5	21.1	3.2	1.4	0.08	11 181
190	Illinois No.6, Victoria, Ill.	HVC	16.1	38.4	8.5	3.1	1.0	0.10	11 960

* 1 Btu/lb = 2.326 kJ/kg

Table 2 Ultimate analysis of coal samples (d.a.f. basis)

PSOC Sample No.	Analyses (%)					
	C	H	N	Cl	S	O
113	88.2	4.6	1.1	0.13	3.3	2.7
116	85.2	4.9	1.3	0.12	4.7	3.8
185	80.8	5.9	1.0	0.09	3.9	8.3
24	79.4	5.4	1.0	0.05	4.3	9.9
26	77.2	5.6	1.1	0.00	7.8	8.3
184	76.5	5.4	1.0	0.05	4.0	13.1
190	75.5	5.2	1.1	0.07	4.1	14.1

thickness of the layer was about 3–4 mm. The boat was then placed in the centre of a tube furnace maintained at the required temperature. The temperature probe, a chromel–alumel thermocouple, almost touched the coal bed. The variation in the probe temperature during an experiment was about $\pm 10^\circ\text{C}$. A current of dry air flowed past the coal sample at a linear velocity of about 20 cm/min. The extent of sulphur removal following the oxidation was monitored either by measuring the total sulphur on the oxidized sample using the Leco sulphur analyser or by absorbing the gases evolved in a known volume of standard acidified dichromate solution. The excess dichromate was iodometrically back titrated. The sulphur removed from the coal was in the form of sulphur dioxide.

RESULTS AND DISCUSSION

The proximate analyses and sulphur distribution according to its form of occurrence in the coal samples, together with their rank and geographical locations in the U.S., are shown in Table 1. Ultimate analyses of these coals are recorded in Table 2. Three Tyler sieve sizes, 40 X 70, 70 X 100, and –100 mesh, were used in this study. The –100 mesh samples had an approximate size distribution of: 29%, 100 X 150 mesh; 21%, 150 X 200 mesh; and 50%, –200 mesh. The samples were obtained by the grinding of whole coals and the separation into size fractions by sieving. For all coals, there was no apparent variation of total sulphur content with size consist; also in all cases the figures reported for total sulphur content varied by less than 5% between size fractions.

Desulphurization runs at 350°C for various levels of exposure time are shown in Table 3. The data are for the 40 X 70 mesh sieve size. The percentage sulphur remaining in the coal and the total weight loss following oxidation times between 15 and 60 min are reported. The amount of sulphur remaining in the treated coal is dependent upon two factors – the amount of sulphur removed and the total weight loss. Sulphur removal is expected to be achieved primarily through oxidation of pyrite. Removal of organic sulphur will be small, if for no other reason than it is not accessible to oxygen except where gasification of the carbon skeleton in the coal occurs. Total weight loss will be accounted for by three main processes: oxidation of sulphur, volatile matter release, and gasification of carbon. In fact, at 350°C gasification of carbon is expected to be

Table 3 Desulphurization of 40 x 70 mesh Tyler fractions in air at 350°C

PSOC Sample No.	Sulphur remaining in coal and weight loss							
	15 min		30 min		45 min		60 min	
	S (%)	Wt loss (%)	S (%)	Wt loss (%)	S (%)	Wt loss (%)	S (%)	Wt loss (%)
113	2.8	—	2.8	—	2.4	1.0	1.7	2.0
116	—	—	3.8	—	3.5	1.0	3.0	3.0
185	—	—	2.6	3.2	2.4	6.0	2.2	8.0
24	3.3	2.5	3.3	2.5	3.2	6.0	3.2	6.0
26	5.0	7.1	5.0	15.7	—	—	4.8	21.0
184	3.1	5.3	3.0	7.0	—	—	2.8	18.0
190	3.0	3.0	3.0	6.7	—	—	2.8	11.0

Table 4 Desulphurization of –100 mesh fractions in air at 400°C

PSOC Sample No.	Total sulphur removed (g/100 g coal) and weight loss (%)									
	10 min		15 min		20 min		30 min		60 min	
	S	Wt loss	S	Wt loss	S	Wt loss	S	Wt loss	S	Wt loss
113	0.25	2.4	0.32	1.0	0.80	3.8	1.2	4.5	1.4	6.0
116	1.2	4.0	1.3	4.0	1.9	4.0	2.1	5.0	2.5	10.0
185	0.32	8.0	0.60	8.5	1.0	11.5	—	—	—	—
24	0.93	6.5	1.1	7.0	1.3	8.2	1.6	11.0	—	—
26	1.4	11.0	1.7	13.0	2.1	15.0	—	—	—	—
184	0.70	7.0	0.86	10.0	1.2	12.7	1.7	19.0	—	—
190	0.65	8.0	—	—	0.97	12.5	1.2	14.0	—	—

Table 5 Removal of pyritic sulphur for desulphurization in air of –100 mesh fractions at 400°C

PSOC Sample No.	Pyritic sulphur (%)	Pyritic sulphur removed (%)				
		10 min	15 min	20 min	30 min	60 min
113	2.0	12.5	16.0	40.0	64.0	67.5
116	2.6	46.0	49.6	71.6	77.6	87.6 (85.0)
185	1.6	20.0	35.0 (32.0)	62.0 (55.0)	—	—
24	1.9	47.9 (42.5)	58.2 (52.5)	67.0 (62.5)	81.4 (74.0)	—
26	4.2	34.5 (28.0)	42.0 (34.0)	49.0 (41.0)	—	—
184	1.4	50.0 (42.0)	61.4 (49.0)	89.3 (73.0)	120 (96.0)	—
190	1.0	62.0 (48.0)	—	92.4 (70.5)	110.0 (90.0)	—

relatively slow and at least counterbalanced by chemisorption of oxygen on the coal. Therefore, to a close approximation weight loss can be attributed to oxidation of pyritic sulphur and VM release. As seen from Table 3, oxidation at 350°C does reduce the amount of sulphur in the coals. However, the extent of reduction for this particle size (or the two smaller sizes which were also studied) at 350°C is not considered sufficient to warrant commercial interest.

Results on oxidation runs at 400°C on –100 mesh samples are summarized in Tables 4 and 5. The extent of sulphur removal for these runs was estimated by absorption of the effluent gases in acidified dichromate. The flow of air was switched to nitrogen for 10 min at the conclusion of each oxidation cycle to ensure that all the sulphur dioxide evolved during oxidation was absorbed in the acidified dichromate. Insignificant weight losses in the coals occurred during the nitrogen purges. In Table 5, pyritic sulphur removal has been calculated on two bases: (1) that all the sulphur removed from coal is pyritic, and (2) that pyritic and organic sulphur are both removed. The amount of organic sulphur removed was estimated as equal to the weight loss of the coal (other than sulphur) times the fraction of the coal which was organic sulphur. This assumes that removal of organic sulphur is contingent upon removal of volatile matter and carbon gasification. Values for pyritic sulphur removal based on this latter assumption are shown in parentheses in Table 5, for cases where a significant difference in figures calculated on the two bases exists. The actual amount of pyritic sulphur removed is expected to be between the two values given in Table 5. Significant variations in the ease of removal of pyritic sulphur from the different coals are noted.

The effect of particle size on the extent of pyritic sulphur removal was studied at 400°C. Two additional sizes, 40 X 70 and 70 X 100, were used for this purpose. Oxidation was carried out for 15 min in this study. Results are shown in Table 6. Sulphur removed was estimated by titrating the absorbed gases. In general, greater removal of pyritic sulphur is accomplished by reduction in particle size. This suggests that the removal of pyritic sulphur, in part, is controlled by the diffusional resistance of oxygen into the microporous structure of the coals. This is in general agreement with reactions involving solid–gas interfaces. The effect of linear gas (air) flow rate was not studied. It is anticipated that a better removal will also be accomplished at higher flow rates.

Vestal and Johnston¹⁸ have recently studied the kinetics of coal desulphurization by hydrogen under non-isothermal conditions. They observed that organic and pyritic sulphur in coals can be removed by hydrogen treatment; however, the complete removal was only possible at about 1000°C. They classified the organic sulphur occurring in coals into three forms. The first two forms show differential removal peaks at 350 and 450°C, respectively. The pyritic sulphur shows the first peak at about 500°C. Desulphurization by pure hydrogen at 400°C for 15 min was studied on –100 mesh samples. Table 7 shows that little reduction in total sulphur was accomplished. Low sulphur removal at 400°C by hydrogen is in agreement with the findings of Vestal and Johnston. It is likely that only the first type of organic sulphur was removed.

Blum and Cindea¹⁶ reported good success when carrying out desulphurization at 380°C using an air/steam mixture in a 15/85 ratio. We studied the effect on desulphurization

Table 6 Effect of particle size on the extent of pyritic sulphur removal at 400°C for 15 min

PSOC Sample No.	Pyritic sulphur removed (%)		
	40 x 70	70 x 100	-100
113	6.8	10.0	16.0
116	10.1	21.0	49.6
185	35.0 (31.0)	47.5 (41.5)	35.6 (32.0)
24	20.2 (16.5)	39.5 (35.0)	58.2 (52.5)
26	28.5 (24.0)	42.8 (36.5)	42.0 (34.0)
184	55.3 (43.4)	65.4 (53.0)	61.4 (49.0)
190	45.0 (32.5)	64.7 (50.0)	—

Table 7 Desulphurization in hydrogen of -100 mesh fractions at 400°C for 15 min

PSOC Sample No.	S _{tot} (%)		Wt loss (%)
	Before	After	
113	3.0	2.5	2.7
116	4.1	3.7	2.5
185	3.3	2.9	5.5
24	3.8	3.1	5.0
26	7.0	6.1	6.2
184	3.2	2.5	5.0
190	3.1	2.8	5.5

by dry and humid air at 400°C. The humid stream contained about 4% moisture by volume. No discernible improvement in sulphur removal using humidified air instead of dry air was noted.

Desulphurization runs on -100 mesh material were conducted at 450°C for 10 min. Extent of removal of pyritic sulphur and weight losses are recorded in Table 8. A com-

Table 8 Removal of pyritic sulphur for desulphurization in air of -100 mesh fractions at 450°C for 10 min

PSOC Sample No.	Pyritic sulphur removed (%)	Weight loss (%)
113	88.2 (86.0)	4.5
116	77.0 (73.5)	5.5
185	108.0 (93.7)	13.5
24	89.0 (77.3)	10.0
26	52.4 (45.2)	16.7
184	107.0 (95.7)	12.0
190	128.0 (~100)	16.5

Table 9 Volatile matter and calorific value of original and oxidized coal samples following desulphurization at 400 and 450°C

PSOC Sample No.	Dry basis					
	Orig.	Volatile matter (%)		Orig.	Calorific value (Btu/lb)	
		400°C (15 min)	450°C (10 min)		400°C (15 min)	450°C (10 min)
113	18.1	21.4	17.0	13 745	12 142	12 580
116	20.6	21.6	18.2	13 233	11 336	12 064
185	38.0	25.9	21.8	12 126	9 974	10 242
24	36.9	33.6	22.7	12 826	10 574	11 098
26	40.7	26.0	24.5	12 773	10 278	9 250
184	34.5	19.2	17.1	11 181	—	—
190	29.4	25.7	23.5	11 960	10 320	10 770

parison of this table with Tables 4 and 5 indicates that much more pyritic sulphur is removed in 10 min at 450°C than in 15 min at 400°C. Volatile matter and calorific values for the coals before and after oxidation runs at 400 and 450°C for exposure times of 15 and 10 min, respectively, are presented in Table 9. The general trend is a decrease in both the VM and the calorific value following oxidation. However, the VM for samples 113 and 116 increased following treatment at 400°C, since chemisorption of oxygen on these lower-VM samples outweighed the loss of volatile matter. The calorific value following oxidation at 450°C is generally higher than at 400°C, probably due to the chemisorption of more oxygen at 400°C than at 450°C.

It is of interest to investigate whether the level of sulphur dioxide concentration in the effluent gases can be increased by recycling the hot gases over a fresh charge of coal without adversely affecting the extent of sulphur removal. A rich stream of sulphur dioxide can directly be used for sulphuric acid manufacture. Thus, a study on oxidative desulphurization by air containing 5 and 10% sulphur dioxide by volume was made. The oxidized samples were analysed for total sulphur. Table 10 summarizes the results.

Table 10 Desulphurization of -100 mesh samples by SO₂-enriched air at 450°C for 10 min

PSOC Sample No.	Total sulphur in treated samples		
	Air	5% SO ₂ -95% air	10% SO ₂ -90% air
113	1.2	1.2	1.3
116	2.2	2.2	2.0
185	1.8	1.7	1.8
24	2.3	2.4	2.4
26	5.0	4.2	3.8
184	1.9	1.5	1.9
190	2.0	2.0	2.0

In the desulphurization runs with 5 and 10% sulphur dioxide in air, the coal samples underwent weight losses similar to those in air alone. Further, the extent of desulphurization is not decreased by the presence of large amounts of sulphur dioxide in the desulphurizing gas. In fact, results on sample 26 show enhanced desulphurization in the presence of sulphur dioxide, which is not understood at this time. It is clear that the sulphur dioxide concentration in the effluent

can be increased to levels suitable for sulphuric acid manufacture by successive recycling.

It emerges from our studies at 350, 400 and 450°C that the desulphurization efficiency increases with increasing temperature. Desulphurization at about 550°C, using a lower oxidation time, was briefly studied by dropping –100 mesh coal through a vertical furnace. The residence time was estimated as 0.4 s. A stream of air flowed through the furnace at 300 cc/min (NTP). Little reduction of sulphur was achieved under these conditions. It is thought that this result, in part, is due to the difficulty which oxygen will have in penetrating into the interior of the coal particle during the diffusion of volatile matter out of the particle.

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