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Biodesulfurization of Coals of Different Rank: Effect on Combustion Behavior

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The emission of sulfur oxides during the combustion of coal is one of the causes, among other air pollution problems, of acid rain. The contribution of coal as the mainstay of power production will be determined by whether its environmental performance is equal or superior to other supply options. In this context, desulfurization of coal before combustion by biological methods was studied. Four Spanish high-sulfur content coals of different rank were inoculated with bacteria isolated from mine-drainage waters and with naturally occurring bacteria inherent in the coals to be treated. Higher levels of desulfurization were obtained in the case of the samples treated with their own accompanying bacteria and when aeration was increased. All the samples were amenable to the biodepyritization processes. However, it is of little value to achieve large sulfur reductions if a decrease in coal combustion performance is obtained in the process. For this reason, a comparison was made between the combustibility characteristics of the original coals and those of the biodesulfurized samples. Results indicated that combustibility was not substantially modified by the overall biological treatment. The benefits of reduced sulfur emissions into the atmosphere ought to be taken into account as part of the general evaluation of the processes.

Introduction

Increasing concern regarding the environmental changes caused by pollution emissions has prompted the adoption of more stringent legislation in all developed countries. The combustion of high-sulfur content coal has been identified as a major source of acid rain, particularly from electricity generation and industrial boilers (1). In the United States, the 1990 Clean Air Act Amendments and in Europe the Large Combustion Plant Directive of 1988 set overall sulfur emission quotas. The Directive called for a reduction in sulfur oxide emissions in Spain of up to 50% by 2003, based on 1980 levels, with stricter controls being forecast for the near future. All over the world there are vast reserves of coal with high levels of sulfur, and this is the case of many of the major Spanish deposits. There is an urgent need, therefore, to find a means of reducing sulfur emissions.

Sulfur emissions can be reduced before combustion by physical, chemical, or biological coal cleaning; during

combustion by sorbent injection; after combustion by flue gas desulfurization; or by the most obvious, though not always possible, method of using low-sulfur coals alone or as components of coal blends. Integrated strategies that take into account several options of sulfur reduction could be also considered, such as combined bacterial and physical coal cleaning (2).

Since the discovery of microorganisms capable of oxidizing and solubilizing coal pyrite (3), intensive research on the applicability of biological processes for sulfur removal has been carried out (4–6). The main process involving coal microbial degradation is the removal of inorganic sulfidic minerals, denoted as pyritic sulfur, although claims have been made on the removal of organic sulfur (7–9) with some researchers having reported 72% removal of organic sulfur by using a bacterial culture designated *Rhodococcus rhodocrous* (10). Genetic engineering has been cited as the possible breakthrough that could considerably enhance the performance of this process (11). The microbial desulfurization of coal has been reviewed by several researchers (12–14); a large-scale demonstration unit has been built and tested in the United States (15), and a pilot-scale plant has been used in Europe (16). Furthermore, the state-of-the-art process has been recently discussed in a symposium on the biological processing of fossil fuels (17).

However, little has been said about the effect of the overall coal biological treatment on the subsequent combustion process (18). In this work, four high-sulfur coals of different ranks, ranging from anthracite to subbituminous, currently used in Spanish power stations were microbially treated. The combustibility characteristics of the parent coals were studied and compared with those of the biodesulfurized coal samples. The main objective of this study was to evaluate the changes in the combustion behavior of coal after exposure to the action of bacterial attack for the removal of pyritic sulfur.

To investigate the thermal behavior of the coal samples, temperature-programmed combustion (TPC), also referred to as burning profiles, and isothermal reactivity tests were employed in this work. Different authors have used characteristic parameters obtained from TPC and isothermal reactivity to predict and compare the combustion behavior of coals. Correlations between TPC results and combustion efficiencies in a drop-tube furnace (19), pilot-scale pulverized coal combustor (20, 21), and industrial scale demonstration boiler (22) have been reported. Similarly, a qualitative reactivity classification of coals obtained by means of isothermal reactivity agreed well with one obtained using an entrained flow reactor (23). These techniques, as stated by Pisupati and Scaroni (24), seem to be quite useful for predicting the relative combustion characteristics of coals when it is difficult or there is not enough sample to test fire in a large installation as is the case in the present work.

Experimental Section

Microbiological Methods. Four different coals varying in rank from anthracite to subbituminous were used in this study, and Table 1 presents analyses of the coals. Proximate analysis and forms of sulfur were made according to ASTM standard tests. For the ultimate analysis, total sulfur was determined in a Leco S144 DR instrument, and carbon, hydrogen, and nitrogen were measured with a Leco CHN600 instrument. Oxygen was calculated by the difference. As the samples experienced different treatments, the nomenclature employed throughout this work is explained in Table 1. The growing of the cultures was carried out in shaken (180 rpm)

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TABLE 1. Main Characteristics of the Parent Coals and Their Biodesulfurized Samples^a

sample ^b	proximate analysis (wt %)			ultimate analysis and forms of sulfur (wt % daf)								total S reduction (%)	pyritic S reduction (%)	CV (MJ/kg, db)
	moisture	ash (db)	volatile matter (db)	C	H	total S	pyritic S	organic S ^c	sulfate S	O ^d				
AN1	0.9	33.0	7.1	87.9	3.6	4.4	2.9	1.4	0.1	2.7				22.6
AN2	1.4	30.0	7.8	88.1	3.6	3.6	2.2	1.4	0	3.3	18.2	24.8		23.6
AN4	1.2	31.2	7.0	88.4	3.6	3.0	1.6	1.4	0	3.6	32.6	45.9		23.0
AN5	1.3	31.5	7.4	88.3	3.6	2.7	1.1	1.6	0	3.9	38.5	62.1		23.0
AN6	1.3	31.9	7.3	88.7	3.7	2.1	0.8	1.3	0	4.1	51.8	72.4		22.9
AN7	1.4	32.3	7.4	87.2	3.6	1.9	0.3	1.6	0	5.9	56.2	91.4		22.8
AN8	1.4	30.5	7.8	88.2	4.1	1.9	0.3	1.6	0	4.1	57.7	89.2		23.4
HV1	0.8	39.7	10.3	87.2	4.0	3.1	2.1	1.0	0	3.6				20.0
HV2	1.2	34.1	8.3	86.3	3.9	2.4	1.2	1.1	0.1	5.2	21.9	40.9		21.9
HV3	1.2	33.4	8.3	87.2	3.9	1.8	0.6	1.1	0.1	5.0	41.9	71.5		21.9
HM1	1.3	8.4	34.8	84.5	5.5	1.8	0.8	1.0	0	6.1				32.1
HM2	1.4	3.6	34.4	84.6	5.6	1.7	0.7	1.0	0	6.1	7.8	11.7		33.6
HM3	1.5	3.5	34.5	84.4	5.5	1.6	0.6	1.0	0	6.4	13.1	26.8		33.6
HM4	1.5	3.2	34.6	84.5	5.5	1.4	0.3	1.1	0	6.5	20.2	58.1		33.7
SU1	5.1	34.9	32.4	61.1	4.7	15.0	5.9	7.0	2.1	18.2				15.6
SU2	4.7	31.6	31.9	63.8	4.5	12.2	4.7	6.7	0.8	18.4	18.7	21.0		17.2
SU4	3.6	26.1	33.7	65.4	4.8	8.4	2.1	6.0	0.3	20.8	44.2	65.2		19.5
SU5	3.6	27.3	33.4	64.6	4.8	7.8	1.5	6.0	0.3	22.2	47.8	74.4		19.1
SU7	3.7	26.9	33.5	64.8	4.7	7.4	1.0	6.0	0.4	22.5	50.7	83.4		19.1

^a db, dry basis; daf, dry and ash free basis. ^b The numbers in the sample column in the table denote the following: 1, parent coal; 2, coal treated in nutrient medium without inoculum for 12 days; 3, inoculated with a bacterial culture isolated from mine-drainage waters (12 days); 4, inoculated with bacterial culture inherent in the coal (12 days); 5, inoculated with bacterial culture inherent in the coal and with additional aeration (12 days); 6, inoculated with bacterial culture inherent in the coal and with additional aeration (19 days); 7, inoculated with bacterial culture inherent in the coal and with additional aeration (25 days); 8, inoculated with bacterial culture inherent in the coal (12 days) and treated in fluidized bed. ^c By difference, i.e., organic sulfur = [total sulfur - (pyritic sulfur + sulfate sulfur)]. ^d By difference, i.e., oxygen (daf) = 100 - (carbon + hydrogen + nitrogen + total sulfur).

100-mL Erlenmeyer flasks. Two types of inocula were used; the first inoculum was derived from the acid drainage of a coal mine, and 10 mL was added to a shake-flask containing 50 mL of Silverman's 9K nutrient medium (25) with 9 g/L FeSO₄ at a temperature of 30 °C and pH 2. After 10 days, a culture rich in bacteria (10⁸ cells/mL) was obtained. Prior to being used, this culture was allowed to acclimatize to the particular coal for a period of 12 days at a pulp density of 15% w/w. The second inoculum was prepared with bacteria inherent in the coals to be treated. In this case, a small amount of coal (100–200 mg) was added to a shake-flask in the same conditions as above. Successive sowings were done by adding 1 mL of the culture to 50 mL of the coal pulp (15% w/w), and the procedure finishes after a bacterial density of 10⁸ cells/mL is obtained.

The desulfurization process was performed in 2-L stirred flasks by adding 10 mL of culture to 1 L of coal pulp with a density of 15% w/w. In the samples denoted as aerated, additional air was supplied at a flow rate of 0.5 L/min. The AN8 sample was biodepyritized in a cylindrical methacrylate fluidized column of 115 cm height and 7 cm i.d. Approximately 2.5 L of coal slurry (15% w/w) was charged into the column and inoculated with 250 mL of the culture. An air flow rate of 500 L/h was used.

Prior to the analyses, the samples were washed with 5 N HCl at 60 °C in order to remove soluble sulfates adhering to the coal grains. Then the samples were filtered under vacuum, and the wet cake was washed with 0.1 N HCl and finally with deionized water.

Temperature-Programmed Combustion (TPC) and Char Isothermal Reactivity Procedures. Coal combustion profiles and char isothermal reactivities to oxygen were obtained using a Setaram TAG 24 differential thermal analyzer. It is well-known that the use of different equipment and conditions of operation lead to significant differences in the results obtained by TGA. Therefore, TPC tests were carried out in this work with 25 ± 0.3 mg of sample, heated to 900 °C at a heating rate of 15 °C min⁻¹ in air flowing at 50 cm³ min⁻¹.

From the combustion profiles, various characteristic parameters can be obtained (26) and are defined as follows: T_v is the volatile matter initiation temperature at which the rate of mass loss is 0.01% s⁻¹ after the loss of moisture and oxygen chemisorption; T_p is the peak temperature at which the maximum rate of mass loss occurs; T_b is the char burnout temperature at which the rate of mass loss is again 0.01% s⁻¹; T_{50} is the temperature of 50% burnoff of organic material (daf); R_p is the rate of mass loss value at the peak temperature. In addition, from the DTA curve that represents the evolution of heat flow during the test, two temperatures can be calculated (27): T_{sh} is the temperature of self-heating at which the rate of heat flow, in the DTA curve, begins to be exothermic; T_{ec} is the end of combustion temperature at which the rate of heat flow is zero.

Isothermal reactivities of the coal samples were carried out using a method based on that described by Jenkins et al. (28) involving the heating of the coal sample in 50 cm³ min⁻¹ of nitrogen to 850 °C at a heating rate of 15 °C min⁻¹. After weight stabilization, the char formed was cooled to 500 °C, and isothermal combustion of the char sample was started by changing the nitrogen for air at the same flow rate. The char reactivity is defined as follows:

$$R = -\frac{1}{W_0} \frac{dW}{dt} = \frac{dX}{dt}$$

where W_0 is the initial mass of char; W is the mass of char at each instant, on an ash-free basis; and X is the conversion at time t , defined by

$$X = \frac{W_0 - W}{W_0}$$

Results and Discussion

Effect of Biodesulfurization on Sulfur Reduction. The reduction percentages of total and pyritic sulfur achieved

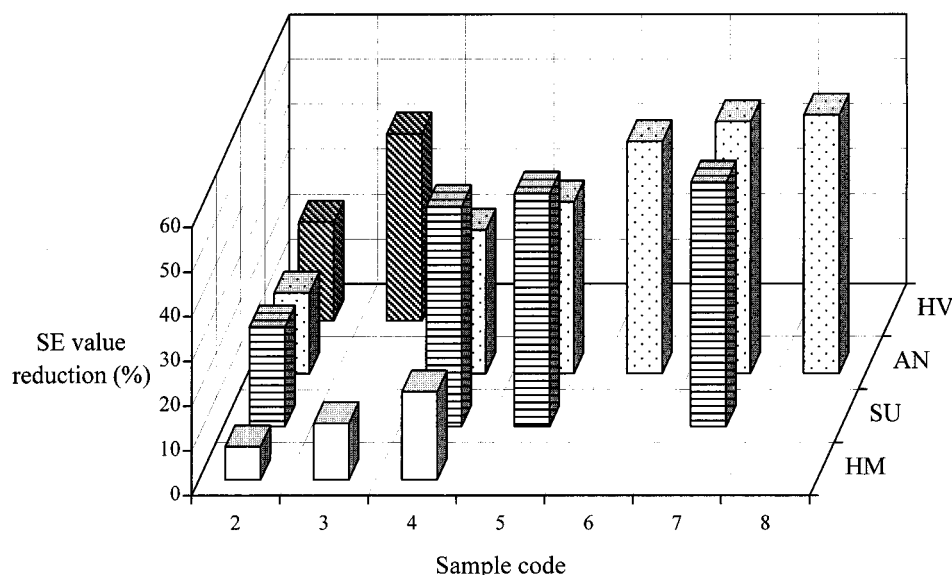


FIGURE 1. Sulfur emission reduction levels of the different coal series.

after the biological treatment of the coals are shown in Table 1. Relatively large reductions were observed in the samples that were not inoculated (AN2, HV2, HM2, and SU2). This could lead to the conclusion that significant sulfur reduction was achieved solely by the use of an acidic medium. However, previous studies that have used sterilized and nonsterilized coal samples (29) have demonstrated that bacteria inherent in the coal exerts an important desulfurizing action, even when coal was not inoculated. Another feature that can be inferred from the results of samples HM3 and HM4 is the higher sulfur reduction levels obtained by coal HM4, produced by using an inoculum derived from its parent coal HM1. The advantages of this procedure over that of using pure cultures are the immediate adaptation of the micro-organism to the coal and the reduced period of latency in the inoculum (30). In addition, the use of an enriched culture as an inoculum, obtained from the coal itself, could be of special interest for application in coal heaps in the open air.

From the results in Table 1, no general conclusion can be drawn with regard to the effect of coal rank on the treatability of coal. However, a significant feature of the results shown in Table 1 is that all of the coals are amenable to biodesulfurization, independent of coal rank and sulfur content. This is in contrast to other types of coal desulfurization such as density and froth flotation processes in which the efficiency often varies depending on the particular coal. Furthermore, it has been stated that biodesulfurization depends on the amount of sulfur in the coal, its composition, and its degree of distribution throughout the coal (31). In this work, it can be seen in Table 1 that, in general, the larger the amount of pyritic sulfur in the parent coal, the higher the mass of pyritic sulfur removed. For example, higher total and pyritic sulfur reductions were reached by the subbituminous coal series when compared with similar treated samples of the anthracite coal series, an exception to this trend being the pyritic sulfur reduction of samples AN7 and SU7 obtained after 25 days. But even in this case the total mass of sulfur removed from sample SU7 exceeded the amount removed from the anthracite sample AN7. The results, therefore, highlight the considerable potential of biodepyritization, particularly for coals with a high pyritic sulfur content in accordance with the results obtained by many other researchers (8, 32).

For environmental purposes, it is useful to relate sulfur to the heating value of the coal. Sulfur contents were therefore expressed on a thermal basis as sulfur emission values (g of sulfur/GJ) and were calculated assuming that all sulfur was

released, after combustion, as sulfur dioxide. It needs to be pointed out however that, during coal combustion, part of the sulfur dioxide may be retained in the coal ash in a natural way (33), thus reducing the theoretically calculated sulfur emission values.

Figure 1 shows the reduction percentages of sulfur emission (SE) values for the different coal series. An increase in the time of treatment caused, as it could be expected, an increase in the reduction of SE values (see samples AN5, AN6, and AN7). Nevertheless, even at the lowest residence time of 12 days, reduction levels of 38.4% for sample AN5 and of 52.2% for sample SU5 were obtained. This compares favorably with the reductions achieved in normal coal preparation plants, which lie between 20% and 30% according to Abbot and Jenkinson (34). In a test performed at larger scale for the anthracite in the fluidized bed reactor previously described, an improvement of nearly 20 points in SE reduction was obtained for the sample AN8 in comparison with that reached by AN5, which was obtained in a stirred flask during the same period of time of 12 days. This agrees with the conclusions obtained from others (16), who found that pilot-scale tests gave higher removal levels than those suggested from smaller scale tests (shake-flask). The biodesulfurization time has important implications when considering the size of a full-scale plant since the required capacity is proportional to the residence time for a given throughput.

Combustibility Behavior of the Biodesulfurized Coals.

Once different biotreated samples were produced, the effect of the overall desulfurization process on coal combustion properties was evaluated. The temperature-programmed combustion profiles for the parent coals are shown in Figure 2. It can be seen that these profiles have quite different characteristics in consonance with the varied rank of the coals. The mass loss at about 100 °C is due to water loss with higher values in the case of the lower rank coal SU1. Thereafter, there is a net gain in mass that takes place at lower temperatures as the coal rank decreases. This is caused by oxygen chemisorption of the organic matter and was not observed in the subbituminous coal due to its already high oxygen content, as indicated in the ultimate analysis presented in Table 1. In addition, coal SU1 also presents two peaks at about 360 and 400 °C before the major one. This is characteristic of low-rank coals and is attributed for some authors to the release of volatile matter (35). After this event, the rate of mass loss reaches a maximum at the peak

TABLE 2. Characteristics Parameters from the Combustion Profiles of the Parent Coals and Their Biodesulfurized Samples^a

coal	T_v (°C)	T_p (°C)	T_b (°C)	T_{50} (°C)	R_p (% s ⁻¹)	T_{sh} (°C)	T_{ec} (°C)
AN1	437	595	789	624	0.103	275	805
AN2	410	590	742	594	0.104	281	749
AN4	415	594	797	616	0.092	271	810
AN5	408	585	787	607	0.089	241	806
AN6	403	590	741	594	0.104	239	750
AN7	397	584	735	587	0.104	230	748
AN8	401	581	775	599	0.093	234	791
HV1	425	586	745	607	0.121	244	751
HV2	380	588	767	597	0.098	226	800
HV3	404	585	762	603	0.112	202	786
HM1	330	523	765	561	0.084	177	773
HM2	322	507	720	533	0.094	182	766
HM3	330	524	782	567	0.079	178	800
HM4	335	536	796	569	0.080	185	812
SU1	254	478	571	431	0.108	199	609
SU2	236	479	591	434	0.108	204	622
SU4	250	428	595	440	0.105	203	629
SU5	236	403	584	422	0.100	196	649
SU7	235	428	591	436	0.107	197	637

^a Codes of the samples are the same as in Table 1. T_v , volatile matter initiation temperature; T_p , peak temperature; T_b , char burnout temperature; T_{50} , temperature of 50% burnoff of organic material; R_p , rate of mass loss at the peak temperature; T_{sh} , temperature of self-heating; T_{ec} , end of combustion temperature.

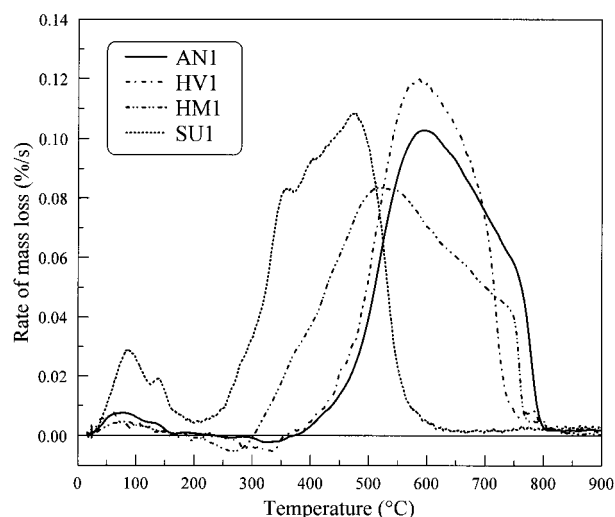


FIGURE 2. Combustion profiles of the parent coals used in the desulfurization processes.

temperature (T_p), which increases with coal rank and presents values of 478, 523, 585, and 595 °C for SU1, HM1, HV1, and AN1, respectively. The peak temperatures of the coals studied in this work are in good agreement with the values found by other authors under similar experimental conditions (26, 36). A peculiar characteristic exhibited by coal HM1 that is not present in the other coals is the delay in combustion rate after the peak temperature. This fact has been attributed to coals that swell and form an impermeable mass that reduces oxygen accessibility (37). The parent coal HM1 is a highly agglomerating coal, as indicated by a value of 7 in its free-swelling index (38).

The combustion profile parameters of the biodesulfurized coal samples are presented in Table 2. As for the effect of the biological treatment on the peak temperatures, it can be seen in Table 2 that the higher rank coal series AN and HV are little affected, with a nearly constant value for the HV series of 587 °C. In the anthracite series, there is a slight decrease in the samples that were aerated. More important are the variations in the subbituminous coals. Lower peak temperatures are indicative of more reactive coals, and it

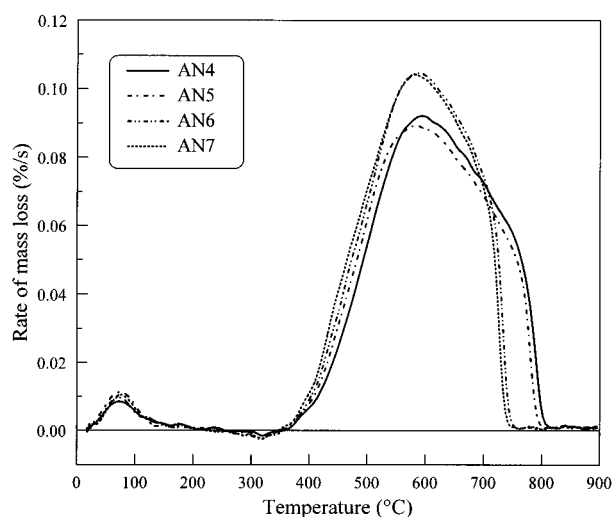


FIGURE 3. Combustion profiles of the anthracite coal series treated for different periods of time.

has been found that more oxidized coals are more reactive (36, 39).

Aeration during biological treatment produced important changes in the combustibility of the anthracite series, as can be observed in Figure 3. There was a displacement of the burnout temperatures toward higher values with a decrease in the time of aeration. Moreover, sample AN4 that was not aerated presented the highest values of burnout and initiation temperatures, as shown in Figure 3. A difference of 62 °C was obtained between the burnout temperature of sample AN4 and that of AN7, the latter having been aerated for 25 days. The lower values of the burnout temperature are indicative of lower residence times in order to achieve a complete combustion and consequently less unburnt carbon in the fly ashes.

Another important parameter, the self-heating temperature, evaluated in this work from differential thermal analysis (DTA), is often regarded as an indicator of the susceptibility of coal to self-heating and spontaneous ignition. It has been observed to increase with decreasing coal rank, with lignite and subbituminous coals being more susceptible to self-

TABLE 3. Reactivities in Air at 500 °C of the Coal Samples Measured at Various Char Conversion Degrees, in Units of $\text{min}^{-1} \times 100^a$

coal	R_{20}	R_{50}	R_{max}
AN1	2.4	1.6	2.4
AN2	3.5	3.1	3.6
AN4	2.4	1.9	2.4
AN5	2.5	2.1	2.6
AN6	2.8	2.6	3.0
AN7	2.5	2.3	2.6
AN8	2.7	2.4	2.7
HV1	1.6	1.1	1.6
HV2	1.3	1.0	1.3
HV3	1.4	1.0	1.4
HM1	1.6	1.2	1.6
HM2	1.6	1.4	1.7
HM3	1.5	1.4	1.6
HM4	1.5	1.4	1.5
SU1	8.2	6.2	8.2
SU2	5.3	4.3	5.3
SU4	5.2	3.8	5.2
SU5	5.0	3.6	5.0
SU7	4.1	3.4	4.2

^a Codes of the samples are the same as in Table 1. R_{20} , reactivity value at 20% char conversion; R_{50} , reactivity value at 50% char conversion; R_{max} , maximum reactivity value.

heating than bituminous coals and anthracites. The values given in Table 2 indicated a marked effect of biodesulfurization on the T_{sh} temperatures of the anthracite series as well as in the semianthracite series, with an important decrease in this parameter in many samples. Once again, the samples that experienced aeration in the anthracite series were the most affected, an increase in aeration time entailing correspondingly lower values of self-heating temperature. This means that the risk of autogenous heating is greater for the anthracite- and semianthracite-treated samples. Nevertheless, these temperature values are still relatively high due to the rank of the parent coals.

Assessment of Chars Combustibility by Isothermal Gasification. Heterogeneous char combustion is the slowest step in the overall coal combustion process and, therefore, has a great influence on coal burning behavior. The rate at which char oxidation proceeds has an important effect on the amount of unburned carbon in fly ash. In this work, the reactivities of the chars produced from the parent coals and their biotreated samples were measured in air at 500 °C. Under these conditions, char oxidation is chemically controlled, and the overall rates of combustion will depend on the reactivity of the chars (40).

The most relevant reactivity parameters for the different coal series are summarized in Table 3, where R_x is the reactivity measured at x percent char conversion, and R_{max} is the maximum reactivity value. From the data of Table 3, it appeared that the influence of the biodesulfurization processes on char reactivities was less for the intermediate rank coals than for the anthracite and subbituminous coal series, following a similar behavior to the combustion profiles discussed above. In general, slight decreases were observed for the HV and HM coal series in comparison with their parent coals. However, the parent coal SU1 was much more reactive than the rest of its coal series, while the anthracite AN1 followed the reverse trend. This behavior is clearly illustrated in Figure 4 where the reactivities at different char conversion degrees are represented for the anthracite and subbituminous coal series. The rate parameters reflected the heterogeneous char reactivity to oxygen, which is related to char physico-chemical properties and the catalytic effect of mineral impurities. According to this, the lower reactivity of the

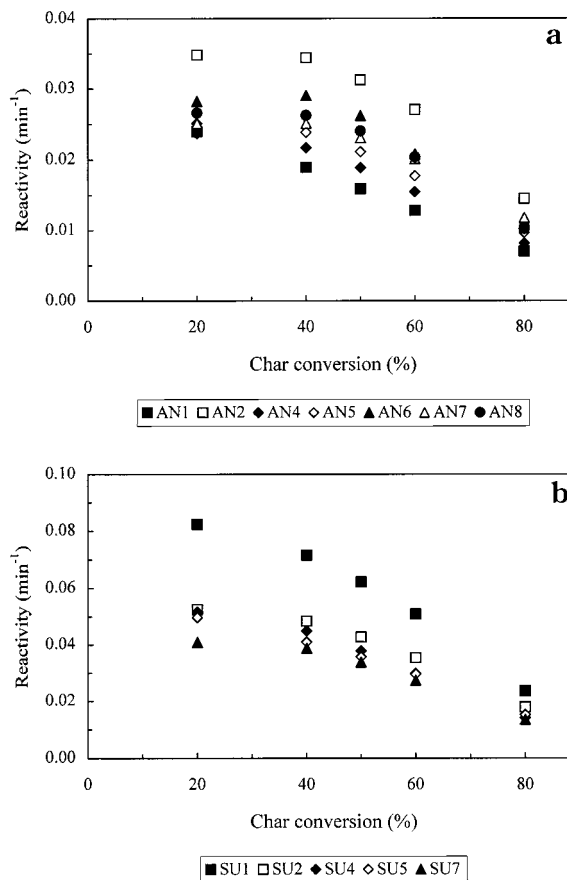


FIGURE 4. Variation of the reactivity at different char conversion degrees for the anthracite (a) and subbituminous (b) coal series.

subbituminous samples in comparison with their parent coal could be due to the marked decrease in their ash content (see Table 1) after the overall desulfurization processes.

From a general point of view, the combustion behavior of biologically desulfurized coals was not substantially affected by the treatment. Only in the case of the subbituminous coal series was a significant reduction in combustibility observed. Nevertheless, even in these samples the combustibility was high enough to guarantee a good combustion behavior. Finally, it should be pointed out that the immediate benefits of reducing sulfur emission levels cancel out and probably outweigh any disadvantage arising from these processes.

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Received for review July 6, 1998. Revised manuscript received October 9, 1998. Accepted November 6, 1998.

ES980679V