

PRODUCTION OF ACTIVATED CHAR FROM ILLINOIS COAL FOR FLUE GAS CLEANUP

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INTRODUCTION

One of the unique properties of activated carbon is that it can remove nearly every impurity found in flue gas including SO₂, NO_x, particulates, mercury, dioxins, furans, heavy metals, volatile organic compounds, and other trace elements [1-6]. No other existing sorbent has that capability. An activated carbon-based process, typically placed after the precipitator and just before the stack, can be used alone or in conjunction with other control methods to remove SO₂ and NO_x from flue gas [7-10]. This technology has been used in Europe and Japan for cleanup of flue gas from both coal combustion and waste incineration. Currently, no U.S. utility employs a carbon-based process to clean flue gas. An ongoing research program [10-19] at the Illinois State Geological Survey (ISGS) has as one of its principal objectives the development of activated char from Illinois coal suitable for cleaning flue gas. This paper summarizes some of our recent efforts to produce activated char for several flue gas cleanup applications, including coal-fired utilities, diesel engine exhaust and waste incinerators.

RESULTS AND DISCUSSION

SO₂ Removal

The initial aim of this study was to identify process conditions for production of activated char with optimal SO₂ removal characteristics [10-16]. Chars with varying pore structure and surface chemistry were prepared from an Illinois hvC bituminous coal (IBC-102) under a wide range of pyrolysis and activation conditions, and tested for their ability to remove SO₂ from simulated flue gas (2500 ppm SO₂, 5% O₂, 10% H₂O, balance He). Table 1 summarizes the results. A novel char preparation method, involving nitric acid treatment followed by thermal desorption of carbon-oxygen (C-O) complexes, was developed to produce activated chars with SO₂ adsorption capacities comparable to those of commercial activated carbons. An attempt was made to relate the observed SO₂ adsorption behavior to the physical and chemical properties of the char. There was no correlation between SO₂ capacity and N₂ BET surface area. A TPD method was used to determine the nature and extent of C-O complexes formed on the char surface. TPD data revealed that SO₂ adsorption was inversely proportional to the amount of stable C-O complex. The formation of stable C-O complex may have served only to occupy carbon sites that were otherwise reactive to SO₂ adsorption. TPD data also revealed that SO₂ adsorption was directly proportional to the number of free adsorption sites on the carbon surface [12-15]. Based on these results, a detailed mechanism for SO₂ removal by carbon has been proposed [19].

NO_x Removal

Carbon can also be used to remove NO_x from flue gas at temperatures between 80 and 150° C. Carbon may act as both a sorbent [20-22] and a catalyst [23-25]; the extent of each depending on the physical/chemical properties of the carbon and flue gas conditions. Sorbent Technologies Corporation (STC) is presently developing a carbon-based process for low-temperature (20-100°C) removal of NO_x from exhaust streams from jet engine test cells and other combustion sources such as diesel engines that would not require the use of ammonia [22]. The carbon filter being developed is a relatively simple NO_x control device that is placed directly into the exhaust duct path. There it substantially removes the NO_x as well as other contaminants such as SO₂, HCl and organics.

The ISGS and STC are working together to develop a carbon for this process. Several carbons prepared at the ISGS were tested by STC. The experimental conditions used were: 10 g of carbon, 1 in. ID fixed bed reactor, 500 ppm NO in air, and a flow rate of 4 L/min. Figure 1 shows that a steam activated Illinois coal char treated with HNO₃ and thermally desorbed at 925°C for 1 h in flowing N₂ (IBC-102, HNO₃-925°C) removed 100% of the NO_x during a one hour test. Recall that this char also adsorbed the greatest amount of SO₂ (Table 1). The NO_x removal efficiency of a similarly treated Calgon F400 carbon (Calgon, HNO₃, 925°C) decreased from 100% to 70% after 1 h. The HNO₃-treated Calgon carbon that was not heat treated at 925°C (Calgon, HNO₃) removed only 10% of the NO_x after 60 minutes illustrating the importance of the thermal desorption step in our char preparation method. Thermal desorption of C-O complexes opens closed porosity and widens pores, but more importantly, creates nascent sites on the carbon surface that react vigorously with NO_x. Figure 2 shows that the NO_x removal performance of the IBC-102, HNO₃-925°C char decreases as flue gas temperature increases from 22°C to 150°C. The NO_x removal efficiencies of IBC-102, HNO₃-treated chars thermally desorbed at 525°C, 725°C and 1075°C were all less than that of the IBC-102, HNO₃-925°C char. It was interesting that the NO_x removal efficiencies of the KOH activated chars listed in Table 1 were comparable to those of the IBC-102, HNO₃-925°C char. These chars contained some leftover potassium, about 0.5% by weight. Potassium has been found to be an excellent catalyst for NO reduction [26-29].

Figure 3 compares the NO_x removal behavior of the IBC-102, HNO₃-925°C char and the Centaur carbon, a catalytic carbon manufactured by the Calgon Carbon Corporation. The HNO₃-925°C char

achieves nearly 100% NO_x removal for the first 8 h compared to only 4 h for the Centaur carbon. For the HNO₃-925°C char, the concentration of NO in the effluent increases from about 3% to 80% between 8 and 15 h, while the Centaur carbon continues to remove 50% of the NO after 20 h. It is interesting to note that NO₂ is generated by both carbons, and that its onset occurs at 15 h (50% of maximum breakthrough). The formation of NO₂ during the low temperature reaction of activated carbon fibers with NO was recently observed [30,31]. It seems that once the carbon surface becomes saturated with C-O, C-N and C-NO functional groups, NO₂ formation is initiated. Eventually, a char would require regeneration as these functional groups accumulate on the carbon surface and block the sites responsible for NO reduction. Preliminary tests suggest that the NO_x removal activity of the spent HNO₃-925°C char can be partially restored by thermal regeneration. Further work is needed to determine the mechanism by which this high activity carbon removes NO_x from flue gas.

Combined SO₂/NO_x Removal

The Research Triangle Institute (RTI) in conjunction with the University of Waterloo developed a low temperature process capable of removing more than 95% of the SO₂ and 75% of the NO_x from coal combustion flue gas [32,33]. The flue gas is cooled to 100°C and the SO₂ is catalytically oxidized to SO₃ by one type of carbon and converted to medium strength H₂SO₄ in a series of periodically flushed trickle-bed reactors. The SO₂ free gas is reheated to 150°C and NH₃ is injected over a different carbon to reduce NO_x to N₂ and H₂O.

In this study, RTI performed SO₂ removal tests on five ISGS chars under conditions simulating those used in the RTI-Waterloo process: 100°C, 2500 ppm SO₂, 5% O₂, 10% H₂O, balance helium, space velocity of 1400 h⁻¹. Each adsorption cycle was followed by a 0.2 L wash with 4.32 N H₂SO₄. The results of nine adsorption/desorption cycles performed with the HNO₃-925°C char are summarized in Table 5. After the first cycle, the 20% breakthrough time (BT), i.e., when 20% of inlet concentration of SO₂ is measured in the effluent, was extended from 3 to 80 minutes, however, BT decreased steadily thereafter. Also, the lowest emission (LE), i.e., the minimum SO₂ concentration detected in the effluent, increased with each subsequent cycle. It should be mentioned that the NO_x removal tests for the HNO₃-925°C char were performed prior to these SO₂ removal tests. The poor performance of this char may be attributed to adsorbed NO, NH₃ and O₂ poisoning SO₂ adsorption sites. Commercial processes typically remove SO₂ before NO_x to avoid production of ammonium sulfate, which can also reduce catalytic activity [3,8]. All subsequent SO₂ removal tests were performed on carbons that were not previously exposed to NO or NH₃. The KOH activated char was also tested (Table 5). A relatively low concentration of SO₂ in the effluent was achieved and the 20% BT was 45 minutes, but after the first cycle, performance decreased dramatically.

Table 6 shows that the SO₂ removal performance of the Centaur carbon was superior to all other carbons tested. Its 5% BT, however, also decreased after the first cycle. The IBC-102, HNO₃-725°C char performed best among the ISGS chars tested. Its initial LE was 10 ppm SO₂ (equivalent to 99% removal efficiency), but this increased to 1200 ppm SO₂ after just the first acid wash. An RTI carbon (not listed in Table 6) showed similar deactivation after the first cycle. The integral feature of the RTI-Waterloo process is its regeneration step involving a water wash with dilute H₂SO₄. One important advantage of doing this instead of thermal regeneration is that it does not consume carbon. Thus, a carbon catalyst in this process could last for many years without having to be replaced. The SO₂ removal performance of all the carbons tested by RTI in this study decreased markedly after the first cycle. Whether a low cost carbon having a high concentration of free sites resistant to poisoning by the dilute acid wash can be developed for this process remains to be determined.

The NO_x removal capabilities of these chars in the presence of ammonia were also tested. The following reaction conditions were used: 40 cm³ carbon, 130-160°C, 500 ppm NO, 690 ppm NH₃, 5% O₂, 10% H₂O and 1400 h⁻¹ space velocity. Table 4 shows that the NO_x removal activity of the HNO₃-925°C char, although highest among the ISGS chars tested, was still significantly lower than that of the carbon catalyst developed and tested by RTI (commercial activated carbon impregnated with a metal catalyst). The RTI carbon, although it achieved better than 99% conversion of NO to N₂, remains quite costly to produce. Table 4 also shows that the five ISGS samples responded differently to variations in reaction temperature. The NO removal activity of the HNO₃-925°C char went through a maximum at 150°C. NO_x conversion with the air-925°C char decreased with increasing temperature, while the steam activated char maintained essentially the same level of activity between 130 and 160°C. The NO_x reduction activity of the HNO₃-725°C char increased from 3% to 22% between 130 and 160°C, whereas that of the KOH activated char decreased from 22% to 7%. The Centaur carbon performed rather poorly at the two temperatures studied. Note that the ISGS chars tested were not representative of chars optimized for this application. Typically, commercial selective catalytic reduction (SCR) processes use higher surface area (1000-1500 m²/g) activated carbons made from bituminous coal. Carbo Tech (Essen, Germany) presently manufactures ton quantities of this carbon for carbon-based SCR processes operating in Europe. Singoredjo et al. [34] recently noticed that NO_x removal with carbon in the presence of ammonia was influenced by a number of factors including the number and type of C-O complexes on the carbon surface, nitrogen content of the char, and accessibility of the pores. The oxygen contents of most of our chars were minimized due to the steam activation or thermal desorption treatments used in their preparation.

Incinerator Flue Gas

Incinerator flue gas typically contains much lower concentrations of SO₂ (20-100 ppm) compared to

those found in coal combustion flue gas (500-3000 ppm), but also contains much higher levels of other pollutants such as mercury, dioxins, furans, heavy metals, and hydrochloric acid. STEAG Aktiengesellschaft (Essen, Germany) has pioneered the cleanup of incinerator flue gas using their patented $\text{I}/\text{a}/\text{c}/\text{t}^{\text{TM}}$ process and a low cost ($< \$300/\text{ton}$), low surface area ($< 300 \text{ m}^2/\text{g}$) activated carbon made from German brown coal [1,2]. A U.S. source of this carbon is currently needed for their processes soon to be installed on waste incinerators in the U.S. A market potential of 80,000 tons/year of activated char (160,000 tons of coal) has been estimated, assuming 10% of U.S. incinerators adopt their technology to meet needs emanating from anticipated regulation of emissions from existing incinerators. This potential market for Illinois coal was of interest to us, so we agreed to produce this type of carbon for STEAG.

The laboratory conditions needed to produce a suitable sorbent from a Colchester (Illinois No. 2) hvC bituminous coal were first identified. Char production runs were performed at the ISGS using a 2 in. ID horizontal tube furnace, 4 in. ID, 4 ft. heated zone, continuous feed rotary tube kiln, and a continuous feed charring oven. With the assistance of Allis Mineral Systems (Milwaukee, WI), the production steps were carried through two levels of scale up, culminating in the production of 610 pounds of activated char in an 18 in. ID, 10 ft. heated zone, externally fired rotary tube kiln. A 1500 pound sample of size-graded bituminous coal having a free swelling index of 4.5 was used as feedstock for the production run. A three-step process, which included preoxidation, pyrolysis and activation, was necessary to process this coal. Details of the conditions used in the production runs are described elsewhere [10,16,17]. Table 7 compares the properties of the ISGS char and the char presently used by STEAG (hereafter referred to as Herdofenkoks). It shows that ISGS char had a N_2 BET surface area of only $110 \text{ m}^2/\text{g}$, but an SO_2 capacity after 4 h greater than that of the Herdofenkoks. The iodine number of the Herdofenkoks was about three times greater than that of ISGS char. (This number relates to the surface area contained in pores greater than 10 \AA .) The mechanical strength of ISGS char, although not as good as Herdofenkoks, was still considered satisfactory for this application. Note that the carbon tetrachloride activity of ISGS char is $51 \text{ mg}/100 \text{ g}$ char, about three times that of the Herdofenkoks. The carbon tetrachloride activity is used as an indicator of carbon performance in vapor phase applications, e.g., VOC removal. Normally, one would not expect a carbon tetrachloride activity to be this high given the N_2 BET surface area of the char ($110 \text{ m}^2/\text{g}$). The observed value of 51 is more typical of carbons having surface areas of $800\text{-}1200 \text{ m}^2/\text{g}$. Evidently, the ISGS char (bituminous coal char activated in CO_2 for 2 h) developed a more extensive microporosity than the Herdofenkoks (brown coal pyrolyzed at 950°C for 0.75 h).

Commercial activated carbons available in the U.S. are believed to be too reactive due to their relatively high surface area ($> 600 \text{ m}^2/\text{g}$) and propensity to adsorb and react with NO_x . The reaction of carbon with adsorbed NO_x is exothermic and can ignite the carbon bed under certain conditions, e.g., in the absence of gas flow. The STEAG $\text{I}/\text{a}/\text{c}/\text{t}^{\text{TM}}$ process requires the use of a low activity char having a surface area less than $300 \text{ m}^2/\text{g}$. A 550 pound sample of low surface area activated char produced in this study was shipped to Essen, Germany where it was installed in a pilot plant unit and subjected to a NO_x self heating test. This involved adsorbing NO_x on the carbon until saturated, shutting off the flow of gas to the adsorber, and measuring the temperature rise of the char bed. The ISGS char passed the test and is the only U.S. material known to have done so. (Figure 1 also shows that the NO_x removal efficiency of this char was less than that of the Herdofenkoks.)

The test unit containing ISGS char was then installed on a slipstream of flue gas from a commercial waste incinerator in Germany. Flue gas velocity through the 800 mm char bed was 0.15 m/s . The ISGS activated char removed more than 99.7% of the dioxins and furans from the incinerator flue gas (Table 8). Also, the mercury, which was present in the inlet gas, was not detected in the exit gas. The removal efficiencies achieved by ISGS char were at least as good as, if not better than, those achieved with Herdofenkoks. The two-week test, however, was not of ample duration to observe complete breakthrough of any of the pollutants listed in Table 8, so there was no information on total adsorption capacity. Typically other pollutants do not breakthrough the bed before SO_2 , so the SO_2 capacity is considered a good measure of the total adsorption capacity of the char. An economic analysis indicates that it would cost between $\$325$ and $\$400$ to produce one ton of ISGS char with a plant designed and constructed to produce 80,000 tons per year, assuming a 20% rate of return on initial investment.

Another potential market for low cost char is the cleanup of coal combustion flue gas. The Mitsui Mining Company Limited (Tokyo, Japan) has developed a combined SO_2/NO_x removal process featuring a two stage adsorber in which flue gas is contacted with activated coke at temperatures between 100 and 200°C [3]. Sulfur oxides are adsorbed onto the activated coke in the first stage resulting in the formation of sulfuric acid. The flue gas moves into the second stage where NO_x reacts with NH_3 to form N_2 and H_2O . The coke from the first stage is thermally regenerated at temperatures between 300 and 500°C . Mitsui has licensed their technology to General Electric and both are working together to develop new markets in the U.S. for their process. (They have also developed a process to clean incinerator flue gas [4].) A carbon having a selling price less than $\$600/\text{ton}$ is needed.

Our low cost char was tested by Mitsui Mining under the following conditions: 140°C , 1000 ppm SO_2 , 200 ppm NO_x . Table 9 compares the properties of ISGS char and Mitsui Mining activated coke. The N_2 BET surface area and SO_2 adsorption capacity of ISGS char is less than that of Mitsui coke. Also, the NO_x removal efficiency of Mitsui coke is significantly greater than that of ISGS char. The surface of the Mitsui coke is said to contain functional groups (C-O, C-N and C-OH) that react more effectively with SO_2 and NO_x . Note that the ISGS char tested by Mitsui Mining was not optimized for this process. These tests were performed only to evaluate the potential of our low cost char in other applications.

SUMMARY

Activated chars were produced from Illinois coal and tested in various flue gas cleanup applications. A low cost, low surface area char was developed for cleanup of incinerator flue gas. Five hundred pounds of the char was tested on a slipstream of flue gas from a commercial incinerator. The char was effective in removing more than 97% of the dioxins and furans present in the flue gas; mercury levels in the effluent were below detectable limits. Higher activity chars that showed excellent potential for both SO₂ and NO_x removal were also produced. The performance of one char compared favorably with that of a commercial carbon catalyst.

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Table 1. Correlation of SO₂ adsorption capacity with surface area and chemisorbed oxygen.

Sample	SO ₂ Capacity ¹ (mg SO ₂ /g char)	N ₂ BET (m ² /g)	CO ₂ BET (m ² /g)	SO ₂ /N ₂ (mg/m ²)	SO ₂ /CO ₂ (mg/m ²)	O ₂ (wt%)	SO ₂ /O ₂
Calgon F400	206	1000	1000	0.21	0.21	0.5	41.2
Calgon Centaur	327	360	...	0.91
IBC-102, N ₂ , 500°C, 0.5 h	19	1.2	270	15.8	0.07	8.8	0.22
IBC-102, N ₂ , 700°C, 0.5 h	33	10	315	3.3	0.10	1.5	2.20
IBC-102, N ₂ , 900°C, 0.5 h	7	1.2	98	5.8	0.07	0.5	1.40
IBC-102 + KOH, N ₂ , 600°C, 0.5 h	157	500	725	0.31	0.22	7.4	2.12
IBC-102 + KOH, N ₂ , 800°C, 0.5 h	176	800	1155	0.22	0.15	5.6	3.14
IBC-102, 500°C, 10% O ₂ , 390°C	13	220	422	0.06	0.03	8.6	0.15
IBC-102, 700°C, 10% O ₂ , 440°C	37	320	490	0.11	0.08	8.9	0.42
IBC-102, 900°C, 10% O ₂ , 500°C	42	230	395	0.18	0.11	5.2	0.81
IBC-102, 900°C, H ₂ O, 860°C	176	220	613	0.80	0.29	1.1	16.0
IBC-102, 900°C, H ₂ O, 860°C; 45% HNO ₃ , 2.5 h, 25°C	...	400	585	0.06	0.04	16.4	...
IBC-102, 900°C, H ₂ O, 860°C; 45% HNO ₃ , desorbed at 525°C	91	460	693	0.20	0.13	5.9	1.54
IBC-102, 900°C, H ₂ O, 860°C; 45% HNO ₃ , desorbed at 725°C	241	500	727	0.48	0.33	1.6	15.0
IBC-102, 900°C, H ₂ O, 860°C; 45% HNO ₃ , desorbed at 925°C	287	550	726	0.05	0.39	0.5	57.4

¹ SO₂ capacity determined after 6 h.

² not determined.

Table 2. SO₂ removal tests.

Cycle	Low Emission (ppm SO ₂)	20% BT (min)
HNO ₃ -925°C		
1	80	3
2	100	80
3	80	60
4	150	25
5	160	35
6	240	30
7	300	20
8	350	15
9	400	10
KOH activated		
1	25	45
2	1800	----

Table 3. SO₂ removal tests.

Cycle	Low Emission (ppm SO ₂)	5% BT (min)
HNO ₃ -725°C		
1	10	60
2	1200	----
Centaur		
1	0	725
2	10	40

Table 4. NO_x reduction with ammonia.

Sample	130°C	140°C	150°C	160°C
HNO ₃ -925°C	20	29	33	30
air-925°C	14	8	3	0
H ₂ O activated	16	14	14	14
HNO ₃ -725°C	3	6	----	22
KOH activated	22	8	7	----
Centaur carbon	0	----	----	4
RTI carbon	99	99	99	99

Table 7. Comparison of Mitsui coke and ISGS char.

Property	Mitsui coke	ISGS char
N ₂ BET surface area (m ² /g)	200	105
CO ₂ BET surface area (m ² /g)	---	142
SO ₂ capacity (mg SO ₂ /g)	100	27.1
DeNO _x efficiency (%)	55	18.8
Bulk density (g/ml)	0.60	0.39
Micro strength (%)	70	48
Ignition point (°C)	420	468

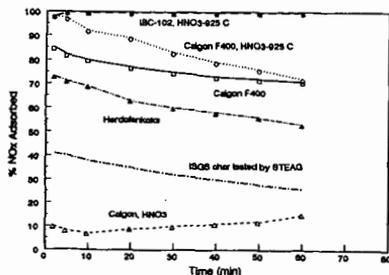


Figure 1. NO_x removal efficiency at 22°C.

Table 5. Comparison of Herdofenkoks and ISGS char.

Property	Herdofenkoks	ISGS char
N ₂ BET Surface Area (m ² /g)	275	110
Iodine Number	349	137
SO ₂ Capacity (wt%, 120°C, 4 h)	2.8	4.2
SO ₂ Capacity (wt%, 120°C, 15 h)	7.6	5.8
Bulk Density (lb/ft ³)	29.8	23.8
Carbon (wt%)	83.1	87.0
Volatile Matter (wt%)	7.7	4.7
Ash (wt%)	8.7	8.3
Mechanical Strength	98.8	78.3
Ignition Point (°C)	405	395
Carbon Tetrachloride Activity (mg/100 mg char)	17	51

Table 6. Pilot plant tests with ISGS char.

Pollutant	Inlet	Outlet	Efficiency (%)
Dioxins, Furans			
Test 1 (ng/m ³)	333.3	0.062	99.98
Test 2 (ng/m ³)	337.9	0.052	99.98
Test 3 (ng/m ³)	282.3	0.789	99.72
Cadmium, Titanium			
Test 1 (mg/m ³)	0.0140	0.0012	91
Test 2 (mg/m ³)	0.0062	0.0012	81
Test 3 (mg/m ³)	0.0052	0.0004	92
Mercury			
Test 1 (mg/m ³)	0.0177	---- ¹	---- ¹
Test 2 (mg/m ³)	0.0384	---- ¹	---- ¹
Test 3 (mg/m ³)	0.0223	---- ¹	---- ¹
Sb, As, Pb, Cr, Co, Cu, Mn, Ni, V, Sn			
Test 1 (mg/m ³)	0.2698	0.0744	72
Test 2 (mg/m ³)	0.0805	0.0347	57
Test 3 (mg/m ³)	0.0634	0.0185	71

¹ below detection limits.

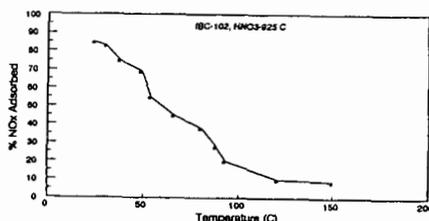


Figure 2. Effect of temperature on NO_x removal efficiency.

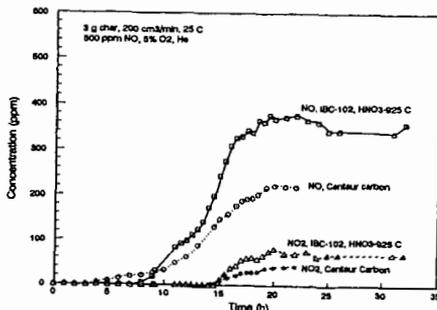


Figure 3. NO_x removal with Centaur carbon and IBC-102, HNO₃-925 C char.