ABSTRACT

One of the primary concerns in coal utilization is the emission of sulfur compounds, especially SO₂. This project deals with catalytic reduction of SO₂ with methane using molybdenum sulfide catalyst supported on different activated carbons: Darco TRS, Norit ROZ-3, and an activated carbon prepared from Illinois coal IBC-110. The work conducted during this quarter has concentrated on catalyst preparation and characterization along with synthesis of activated carbon from IBC-110 coal, as well as, construction of the apparatus for catalytic tests of SO₂ reduction with methane. It was found that Darco TRS supported catalysts have larger surface area than the pure activated carbon, whereas the impregnation of Norit ROZ-3 did not significantly change the BET surface area. Also, the synthesis of activated carbon support from IBC-110 is in progress.
EXECUTIVE SUMMARY

The high-sulfur contents, both pyritic and organic, of Illinois's bituminous coals present significant hurdles for either effective, economical, or environmentally acceptable utilization. Therefore, it is imperative that prior to subjecting the Illinois coal to any process they either have to be physically cleaned or the sulfur compounds found in the flue gases from coal have to be adsorbed or decomposed. The choice of suitable sorbent for flue gas processing depends on many factors such as coal properties, process operating conditions, and flue gas composition. Catalytic process to decompose SO$_2$ is alternative to adsorption techniques.

The goal of this project is to evaluate the capability of molybdenum sulfide supported on an activated carbon to act as a catalyst for SO$_2$ reduction with methane as the reactant. The specific objectives of this project are to: (1) synthesize the catalysts supported on different activated carbons, (2) determine the ability of methane to catalytically reduce sulfur dioxide at a wide range of temperatures, and (3) determine the mechanism of SO$_2$ reduction.

The work conducted during this past quarter includes catalysts preparation and characterization, preparation of activated carbon from IBC-110 coal, and construction of the apparatus for SO$_2$ reduction with methane. Darco TRS and Norit ROZ-3 activated carbons were used as the catalyst supports. MoS$_2$ loadings of 10%, 15%, and 20% were used in the preparation. It was found that the BET N$_2$ surface area of Darco TRS supported catalysts increased during preparation. In contrast, the BET surface area of Norit ROZ-3 support did not change during catalyst preparation. It is suggested that the segregation phenomenon affects the surface area enhancement for Darco TRS support.

The synthesis of activated carbon from IBC-110 coal is in progress. The apparatus for catalytic SO$_2$ reduction with methane was constructed and tested.
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OBJECTIVES

The goal of this project is to evaluate the capability of molybdenum sulfide supported on an activated carbon to act as a catalyst for SO₂ reduction with methane as the reactant. The specific objectives of this project are to:

1. synthesize the catalysts supported on different activated carbons,
2. determine the ability of methane to catalytically reduce sulfur dioxide at a wide range of temperatures, and
3. determine the mechanism of SO₂ reduction.

INTRODUCTION AND BACKGROUND

Removal of sulfur containing gases from effluents of coal processes has received considerable attention in recent years. In a definitive work, Westmoreland and Harrison studied the desulfurization potential of 28 metal oxides. Since then the work has mainly concentrated on only a few metal oxides. Principle attention has been given to Zn, Fe, Cu-based sorbents with some minor attention given to Mn, V, and Ca. In a continuation of their original work, Westmoreland and Harrison established the reaction order between H₂S and selected metal oxides as ZnO > CaO > V₂O₅. The sulfur adsorption capabilities of Zn, Fe, and Mn oxides were studied by Furimsky and Yumura, but Gibson and Harrison showed that the thermodynamic properties of ZnO allow it to adsorb H₂S at 500-700°C and that it exhibits a high theoretical sulfur capacity, 393 gms of S per kg of ZnO. Rao and Kumar also concluded that ZnO is a desirable sorbent for H₂S because of its high reactivity and high equilibrium constant for the ZnO-H₂S reaction and its ability to be regenerated in combination with Fe as zinc ferrite, ZnFe₂O₄, has received attention as a sorbent for H₂S. Grindley found that zinc ferrite, formed by combining the individual oxides, maintains the favorable thermochemistry of ZnO, reacts rapidly with H₂S, and is capable of multiple sulfidation-regeneration cycles. In a more detailed study, Focht, Ranade and Harrison studied zinc ferrite as single cylindrical pellets in a microbalance reactor. They found that zinc ferrite, in the form of ZnO plus Fe₂O₃, is capable of rapid and complete reaction with H₂S in the temperature range 500-700°C. However, in strongly reducing atmospheres and high temperatures, further reduction of Fe₂O₃ to FeO occurs and produces a negative effect on sulfidation kinetics.

Copper oxide, CuO, both alone and supported on various carriers has been studied. Kyotani, Kawashima, Tomita, Palmer, and Furimsky found that CuO alone formed sulfide surface layers, but that CuO dispersed on a support, such as silica, made more efficient use of its sorptive capacity. Additionally, they observed that oxidation of H₂S to SO₂ on admission of hot gases to the fixed bed of sorbent was common to all, but that admission of steam to the hot gases suppressed SO₂ formation. CuO alone and in combination with Fe₂O₃ as well as supported on Al₂O₃ were studied by Tamhankar, Bagajewich, Gavalas, Sharma, and Flytzanis-Stephanopoulos. Comparison of these sorbents was made using breakthrough curves as several sulfidation cycles. They concluded that combination of CuO with alumina may yield...
rather stable and efficient sorbents. This favorable combination of CuO and alumina was confirmed by Sick and Schwerdtfeger\textsuperscript{10}. But superior sorbent activity has been reported recently by Voecks and Sharma\textsuperscript{11} from the use of mixed copper and molybdenum oxides supported in the pores of zeolites. This unique sorbent was found to remove H\textsubscript{2}S from a mixture of gases rich in hydrogen and steam at temperatures from 256-538°C. Zeolites were chosen as support material because of their porosity, rigidity, alumina content, and variety of both composition and form. Most of the sulfidation reactions involve the conversion of CuO to Cu\textsubscript{2}S; molybdenum sulfides were not seen\textsuperscript{11}. Vanadium oxide supported on alumina was studied by Bagajewicz, Tamhankar, Stephanopolus, and Gavalas\textsuperscript{12} at 650-700°C in a flow reactor. In the presence of water vapor, low sulfur capacity was observed. Adsorption of H\textsubscript{2}S, SO\textsubscript{2}, and their mixtures on g-alumina, g-alumina doped with NaOH, sodium Y zeolite, and hydrogen Y zeolite as a part of the Claus reaction has been studied extensively in the pioneering work of Dalla Lama and co-workers\textsuperscript{13}. All catalysts showed physical adsorption of both reactants with strong hydrogen bonding to surface OH groups. This may suggest that the role of the catalyst is primarily to bring the reactants together in suitable orientation. In the classic Claus reaction, hydrogen sulfide is oxidized by oxygen according to Eq. (1):

\[
2\text{H}_2\text{S} + \text{O}_2 \rightarrow \text{S}_2 + 2\text{H}_2\text{O} \quad (1)
\]

The modified Claus process first converts part of the H\textsubscript{2}S to SO\textsubscript{2} and then oxidizes the rest of H\textsubscript{2}S by SO\textsubscript{2}, according to Eq. (2):

\[
2\text{H}_2\text{S} + \text{SO}_2 \rightarrow \frac{3}{2}\text{S}_2 + 2\text{H}_2\text{O} \quad (2)
\]

Subsequently, contributions to explore the mechanism of this reaction came from several groups, e.g. Slager and Amberg\textsuperscript{14}, Förster \textit{et al.}\textsuperscript{15-17}, Karge \textit{et al.}\textsuperscript{18}, Lavaley \textit{et al.}\textsuperscript{19-23}, and Datta \textit{et al.}\textsuperscript{24-26}. Since both H\textsubscript{2}S and SO\textsubscript{2} are constituents of the flue gases, it is interest to take this reaction into consideration in proposed studies.

Recently, advanced separation technologies are being developed to improve the emission control process. Membrane gas separation is one of the technologies under development\textsuperscript{27}. This process becomes a highly efficient alternative to the wet limestone scrubbing. Liquid membranes that exhibit very high species permeability and selectivity are appealing in particular. The liquid membrane called hollow-fiber-contained liquid membrane (HFCLM) has been investigated by Majumdar \textit{et al.}\textsuperscript{28,29} In this configuration, an aqueous liquid functioning as a membrane is kept between two sets of microporous hydrophobic hollow fibers that are tightly packed in a permeator cell.

The reduction of sulfur dioxide with methane is another important reaction that has to be taken into account in the discussion of the treatment of flue gases. The primary reaction between SO\textsubscript{2} and CH\textsubscript{4} is:

\[
2\text{SO}_2 + \text{CH}_4 \rightarrow 2\text{H}_2\text{O} + 2\text{S} + \text{CO}_2 \quad (3)
\]
Along with the primary reaction products, a number of undesired by-products is also possible. These include H₂S, COS, CO, and elemental carbon. Therefore, an effective catalyst for this reaction system is one that has high selectivity for elemental sulfur and carbon dioxide. The reduction of SO₂ was implemented in industry which used alumina as a catalyst. Mulligan and Berk examined the use of pure crystalline MoS₂, WS₂, and FeS as catalyst for the same reaction. These authors found that MoS₂ supported on alumina has a high selectivity for sulfur and CO₂, and good stability at high temperature.

**EXPERIMENTAL PROCEDURE**

**Materials**

The activated carbon samples (DARCO TRS and Norit ROZ-3) were obtained from the American Norit Company, Inc. at Atlanta, GE. These sorbents are thermally activated from lignite coal and wood, respectively.

**Catalysts Preparation**

The molybdenum sulfide (MoS₂) catalysts supported on activated carbons were prepared by impregnation of the activated carbons with the solution of ammonium molybdate. Three different loadings were prepared: 10%, 15%, and 20%. To determine the appropriate loadings for the catalysts, the following information was determined: (a) For every 1235.86 grams of ammonium molybdate(VI) tetrahydrate there are 671.58 grams of molybdenum. This was determined by the molecular weights and compositions. Ammonium molybdate molecular weight is equal to 1235.86, and the chemical composition of ammonium molybdate is \((\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot4\text{H}_2\text{O}\). The molecular weight of molybdenum is 95.94. (b) 5 grams of each catalyst were synthesized. (c) From this information the appropriate amount of ammonium molybdate was determined for each individual loading.

For the 20% loading, 1 gram of molybdenum was needed for each 5 grams of carbon. To obtain this amount of molybdenum, 1.840 grams of ammonium molybdate was used. This value was calculated using Eq. 4:

\[
\frac{(x)\text{ grams of } \text{AM}}{1235.86\text{ grams of AM total}} = \frac{(1.0)\text{ grams of Mo}}{671.58\text{ grams of Mo total}}
\]

where _ is equal to the amount of ammonium molybdate (AM) needed. The 1.0 gram of Mo represents the desired amount of molybdenum for the appropriate 20% loading. Table 1 shows the data necessary for all Mo loadings:
Tables 2 and 3 show the actual numbers obtained when the individual samples were weighed:

Table 1. Calculated % Molybdenum Loading Values

<table>
<thead>
<tr>
<th>Loadings (%)</th>
<th>Activated Carbon (g)</th>
<th>Molybdenum (g)</th>
<th>Ammonium Molybdate (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>5</td>
<td>1.0</td>
<td>1.840</td>
</tr>
<tr>
<td>15</td>
<td>5</td>
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</tr>
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<td>10</td>
<td>5</td>
<td>0.5</td>
<td>0.920</td>
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Table 2. Darco TRS Actual % Loading Values

<table>
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<th>Loadings (%)</th>
<th>Activated Carbon (g)</th>
<th>Molybdenum (g)</th>
<th>Ammonium Molybdate (g)</th>
</tr>
</thead>
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<tr>
<td>19.992</td>
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<td>0.9996</td>
<td>1.8395</td>
</tr>
<tr>
<td>14.999</td>
<td>5.0005</td>
<td>0.7500</td>
<td>1.3800</td>
</tr>
<tr>
<td>9.992</td>
<td>5.0002</td>
<td>0.4996</td>
<td>0.9194</td>
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</tbody>
</table>

Table 3. Norit ROZ-3 Actual % Loading Values

<table>
<thead>
<tr>
<th>Loadings (%)</th>
<th>Activated Carbon (g)</th>
<th>Molybdenum (g)</th>
<th>Ammonium Molybdate (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>19.999</td>
<td>5.0008</td>
<td>1.0001</td>
<td>1.8404</td>
</tr>
<tr>
<td>15.008</td>
<td>4.9993</td>
<td>0.7503</td>
<td>1.3807</td>
</tr>
<tr>
<td>9.999</td>
<td>5.0022</td>
<td>0.5002</td>
<td>0.9204</td>
</tr>
</tbody>
</table>

After each sample was weighed, the proper amounts of ammonium molybdate were added to 200 ml of double distilled water. When the ammonium molybdate was completely dissolved, the mixture was set on a low stirring speed. While the mixture was being stirred, activated carbon was slowly added. The mixture was then allowed to stir at room temperature for eight hours. After that, the sample was filtered and placed in the oven at 95°C for 16 hrs in order to remove ammonia and water and to dry. When the drying was completed, Mo supported catalyst precursor was gently pulverized by use of a mortar and pestle.

The next step in the process for making the catalysts was the sulfidation. This step entailed putting the MoO₃ and supporting activated carbon into a quartz tube plug-flow reactor. The
catalyst precursor was supported in the quartz tube by glass wool. Only about one-third to one-half of the whole amount of the sample was loaded into the reactor on any given run, so no plugging problems would occur. The reactor was then heated up to 400°C. After the furnace reached the set temperature, a slow flow of 7.3% H2S in H2 was introduced. This flow was continued for a period of 30 min. The reaction that took place was:

\[ \text{MoO}_3 + \text{H}_2\text{S} \rightarrow \text{MoS}_2 + \text{H}_2\text{O} \]

After sulfidation, the reactor was flushed with He at 400°C, for 2 hours. This was done in order to remove the excess of H2S from the activated carbon support. Then the reactor was allowed to cool slowly. When completely cool the catalyst, MoS2 supported on activated carbon was removed and stored.

**Activated Carbon Support from Illinois Coal IBC-110**

Activated carbon sample is prepared according to the procedure developed by Dr. W. O'Brien from the Department of Mechanical Engineering and Energy Processes at SIU-C. The IBC-110 coal from The Illinois Coal Bank is used in this step. The charring and activation process is carried out in a quartz-tube reactor system (1 inch in diameter) surrounded with the heating furnace controlled with an Omega temperature programmer at 600°C and 1000°C, respectively. First, the coal samples are crashed and sieved to isolate the fraction of 100 mesh. Then, devolatilization is performed under flowing N2 at 600°C. Additionally, another devolatilization is performed at 750°C in flowing 2% O2 and N2 mixture and 5% steam in N2. Gas activation process is done using the mixture of 5% steam and 4% of O2 in nitrogen at 750°C.

The preparation of activated carbon from the Illinois basin coal started by riffle-sifting 20 lbs of IBC-110 into 4 equal 5 lbs. samples. The use of riffle-sifting allows for a better sampling of the supplied coal. The next step was to grind the coal to the given size range. One of the 5 lbs. coal lots was chosen, the other three lots were subsequently stored away. The chosen 5 lbs. of coal was then passed through a simple motor driven grinder twice. This ground coal was then sifted to the size range of 60 mesh < 100 mesh. The majority of this coal was too large to go through the 60 mesh, so the remaining coal was run through a ball mill at 45 second intervals and then sifted until the entire lot could be sized. This preparation is in progress.

**Characterization of Activated Carbons and MoS2 Catalysts**

Each activated carbon, as well as, each MoS2 catalyst had a complete characterization performed using a NOVA-1200 BET Surface Area Analyzer. The BET analysis was performed with nitrogen as the sorbent. Approximately 0.2 grams of the catalyst or catalyst's support was loaded into the sample cell. The cell was then set into an outgas station where it was heated to 200°C under flowing nitrogen. This process was carried out for 24 hours to remove air and water that may have adsorbed onto the sample. After the outgassing was
completed, the cell was weighed. Next, the sample cell was placed in the analyzer station, where the BET surface analysis was carried out at the temperature of 77 K.

RESULTS AND DISCUSSION

Apparatus for SO$_2$ reduction with methane

In the present quarter, the system for running SO$_2$ reduction with methane was constructed. Figure 1 shows the schematic diagram of the apparatus. The main part of this system is a quartz fixed-bed catalytic reactor that is 0.5 inch in diameter and 21 inches in length. To facilitate tubing connection and disconnection, Swagelok connectors are used at both inlet and outlet ends of the reactor.

Three main lines flow into the system, each transporting an individual gas for the process. The three gasses are SO$_2$, CH$_4$, and He. Each line will be traced through the system to better show the specifics of the reactor. He gas flows through a pressure regulator, reducing the pressure to 70 psi, into a typical needle valve. After this valve the tubing was reduced from one-half to a one-eighth inch brass line. The one-eighth inch line is connected to a Sierra Side Trak III 0-50 sccm mass flow controller. The controller is then in turn electronically controlled by a Sierra model 920C "blackbox" on channel 1. Channel 1 has a range of 0-150 sccm. The next connection after the mass flow controller is to a straight toggle valve which delivers the flow to a Whitey needle valve. From this needle valve the He combines with the other gases in a series of tees before entering the by-pass valve system.

CH$_4$ entered into a Sierra Side Trak III 0-150 sccm type mass flow controller. This controller electronically connected to the same Sierra model 920C "blackbox" as above on channel 2. Channel 2 has a range of 0-50 sccm. The flow from the mass flow controller passed through a Whitey ball valve, which when open is connected to the other flows in a series of tees.

SO$_2$ lines and metering valves are made from stainless steel. The SO$_2$ is supplied by tank (liquid SO$_2$) having the pressure of 35 psi. The SO$_2$ tank flow was controlled by a single stage needle valve which is connected to a Whitey micrometer vernier valve. From the micrometer vernier valve the gas passed through a capillary tube, approximately one-sixteenth of inch. This line goes to a Sierra Top Trak mass flow meter, with a range of 0-50 smlm. After the meter, the flow mixed with the other gas flows in a series of tees and then connected to the by-pass system.

The by-pass system is made up of two 3-way Whitney ball valves holding each of the two ball valves in either the furnace or by-pass positions controlled the flow. The flow by-passes the furnace, so that the initial concentration of SO$_2$ could be obtained. The connections from the 3-way ball valves to the furnace is Swagelok, however, the material used is Teflon. The Teflon tubing used is one-eighth inch line, which connects to one-half inch Teflon connectors. These connectors are fixed to an one-half inch diameter quartz tube. The quartz tube was plugged at one end with quartz wool to keep the catalyst in place. Quartz wool is utilized due
to the high temperature range, 450-600°C will be used in the catalytic experiments. To operate at and maintain these temperatures a General Signal Lindberg programmable furnace is used. The operating range for this particular furnace is 0-1200°C.

The products of the reaction will be analyzed using GowMac model 550 P gas chromatograph equipped with the thermal conductivity detector (TCD) and 2m Porapak Q stainless steel column.

**Catalysts Characterization**

Table 4 shows the BET surface areas and mean pore diameters of both catalyst supports (Darco TRS and Norit ROZ-3 activated carbons) and MoS₂ catalysts. Additionally, Figures 2-5 show the pore size distribution curves for Darco TRS and MoS₂ impregnated Darco TRS catalysts and Figures 6-9 present the same curves for Norit ROZ-3 and molybdenum sulfide impregnated on Norit ROZ-3.
As shown in Table 4, the surface area of Darco TRS activated carbon increases after impregnation with molybdenum followed by sulfidation. In contrast, the surface area of Norit ROZ-3 activated carbon remains practically constant during catalyst preparation. These two catalyst supports have different mean pore diameters which essentially do not change during impregnation. It may be suggested that a surface segregation takes place during the preparation of MoS₂ catalysts supported on Darco TRS resulting in the formation of the new surface layer of molybdenum sulfide. This may result in the surface area increasing. For Norit ROZ-3 support, which has large surface area, molybdenum sulfide is probably located inside the pores as clusters, without affecting the total surface area of the support.

Segregation in catalysis is an important phenomenon. Its influence on catalytic activity and selectivity has been demonstrated for both metallic and nonmetallic systems. Segregation controls the catalytic site density related to anion vacancies, and therefore, can control the mechanism of the catalytic process, resulting in the different path along which the molecules may react. The real proof of this hypothesis will be performed during the catalytic tests of SO₂ reduction in the next quarter.

**Preparation of Activated Carbon from IBC-110 coal**

The preparation of activated carbon from the Illinois basin coal started by riffle-sifting 20 lbs of IBC-110 into 4 equal 5 lbs. samples. The use of riffle-sifting allows for a better sampling of the supplied coal. The next step was to grind the coal to the given size range. One of the 5 lbs. coal lots was chosen, the other three lots were subsequently stored away. The chosen 5 lbs. of coal was then passed through a simple motor driven grinder twice. This ground coal was then sifted to the size range of 60 mesh < 100 mesh. The majority of this coal was too

<table>
<thead>
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<th>Material</th>
<th>BET Surface Area m²/g</th>
<th>Mean Pore Diameter Å</th>
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</thead>
<tbody>
<tr>
<td>Darco TRS</td>
<td>218.7</td>
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</tr>
<tr>
<td>10% MoS₂/TRS</td>
<td>519.6</td>
<td>19.8</td>
</tr>
<tr>
<td>15% MoS₂/TRS</td>
<td>497.6</td>
<td>19.9</td>
</tr>
<tr>
<td>20% MoS₂/TRS</td>
<td>481.4</td>
<td>19.4</td>
</tr>
<tr>
<td>Norit ROZ-3</td>
<td>926.5</td>
<td>10.7</td>
</tr>
<tr>
<td>10% MoS₂/ROZ-3</td>
<td>867.1</td>
<td>9.9</td>
</tr>
<tr>
<td>15% MoS₂/ROZ-3</td>
<td>895.5</td>
<td>10.4</td>
</tr>
<tr>
<td>20% MoS₂/ROZ-3</td>
<td>902.6</td>
<td>10.2</td>
</tr>
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large to go through the 60 mesh, so the remaining coal was run through a ball mill at 45 second intervals and then sifted until the entire lot could be sized. This preparation is in progress.

CONCLUSIONS AND RECOMMENDATIONS

1. The apparatus for catalytic reduction of SO$_2$ with methane was constructed and tested.

2. Six molybdenum sulfide catalysts having three different loadings (10%, 15%, and 20%) supported on Darco TRS and Norit ROZ-3 activated carbons were synthesized and characterized.

3. The surface area of Darco TRS supported MoS$_2$ catalysts increased during impregnation. In contrast, the surface area of Norit ROZ-3 supported catalysts remains essentially constant. Both catalyst supports have basic differences in average pore size diameter.

4. Surface area increase for Darco TRS catalyst support during preparation may be attributed to the surface segregation of MoS$_2$ sublayer.

5. Synthesis of activated carbon support from IBC-110 coal is in progress.

DISCLAIMER STATEMENT

"This report was prepared by Tomasz Wiltowski of Southern Illinois University at Carbondale with support, impart by grants made possible by the U.S. Department of Energy Cooperative Agreement Number DE-FC22-92421 and the Illinois Clean Coal Institute. Neither Tomasz Wiltowski of Southern Illinois University at Carbondale nor any of its subcontractors nor the U.S. Department of Energy, Illinois Department of Energy & Natural Resources, Illinois Coal Development Board, Illinois Clean Coal Institute, nor any person acting on behalf of either:

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REFERENCES

1. Westmoreland, P.R., and D.P. Harrison, 1976, *Envir. Science and Technol.*, 7, 659


FIGURE 2. Pore size distribution curve for DARCO TRS activated carbon.
FIGURE 3. Pore size distribution curve for 10% MoS2 supported on DARCO TRS activated carbon.
FIGURE 4. Pore size distribution curve for 15% MoS₂ supported on DARCO TRS activated carbon.
FIGURE 5. Pore size distribution curve for 20% MoS₂ supported on DARCO TRS activated carbon.
FIGURE 6. Pore size distribution curve for NORIT ROZ-3 activated carbon.
FIGURE 7. Pore size distribution curve for 10% MoS$_2$ supported on NORIT ROZ-3 activated carbon.
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DVR (Desorption)

![Pore size distribution curve](image)

**FIGURE 8.** Pore size distribution curve for 15% MoS₂ supported on NORIT ROZ-3 activated carbon.
FIGURE 9. Pore size distribution curve for 20% MoS\textsubscript{2} supported on NORIT ROZ-3 activated carbon.