ABSTRACT OF THE DISCLOSURE

Coal is extracted in quinoline by treatment of a coal-quinoline slurry with ultrasonic irradiation at ambient temperature. The quinoline may then be removed from the solubilized coal fraction by conversion to a water-soluble quinoline salt. The solubilized fraction may be used for production of gasoline, aromatic chemicals, carbon black, electrode carbons, low-sulfur and -ash fuels for power plants, etc.

This invention relates to solubilization or extraction of coal to form products that are low in both sulfur and ash content. These products may be used directly as fuels, e.g., in power plants, or they may be further processed into gasoline, chemicals, carbon black, etc.

Processes for extraction of coal are well known and a wide variety of solvents and reaction conditions have been employed in prior art processes. However, the economics of these processes have generally not been favorable to extensive commercial utilization, primarily due to inefficient extraction and high processing costs.

It has now been found, according to the process of the invention, that the use of quinoline as solvent, when exposed to ultrasonic irradiation, results in an efficient and economical extraction of coal. Quinoline has previously been used in extraction of carbonaceous fuels; however, the use of high temperatures, i.e., about 300° C., was necessary for efficient extraction. In the process of the invention, on the other hand, the extraction may be carried out at ambient temperature. The economics of the process is thereby considerably improved as a result of the reduction or elimination of the required amount of thermal energy.

Quinoline has been found to be unique in exhibiting efficient extraction at ambient temperature. The superiority of this solvent as compared to other conventional solvents is illustrated in the example below.

According to the process of the invention, a slurry of the coal in quinoline is subjected to ultrasonic irradiation at ambient temperature. The initial slurry suitably consists of about 10 to 40 weight percent of coal and is prepared by conventional means such as grinding or pulverizing the coal to a particle size of about 36 to 44 microns and introducing the finely divided coal into the quinoline by any conventional means. The process of the invention is generally most effective with high-volatile-A bituminous coals; however, other coals such as high-volatile-B, high-volatile-C bituminous, andannel may also be treated according to process of the invention. Pure quinoline is not necessary for efficient extraction, crude quinoline base fractions (water free) usually being satisfactory.

Ambient temperature, i.e., about 30° C. is generally optimum in the process of the invention. However, temperatures in the range of about 20° C. to 40° C. usually result in effective and economical extractions. Atmospheric pressure is satisfactory. The extraction is preferably carried out in an air atmosphere. Inert gases may be used but generally show no advantages.

Irradiation of the coal-quinoline slurry may be carried out in any suitable apparatus such as a tank-type ultrasonic cleaner. Frequency of the radiation will range from about 30 to 90 kHz. Optimum power and duration of the irradiation will depend on the frequency employed, the type of coal, particle size of the coal, size and configuration of the reaction vessel, amount of quinoline employed, etc., and are best determined experimentally; however, a power of about 0.5 watt or less per square centimeter of slurry and an irradiation time of about 4 to 6 hours is usually satisfactory. The radiation may be supplied by any conventional ultrasonic generator such as Model 040015, Ultrasonic Industries, Inc., cleaner.

Following irradiation, the solubilized coal and excess quinoline are removed from the undissolved coal residue by conventional means such as centrifugation or filtration. The residue is then preferably washed with additional quinoline; the washings are then added to the solubilized coal-quinoline mixture.

Quinoline is then recovered from the solubilized coal-quinoline mixture by conversion of the quinoline to a water-soluble salt. This is readily accomplished by addition of water and sufficient acid to give a mixture having a pH of about 1.5 to 2.0. The volume of water added is suitably about 5 to 10 times the volume of quinoline used to form the original slurry. Concentrated hydrochloric acid is the preferred acid; however, 1:1 HCl may also be used. At a pH in the above range the quinoline is converted to a water-soluble salt, leaving the solubilized coal product suspended in the quinoline-acid-water mixture. The coal product is then separated by filtration or centrifugation and is preferably washed with water to remove any occluded quinoline or acid. The quinolinedic acid mixture can then be converted to quinoline by neutralization with sodium hydride and the quinoline then reused in the extraction process.

The invention will be more specifically illustrated by the following example.

EXAMPLE

A commercial ultrasonic generator operating at a frequency of 80 kHz, with a total output of 1,800 watts was used to irradiate one-half gram samples of Pittsburgh seam coal (<325 mesh) in 5 ml. of solvent for 4 hours at ambient temperature (30° C.) in an argon atmosphere. In a single experiment with quinoline, the irradiation time was extended to 24 hours. After irradiation, the solvent-extract mixture was removed from the coal residue by centrifugation. The coal residue was then washed with two 5-ml. portions of solvent; the washings were added to the solubilized coal-quinoline mixture. Concentrated hydrochloric acid was added to the solubilized coal quinoline mixture until a pH of 1.5 was obtained. Seventy-five ml. of distilled water were added to separate the solvated coal from the soluble quinoline-hydrochloride. The solvated coal was removed from the mixture by filtration and washed with 500 ml. of distilled water to remove all traces of occluded quinoline hydrochloride.

The amount of coal solvated by various solvents is shown in the following table:

<table>
<thead>
<tr>
<th>Solvent</th>
<th>Weight percent coal solvated</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quinoline</td>
<td>49</td>
</tr>
<tr>
<td>Pyridine</td>
<td>19</td>
</tr>
<tr>
<td>Formamide</td>
<td>10</td>
</tr>
<tr>
<td>N,N-dimethylformamide</td>
<td>&lt;1</td>
</tr>
<tr>
<td>1,3,4-triethylborohydrazine</td>
<td>4</td>
</tr>
</tbody>
</table>

As seen from the above table, approximately half of the coal was solvated after 4 hours of irradiation in quinoline, 2½ times the yield with pyridine. Yields were considerably less with formamide, N,N-dimethylformamide, and the
hydroaromatic, 1,2,3,4-tetrahydronaphthalene. Seventy-nine percent of the coal was solubilized in quinoline when the irradiation time was increased from 4 hours to 24 hours. By contrast, mechanical agitation of a coal-quinoline slurry for 24 hours at ambient temperature solubilized only 10 percent of the coal.

What is claimed is:

1. A process for extraction or solubilization of coal comprising forming a slurry of the coal in quinoline and subjecting the coal-quinoline slurry to ultrasonic irradiation at a temperature of about 20 to 40°C.
2. The process of claim 1 in which the coal comprises about 10 to 40 weight percent of the coal-quinoline slurry.
3. The process of claim 1 in which the frequency of the ultrasonic irradiation is about 30 to 90 kHz.
4. The process of claim 3 in which the frequency is about 80 kHz.

5. The process of claim 1 in which the power of the ultrasonic irradiation is about 0.5 watt or less per square centimeter.
6. The process of claim 1 in which the temperature is about 30°C.
7. The process of claim 1 in which the quinoline is removed from the solubilized coal by formation of a watersoluble salt.

References Cited

UNITED STATES PATENTS
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