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(54) **Coal enhancement process.**

(57) The application relates to enhancing coal by removing the gangue by slurrying crushed run of the mine - (ROM) coal with a heavy medium and thereafter allowing the solids to settle or float according to their inherent densities. The float solids are separated from the heavy medium as one stream and the sink solids as a second stream. These solids are substantially freed of the heavy medium by washing with hot (eg. 90°C to 100°C) water and the vapor azeotrope which forms from the water and heavy medium mixture is removed. The heavy medium content of the solids can be reduced to less than about 400 parts by weight of heavy medium per million parts by weight of solids.

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COAL ENHANCEMENT PROCESS

This invention concerns the separation of coal from gangue.

There are many patents and literature references, too numerous to mention, which disclose techniques as well as chemicals useful to separate coal from the gangue. The most widely used of these techniques employs the sink-float principle using magnitite as the heavy medium. The literature, however, teaches literally hundreds of compounds, most halogenated hydrocarbons, as equally useful.

So far the substitution of this later genus of compounds has not had much, if any, commercial success although several pilot plants are now running using perchloroethylene. One disadvantage associated with the use of this later class of compounds is that the coal retains a considerable amount of the halogenated compound and if it is not removed by stringent heating, its retention, on burning, causes excessive corrosion in boilers and furnaces.

Representative of recent patents disclosing equipment employed in heavy media separations are U.S. Patent Numbers 3,348,675, 2,150,917, 2,150,899 and 2,151,578. In addition, sales and promotional literature of Otisca and McNally show licensed processes.

Surprisingly, the present process removes a considerable amount of the heavy media, usually a halogenated hydrocarbon, and permits its recycle.

Thus, in a process for treating coal by the heavy media technique wherein the float and sink solids each individually are recovered and freed of liquid, then dried to remove the residual heavy media retained thereon, the improvement comprises: treating each solids portion with hot water, at the temperature of from the heavy medium water azeotroping point to the boiling point of water, for a time sufficient to remove a substantial portion of the heavy media or until the water comes off at its boiling point, and thereafter separating the solids from the water.

In accordance with the present invention a halogenated hydrocarbon--the bromo, chloro, fluoro and mixed halogen hydrocarbons, particularly tetrachloroethylene (perchloroethylene), trichloroethylene, 1,1,1-trichloroethane (methylchloroform), or methylene chloride (dichloromethane)--is slurried with a crushed - [preferably 1/16 to ½ inch (0.16 to 1.27 cm)] run of the mine (R.O.M.) coal for a short period of time, usually from 15 to 10 minutes, at temperatures of from -10°C to 90°C. The slurry is allowed to settle under substantially quiescent or mild agitation conditions. The coal rich particles will float free of the gangue rich particles which settle. Each body of settled and floated particles are removed for separate treatment. Following such separation, each body of particles is independently treated with hot (preferably 90° to 100°C) water for a time sufficient to azeotrope off the separating liquid, i.e., the chlorinated solvent. This time is usually from several seconds to one hour, depending on the nature and the size of the particles. Such treatment reduces the retained liquid to between 100 to 19,000 parts by weight of liquid per million parts by weight of coal. Many coals can be freed of solvent to less than 400 parts by weight of liquid per million parts by weight of coal usually in ½ to 5 minutes contact. This is equivalent to less than 0.8 pound - (0.36 kg) of liquid per ton of coal. Mild agitation may accompany the hot water treatment.

Representative results of processes which use steam or hot air show residual liquid in the order of 0.095% (950 ppm) at 180°F (83°C) steam; while oven heating for one hour at 100°C leaves 0.655 percent - (6550 ppm) liquid, at 175°C leaves 0.08 percent (800 ppm) liquid retained or at 200°C for one hour leaves 0.02 percent (200 ppm) liquid retained compared to the 400 parts using only 98°C water and only one minute contact in accordance with the present invention. Thus it is seen that, unexpectedly, the use of hot water vis-a-vis steam, hot air or ovens (kilns) removes more of the retained heavy media quicker than these prior art higher temperature processes. The invention allows essentially full recovery of the halogenated solvent. Both the steam and hot air (ovens) make it very difficult to have a good recovery of the halogenated solvent because substantial amounts of it generally escape to ambient air. The ability to have a quick solvent removal is beneficial because the solvent may permiate the tars in a coal with time.

Example 1

To illustrate the effect hot water has on removing the heavy media from coal, 200 grams of Aryshire coal, run of the mine (R.O.M), ½ inch (1.27 cm) to pan, 3½ percent less than 100 mesh, was added to a liter of perchloroethylene with mixing for 5 minutes. Mixing was rapid but insufficient to attrite any appreciable amount of the coal. Following cessation of mixing the slurry was allowed to settle and about ¾ of the liquid, containing most of the coal, was separated and the liquid filtered away. This float filter cake portion, filtered free of the body of liquid perchloroethylene, was slurried into hot water at 99°C. The temperature dropped

to between 88°C and 93°C due to mixing the wetted coal which was at ambient temperature (23°C) with the hot water. Heat was applied to the container to maintain the temperature of the slurry at 88°C and heating was continued until the slurry temperature rose to 100°C at which temperature about 10 ml of water substantially free of perchloroethylene was distilled over. Thereafter the coal was filtered free of the water and analyzed.

TABLE I

Analysis showed the coal had
water 9%
perchloroethylene 1.3%
ash 6%
sulfur 3.4%
The original coal had
ash 11.7%
sulfur 4.5%
water 6.3%
The bottom portion (sink) of the sink float step treated in the same manner as the float had an analysis of
ash 42%
sulfur 6.9%
perchloroethylene 0.5%

The above analysis illustrates that the hot water treatment effectively removes perchloroethylene from coal.

In comparison the same coal, treated by the same sink float technique above using perchloroethylene as the heavy medium, when dried in a hot air convection oven at 120°C air had a retained perchloroethylene content of 1.44 percent in the float portion after ½ hour, 1.10 percent after 1 hour, 0.96 percent after 2 hours, 0.86 percent after 3 hours and 0.68 percent after 4 hours.

Example 2

Ayrshire coal (R.O.M. from Amax, Indiana) was processed by a sink float method in one of the following solvents then freed of residual solvent by the hot water treatment of Example 1. Comparison is also reported using the convection oven drying technique. The following table lists the results of the hot water heavy media removal technique:

TABLE II

	Perchloroethylene	trichloroethylene	1,1,1-trichloroethane
% solids in float	82	71	64
% solids in sink	18	29	36
% solvent in float solids	1.5	1.8	1.8
water temperature, °C	97	97	97
time of slurry in hot water	15 minutes	15 minutes	15 minutes
ppm solvent after hot water treament	1908	1675	1413
% ash orig/float/sink	11.7/5.9/41.9	11.7/4.9/38.6	11.7/2.7/21.6
% sulfur orig/float/sink	4.5/3.4/6.9	4.5/3.3/4.7	4.5/2.5/4.5
Specific Gravity of Solvent, 25/25°C	1.62	1.32	1.46

Thus it is seen that the hot water treatment is equivalent to treatment with steam or hot air but equivalency can be achieved in shorter periods of time. The more important advantage is the ability to recover greater than 99 percent of the chlorinated solvent with the hot water treatment.

Example 3

Cammeron Coal (R.O.M., Perma Mining, S.E. Colorado) was processed by the sink float technique, the sink and float separated and each filtered to remove the solids with only retained solvent. The resulting cakes were each slurried in hot water to remove the residual solvent (heavy medium). The results employing different solvents as the heavy media are set forth below:

TABLE III

	Perchloroethylene	Trichloroethylene		1,1,1-Trichloroethane	
% sink/float	2/98	6/94	6/94	44/56	20/80
% solvent on float		6	1		
temperature, sink/float treatment	ambient	ambient		25°C	-5°-0°C
temperature hot water, °C	100	100	97	100	100
time to dry, min.	10	10	1/2	1	1
% solvent remaining after hot water treatment (ppm)	0.14 (1400ppm)	0.061 (614 ppm)	0.042 (416 ppm)	- -	<0.010 (<100 ppm)
% ash in orig. coal	12-15	12-15	12-15		
% ash after hot water treatment in float	8.72*	8.35*	8.52*		7.72*

*pretreated ROM had an ash content of 12-15%

Example 4 (Comparative)

In a comparative run, the same coal from Example 3 was treated by the sink/float technique using trichloroethylene was subjected to convection oven heating at 120°C and measurements of residual solvent made at ½, 1, 2, 3, 4, and 8 hours. The results are set forth below.

TABLE IV

Original residual solvent content after filtering	3.9% (38,275 ppm)
Solvent content after	
1/2 hour	262 ppm
1 hour	270 ppm
2 hours	263 ppm
3 hours	281 ppm
4 hours	172 ppm
8 hours	157 ppm

Again, the ability of the hot bath treatment to recover 95 to 100 percent by weight of the solvent in less than 10 minutes is shown where the oven treatment does not recover 90 percent by weight of the solvent in a practical manner in less than ½ hour.

Example 5

Rocky Mountain Energy Prospect Point Mine, labeled Leucite Hills, Wyoming, greater than 8 mesh coal from ground and screened R.O.M. coal, was subjected to heavy media separation using perchloroethylene as the heavy media. The specific gravity of each screen aliquot of coal was measured, the float/sink from each screening filtered, and the cakes subjected to the hot water treatment of the present invention. The results obtained when the specific gravity varied are set forth below.

TABLE V

(Specific gravity)	ROM	1.6	1.5	1.4	1.3
% sink/float	0/100	1/99	3/97	22/78	30/70
Desolventized Float					
% Ash	8	8	--	3.9	3.3
ppm residual solvent	--	--	--	650	600
Desolventized Sink					
ppm residual solvent				500	10

It is thus seen that in most instances the hot water treatment, water at or near its atmospheric boiling point, removes in shorter treatment periods more of the residual heavy medium halogenated solvent from the coal than the conventional steam, hot air or kilns.

Claims

1. A process for treating coal by the heavy media technique wherein the float and sink solids portions each individually are recovered and freed of liquid, then dried to remove the residual heavy media retained thereon, characterised in that at least one of the solids portions is treated with hot water, at the temperature of from the heavy medium water azetroping point to the boiling point of water, for a time sufficient to remove a substantial portion of the heavy media or until the water comes off at its boiling point, and thereafter separating the solids from the water.

2. A process as claimed in Claim 1, wherein both the float and sink portions are subjected to the said hot water treatment.

3. A process as claimed in Claim 1 or Claim 2, wherein the heavy media is recovered from the water/media vapors by condensation.

4. A process as claimed in any one of the preceding claims, wherein prior to separating the solids from the water, the azeotrope is condensed to recover the heavy media from the condensate.

5. A process as claimed in any one of the preceding claims, wherein the heavy media is a halogenated hydrocarbon.

6. A process as claimed in Claim 5, wherein the halogenated hydrocarbon is tetrachloroethylene, trichloroethylene, 1,1,1-trichloroethane or methylene chloride.

7. A process as claimed in any one of the preceding claims, wherein the hot water is at a temperature of 90 to 100°C.

8. A process as claimed in any one of the preceding claims, wherein the time of water treatment is less than one hour.

9. A process as claimed in Claim 8, wherein said water treatment time is 0.5 to 5 minutes.

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EUROPEAN SEARCH REPORT

Application number

EP 86 30 1033

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
A	US-A-4 076 505 (R.M. DESSAU) * Column 3, line 59 - column 4, line 10; column 5, line 61 - column 6, line 7; column 6, line 53 - column 7, line 2 *	1,3-5	B 03 B 5/30 B 03 B 5/34
A	US-A-3 365 395 (D. McDONALD) * Column 2, lines 43-53; column 5, lines 28-44; column 7, line 60 - column 8, line 17; figures 2,3 *	1,5,6	
A	US-A-3 275 549 (C.R. CRABB) * Column 4, lines 15-27 *	1,5,6	
A	US-A-4 324 560 (A.G. FONSECA) * Column 3, lines 19-55; column 8, lines 1-28; figures 1,2 *	1-6	
E	US-A-4 579 650 (R.J. NANKEE et al.) * Whole document *	1-9	
A	US-A-4 198 289 (G.R.B. ELLIOTT et al.)		
A	US-A-3 347 370 (C. RAMPACEK et al.)		

TECHNICAL FIELDS SEARCHED (Int. Cl.4)

B 03 B
B 01 D

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	24-09-1986	LAVAL J.C.A

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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T : theory or principle underlying the invention
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