Nov. 4, 1930. R. LESSING 1,780,830

TREATMENT OF CARBONACEOUS MATERIALS

Filed May 23, 1927 2 Sheets-Sheet 1

Fig. 1.

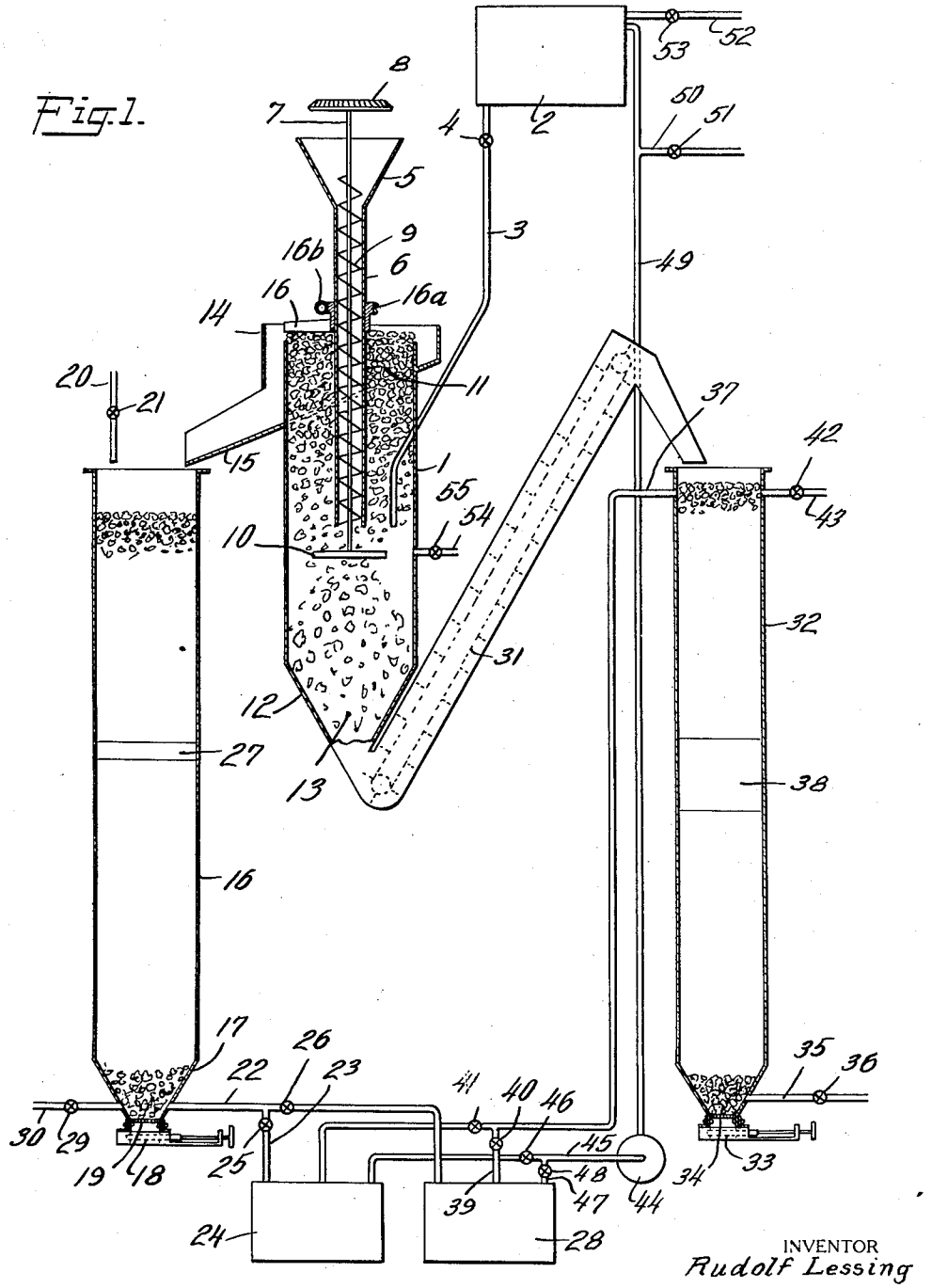

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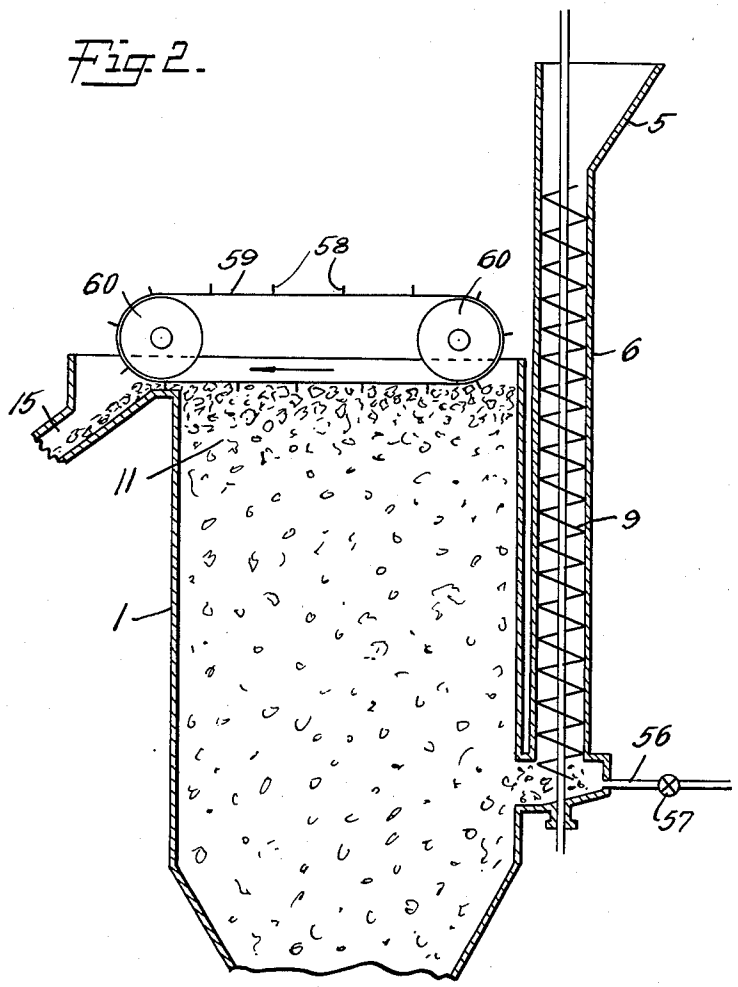
Fig. 2.



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UNITED STATES PATENT OFFICE

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TREATMENT OF CARBONACEOUS MATERIALS

Application filed May 23, 1927, Serial No. 193,610, and in Great Britain May 27, 1926.

This invention relates to improvements in the separation of coal material more particularly for the obtaining therefrom of a portion having a low proportion of ash-forming ingredients.

The ash-forming ingredients of coal are not uniformly distributed throughout the coal but some portions of a body of coal have more than the average amount of ash-forming ingredients and some have considerably less. Thus a coal which has an average ash content of say 12% may contain a substantial proportion of coal containing but 2% or less of ash. The present process renders commercially possible the separation from coal in bulk of a low ash forming component and a high ash forming component in an improved manner. In this process advantage is taken of the difference in specific gravity of those components whereby they can be separated by flotation of the lighter fragments in an aqueous solution whose specific gravity lies between the specific gravities of the components.

By the present invention, clean coal having a lower proportion of ash forming ingredients than any commercially available at present, so far as I am aware, may be produced.

In order to render the separation economically practicable it is necessary to recover the dissolved salt and, moreover, to recover it with the least possible dilution. An important feature of the present invention, therefore, consists in removing the lighter material from the bath and washing it with a continuous unbroken body of water or weak solution.

The displacement of the adherent liquor may be effected in an upward or downward direction but in either case the principle is the same. The washing liquid is allowed to ascend or descend as a continuous body in contact with the coal to be washed. The more concentrated solution displaced thereby is removed at the same rate and in this way the adherent solution is removed from all sides of the coal particles with the least possible dilution. There should be no broken films or boundaries between the washing liquid and the liquid displaced thereby and as little mixing as possible should be allowed to take pace at the boundary.

The tendency of the lighter portion (the float) to rise causes the solid particles to pack in an upward direction and accordingly downward displacement of the bath liquor counteracts this tendency and thus prevents clogging. On the other hand, the tendency of the heavier portion (the sink) to fall causes the solid particles to pack in a downward direction and it has now been found that this can satisfactorily be overcome by washing the sink by upward displacement of the bath liquor. If it is desired to remove the bath liquor from the float by upward displacement, it will be necessary to prevent the float from being carried out of the drainer by suitable means such as a grid which will confine the solid particles but will allow the liquor to pass therethrough. The sink may be washed by downward displacement if desired although this may give rise to tighter packing of the material and consequent slower drainage. On the other hand, less dilution of the stronger liquors by the weak is liable to take place when the latter are superimposed on the former, i. e., with downward washing.

Drainage may be assisted by pressure or suction and it might be advisable to vibrate the drainer or its contents, e. g., by tapping or hammering the vessel or by plunger action applied to the contents. The beneficial effect of such vibration is due to the separation of the particles which allows liquids and possibly fine dust to pass freely between them, thus assisting the draining operation and shortening the time required. Excessive vibration should be avoided since this might cause diffusion of water or weak solution into the strong solution.

It is preferable to remove the greater part of the dust from coal or slack prior to the flotation treatment, but this is not so essential when handling large coal or nuts. It is preferred to remove the finely divided material by passage through an air sifter or by other operations carried out on the air dry coal, but in some cases I may treat the raw

coal, by elutriation in an upward current of water whereby the fines are removed.

Fines which may be present in the sink when it is fed into the drainer and which might hinder the draining or washing, can be effectively removed by passing an upward current of bath liquor through the sink prior to washing.

Calcium chloride solution may be employed as the separating liquid although other substances such as calcium nitrate can also be used. The solution should have a decidedly alkaline reaction.

Another important feature of the present invention consists in supplying the coal material to be separated below the surface of the liquid and preferably agitating the liquid at the point of introduction to facilitate disaggregation of discrete particles of different specific gravities.

There is a considerable advantage in removing the greater part or as much as is practicable of the dust which, for example, passes through a screen having 50 meshes to the linear inch.

It is particularly desirable that not more than 2% and preferably less than 1% of such dust is present.

These data as to fineness and proportions are indicative, but I may add that the material passing a screen having 50 meshes to the linear inch consists mainly of fusain and finely divided clay and other materials. Some of these materials are highly porous and others usually colloidal and I have found that they considerably impede the flow of liquids between the particles which probably explains the considerable advantage obtained by removing particles of such fineness. It is therefore a particularly important feature of the invention first to remove the dust in order to facilitate the separation of all coal of low ash content from the specifically heavier mineral matter or to divide the coal itself into bright coal of very low ash content and a dull coal of higher ash content and to facilitate the draining and washing of the products. Products which may contain 2% or less of ash and are therefore of unusual purity may thus be obtained. Again, when treating bituminous coal, it is possible to separate the coking from the non-coking or feebly coking portion.

I have found that with coals from which the dust has not been entirely removed before separation according to my process, it may be of advantage to withdraw a portion of the bath liquor periodically or continuously and to separate from it by filtration those fine particles which do not readily rise or sink in the bath solution. The clear liquor obtained may be returned to the bath, after having been concentrated to the requisite strength, if necessary. By this modification of my invention, draining and washing of the float and sink, by whatever method it may be carried out, will be facilitated.

In the accompanying drawings is shown a preferred form of apparatus for carrying out the invention. Fig. 1 is a diagrammatic elevation, partly in section, of an installation, and Fig. 2 is a diagrammatic elevation, partly in section, showing a modified form of the separating vessel.

In the drawing, 1 is the separating vessel which contains a solution of calcium chloride of suitable specific gravity, which, for the separation of bituminous coal into components of high ash content and low ash content, may be, for example, of specific gravity 1.35. This solution is delivered from the supply tank 2 through a pipe 3 having control valve 4. The coal to be treated is contained in a hopper 5 which is situated above the vessel 1 and is delivered through the chute 6 which terminates at a point about midway between the top and bottom of the vessel 1. A driving shaft 7 extends from the chute, being driven from a suitable source of power through a gear 8 or other suitable drive. A screw conveyor is arranged on this shaft to feed the coal into the bath. The shaft 7 extends to the lower end of the chute and is provided with a paddle 10 to agitate the bath at the point of introduction of the coal.

The particles of coal which are of low ash content, have a lower specific gravity than the solution, and, therefore tend to float, travelling upwards forming a layer 11 which may be of pudding-like consistency, at the top of the vessel. The depth of this layer may be controlled by controlling the rate at which the coal is delivered in to the bath, and the rate at which the clean coal, which may be referred to as the float, is removed from the top of the vessel. The bottom of the vessel 1 slopes inwardly as shown at 12, forming a hopper in which the coal of high ash content sinks and collects as indicated at 13.

The top of the vessel 1 is surrounded by a launder 14 having a sloping bottom 15. As the top of the float rises beyond the top of the vessel as indicated at the right, the coal falls into the launder or may be mechanically removed from the top of the layer. As shown, a revolving blade 16 is mounted over the vessel and is carried by a hub surrounding the chute 6 and provided with a worm wheel 16^a, meshing with the worm 16^b driven from a suitable source of power. This blade is adapted to remove that portion of the layer 11 projecting beyond the top of the vessel 1 whence the clean coal and adherent calcium chloride falls on the sloping bottom of the launder 15 and is delivered to a shaft or tower 16. The shaft or tower is provided with a funnel shaped lower portion 17 closed by a slide valve 18. Above the slide valve a grid 19 is removably supported and the

clean coal delivered to the tower forms a column resting on this grid. When the coal is to be washed to recover the calcium chloride, water is delivered to the top of the tower through a pipe 20 having a control valve 21. An outlet pipe 22 is arranged in the bottom of the shaft or tower 16 and this pipe is provided with a branch pipe 23 communicating with a vessel or receptacle 24. The branch pipe is provided with a control valve 25 and beyond the branch pipe the pipe 22 is provided with a control valve 26. When the valve 21 is opened, the valve 25 is also opened and the valve 26 closed so that the liquid from the bottom of the tower will enter the receptacle 24. The water is carefully superimposed on the top of the clean coal and adherent calcium chloride, and is allowed to descend slowly as a continuous or unbroken body in contact with the coal to be washed. The calcium chloride solution from the bottom of the tower is removed to the tank 24, at the same rate of speed that the water is delivered to the top of the tower, the concentrated solution being thus displaced by the water. There should be no broken films or boundaries between the washing water and the concentrated solution displaced thereby and in this way the adherent solution is removed from all sides of the coal particles with the least possible dilution. A relatively sharp line of demarcation will exist between the washing water and the concentrated solution; the water will tend to mix with the heavier solution only to a very slight extent. When the washing reaches the point where this weaker solution is flowing through the pipe 22 the valve 25 may be closed and the valve 26 opened to deliver the less concentrated liquor to a tank 28. When water begins to flow from the bottom of the tower, a valve 29 in pipe 30 is opened and the wash water run to waste. After the coal has been washed it may be removed from the tower by opening the slide valve and displacing the grid. The grid may be displaced by inserting a tool or instrument through the bottom of the tower and removing it from its support.

Instead of providing grids 19 and 34, at the bottom of the drainers, the pipes 22, 30, 35 and 37 may be protected by grids arranged at the side of the cones 17. Further inlet or outlet pipes suitably protected by grids may be provided in the walls of the drainers 16 and 32 at intermediate points in the height to assist in draining, washing or drawing off samples for testing.

The coal of high ash content, which may be referred to as the sink, collects at the bottom of the vessel 1 and is removed by a bucket elevator 31 to a tower 32, similar to the tower 16. The lower end of this tower is also provided with a slide valve 33 and a grid 34 to support the sink. In this tower the calcium chloride solution is removed by upward displacement. The wash water is delivered to the bottom of the tower through a pipe 35 having a control valve 36. The principle of operation is the same as in the downward displacement of the adherent solution from the float. The calcium chloride solution is removed from the top of the tower through a pipe 37 at the same rate of speed that the water is fed so that as little mixing as possible occurs at the boundaries between the washing water and the concentrated solution. During the upward displacement the line of demarcation between the water and the solution is not as clear and a rather larger volume of weak solution will result. The pipe 37 is provided with a branch pipe 39 having a control valve 40 and beyond the branch pipe it is provided with a control valve 41. During the first part of the washing process when strong solution is being removed from the top of the tower, the valve 40 is closed and the valve 41 opened so that the concentrated solution will pass through the pipe 37 to the tank 24. When the diluted solution begins to pass through the pipe, 37, the valve 41 is closed and the valve 40 is opened to collect the diluted solution in the tank 28. When water reaches the top of the tower both valves are closed and a valve 42 in the pipe 43 is opened to remove the wash water.

Fig. 2 shows a modified construction in which the coal is delivered from hopper 5, through chute 6 situated on the side of the vessel 1 by means of the screw conveyor 9. Agitation of the bath at the point of introduction is effected by a jet of liquid supplied through a tube 56 controlled by a valve 57. The separating vessel in this case is of square or rectangular section, and the float 11 is removed by scrapers 58 mounted on a belt or band 59 which is actuated through the driving wheels 60. The float can thus be automatically and continuously removed to the chute 15 whence it is delivered to the draining tower.

The tank 24 is connected to a pump 44 by a pipe 45 having a control valve 46. The tank 28 communicates with the pipe 45 through a branch pipe 47 having a control valve 43. The outlet side of the pump is connected to the supply tank 2 by a pipe 49 so that the strong or concentrated calcium chloride solution from the tank 24 may be returned directly to the supply tank. The pipe 49 is provided with a branch pipe 50 having a control valve 51 by means of which the weaker solution from the tank 28 may be delivered to a concentrator before it is returned to the supply tank. The strong solution may also be conveyed to the concentrator through pipe 50 if it requires concentration. The supply tank is provided with a feed pipe 52 having a control valve 53. The main receptacle 1 may be provided with an outlet pipe 54 hav-

ing a control valve 55 through which a portion of the liquid from the bath may be removed for filtering.

The method of separating the coal of low ash content will be apparent from the foregoing description. The coal is floated and removed to the tower 16 where the adherent calcium chloride solution is removed from the coal in the manner heretofore described. The solution is also recovered from the sink in the tower 32 and in practicing the process the loss of calcium chloride has been found to be less than 1% of the weight of the coal treated. This low loss renders the process commercially practical in the separation of coal of low ash content. The alkalinity of the calcium chloride solution may be maintained by adding the requisite quantity of lime to the solution in the concentrator or in the storage tank.

The process may be performed either intermittently or continuously. When practiced continuously a number of the shafts or towers 16 are provided and are successively connected to the bath for charging. After one tower has been charged it is washed and drained during which periods other towers will be charged. The liquid may be filtered at intervals to separate any fines which remain suspended by removing a portion of the liquid through the pipe 54 and conveying it to a filter. Drainage may be assisted in the towers 16 and 32 by pressure or suction and the tops of the towers may be closed after charging by suitable covers. The specific gravity of the solution of calcium chloride will vary according to the coal treated and the degree of purity desired. I have found in the case of Welsh anthracite a bath having a specific gravity of 1.4 will yield a product having no more than 2% of ash.

In washing the coal weak liquors of descending strength may first be employed to displace the strong liquor. The weak liquor may then finally be removed by water. The strong liquor, weak liquor and water may be drawn off separately from suitable outlets on the drainer.

The invention has been described with reference to the employment of a solution of calcium chloride. Other substances may be employed, and I have found that for some purposes calcium nitrate, for example is particularly suitable since solutions of higher specific gravity may be employed. Thus it is possible to treat the sink from the foregoing process for a further separation of components of different specific gravities with a solution of higher specific gravity. The float may be likewise further treated with the aid of a solution of lower specific gravity.

The clean coal or float from the above described process is especially suitable for the manufacture of high quality fuel briquettes. If it is required to dry the float, this may be continuously washed and continuously dried without interruption. The material, after drying by heat may then be treated with a binding agent and briquetted without intermediate cooling.

In a further modification of the invention, the washing may be incomplete or after washing predetermined quantities of the coal calcium chloride or other catalyst, such as ferric chloride or ferrous sulphate, may be added by displacement of a portion of the adhering liquor with the body of a solution, colloidal solution or suspension of the catalyst of such concentration that the film left on the surface of the particles after draining contains the catalyst in a predetermined proportion to the clean coal.

The float may be used for the process of hydrogenation at high pressure, whether catalysts are added or not.

The raw material used for the flotation process is preferably one in which the distribution of the ash is such that one fraction can be separated containing the majority of the ash-forming bodies while another large fraction can be separated, which upon incineration yields very little ash. In other words, I prefer to treat the raw material in which a large fraction thereof contains a very low percentage of ash-forming bodies. The method, however, is applicable to any carbonaceous material which is not homogeneous as regards its specific gravity but contains fragments which are of relatively high average specific gravity and fragments which are relatively low average of specific gravity. Thus, as already stated, certain bituminous coals may be separated into a good coking portion and a feeble coking portion.

I claim:

1. In the process of separating coal into its heavier and lighter components by delivering it into a bath of calcium chloride solution having a specific gravity intermediate between the specific gravities of the components to be separated so that the fragments of said heavier component sink and the fragments of said lighter component float, removing the floated material and removing bath liquor therefrom by displacement with a continuous unbroken body of washing liquor until a predetermined small quantity of calcium chloride remains.

2. In the process of separating coal into its heavier and lighter components by delivering it into a bath of calcium chloride solution having a specific gravity intermediate between the specific gravities of the components to be separated so that the fragments of said heavier component sink and the fragments of said lighter component float, removing the floated material and removing bath liquor therefrom by downward displacement with a continuous unbroken body of water until a

predetermined small quantity of calcium chloride remains.

3. In the process of separating coal into its heavier and lighter components by delivering it into a bath of calcium chloride solution having a specific gravity intermediate between the specific gravities of the components to be separated so that the fragments of said heavier component sink and the fragments of said lighter component float, removing from the coal to be treated the greater part of the dust contained therein before delivery to the bath, removing the floated material from the bath and removing bath liquor therefrom by downward displacement with a continuous unbroken body of water until a predetermined small quantity of calcium chloride remains.

4. In the process of separating coal into its heavier and lighter components by delivering it into an aqueous bath having a specific gravity intermediate between the specific gravities of the components to be separated so that the fragments of said heavier component sink and the fragments of said lighter component float, removing the floated material, removing bath liquor therefrom by displacement with a continuous unbroken body of washing liquor and displacing the last portion of the bath liquor with a solution of a substance which acts as a catalyst during subsequent combustion.

5. In the process of separating coal into its heavier and lighter components by delivering it into an aqueous bath having a specific gravity intermediate between the specific gravities of the components to be separated so that the fragments of said heavier component sink and the fragments of said lighter component float, removing from the coal to be treated the greater part of the dust contained therein before delivery to the bath, removing the floated material, removing bath liquor therefrom by displacement with a continuous unbroken body of washing liquor and displacing the last portion of the bath liquor with a solution of a substance which acts as a catalyst during subsequent carbonization.

6. In the process of separating coal into its heavier and lighter constituents by delivering it into a bath of calcium chloride solution having a specific gravity intermediate between the specific gravities of the components to be separated so that the fragments of said heavier component sink and the fragments of said lighter component float, the step which consists in employing an alkaline solution of calcium chloride.

7. In the process of separating coal into its heavier and lighter components by delivering it into a bath of aqueous solution having a specific gravity intermediate between the specific gravities of the components to be separated so that the fragments of said heavier component sink and the fragments of said lighter component float, removing at least one of said components, freeing it from bath liquor by displacement with a continuous unbroken body of washing liquid, returning at least a portion of the displaced liquid to the bath substantially undiluted, concentrating the remainder of said bath liquor recovered and returning it to said bath.

8. In the process of separating coal into its heavier and lighter components by delivering it into a bath of aqueous solution having a specific gravity intermediate between the specific gravities of the components to be separated so that the fragments of said heavier component sink and the fragments of said lighter component float, removing from the coal to be treated the greater part of the dust contained therein before delivery to the bath, removing at least one of said components, freeing it from bath liquor by displacement with a continuous unbroken body of washing liquid, returning at least a portion of the displaced liquid to the bath substantially undiluted, concentrating the remainder of said bath liquor recovered and returning it to said bath.

9. The process of separating coal into its heavier and lighter components which consists in delivering it into a bath of aqueous solution having an alkaline reaction and having a specific gravity intermediate between the specific gravities of the components to be separated so that the fragments of said heavier component sink and the fragments of said lighter component float, and then removing at least one of said components, freeing it from the bath liquor by displacement with a continuous unbroken body of washing liquid, returning at least a portion of the displaced liquid to the bath substantially undiluted, concentrating the remainder of said bath liquor recovered and returning it to said bath.

10. In the process of separating coal into its heavier and lighter components by delivering it into a bath of calcium chloride solution having a specific gravity intermediate between the specific gravities of the components to be separated so that the fragments of said heavier component sink and the fragments of said lighter component float, removing at least one of said components, freeing it from bath liquor by displacement with a continuous unbroken body of washing liquid, returning at least a portion of the displaced liquid to the bath substantially undiluted, concentrating the remainder of said bath liquor recovered and returning it to said bath.

11. In the process of separating coal into its heavier and lighter components by delivering it into a bath of calcium chloride solution having a specific gravity intermediate between the specific gravities of the components to be separated so that the fragments of said heavier component sink and the frag-

ments of said lighter component float, removing from the coal to be treated the greater part of the dust contained therein before delivery to the bath, removing at least one of the components, freeing it from bath liquor by displacement with a continuous unbroken body of washing liquid, returning at least a portion of the displaced liquid to the bath substantially undiluted, concentrating the remainder of said bath liquor recovered and returning it to said bath.

12. The process of separating coal into its heavier and lighter components which consists in delivering it into a bath of alkaline calcium chloride solution having a specific gravity intermediate between the specific gravities of the components to be separated so that the fragments of said heavier component sink and the fragments of said lighter component float, removing at least one of said components, and freeing it from the bath liquor by displacement with a continuous unbroken body of washing liquid, returning at least a portion of the displaced liquid to the bath substantially undiluted, concentrating the remainder of said bath liquor recovered and returning it to said bath.

13. The process of separating coal into its heavier and lighter components which consists in delivering it into a bath of alkaline calcium chloride solution having a specific gravity intermediate between the specific gravities of the components to be separated so that the fragments of said heavier component sink and the fragments of said lighter component float, removing from the coal to be treated the greater part of the dust contained therein before delivery to the bath and removing at least one of said components, freeing it from the bath liquor by displacement with a continuous unbroken body of washing liquid, returning at least a portion of the displaced liquid to the bath substantially undiluted, concentrating the remainder of said bath liquor recovered and returning it to said bath.

14. The process of separating coal into its heavier and lighter components which consists in delivering it into a separating bath of calcium chloride solution having a specific gravity intermediate between the specific gravities of the components to be separated so that the fragments of said heavier component sink and the fragments of said lighter component float, removing at least one of said components and freeing it from the bath liquor by displacement with a continuous unbroken body of washing liquid, returning at least a portion of the displaced liquid to the bath substantially undiluted, concentrating the remainder of said bath liquor recovered with the addition of a material giving an alkaline reaction to maintain the required alkalinity and returning liquor so concentrated to said separating bath.

15. The process of separating coal into its heavier and lighter components which consists in delivering it into a bath of aqueous solution having a specific gravity intermediate between the specific gravities of the components to be separated so that the fragments of said heavier component sink and the fragments of said lighter component float, by introducing it below the surface of the bath, then removing at least one of said components, freeing it from the bath liquor by displacement with a continuous unbroken body of washing liquid, returning at least a portion of the displaced liquid to the bath substantially undiluted, concentrating the remainder of said bath liquor recovered and returning it to said bath.

16. In the process of separating coal into its heavier and lighter components by delivering it into a bath of calcium chloride solution having a specific gravity intermediate between the specific gravities of the components to be separated so that the fragments of said heavier component sink and the fragments of said lighter component float, removing from the coal to be treated the greater part of the dust contained therein before delivery to the bath, continuously removing, filtering and returning to the bath a portion of the liquor in order to avoid accumulation of fine material in the bath, removing at least one of said components, freeing it from the bath liquor by displacement with a continuous unbroken body of washing liquid, returning at least a portion of the displaced liquid to the bath substantially undiluted, concentrating the remainder of said bath liquor recovered and returning it to said bath.

In testimony whereof I affix my signature.

RUDOLF LESSING.