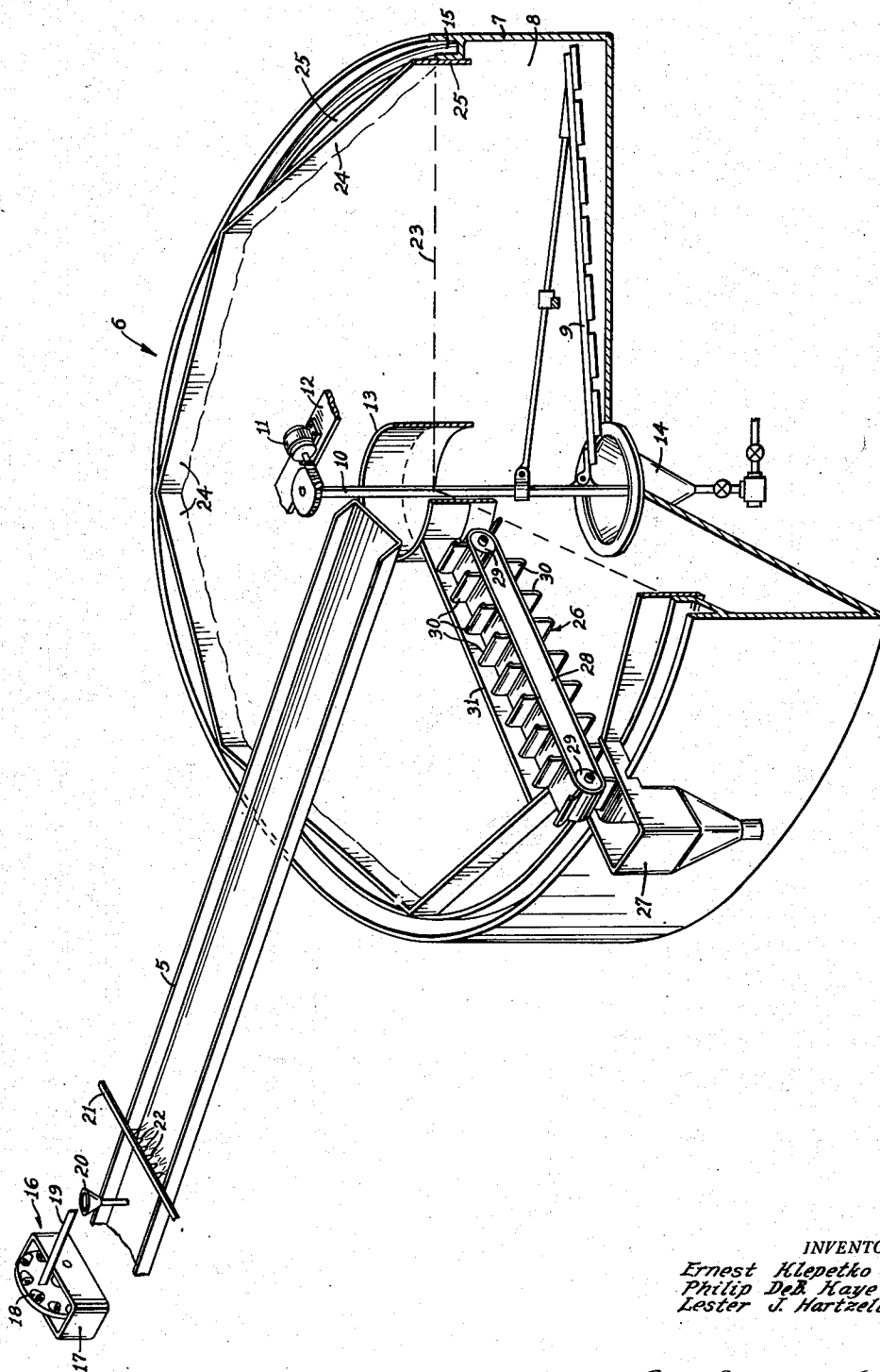


April 8, 1952

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RECOVERY OF RESINS FROM COAL

2,591,830

Filed July 25, 1946



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## UNITED STATES PATENT OFFICE

2,591,830

## RECOVERY OF RESINS FROM COAL

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Application July 25, 1946, Serial No. 686,132

3 Claims. (Cl. 209—166)

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This invention relates to the recovery of resins from resin-bearing coal, and provides an improved method and improved apparatus for recovering resins from such coal with a minimum investment in plant equipment while at the same time achieving good recovery of the resin in a concentrate of good quality. These results are attained in accordance with the invention by flotation recovery of the resin concentrate in the apparatus customarily employed in coal wash-

Some coals, such as some of the Utah coals, contain valuable fossil resins which, when purified, are suitable for uses similar to those for which lac resins are commonly employed. For example, these coal resins, when purified, may be used in the preparation of insulating and other varnishes, printing inks, resinous waterproofing agents, special rubber compositions, rubber cements, and other resin-containing products.

Various procedures have been proposed heretofore for recovering resins from resin-bearing coal. Probably the most satisfactory procedure involves flotation of a resin concentrate from the coal with the aid of one or more flotation agents capable of promoting flotation of the resin. United States patent to Green No. 1,773,997 describes a coal-resin flotation process using as flotation agent one of the higher alcohols, turpentine, cresol, one of the pine oils, or the like. As is the case in flotation processes for beneficiating other materials, Green proposed conducting his flotation operation in special flotation apparatus, that is, in apparatus falling in the category of flotation machines and designed specifically and solely for use in carrying out flotation operations. Use of such apparatus for recovering resins from coal necessitates developing and incorporating a special flotation circuit in the coal preparation plant in which the resin is recovered. It is not a simple matter to develop and incorporate this flotation circuit in the ordinary coal washery, because it must be designed and built so as not to impair or particularly increase the cost of recovering the washed coal products. The coal is the material of primary value. The resin flotation operations often must be regarded as concerned with the recovery of a by-product that, while valuable, does not justify decreasing the economic value of the coal itself, either by degrading the quality of the recovered coal or by increasing the cost of recovering it in marketable form.

We have discovered that if resin is present in the coal, it may be recovered by a flotation op-

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eration conducted in the usual coal-washing apparatus, and without the use of a special flotation circuit. In a typical coal preparation plant, run-of-mine coal is subjected to a primary crushing operation to break up big lumps, the coarse crushed coal is classified by screening into the various commercial sizes and is washed free of fines and slimes, and the fine coal particles present in the run-of-mine coal and unavoidably produced in the crushing operation are freed of slimes to recover a marketable fine coal product. In treating the fine coal, a pulp of the coal in water is prepared, and this pulp is introduced into sedimentation apparatus from which a clean fine coal product, substantially free of slimes, is recovered.

In accordance with our invention, a reagent capable of promoting flotation of the resin is incorporated in the pulp prior to its introduction into the sedimentation apparatus. For example, the flotation reagent may be added to the pulp as it flows through a launder by which it is carried to the sedimentation apparatus. Advantageously the pulp also is aerated prior to its entry into the sedimentation apparatus, as such aeration has been found to improve recovery of the resin. In the sedimentation apparatus the resin floats to the surface of the pulp, and it may readily be removed therefrom as a resin concentrate, for example, by skimming it to a resin concentrate discharge.

We have discovered that it is possible to effect a substantial recovery of resin in the form of a resin concentrate from the pulp in sedimentation apparatus, even without adding any flotation reagent to the pulp and without aerating it. However, the resin recovery is greatly increased by incorporating in the pulp a flotation reagent capable of promoting flotation of the resin, and by aerating the pulp, as described above.

The invention contemplates some modification in conventional coal-washery apparatus for the purpose of carrying out the new method. Thus the invention contemplates the provision of reagent-feeding means located so as to introduce the desired flotation agent into the coal-water pulp as it flows through the launder to the sedimentation apparatus, or at some other point prior to introduction of the pulp into this apparatus. Advantageously, a conduit also is provided for introducing air into the pulp prior to its entry into the sedimentation apparatus, and most advantageously at a point only a short distance downstream from the point at which the flotation reagent is added. The sedimentation vessel is pro-

vided with a resin concentrate froth discharge outlet, and advantageously also with a skimmer for skimming the resin concentrate froth from the surface of the pulp in the sedimentation vessel to the froth discharge. A retaining baffle preferably is provided to prevent the resin-bearing froth from overflowing with the coal slimes. A deflection baffle also may be provided to deflect the resin concentrate froth toward the froth discharge outlet or toward the skimmer.

An advantageous embodiment of the invention is described below with particular reference to the accompanying drawing, in which the single figure is a perspective, partly in section, of a type of sedimentation apparatus commonly used in coal washeries, but modified in accordance with the invention for the purpose of carrying out the new method.

Run-of-mine resin-bearing coal received at the coal preparation plant generally is first subjected to a primary crushing operation to break the large lumps to commercial size. The crushing operation has not been conducted heretofore for the purpose of liberating the resin from the coal, as it has not been regarded economic practice to degrade the coal by fine crushing solely to recover its resin content. However, the resin is much more friable than the coal, and it usually occurs along seams within the coal. Consequently, when the coal is crushed to reduce it to market sizes, it tends to break along the resin seams. Fines that are unavoidably produced in breaking the coal (both at the mine and in the crushing plant), therefore became enriched in resin content. For example, whereas run-of-mine coal may contain only 2% or 3% of resin, the fines (i. e., the minus  $\frac{1}{4}$  inch material) will contain two or three times this amount of resin. The coal fines constitute roughly 10% to 15% of the amount of coal received at the coal preparation plant, and the better equipped coal washeries have the necessary apparatus to recover a marketable sized coal product from these fines. Since coal washeries begin adding water at the very first stages of the breaking, and add various amounts along the line during the further crushing and screening, it is evident that all fines collect in a coal-water pulp from which it is desirable to recover commercial size coal fines, as by passing the pulp to sedimentation apparatus such as a hydroseparator, a drag classifier, or similar apparatus, wherein the coarser components of the coal fines are caused to settle substantially free from the coal slimes. Due to the continuous flow of pulp into the sedimentation apparatus and movement of rakes in the apparatus, the pulp is agitated properly to promote separation of the slimes (which overflow at the top of the apparatus) from the coarser coal particles (which are discharged at the bottom of the dewatering apparatus, or are mechanically moved up from the bottom to a sand discharge outlet).

The method provided by the invention for recovery of the coal resin is readily applied in connection with the washing of the coal fines. As shown in the drawing, a launder 5 is provided for conducting the previously prepared pulp of coal in water to a sedimentation vessel in the form of a hydroseparator 6. The hydroseparator, for the most part of conventional construction, comprises a large tank 7 in which a fairly large body of the coal-water pulp 8 is very gently agitated and is given a slow rotary motion by means of a plurality of rake arms 9 (only one

rake arm is shown in the drawing). The rake arms are carried on a central column 10 that is rotated by a motor 11 mounted on the usual bridge structure (a fragment 12 of which is shown in the drawing) extending across the tank. The launder 5 discharges pulp into a central feed well 13, which serves to prevent excessive agitation of the main body of pulp 8 by the incoming stream of pulp, and which insures delivery of the incoming pulp at the optimum level below the surface of the pulp. The body of pulp within the tank is sufficiently quiescent so that the larger coal particles settle quite readily to the bottom of the tank, and they are slowly carried by the rake arms 9 to the central sand discharge 14. The current created by the flow of pulp in the apparatus, however, plus the movement of the rake arms 9, agitate the pulp sufficiently to hinder settling of the coal slimes, and these overflow continuously with much of the water into an overflow launder 15. In most coal washeries the slime overflow is sent to a slime-treatment plant for the recovery of very fine size coal.

In employing the apparatus shown in the drawings in accordance with the invention, a flotation reagent capable of promoting flotation of the resin from the coal (such as one of the higher alcohols, pine oil, or the like) is added by a reagent feeder 16 to the pulp as it flows through the launder to the hydroseparator. The reagent feeder may be of any suitable construction. As shown, it comprises a tank 17 containing the reagent, and a rotating bucket elevator 18 which discharges through a trough 19 and funnel 20 into the pulp. The rate at which reagent is delivered to the pulp is governed by the speed at which the bucket elevator rotates.

A short distance downstream from the point at which the flotation reagent is added to the pulp in the launder, a conduit 21 is provided with branch pipes 22 for introducing air into the pulp. We have found that aeration of the pulp as it flows through the launder is of substantial value in promoting flotation of the resin.

When the reagent-treated and aerated pulp enters the hydroseparator, a light fluffy froth of resin concentrate accumulates on the surface 23 of the body of pulp 8. Baffle boards 24, which extend a short distance above and below the surface of the body of pulp near the periphery of the tank but which for the most part are spaced from the overflow weir 25, of the launder 15, prevent this froth from overflowing with the slimes. These baffles, however, do not materially impede the slimes overflow.

A skimmer 26 is provided for skimming the resin concentrate froth from the surface of the pulp to a froth discharge 27. The skimmer shown in the drawings comprises a belt or other flexible member 28 supported on pulleys 29 and carrying a plurality of skimming blades 30. One of the pulleys 29 may be motor-driven in the proper direction so that the skimmer blades move the froth along the surface of the pulp to the froth discharge.

The slow rotation imparted to the body of pulp in the hydroseparator by the rake arms 9 serves to carry the froth rising to the surface of the pulp at any point in the tank eventually under the skimmer 26. Assuming clockwise rotation of the rake arms, a froth-deflecting baffle board 31 may be mounted as shown at the left side of the skimmer so that froth brought under the skimmer by rotation of the pulp is deflected toward the froth discharge and cannot be car-

ried beyond the skimmer. The baffle 31 effectively causes the froth to pile up under the skimmer and so facilitate its removal.

The products produced by the hydroseparator modified and operated as described above are (1) a de-slimed and largely dewatered fine sized coal product withdrawn through the spigot at the base of the sand discharge 14, (2) a slime-coal slurry withdrawn from the peripheral launder 15, and (3) a resin concentrate of good quality withdrawn through the resin discharge 27. The first two products are those ordinarily produced in a coal washery hydroseparator, and they are produced in the modified apparatus in substantially the same manner and at substantially the same rate as heretofore. In addition, however, the third product, the resin concentrate, is effectively recovered with but inexpensive modifications to the apparatus and with no extra treatment charge other than for reagents and aeration of the pulp as it enters through the launder 5.

The foregoing specific embodiment relates particularly to modifications made in a hydroseparator type of sedimentation apparatus in order to effect resin recovery in accordance with the invention. It is understood, however, that other types of sedimentation apparatus often are employed in coal washeries and also lend themselves to resin recovery in accordance with the invention. Sedimentation apparatus usually includes some form of vessel in which a fairly large body of the pulp is maintained and is very gently agitated. In any such apparatus it is a simple matter to provide for skimming a resin concentrate from the surface of the body of pulp therein. Likewise, whatever the form of sedimentation apparatus used, it is a simple matter to add the flotation reagents and aerate the pulp as the pulp flows to the apparatus. Consequently, the invention may be employed for the recovery of resins from resin-bearing coal in any coal washery sedimentation apparatus for the treatment of coal fines, such, for example, as drag or rake classifiers, screw-type classifiers, thickeners, or the like.

We have found that some resin floats and may be recovered by skimming from the surface of the pulp in the sedimentation apparatus even without adding any flotation reagent to the pulp and without aerating. However, the resin recovery is greatly increased by the addition of a suitable flotation reagent to the pulp before it enters the sedimentation apparatus, and the resin recovery is still further increased by aeration of the pulp as above described. As an example of this effect, we were able to obtain a resin recovery from the surface of untreated pulp in a hydroseparator at the rate of about 100 pounds per hour. Upon adding a flotation reagent to the pulp as it entered the hydroseparator and by aerating the pulp, but without any other change in the operating conditions, the resin recovery was increased to over 250 pounds per hour.

In our copending application Serial No. 677,394, filed June 17, 1946, now Patent No. 2,506,301, we have described a flotation process for the recovery of resin from coal in which the pulp density is maintained below about 15% solids, in order to produce a resin concentrate that is especially well-suited for subsequent refining by solvent extraction. In carrying out the present invention, it is feasible and advantageous to control the pulp density of the feed to the

hydroseparator as described and claimed in our said copending application.

Resin recoveries by flotation in accordance with the present invention are not so high as can be obtained if the pulp of resin-bearing coal is subjected to flotation in properly designed flotation machines, incorporated in a special flotation circuit for the pulp. However, the recoveries that can be obtained are very good, and since the method of the present invention is carried out in apparatus generally available in coal washeries, with only inexpensive modifications to the apparatus and with no changes in the washing circuit, the invention lends itself readily to the recovery of resins from resin-bearing coal in existing coal washeries, and from coals in which the value of the resin does not justify installation of special resin recovery equipment.

The quality of the resin concentrate is good. It may contain a somewhat higher percentage of non-resin contaminants than the concentrate produced in a specially built flotation plant employing rougher and cleaner flotation circuits, but it is nonetheless readily amenable to refining by solvent extraction as described in our copending application Serial No. 677,393, filed June 17, 1946 now Patent No. 2,506,300, or by other appropriate means.

We claim:

1. The method of washing raw slimy resin-bearing coal to prepare a clean coal product free from coal slimes, and simultaneously recovering the resin content of the coal in the form of a resin concentrate, which comprises flowing a stream of an aqueous pulp of the raw coal to a substantially quiescent body thereof, incorporating a frothing agent in the flowing stream of coal pulp, aerating the pulp, holding the pulp in said substantially quiescent body for a substantial period of time with the result that a resin concentrate froth collects at the surface of said body while fine coal particles settle from the coal slimes to the bottom thereof, withdrawing a fine coal product substantially free of slimes from the lower portion of said body, skimming the froth of resin concentrate from the surface of said body, and separately withdrawing the bulk of the aqueous phase of the pulp with the coal slimes suspended therein from said body at a level just below the level at which the froth is skimmed.

2. The method of washing raw slimy resin-bearing coal to prepare a clean coal product free from coal slimes, and simultaneously recovering the resin content of the coal in the form of a resin concentrate, which comprises flowing a stream of an aqueous pulp of the raw coal to a substantially quiescent body thereof, incorporating a frothing agent in and simultaneously aerating the stream of coal pulp as it flows to said substantially quiescent body, holding the thus treated pulp in said body for a substantial period of time with the result that a resin concentrate froth collects at the surface of said body while fine coal particles settle from the coal slimes to the bottom thereof, withdrawing a fine coal product substantially free of slimes from the lower portion of said body, skimming the resin concentrate froth from the surface of said body, and withdrawing the bulk of the aqueous phase of the pulp with the coal slimes suspended therein from said body at a level just below the level at which the froth is skimmed.

3. In the washing of raw slimy resin-bearing coal, involving flowing a stream of a pulp of the raw coal in water into a substantially quiescent

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body of such pulp, holding the pulp in such body for a substantial period of time, withdrawing a fine coal product substantially free of slimes from the bottom of said body, and collecting a slurry of coal slimes by overflow from said body, the improvement which comprises incorporating a frothing agent in the stream of pulp as it flows to said quiescent body and aerating said pulp with the result that a resin concentrate froth accumulates on the surface of said body, preventing said froth from overflowing with the slurry of coal slimes, and skimming said froth from said body at a level just above the overflow level of the slurry of coal slimes.

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