

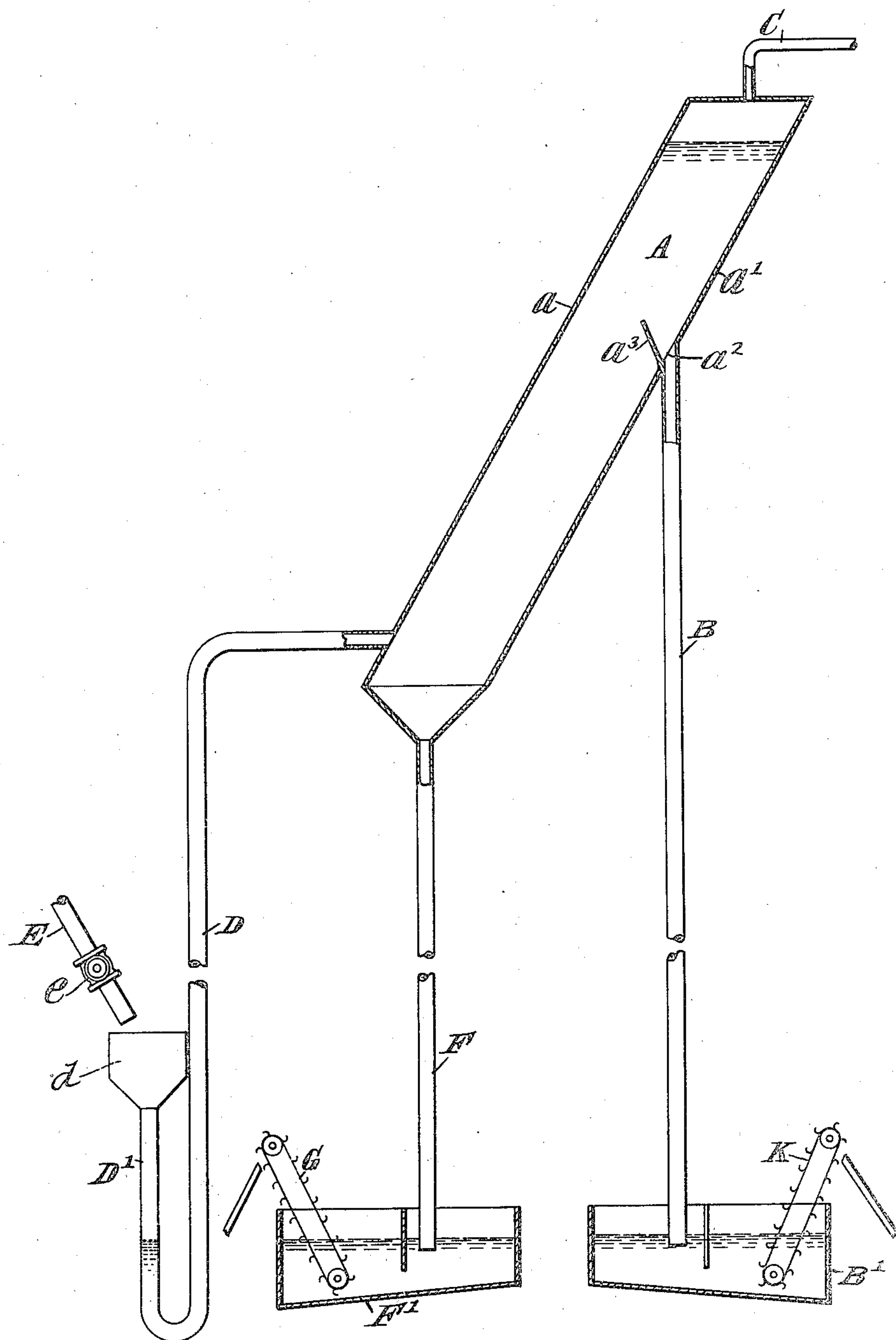
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PROCESS AND APPARATUS FOR SEPARATING CARBONACEOUS MATERIAL

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# UNITED STATES PATENT OFFICE.

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PROCESS AND APPARATUS FOR SEPARATING CARBONACEOUS MATERIAL.

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*To all whom it may concern:*

Be it known that we, BYRON E. ELDRED and ROBERT N. GRAHAM, citizens of the United States, residing, respectively, at Great Neck, county of Nassau, and Long Island City, county of Queens, State of New York, have invented certain new and useful Improvements in Processes and Apparatus for Separating Carbonaceous Material, fully described and represented in the following specification and the accompanying drawings, forming a part of the same.

This invention relates to an improved process and apparatus for the separation of carbonaceous materials from mixtures of the same with inorganic materials.

The object of the invention is to provide a flotation process and apparatus for carrying out the same, which will permit of continuous operation, and give a product of great purity.

The drawing is a diagrammatic view of an apparatus embodying one part of the invention.

In carrying out the process the mixture to be treated is entered into a body of water above whose surface a high vacuum is maintained. The carbonaceous material floats upward and finally accumulates above the surface. After remaining for a time, which is dependent upon the vacuum used, the carbonaceous material sinks in the water and is suitably caught and carried off.

It is to be understood that the term vacuum is used to designate a reduction of pressure below atmospheric and by the term high vacuum is meant such a reduction of pressure as will cause the floated material to sink back into the water within such a reasonable time as will allow the process to be carried out conveniently.

In practice it has been found that when the so-called vacuum is less than 20 inches (mercury) the floated material is likely to stay afloat almost indefinitely so that for the best commercial results it is advisable to use a vacuum which is greater than this, and as a vacuum of 28 inches can be obtained readily and gives efficient results, this has been employed by us in carrying out the process.

In order to keep the sinking carbonaceous material from encountering the rising material, it is advantageous to carry out the

process in such a way as to conduct the rising material along one path and allow the sinking material to traverse another path away from the first path.

In order to carry out the process efficiently the following apparatus has been devised.

Referring to the drawing, A is a vessel for containing water which vessel as shown is arranged at an angle so that it has a wall  $\alpha$  arranged to overhang a considerable part of the body of water so that any carbonaceous material rising toward the surface will be directed by the wall  $\alpha$  along an inclined path toward the surface of the liquid. The opposite wall  $\alpha'$  which, in this case, is inclined also in a direction similar to wall  $\alpha$ , is provided, at a point about in a vertical line with the intersection of the wall  $\alpha$  and the surface of the liquid with a discharge outlet  $\alpha^2$  at one side of which and below the outlet, is a partition wall or deflector  $\alpha^3$  which directs any rising carbonaceous material away from the discharge outlet and also serves to prevent any sinking carbonaceous material from passing below the discharge outlet  $\alpha^2$ .

To the discharge outlet is connected a discharge pipe B for purified material, whose lower end terminates in a sump B', the discharge pipe having its lower end at least about 30 feet below the level of the water in the vessel A so as to form a barometric leg.

The upper end of the vessel A extends above the water level in order to provide a space above said level into which space the floating carbonaceous material may rise.

A vacuum pipe C connected to a suitable air pump (not shown) serves to maintain a vacuum in the space above the water level.

Suitable means are provided for introducing the mixture of materials to be treated into the vessel A without breaking the vacuum therein. In the present example this means comprises a water leg or barometric tube D extending downward from a point somewhat above the bottom of the vessel A to a point more than 30 feet below the water level therein and provided with an upturned end D' into which the mixture of material may be fed, together with water, so as to supply continuously a stream of water and the mixed materials to the interior of the vessel. For the purpose of doing this conveniently the upwardly bent end of the tube



D is provided with a hopper *d*. Delivering to the hopper is a water supply pipe E provided with a valve *e*.

The level of water in the upturned pipe D' is about 30 feet below the water level in the vessel A.

The bottom of the vessel A is hopper shaped and connects to a discharge pipe F which terminates in a sump F' more than 30 feet below the water level in the vessel A.

The operation of the apparatus is as follows:

The vessel A is filled with water to the desired level, in which case the various pipes are also filled, the desired vacuum being established above the surface of the water in vessel A.

The comminuted material to be treated is fed with water to the hopper *d*, the valve *e* in the water supply pipe E being opened to admit a supply of water to the hopper. This causes a flow of water bearing the mixture to the interior of the vessel and also a gradual discharge of water through pipes B and F to the respective sumps.

The comminuted material, as soon as it enters the vessel A, tends to separate. The carbonaceous material floats upward, while the accompanying gangue or other refuse sinks downward and passes out through the pipe F to the sump F' from which it may be removed by any suitable means, as for example, by a conveyor G. The carbonaceous material as it floats upward is deflected by the wall *a* of the vessel, and by the partition wall or deflector *a*<sup>3</sup> toward the surface of the water. It finally accumulates above the surface as a floating mass. After remaining in the high vacuum of the space above the water level, it loses its buoyancy and eventually sinks again. Its downward movement is toward the outlet *a*<sup>2</sup>, through which it passes and finally reaches the sump, B' through the discharge pipe B.

It may be removed from the sump by any suitable means, as for example by a conveyor K.

It has long been known that certain carbonaceous materials, such for example, as charcoal, have the property of absorbing or occluding gases to a large extent. The results of experiments made by us indicate that graphite as well as coal, has the property of occluding gas to a considerable extent, so that when such graphite which has previously been exposed to air or other gas is dropped into water under a pressure less than atmospheric, the absorbed gas will expand and when its expansion is sufficient, the graphite will rise toward the surface of the water.

The amount of expansion of the gases will depend upon the extent to which the pressure in the chamber containing the water has been reduced. With a sufficient vacuum the expansion will always be sufficient to float

the graphite. Furthermore, the surface tension of the water is reduced in a vacuum, so that as the graphite reaches the surface it is not restrained by the surface tension of the water, or at least not to any disadvantageous extent, and therefore is readily lifted above the surface by portions of graphite rising to the surface beneath it.

We have found that carbonaceous materials, particularly graphite, differ from metallic materials in that so-called flotation oils, for example, pine oil, which has been proven the best flotation agent for the usual air flotation processes with metals and metallic ores, is detrimental if used with the present process for carbonaceous materials. Pine oil produces froth and carries fine gangue which prevents the recovery of a high grade product. Concentrates which have a surface film of dried pine oil do not float satisfactorily.

Our process is distinguished by the fact that mica and silica are not floated per se, and it is only where particles of such mica or silica adhere to the graphite or are caught above a mass of graphite on its way to the surface, that mica or silica can contaminate the graphite. The expression mass of graphite is used advisedly because single flakes do not appear to float above the surface readily. The graphite particles appear to coalesce in irregular bodies of greater or less magnitude and then these bodies readily float to and through the surface. Another effect is that the floating mass of graphite assumes a peculiar form, the particles appearing as adherent in broken layers or strata with voids of such magnitude in the aggregate that the coherent mass of graphite now possesses a density less than water and readily floats.

Very high percentage concentrates containing, for example, over 90% graphite are obtainable by this process, and these concentrates may then be subjected to a further treatment by the same process as before, with or without an intermediate washing step, whereby there is obtained a product of still greater approach to complete purity. Finally the product may be subjected to the action of hydrofluoric acid and then washed, so that what is a commercially pure product is finally obtained.

What is claimed is:

1. The process of separating carbonaceous material from mixtures containing the same, which consists in entering the mixture into a body of water whose surface is subjected to a vacuum, whereby the carbonaceous material floats at the surface and above it, maintaining the floated material for a sufficient time and under sufficient vacuum to destroy its buoyancy, whereby it sinks, and collecting the sunken material separate from the rising material.



2. The process of separating carbonaceous materials from a mixture containing the same, which consists in entering the mixture into a body of water whose surface is  
5 subjected to a vacuum, whereby the carbonaceous material tends to float, directing the rising material toward the surface of the water, maintaining it for a sufficient time and under a sufficient vacuum to destroy its  
10 buoyancy whereby it sinks, allowing it to sink along a path out of the path of the rising material, and then collecting it.

3. The process of separating carbonaceous material from mixtures containing the same,  
15 which consists in entering the material into a body of water whose surface is subjected to a vacuum greater than 20 inches of mercury, whereby the carbonaceous material floats at and above the surface, allow-  
20 ing it to remain under this vacuum for a sufficient time to destroy its buoyancy and sink, and collecting the sunken material separate from the rising material.

4. In a flotation apparatus, the combina-  
25 tion, with a vessel arranged to contain a body of water and to provide a space above the level of the water, said vessel having a sloping surface arranged to direct rising carbonaceous material to the surface of the

water, of a discharge pipe opening into the  
30 vessel beneath the surface of the water, and arranged to receive sinking carbonaceous material, means for maintaining a vacuum in the space above the surface of the water, and means for supplying the comminuted  
35 mixture to be treated to the interior of the vessel below the surface of the water and out of a vertical line intercepting said surface.

5. In a flotation apparatus, the combina-  
40 tion, with a vessel arranged to contain a body of water and to provide a space above the level of the water, said vessel having an inclined wall overhanging the body of water, substantially as described, and an  
45 opposite wall provided with a deflector and a discharge outlet below the surface of the water, arranged to receive sinking carbonaceous material, means for leading off the  
50 material from said discharge opening, means for supplying the comminuted material to the body of water, and means for maintaining a vacuum above the surface of the  
water.

In testimony whereof we have hereunto  
55 set our hands.

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