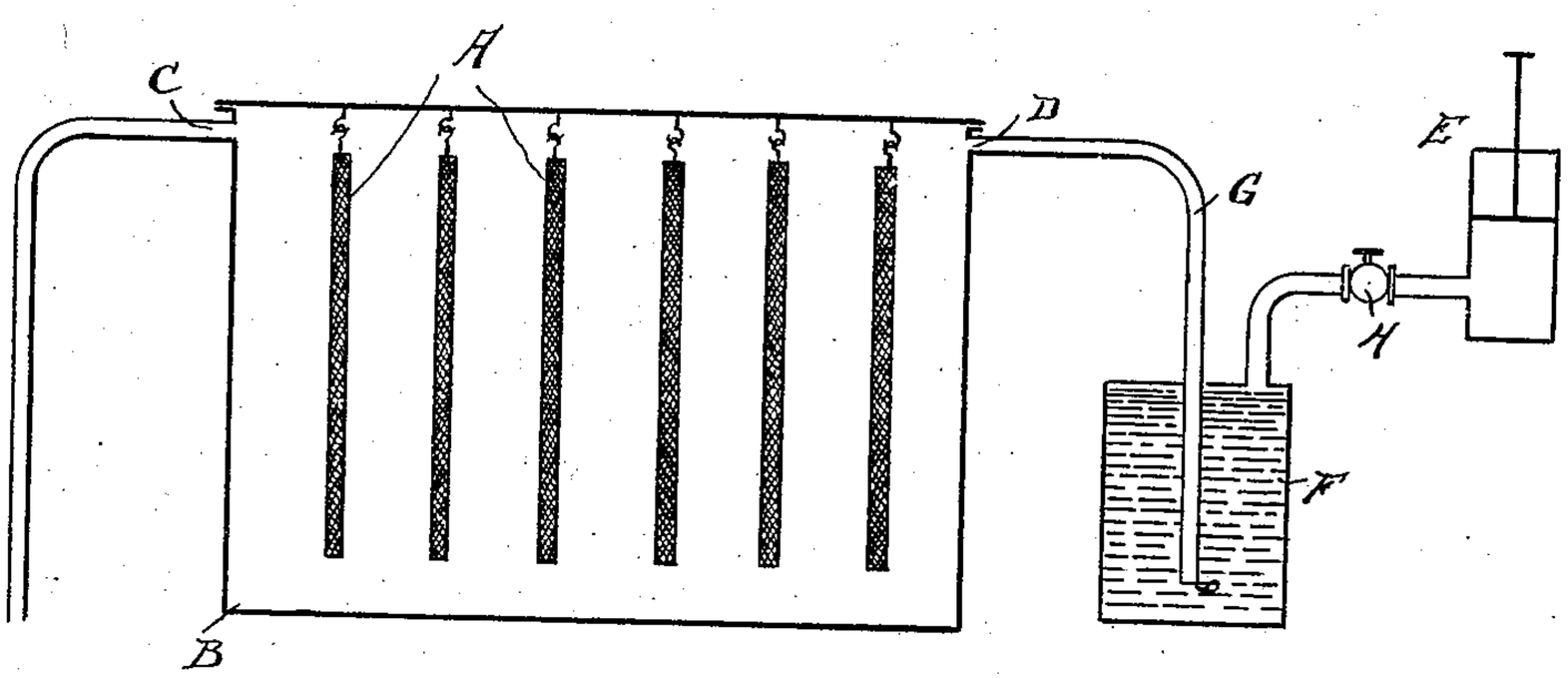
No. 710,005. Patented Sept. 30, 1902.

R. H. PAGE.

PROCESS OF MAKING ORGANIC PEROXIDS.

(Application filed Sept. 10, 1901.)

(No Model.)



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PROCESS OF MAKING ORGANIC PEROXIDS.

SPECIFICATION forming part of Letters Patent No. 710,005, dated September 30, 1902.

Application filed September 10, 1901. Serial No. 74,904. (No specimens.)

To all whom it may concern:

Be it known that I, RALPH H. PAGE, a citizen of the United States, residing at Detroit, in the county of Wayne and State of Michigan, have invented certain new and useful Improvements in Processes of Making Organic Peroxids, of which the following is a specification, reference being had therein to the accompanying drawings.

The invention relates to the art of producing organic peroxids—such, for instance, as benzoyl-acetyl-peroxid.

Heretofore, so far as I am aware, the peroxids referred to have only been produced in an experimental way and by a process in which the yield of the product has been necessarily small, due to the large number of by-products formed.

The present invention consists in an improved method of manufacture wherein a minimum number of by-products are produced and the yield of the article increased to such an extent as to permit of the latter being commercially manufactured.

In carrying out my process the materials employed are an aromatic aldehyde and the anhydrid of a fatty acid, and, briefly stated, the process consists in mixing the materials referred to and gradually oxidizing the mixture until the product in the form of crystals is produced.

More particularly, the steps of the method are as follows: An aromatic aldehyde, such as benzaldehyde, and an anhydrid of a fatty acid—as, for instance, acetic anhydrid—are mixed in preferably, though not necessarily, equal parts, by weight. The mixture is then subjected to the restricted action of an oxidizing agent, preferably air, and to obtain the best results the mixture is treated by the agent while in film form. The films may be made in any suitable manner, such as by a thin layer of the mixture spread upon plates or disks, or preferably by using a sheet or sheets of absorbent material, such as muslin or unsized paper, which is saturated with the mixture. These thin layers or saturated sheets are exposed to the restricted action of an oxidizing agent in such a manner that the oxidation is slowly or gradually effected, as I find by such restricted or slow oxidation the formation of by-products is reduced very greatly.

In practice a number of saturated strips or sheets of absorbent material (designated by the reference-letter A in the drawings) are suspended within a suitable oxidizing-chamber B, preferably having transparent walls to admit daylight, closed except the restricted inlet and outlet openings C and D, as shown. To restrict the oxidation of the mixture, means may be employed for admitting the air gradually within the chamber. I have shown as the preferable mechanism a pump E, connected by a suitable pipe to the top of a receptacle F, filled with a liquid and in which projects a pipe G, leading from an outlet in the chamber B to near the bottom of the receptacle F. Upon the operation of the pump air is drawn from the chamber B and through the filled receptacle, fresh air entering through the inlet-port C. The only object of the receptacle F is so that the air-bubbles may be seen rising through the liquid, and thus will indicate the speed at which the air is being drawn from the chamber.

H is a valve in the pipe G, by means of which the air admitted to the oxidizing-chamber may be controlled.

Any other form of gage and controlling device may be used, the one shown being the form I have heretofore used.

The mixture on the absorbent sheets in the oxidizing-chamber will be very gradually oxidized by the air therein, and the air is very slowly changed, the pump withdrawing part of the air, which is replaced by air coming in the inlet-pipe. The air is withdrawn very slowly. With an oxidizing-chamber eighteen inches by fifteen inches I have obtained good results by drawing out the air, so as to have a bubble a second or less passing up through the fluid in the receptacle F and allowing the oxidation to proceed for forty-eight hours, more or less. This slow oxidation of the mixture produces the organic peroxid, in this case benzoyl-acetyl-peroxid, in crystals on the sheets, and these crystals may be collected from the sheets in any of the several well-known methods.

In the claims I have referred to "restricted" oxidation. By that I mean oxidation which proceeds less actively than would be the case if the sheets or films were exposed to oxidation in the open air. If the oxidation is allowed to take place in the open air, while a very small per cent. of the peroxid crystals is formed a much larger per cent. of by-products are formed, usually benzoic acid and acetic acid; but by restricting the oxidation the peroxid crystals are produced in excess of the by-products.

Where I use the films formed by the saturated strips or sheets of fibrous material, I find that fairly good results can be obtained by hanging those sheets in the open air much better than by the use of any other form of film; but to take the same films and to place them in the chamber and retard the oxidation I obtain still better results.

I assume that the improved result obtained by the use of the films of absorbent sheets is due to the fact that the material is distributed in such fine particles or layers over the fibers of the sheets that the heat produced by the oxidation radiates so quickly that it does not have time to chemically produce the by-products, such as benzoic acid, which would be produced in the open air with thicker films.

What I claim as my invention is—

1. The process of making organic peroxids, which consists in mixing an aromatic aldehyde and the anhydrid of a fatty acid and then oxidizing the mixture and limiting the action of the oxidizing agent to obtain a restricted oxidation for the purpose set forth.

2. The process of making organic peroxids which consists in subjecting a mixture of aromatic aldehyde and anhydrid of a fatty acid in film form to the action of a restricted supply of a gaseous oxidizing agent.

3. The process of making organic peroxids which consists in saturating a sheet or strip of absorbent material with a mixture of aromatic aldehyde and anhydrid of a fatty acid, and subjecting the saturated strip or sheet to retarded oxidation, for the purpose set forth.

4. The process of making organic peroxids which consists in saturating a sheet or strip of absorbent material with a mixture of aromatic aldehyde and anhydrid of a fatty acid, and oxidizing the mixture while supported upon those strips.

In testimony whereof I affix my signature in presence of two witnesses.

RALPH H. PAGE.

Witnesses:
M. B. O'DOGHERTY,
H. C. SMITH.