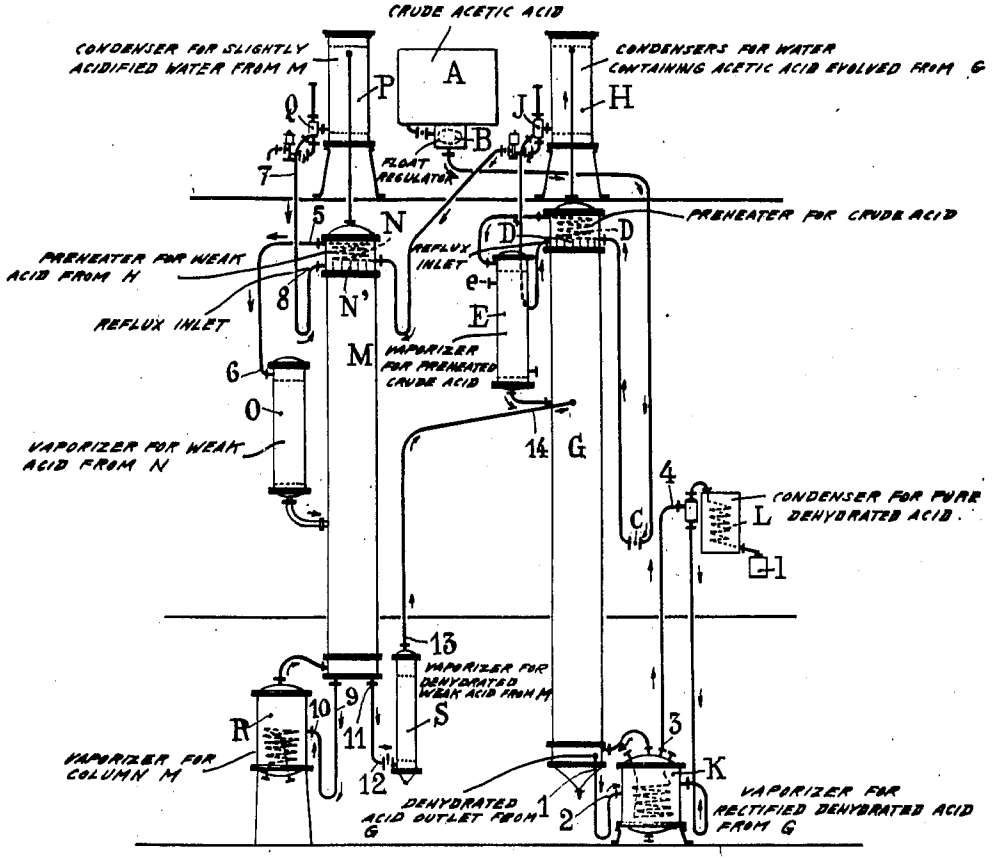
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RECTIFICATION OF ACETIC ACID

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UNITED STATES PATENT OFFICE.

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RECTIFICATION OF ACETIC ACID.

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In whatever way acetic acid is prepared industrially, by mycoderma aceti, by the carbonization of wood or by synthesis, this crude acid is impure and aqueous and it is purified by rectification.

This rectification has this particular feature, that the acetic acid, boiling at about 117° is a tail product relatively to the water, and that consequently the refining effect of the plates is exerted upon the water to be eliminated with the sole purpose of ensuring that this water will take away with it as little acid as possible. It is therefore of importance to make this operation as economical as possible in steam, and it is only by making it continuous that this can be attained. Moreover in a rectifier charged periodically there is inevitably obtained a gradually increasing concentration in acetic acid of the head liquors until a moment arrives when anhydrous acid is obtained, and all these products have then to be passed once more through the rectifier, in order to avoid losses in acetic acid.

There is another advantage in favour of continuity, which is that the apparatus made of copper which is usually employed is only violently attacked by acetic acid in the presence of air. Once the continuous operation is started there is no more air in the apparatus so that the action of the acid is avoided, while for the discontinuous operation, for each stop, that is to say each day, an entry of air takes place during the emptying and recharging period.

The apparatus which is described hereinafter is provided for the purpose of obtaining a continuity in the rectification of acetic acid, commencing with crude acids, having already a certain amount of concentration.

A is the vat of crude acid to be rectified, B the float regulator for the supply, C the supply tap. The liquid is pre-heated in a spiral tube D placed in the head of the first plate column G. H is the cooling condenser.

The water containing traces of acetic acid condensed in H is led away through the test device J, some of which is utilized for the reflux being reheated upon the chimney plate D′ from whence it descends through the column G.

With regard to the pre-heated crude acid, this enters at the top of the tubular heater E which is a stream action tubular heater. This means that the liquid flows through the interior of the tubes and vaporizes there under the influence of the live steam admitted through *e*.

This heating system is provided for the purpose of obviating the use of the steam regulator of the Savalle type which would be dangerous with a liquid of this nature. It is to be understood that with this apparatus a weight of rectifying steam is produced which is exactly equal to the weight of the supply, increasing or decreasing exactly in the same proportions as this supply. It provides therefore of itself an excellent regulation of the rectification.

The degree of concentration at which the water containing a small quantity of acetic acid issues at J depends essentially upon the number of plates, that is to say the more or less perfect working of the column G. For a same hourly weight of crude acetic acid to be dehydrated to glacial acetic acid, the amount of steam used, as is appropriate, proportions itself naturally to the volume of the crude acid, as required, since an extraction of water proportionate to the dilution of the crude acid has to be made.

At the base of the dehydration column G a very concentrated or even completely dehydrated crude acid is obtained. It passes gradually through the siphon pipe 1—2 into the caldron K. This caldron provides at the same time the acid vapour necessary for the rectifier G, and through the tube 3—4, the pure acid which is cooled in the silver spiral tube L, passing out through the testing device *l*.

It is advantageous to provide, before the inlet into the spiral, a vessel 4 for separating out the drops carried along mechanically, for they are contaminated with copper.

The water containing a small quantity of acetic acid which issues from the test safe J, supplies the left hand column M, which is a recuperating column. For this purpose, the said water is pre-heated in the spiral N, and then passes through the pipe 5—6 to the stream action heater O, which is similar to the heater E. The water is entirely vaporized into steam and enters the column M about half way up. Here the object is to make the water as poor as possible in acetic acid pass out at the top; P is the cooling condenser, Q the testing device of the

water condensate which is very poor in acetic acid.

The excess of water containing a small quantity of acetic acid, returning along pipe 7—8, is reheated upon the chimney plate N′ and forms the reflux of the rectifying plates.

At the base of the column M, the siphon pipe 9—10 leads the concentrated acetic acid into the heating caldron R which provides all the necessary heat to the column M.

Here also must be provided an outlet of concentrated acid which will have to return, through the pipe 13—14 to the first rectifier G. In order to be able to regulate more easily the magnitude of this extraction, which is essential, a second heater S which is tubular is provided as shown in the drawing. It is supplied through the pipe 11—12, and at 12 is provided a regulating cock. Once the liquid has entered S, it is completely vaporized and this vapour thus passes naturally into the column G. It will be arranged for this acid vapour to be of about the same acetic concentration as the crude supply liquid.

If the crude liquids are to be dealt with which are very poor in acetic acid, such as pyroligneous liquids from wood carbonization works (approximately 8% of acid), or else vinegar obtained by means of mycoderma aceti, these poor liquids would be supplied into the column M and no longer into G.

The operation would be carried out in M in such a way as to obtain water containing as little acid as possible at the outlet from P any vapours of acetic acid which might be carried over being bubbled through water containing carbonate of soda in solution, or else milk of lime, or any alkali.

The rectifier G would become a finishing column. It would be supplied exclusively by the vapours from the heater S.

What I claim:

1. Apparatus for the continuous rectification of crude acetic acid comprising a first rectification column adapted to be continuously fed with crude acetic acid, means for preheating the crude acetic acid, and means for vapourizing the preheated acid fed to the said column, a condenser communicating with the top of said column to condense water containing a small quantity of acetic acid evolved from said column, a pipe from said condenser to convey condensate to said column to provide reflux therefor, a vapourizer for the rectified dehydrated acid communicating with the bottom of said column, a condenser communicating with said vapourizer, a liquid separator interposed between said condenser and vapourizer, and a receiver for the pure dehydrated acetic acid from said condenser.

2. Apparatus for the continuous rectification of crude acetic acid comprising a first rectification column adapted to be continuously fed with crude acetic acid, means for preheating the crude acetic acid, and means for vapourizing the preheated acid fed to the said column a condenser communicating with the top of said column to condense water containing a small quantity of acetic acid evolved from said column, a pipe from said condenser to convey condensate to said column to provide reflux therefor, a vapourizer for the rectified dehydrated acid communicating with the bottom of said column, a condenser communicating with said vapourizer, a liquid separator interposed between said condenser and vapourizer, a receiver for the pure dehydrated acetic acid from said condenser, a second rectification column communicating with and adapted to receive weak acetic acid evolved from the first rectification column, means for preheating and means for vapourizing the said weak-acid supplied to said second column, a condenser communicating with the top of said second column, a pipe from said condenser to convey condensate to said second column to provide reflux therefor, a vapourizer communicating with the bottom of said second column to supply hot vapours thereto, a second vapourizer communicating with the bottom of said column to vapourize the concentrated acetic acid therefrom and a pipe for conveying said concentrated acid vapours from said vapourizer to the first mentioned rectification column.

3. Apparatus as claimed in claim 2 in which the vapourizers for the preheated acid supplied to the columns constitute regulators for regulating the heating of the acid in proportion to the degree of hydration thereof.

In testimony whereof I have signed my name to this specification.

GEORGES FELIX LEGENDRE.