July 24, 1962 SHIGERU SAITO 3,045,461

APPARATUS FOR ACETYLATING CELLULOSE FIBERS IN GASEOUS PHASE

Filed July 1, 1960

FIG. 1.

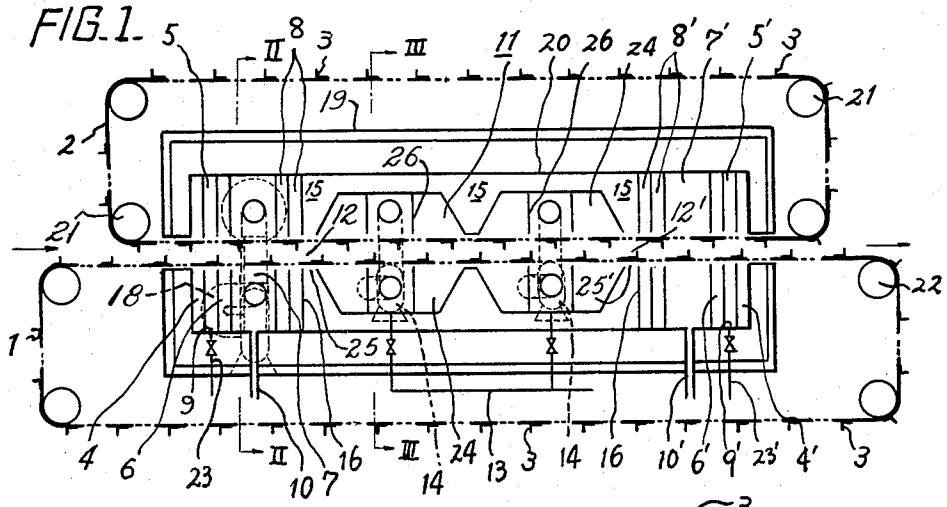


FIG. 2.

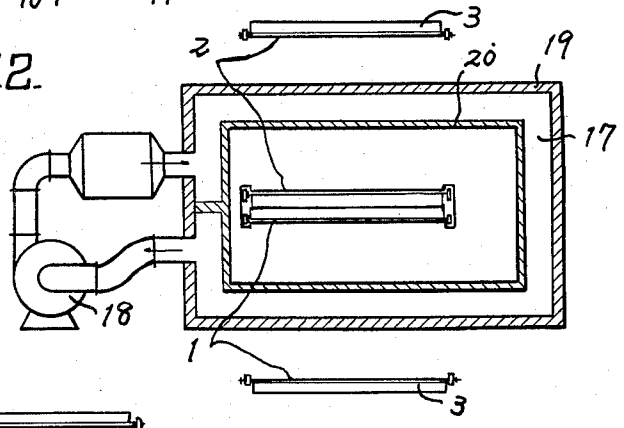
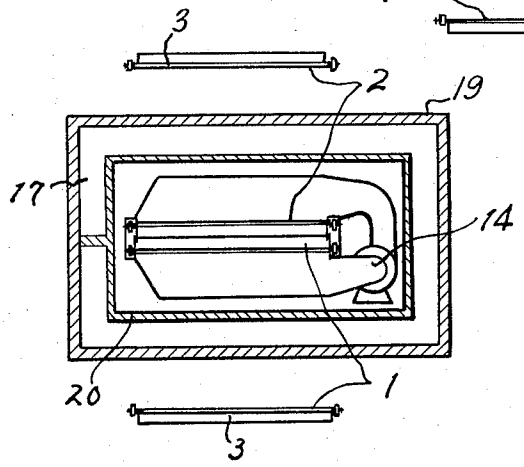


FIG. 3.



United States Patent Office 3,045,461 Patented July 24, 1962

3,045,461

APPARATUS FOR ACETYLATING CELLULOSE FIBERS IN GASEOUS PHASE

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Filed July 1, 1960, Ser. No. 40,286
Claims priority, application Japan Feb. 24, 1960
4 Claims. (Cl. 68—5)

The present invention relates to an apparatus for continuously acetylating cellulose fibers, particularly in gaseous phase, such as cotton fiber, viscose rayon, or copper-ammonium rayon or the like.

In usual fiber acetylating apparatus, acetalizing apparatus, etc., the reaction between the tows, filaments, staples, yarn or fabrics of natural or artificial fibers and the reaction vapour of organic gas, such as acetic anhydride, acetic acid, etc. is performed in a hermetically sealed chamber. Accordingly, there has been a serious disadvantage in that said reaction could not be effected continuously.

The elimination of the above mentioned disadvantage has been made by introducing an apparatus, wherein a gas-seal means is provided in an inlet and outlet portions for fibers respectively. Across said means, inert gas is forced in which does not participate in the reaction, thereby preventing reaction gas from dispersing outwardly. In such an apparatus, the dispersion of the reaction gas filling the reaction apparatus is prevented by thus formed gas seal.

An acetylating apparatus for cellulose fibers has been introduced according to the present invention, for a continuous mass-production of acetate fibers of uniform quality obtained by acetylation from cellulose fibers by using organic gases, in a safety operation and with low consumption of chemicals, characterized in that an upper and a lower endless belt each equipped with stays over all the outer peripheral surface are respectively united together outside the apparatus and so positioned as to pass through a reaction vessel proper, said reaction vessel proper comprising a reaction zone, which consists of a fiber inlet, a fiber outlet, a pair of dehumidifying means, a zone for preventing leakage of reaction gas, a gas reservoir, a zone for preventing the dilution of reaction gas and a uniting portion as well as a reaction chamber, whereby dehumidified air or dehumidified inert gas is fed in said dehumidifying means, and acetic anhydride vapour or acetic acid is fed in said reaction zone to effect acetylation on cellulose fibers and discharge waste gas out of said gas reservoir.

According to this invention, said cellulose fiber is introduced between the two endless belts provided above and below the apparatus, said two sets of endless belts traveling in the reaction zone with said cellulose fiber held between the two sets of endless belts, through the inlet to the acetylating apparatus, by revolving said endless belts. Dehumidified air or dehumidified inert gas, such as nitrogen gas, etc. is introduced through the inlet at an adequate velocity for preventing the reactional gas from dispersing into circulating atmospheric air, and then acetylation is effected in the reaction zone with mixed vapour of acetic anhydride, acetic acid, etc. Further, in order to remove the heat generated in the reaction, said fibers held between the endless belts are subjected to a strong blowing-off operation by reaction gas, during acetylation. Thus, the fiber having been converted into cellulose acetate from the acetylating reaction is led out of the apparatus through the outlet portion constructed entirely similarly as the inlet. The dilution of the vapour concentrations of acetic anhydride, acetic acid, etc. due to the introduction of atmospheric air into the reaction zone, and the

lowering of reaction velocity as well as the danger of explosion are avoided. Further, the apparatus proper is arranged to be enclosed by compulsorily circulating hot blast in order to prevent the gases such as acetic anhydride, acetic acid, etc. from condensing due to cooling from outside of the apparatus proper.

Further, according to the present invention, said two sets of upper and lower belts ordinarily formed of metal gauzes or screening are provided with stays around the outer periphery, which act as buffers to the gas flow through said inlet and outlet portions. Moreover, as said upper and lower endless belts are respectively united together outside the reaction apparatus, the operation and upkeep of the apparatus are advantageously performed and the reaction gas is fed in the reaction chamber. Then, said acetylation of fiber is advantageously accelerated and simultaneously the reaction heat is removed, since the cellulose fiber held between said upper and lower endless belts is vigorously blown through from below to above and from above to below.

Further, according to this invention, as the reaction gas flows into the gas reservoir at a velocity below 0.5 m./sec. through the gas dilution preventing zone, independently of the size, shape, etc. of the apparatus, no other gas can flow into the uniting zone as well as into the reaction chamber after reverse flow through said gas dilution preventing zone. Accordingly, as the concentration of gas in the reaction chamber and uniting zone can be held at 100%, the apparatus of this invention is safe and economically advantageous.

According to the present invention, it is obvious from the foregoing description, that as dehumidified air or dehumidified inert gas, such as nitrogen gas, etc. is rendered to flow into the gas reservoir at the velocity below 0.5 m./sec. through the reaction gas leakage preventing zone, thus the reaction gas can perfectly be prevented from leaking out of the apparatus, after reversely flowing through said gas leakage preventing zone. Accordingly, it is remarkably advantageous with respect to the operational atmosphere and to the consumption of chemicals.

Furthermore, according to the present invention, said dehumidified air or dehumidified inert gas, for instance, nitrogen gas, etc. flows out of the apparatus at the volocity below 0.5 m./sec. through the moisture preventing zone hence, no atmospheric air containing moisture can leak into the gas reservoir thus, the loss of chemicals can be prevented and the acetylating reaction can advantageously be effected with safety.

In the following, the present invention is described in further detail with respect to one embodiment of this invention by reference to the accompanying drawings, of which—

FIG.1 is a sectional plan schematically showing a cross section of the apparatus of the invention;

FIG. 2 is a sectional side view of the apparatus cut along the II—II line of FIG. 1; and

FIG. 3 is a sectional side view thereof cut along the III—III line of FIG. 1.

Referring to the drawings, **1** is a lower endless belt; **2** is an upper endless belt; those belts are ordinarily composed of metal gauzes, ec. All over the outer peripheral surfaces of respective belts, stays **3** as shown in FIGS. 2 and 3 are arranged equidistantly, said upper endless belt **2** and said lower endless belt **1** form respectively an endless conveyor belt, in that the upper belt **2** is passed over four guide pulleys **21** and the lower belt **1** is similarly passed over four guide pulleys **22**. Thus, said endless belt revolves by a driving force from a suitable driving source not-illustrated, and cellulose fibers, such as cotton, viscose rayon or copper ammonium rayon, etc. are conveyed as held between said two endless belts from the left to right in the direction of the arrow in FIG. 1. In this case,

said cellulose fibers is fed continuously on the left side of the lower endless belt 1, which is then held between the upper endless belt 2 and finally discharged again out of the lower endless belt 1, through inlet 4, reaction zone 11 and outlet 4'. In the said condition, cellulose fibers is submitted to acetylation in reaction zone 11, said fibers being either staple and long fibers in the form of tows, thread or fabric.

As illustrated in FIG. 1, the reaction vessel 20 comprises an inlet 4 and an outlet 4', moisture preventing zones 5, 5', gas-leakage-preventing zones 6, 6', gas reservoirs 7, 7', gas-dilution-preventing zones 8, 8' and a reaction zone 11, whereby the said dehumidifying zones 5, 5', gas-leakage-preventing zones 6, 6' and the gas-dilution preventing zones 8, 8' are divided by baffle plates 61 encircling the upper and lower endless belts 1 and 2 respectively. In respective connecting zones for the moisture preventing zones 5, 5' and the gas leakage prevention zones 6, 6' inlets 9, 9' are provided for dehumidified air or dehumidified inert gas, for instance, nitrogen gas, etc., through which said dehumidified air or inert gas is introduced by an appropriate means from outside via feed pipes 23, 23'. Gas reservoirs 7, 7' are connected with the discharge pipes 10, 10' for excess gas, said gas being withdrawn by an unillustrated fan having an exhaust pressure enough to enable said gas to pass through the recovering step. The reaction zone 11 comprises an outer chamber 15 and a reaction chamber 24 disposed inwardly of chamber 15. The reaction zone 11 is completely enclosed, except for the inlet 12 and outlet 12', through which the upper and lower endless belts 1 and 2, respectively, are transmitted to said reaction zone 11. The number of sealed reaction chambers 24 is only two in this particular embodiment. In the actual operation, however, one or more can be used, and said number can suitably be chosen. In this case, the inlet and outlet 12, 12' are passages respectively for the upper and lower endless belts 1, 2.

In the reaction chamber 24, the reaction vapour of acetic anhydride or acetic acid, which is generated at any suitable location (unillustrated) is fed respectively through feed pipe 13. Large clearances 25, 25' are formed between the gas-dilution-preventing zones 8, 8' and the inlet 12 as well as outlet 12', said clearances 25, 25' being in communication through a by-pass pipe (not illustrated). In the illustrated embodiment, instead of the bypass pipe, a space designated as an outer chamber 15 is provided inside the apparatus body 20, which chamber provides communication between the clearances 25, 25'. Moreover, baffle plates 26 are provided in said reaction chamber 24, with a similar purpose as baffle plates 16 in the gas-dilution-preventing zone.

The reaction gas thus introduced in the reaction chamber 24 through feed pipe 13 is forced up or down through the cellulose fibers tightly held between the upper and lower endless belts 1 and 2 by means of fan 14 as shown in FIG. 3, to accelerate the acetylation of said fiber and simultaneously remove the reaction heat. In this case, in reaction chamber 24, respective and independent fans 14 are provided for flowing alternately from above and below said reaction chamber.

The reaction chamber 24 is filled with reaction gas which flows finally out of the reaction chamber through the inlet 12 and outlet 12' and fills also uniting zone 15, and further passes through the gas-dilution-preventing zones 8, 8' and flows into the gas reservoirs 7, 7'. On the other hand, the dehumidified air or inert gas fed through inlets, 9, 9' provided in the moisture preventing zones 5, 5' is discharged in about a half quantity thereof from inlet 4 out of the apparatus through the moisture preventing zone and the other half quantity thereof flows into the gas reservoir 7, 7' like said reaction gas through the gas-leakage-preventing zones 6, 6'.

In the gas circulating system, it has become evident that an overall equilibrium of the circulated gas can be obtained, independent of the size and shape of the apparatus.

The reaction gas is caused to pass through the reaction gas-dilution-preventing zones 8, 8' at a velocity below 0.5 m./sec. and flow into the gas reservoirs 7, 7', thereby preventing other gases from flowing into the uniting zone 15 and the reaction chamber 24, while dehumidified air or dehumidified inert gas is caused to flow into the gas reservoirs 7, 7' at a velocity below 0.5 m./sec. through the moisture preventing zones 5, 5' and inlet 4 and flow out of the apparatus, thereby preventing gas reservoirs 7, 7' from moisture-bearing atmosphere leaking into said reservoirs.

Thus, it is possible to keep the concentration of reaction gas in the reaction zone 11 at 100%. Accordingly, is it safe and economically advantageous since the consumption of chemicals, and simultaneously the diminution of chemicals can be most effectively prevented. Such important advantages as described above can be accomplished.

Furthermore, according to the present invention, as a violent circulation of gas occurs as described before in the reaction chamber 24, the gas stream is directed often from the inlet 12 to outlet 12' of said chamber, or sometimes an impactive stream occurs in the direction from outlet 12' to inlet 12. Said impactive stream is absorbed by the uniting zone 15 having substantially no resistance to a gas stream, with the result that because of being short-circuited among inlet 12, reaction zone 11, outlet 12' and uniting zone 15, said impact stream does not force through baffle plates 16 and stays 3, and further does not run out through gas reservoirs 7, 7' and gas-leakage-preventing zones 6, 6' and moisture preventing zones 5, 5'.

On the other hand, in the reaction apparatus proper 20 as shown in FIGS. 2 and 3, hot blast held at a higher temperature than the due point of reaction vapour is fed through fan 18 to circulate therein and simultaneously the apparatus is perfectly covered with a blast chamber 17 for forced circulation around the periphery and, moreover, said blast chamber 17 is perfectly wrapped with insulating material 19 to reduce its heat loss, in order to avoid the condensation of vapour of acetic anhydride and acetic acid by cooling on the wall of the apparatus proper.

While I have shown and described the preferred embodiment of my invention, I wish it to be understood that I do not confine myself to the precise details of construction herein set forth by way of illustration, as it is apparent that many changes and variations may be made therein by those skilled in the art without departing from the spirit of the invention or exceeding the scope of the appended claims.

What I claim is:

1. Apparatus for acetylating cellulose fibers in gaseous phase comprising a first wire screen endless belt and a second wire screen endless belt spaced below and adjacent said first endless belt, each endless belt being provided with a plurality of stays on the outer surface thereof, the upper belt stays being arranged alternately with the lower belt stays, said endless belts being adapted to carry cellulose fibers, means forming a reaction vessel having an inlet and an outlet through which the endless belts pass, said reaction vessel having a moisture preventing zone and a reaction chamber, dehumidified gas supply means operatively connected to said moisture preventing zone, acetic vapor supply means operatively connected to said reaction chamber whereby dehumidified gas supplied to said moisture preventing zone provides a seal preventing moisture laden atmospheric air from entering the reaction vessel and acetic vapor supplied to said reaction chamber effects acetylation of cellulose fibers held between the endless belts passing through the reaction vessel.

2. Apparatus for acetylating cellulose fibers in accordance with claim 1 wherein fans are mounted in the reaction chamber above and below the endless belts for

forcing the acetic vapor through the cellulose fibers to accelerate the acetylation of the fibers and to remove the reaction heat therefrom.

3. Apparatus for acetylating cellulose fibers in accordance with claim 1 wherein axially spaced baffle plates are mounted within the reaction vessel adjacent the endless belts whereby the baffle plates and stays prevent the escape of acetic vapor from the reaction vessel.

4. Apparatus for acetylating cellulose fibers in accordance with claim 1 wherein the reaction vessel is enclosed by a hot blast chamber to prevent condensation of the acetic vapor.

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