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Eickemeyer

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[54] **APPARATUS FOR PROCESSING DEGRADATION OF CELLULOSIC MATERIALS**

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[30] **Foreign Application Priority Data**

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[51] **Int. Cl.** **C13k 1/02**

[58] **Field of Search** ..... 127/1, 37; 23/272, 272.6 R, 23/312 A

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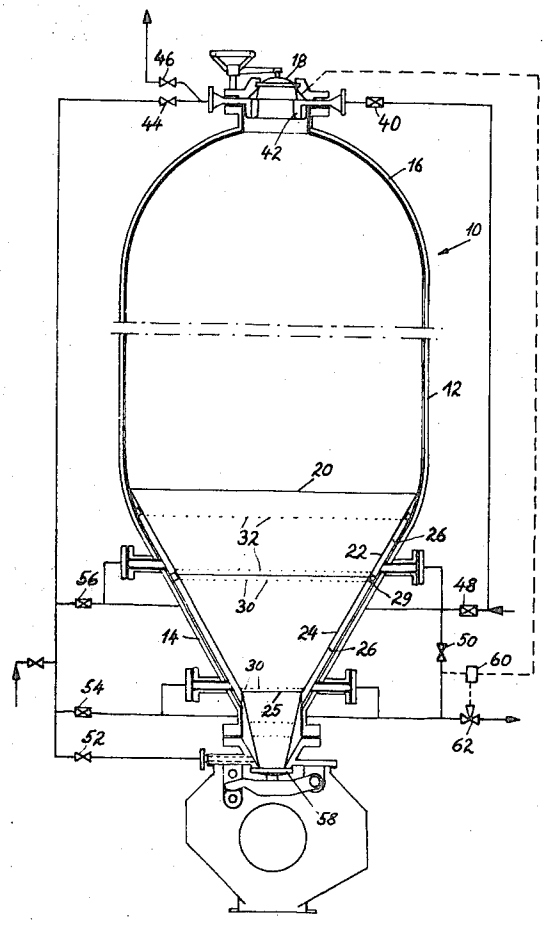
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[57] **ABSTRACT**

An apparatus, for decomposing cellulosic materials, in the form of a percolator vessel, has a conical filter disposed with apertures which is mounted on the lower portion of the vessel. A system of liquid and steam valves controlling the inlets and outlets to the percolator vessel is used to treat cellulosic material in a series of steps with resulting decomposition of almost the entire load of material initially placed into the percolator.

14 Claims, 3 Drawing Figures



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SHEET 1 OF 2

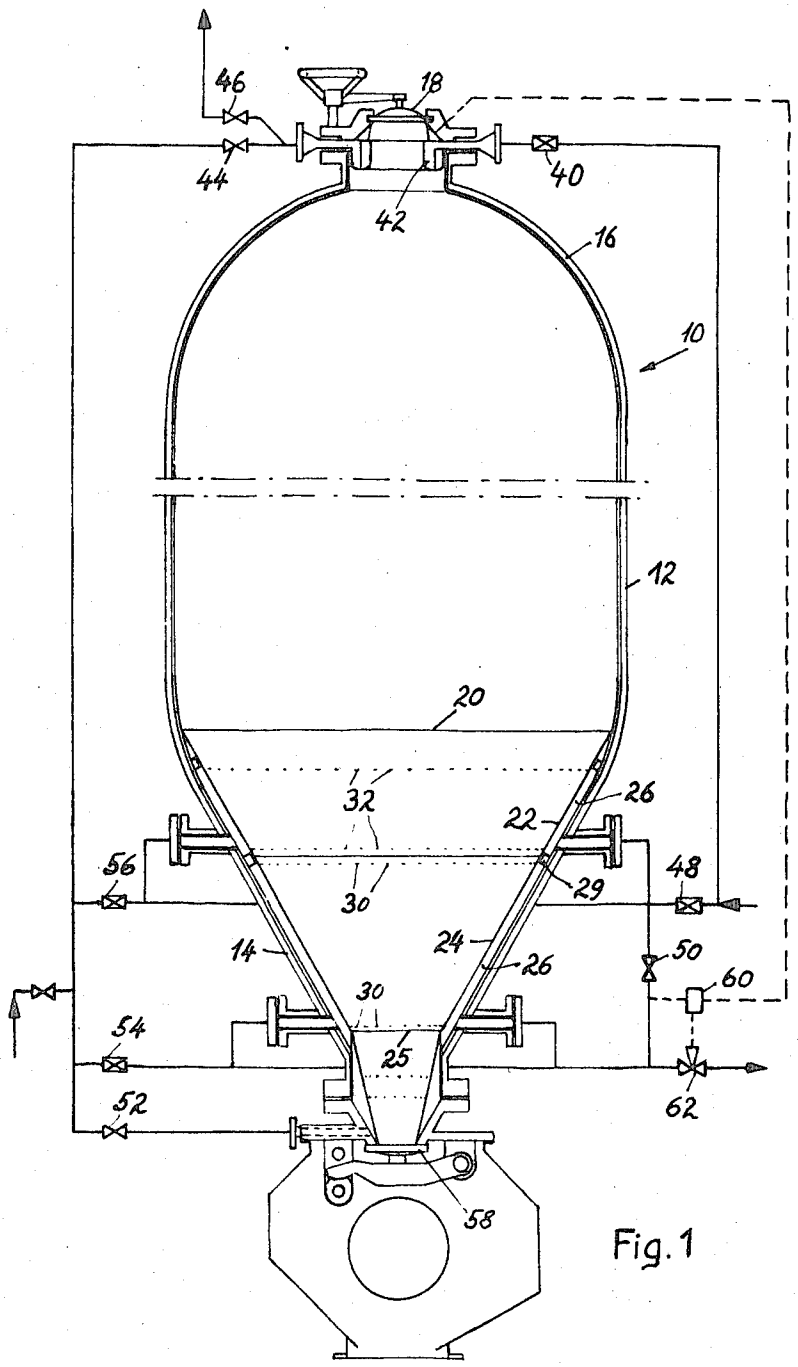
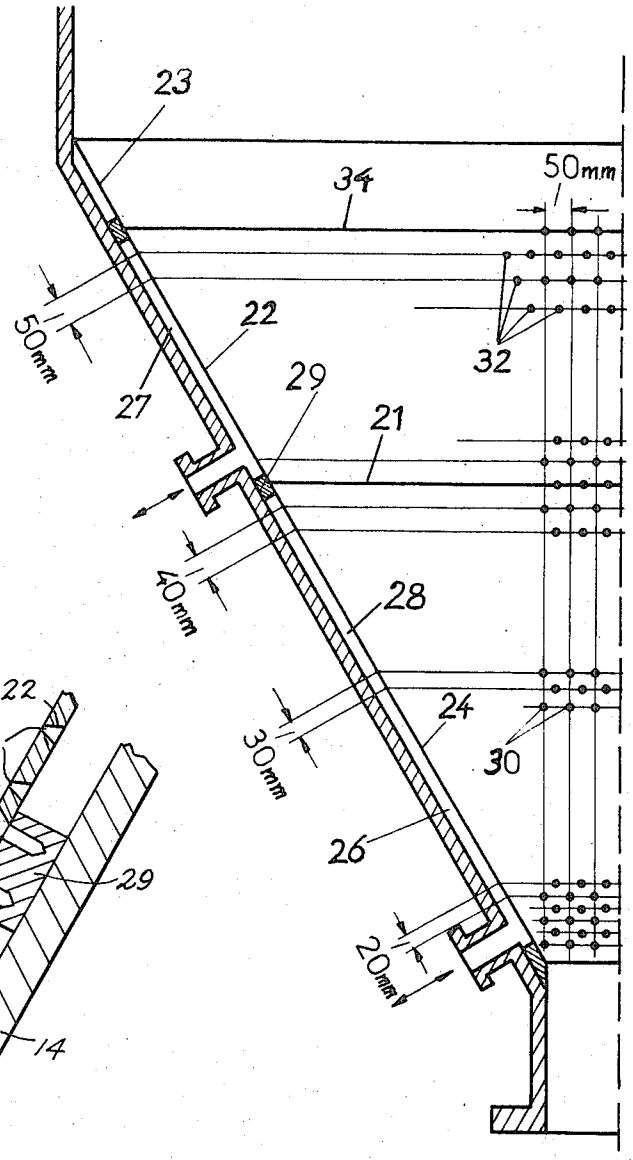


Fig. 1

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Fig. 2



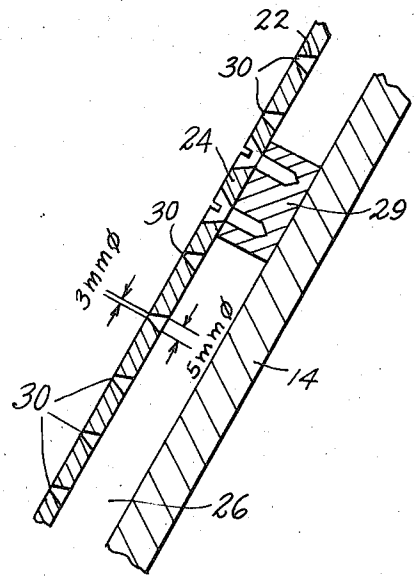


Fig. 3

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APPARATUS FOR PROCESSING DEGRADATION OF CELLULOSIC MATERIALS

CROSS-REFERENCE TO RELATED APPLICATIONS

This application is a division of my copending application, Ser. No. 767,669, filed Oct. 15, 1968, now U.S. Pat. No. 3,640,768.

BACKGROUND OF THE INVENTION

The invention relates to an apparatus for the chemical degradation of cellulosic materials in a percolator.

The conventional apparatus of this type are designed in a manner that the cellulosic material is first subjected to heat by means of steam in order to dispel the air from the percolator. It has also been proposed to evacuate the percolator prior to heating in order to remove at least the major part of the air. The reaction liquid is then added subsequently to the initial heating zone. The liquid is usually preheated to a temperature above 100°C. Very substantial amounts of liquid are necessary to effect the impregnation of the material up to saturation because of the absorptive power of cellulosic materials.

The difficulty with the apparatus of the prior art is that in their use certain portions of the material are left which are not completely saturated with liquid. The well known Scholler percolation process as disclosed for instance in German patent 640,775 therefore uses a multiplicity of liquid charges of equal acid concentration in succession. The probability of an insufficient impregnation with liquid in this case is usually eliminated after a few successive charges.

However, there are problems with the rather high amount of liquid that is necessary and the concentration obtained in the final product.

SUMMARY OF THE INVENTION

The present invention uses an apparatus in the form of percolator vessel. An annular filter, subdivided into two sections, is mounted on the interior of the percolator vessel. The lower portion of the percolator vessel as well as the filter sections are conical and are spaced so as to provide a uniform distance between the interior surface of the vessel and the exterior surface of the filter. A horizontal sealing ring fills this intersurface space so that the materials flowing into or out of the individual filter sections can be controlled independently by means of a series of steam and liquid valves.

Each filter section is provided with a multiplicity of apertures. These apertures diverge from the interior to the exterior of the filter. By properly selecting the ratio between the combined cross sectional areas of the apertures at their narrowest, or interior ends, and the cross sectional area of the filter at its narrowest, or lowest point, best results are obtained. It has been found that a combined aperture area of 0.2 to 4 percent of the narrowest filter cross-sectional areas has given satisfactory results.

The size and the spacing arrangements of the aperture can also enhance the performance of the apparatus. Apertures should have minimum diameters of between 2 and 5 millimeters at the narrowest ends, preferably between 3 and 4 millimeters. To further optimize the efficiency of the apparatus, the apertures should be more closely spaced near the bottom of the filter so that the lower section has 50 to 67 percent more apertures than the upper section.

The novel features which are considered as characteristic for the invention are set forth in particular in the appended claims. The invention itself, however, both as to its construction and its method of operation, together with additonal objects and advantages thereof, will be best understood from the following description of specific embodiments when read in connection with the accompanying drawing.

BRIEF DESCRIPTION OF THE DRAWING

FIG. 1 shows a percolator vessel in schematic manner in accordance with one embodiment of the present invention;

FIG. 2 is an enlarged fragmentary section of the percolator vessel of FIG. 1 showing the details of a filter used in conjunction with the percolator;

FIG. 3 is a further enlarged fragmentary section through the filter of the percolator.

DESCRIPTION OF THE PREFERRED EMBODIMENT

Referring to FIG. 1, a percolator vessel 10 is shown to consist of an upper cylindrical portion 12 and a lower conical portion 14. The upper cylindrical portion 12 is capped by a top dome 16 which is generally spherically shaped and is open in its central top portion. A rapid action closure 18 seals the opening in dome 16.

The suitability of the present apparatus results from the use of an annular filter 20 having two sections 22 and 24. Filter sections or portions 22 and 24 are each conically-shaped but of different cross sectional areas. The angular inclinations of the conical filter sections 22 and 24 can be selected to be about 60 degrees. The filter sections 22 and 24 are constructed to approximate the dimensions of the lower conical portion 14, with the upper filter portion 22 being of greater cross-sectional area than the lower filter portion 24. The upper and lower filter portions 22 and 24 are mounted on the interior surface of lower conical portion 14 by means of cross members 26. Such mounting provides an enclosed annular space between the exterior surface of the filter and the interior surface of the lower conical portion. This space acts as a convenient duct for transporting liquids and steam to and from the filter portions 22 and 24 in relation to a duct and valve system to be described. A sealing ring 29, which spans the common circumference 21 of the upper and lower filter portions 22 and 24, separates the interwall space likewise into an upper and lower portions, 27 and 28 respectively, and this serves to isolate each filter portion and subject it only to the conditions existing in the interwall space adjacent to it.

The filter sections 22 and 24 have apertures 30 arranged in horizontal rows 32. The apertures 30 have diverging diameters which increase from the interior of the filter 20 to the exterior or periphery thereof as shown in FIG. 3. The minimum diameter of the apertures 30 is between 2 and 5 millimeters, preferably between 3 and 4 millimeters. The apertures 30 are distributed over nearly the entire filter surface. The combined cross-sectional areas of all the apertures 30 should be between about 0.2 and 4 percent, preferably 0.5 and 1.5 percent, of the cross-sectional area of the lower filter section 24 at the narrowest point 25, which would

correspond to the opening at the bottom of filter section **24.**

Although the above description involves the use of two filter sections, it is however, possible to use more than two sections. In such a case, it may be desirable, for more control, to seal the sections against each other, as described above.

The apertures **30** are also to be spaced, with respect to each other, to enhance the operation of the filter portions **22** and **24.** As described above, the apertures are aligned on horizontal rows **32.** FIG. **2** illustrates more specifically the preferred arrangement of apertures **30.**

The horizontal rows **32** are not spaced equidistantly over the entire filter surface but their vertical distances decrease in a downward direction. Thus, the uppermost two rows are space 50 millimeters apart as measured along the filter's peripheral surface. This distance gradually decreases to 40 millimeters near the mating point **21** between the upper filter section **22** and the lower filter section **24** and finally becomes equal to 20 millimeters at the lower end **25** of the lower filter section **24.** This gradual decrease should generally be such that the distances near the bottom are equal to about one-half to one-third of the maximum distance existing at the top.

The horizontal spacing of the apertures **30** is selected on the basis of the filter dimensions. Thus, the horizontal spacing selected for the apertures **30** in the top row **34** must be such that the combined cross-sectional areas of all the apertures **30** should be between about 0.2 and 4 percent, preferably 0.5 and 1.5 percent, of the cross-sectional area of the lower filter section **24** at its narrowest point **25.** The horizontal spacing between apertures **30** in the top row **24** is shown to be 50 millimeters. The initial vertical spacing, as discussed above, is made equal to the horizontal spacing selected for the top row.

In addition, the apertures **30** are so situated that they are horizontally displaced from the apertures on adjacent rows so that the inter-aperture distances on the surface of the filter **20** are made as equal as possible.

With this arrangement, the lower filter portion **24** has about 50 to 67 percent more apertures than the upper filter portion **22.** This results in desirable flow conditions.

The upper part **23** of the upper filter section **22** does not contain any apertures in its surface. This serves both to prevent undesirable peripheral circulation of the steam and liquid and also to prevent an edge flow at this height in the filter **20.**

A valve and duct system is used to feed and empty the percolator vessel **10** at suitable steps during the decomposition process, to be described. Thus, inlet valve **40** feeds fluid into distributor member **42** and inlet valve **44** does the same with steam. Valve **46** is an outlet drive for steam which is to be removed from the percolator **10** during the process. Valves **48, 50** and **52** are shut off valves while valves **54** and **56** regulate the supply of steam passing through the percolator **10.** Opening **58,** at the bottom of the percolator **10,** is an outlet for the contents of the percolator.

The use of the present invention involves as a first step the introduction of the reaction liquid and steam at a temperature of at least 100° whereby a reaction liquid steam mixture rises upwardly in the percolator. As a result, the open space between the cellulosic particles is completely taken up and in addition the reaction liquid will penetrate into the individual particles and the displaced air in each case will rise towards the top. After complete covering of the charge with liquid, one may permit the charge to stand for a few minutes to accomplish complete saturation. The amount of liquid which is in excess of the saturation of the cellulosic material and which is in the interstices between the cellulosic particles is then drained downwardly through the filter. It is only after this step that the remaining material which is now just saturated with liquid is heated by the admission of steam through the filter and is thus brought up to the reaction temperature.

This kind of procedure results in a substantial saving in the amount of steam and, on the other hand, because of the lower amounts of liquid required, permits a higher concentration of the sugars to be obtained in the same reaction time.

There results also a substantially lower flow resistance for the steam which arises from the bottom to the top and thus a lower pressure and temperature differential from bottom to top.

If the purpose of the process is the saccharification of the cellulosic material, the reaction liquid will be a suitable mineral acid solution of which the ion concentration in the first stage where the saccharification of the hemicellulose is effected may amount to about that of a 1 percent concentrated sulfuric acid, while in the second stage where the cellulose is subjected to saccharification after the sugars formed from the hemicellulose have been washed out at a reduced temperature and reduced acid concentration, acid may be used of which the ion concentration is equivalent to that of a 3–5 percent sulfuric acid.

Preferably, a preliminary hydrolysis of the hemicellulose of the pentosan-containing starting material is effected followed by an alkali conversion of the cellulose. The process of the invention contemplates that in this case a reaction liquid is used for at least part of the conversion of the hemicellulose consisting of dilute material acid solution having a hydrogen ion concentration corresponding to about 1 percent sulfuric acid. The reaction temperature should be below 140°C. This will be followed by a washing out of the sugars formed from the hemicellulose at a reduced temperature and acid concentration by means of a plurality of liquid charges of reduced sugar concentration. These charges may be obtained from previous percolation runs. The last wash is effected with water. After that, a new reaction liquid is added which contains solvents for the lignin such as sodium hydroxide, sodium sulfide and sodium carbonate in a concentration to provide for the necessary reagent in the cellulosic material after expulsion of the excess liquid. A reaction temperature in this stage is used of at least 140°C. The soluble alkali lignin is then again washed out by means of a plurality of liquid charges of decreasing temperature and decreasing concentration of alkali lignin and other unspent reagent. These liquid charges are again obtained from previous percolation runs. Finally, there follows a wash with water. The necessary temperature is accomplished by means of steam which is introduced in each case between two charges. As a result, pure cellulose will remain in the percolator.

Preferably, nitric acid of a hydrogen ion concentration as indicated is used, at least in case of the hemicellulose saccharification, that is during the preliminary

hydrolysis, but possibly also during the hydrolysis of the cellulose.

The fresh reaction liquid which is necessary for the conversion of the cellulose may contain alkali lignin obtained from a previous percolation run in addition to fresh solvents for the lignin.

Preferably, the fresh reaction liquid consists at least partially of one or several discharges resulting from previous lignin solution percolations to which the necessary additional fresh chemical may be added. After starting the reaction with the cellulosic material which is saturated with acid, the subsequent reaction time necessary to accomplish the saccharification of the hemicellulose or cellulose is of course accompanied also by decomposition of the formed sugar under the conditions of the reaction. As a result, a certain flattening out of the possible maximum sugar concentration occurs after a certain reaction time. The sugar concentration at a continuation of the reaction would decrease.

It is therefore an additional feature of the invention that the reaction time is selected so that the formed sugar has at least 70 percent, and at most 90 percent, of its possible maximum concentration. When this point is reached the sugar is then washed out at a reduced temperature and acid concentration. New reaction liquid is subsequently added, particularly in cases of the saccharification of the cellulose followed again by washing out at reduced temperature and acid concentration.

The elution or washing out of the formed sugar can be effected by a plurality of liquid charges as indicated.

The temperature is preferably 130°–140°C for the conversion of the hemiceulose with a reaction time of about 40 minutes. In the first stage of the conversion of the cellulose, the temperature is 160°–170°C with a reaction time of 40 minutes, and in the second stage of the conversion of the cellulose the reaction temperature is between 170° and 190°C prefereably 180°C with a reaction time of 30 minutes.

A suitable apparatus for operating the described process is a percolator as described above.

hemicellulose 1 preferably

33 tons of beech shavings containing about 26 tons dry wood substance were placed in a percolator **10** having a contents of 100 m^3 and a diameter of 3.2 m in its upper cylindrical portion **12**, while the lower conical portion **14** had an inclination of 60° and a total height of 2.7 m. The percolator **10** also was provided with a top dome **16** in which there were disposed rapid-action closures **18**, a liquid valve **40** and a steam inlet valve **44**, as well as a steam outlet valve **46** and a distributor **42** for both the liquid and the steam. The beech shavings were then subjected to a preliminary hydrolysis to xylose and to a main hydrolysis to dextrose followed by a processing of the lignin residue. The percolator **10** consisted of copper plated sheet steel and was adjusted to withstand an operation pressure of 13 atmospheres above atmospheric pressure.

In the lower conical portion **14**, a vertically subdivided filter **20** consisting of filter sections **22, 24**, each composed of perforated frusto-conical cooper sheets was installed which was supported by cross-members **26**. The upper filter portion **22** had a height of about 700 mm measured in the wall portion of the conical section **14** of the percolator **10** and was sealed against the lower filter portion **24** which had a height of about 1400 mm (measured again in the conical wall portion) by a sealing ring **29**. The upper filter section **22** had apertures of conical form arranged in horizontal rows **32**. The apertures had a diameter at the center of about 3 mm and at the periphery of about 5 mm with a horizontal distance from each other of 50 mm. The rows **32** of apertures **30** in the top levels were spaced by 50 mm which was reduced at the lower edge of the upper filter section **22** to about 40 mm. The apertures **30** of the individual rows **32** were offset against each other.

The lower filter section **24** likewise had apertures **30** arranged in horizontal rows **32** and having the same diameters and same horizontal distances. At the upper edge of the lower filter section **24** the distance of the aperture rows **32** was about 40 mm which was reduced gradually up to the lower edge to about 20 mm. The apertures of the individual rows **32** again were offset against each other.

In this manner, there were obtained about 50 percent more apertures **30** in the lower filter section **24** than in the upper filter section **22**.

It is also possible to use percolators which are steel-plated with a nickel-chromium, non-corroding steel ("V 4 A") which are similar to the conventional cellulose boilers. The filter **20** in the conical portion **14** in this case likewise is formed from V 4A sheet steel. This type of filter is particularly useful for obtaining pure cellulose after carrying out the preliminary hydrolysis.

The different stages of the process carried out in the above-described apparatus will now be discussed:

I. First Stage — Preliminary Hydrolysis of Hemicellulose

In order to effect the complete covering of the wood charge consisting of beech wood shavings, about 63 m^3 of sulfuric acid of a concentration of about 1 percent were used. The initial heating of the charge was effected by means of steam admitted through the valve **54** and the lower filter section **24**. The heating was carried out to a temperature of about 100°C and a brief circulation of the steam was effected by opening the upper outlet valve **46**. Dilute sulfuric acid was then admitted at a temperature of about 90°C during a period of about 12 minutes through the upper filter section **22** of the filter **20** by means of opening the valve **48** and closing the valve **50**. Simultaneously, steam was introduced through the lower filter section **24** while throughout this operation a small amount of steam was permitted to escape through the upper outlet valve **46**. Thus, a mixing temperature of at least 100°C was obtained right above the liquid filled percolator zone.

After covering the charge with liquid the upper steam outlet valve **46** was closed and steam was admitted through the upper inlet valve **44** into the upper section of the percolator until a pressure of about 1 atmosphere above atmospheric was reached, which was then maintained at a constant value by adjusting further admission of steam. The excess liquid of about 25 m^3 was drained through both filter sections by opening the valve **50** and by means of the control valve **62**, while a pressure difference was maintained between the percolator top section and the space behind the filter (impulse control device **60**) between zero at the beginning of the discharge and increasing gradually to 0.5 atmospheres at the end of the discharge, which altogether lasted for about 15 minutes. The charge which now was saturated with dilute sulfuric acid was then heated,

after closing the control valve **62** and opening the valves **54** and **56**, to a temperature of 135°C by admitting steam through the two filter sections. The temperature corresponded to a saturated steam pressure in the percolator upper part **12** which resulted from maintaining a pressure difference of at least 0.8 atmospheres between the lower and the upper portion by means of the impulse control device **60.**

After reaching this pressure in about 15 minutes, steam was passed through the entire vessel for a few minutes by a slight opening of the upper outlet valve **46** in order to obtain a completely uniform heating of the charge. The outlet **46** was subsequently closed and the desired pressure in the upper section of the percolator **12** was maintained for the reaction time of about 40 minutes by specific additions of steam through the filter **20** (valve **54** and possibly valve **56**). This means that preferably the steam was introduced only through the lower filter section **24** (valve **54**) whenever a comparatively small amount of steam was necessary in order to maintain the reaction temperature.

Thereafter, a liquid charge of about 32 cubic meters containing a xylose concentration of about 8 percent and having a temperature of about 90°C was pumped into the percolator **10** from above through the valve **40.** This resulted in a substantial cooling and development of steam in the cake and provided for a mixing temperature of about 120°C corresponding to a pressure of 1 atmosphere above atmospheric pressure.

Thereafter, steam was introduced from the top and the pressure was increased from 1 atmosphere above atmospheric to 2 atmospheres and this pressure was maintained by introducing further controlled amounts of steam. The liquid thereby was forced into the cake and caused condensation of the steam which was at a higher temperature and which was still left in the pores of the material. This resulted in an increased extraction effect.

The liquid was then drained through the valve **62** by adjusting the pressure difference between the upper percolator part **12** and the space behind the filter sections to a value of initially zero, and then increasing during the reaction time of about 15 minutes to a value of 0.5 atmospheres.

A new liquid charge was thereafter admitted in an amount of 32 m^3 having a xylose concentration of about 6 percent and a temperature of about 90° through the valve **40.** This resulted in a further reduction of the mixing temperature to about 110°C. The further process was similar to the above-described operation and also appears from the following schedule:

TABLE I

Liquid Charge No.	Xylose concentration wt.-%	Amount m^3	Discharge No.	Xylose concentration wt.-%	Amount m^3
1	8	32	1	9.3	33
2	6	32	2	8.5	34
3	4	32	3	6.5	34
4	3	25	4	5	26
5	—	25	5	4	27
6	—	20	6 tg 2.5		21

The first discharge and part of the second discharge constituted the so-called extraction amounting to 5.9 t extract having a xylose contents of 4.7 t in an about 9 percent solution. This corresponded to a yield of 18 percent of the beech wood dry substance. This extract could then be processed further to the desired final products.

The remaining discharges were saved for making the extraction charges **1** to **4** for the next run. They were stored in storage vessels. The fifth and sixth charge preferably consisted of de-ionized water.

II. Second Stage — Hydrolysis of Cellulose

After completion of the preliminary hydrolysis, which at the end proceeded with a temperature of only slightly above 100°C, a new charge was added in an amount of 40 m^3 at a temperature of about 90° and consisting of about 4 percent sulfuric acid. This charge was added from below through the upper section of the filter **22** while at the same time steam was introduced through the lower filter section **24** in order to obtain a mixing temperature of at least 100°C.

After permitting the mixture to stand for a few minutes, the steam percolator pressure in the upper section **12** was increased to 1 atmosphere above atmospheric by a heavy addition of steam from the top. This pressure was then maintained by admitting controlled additional amounts of steam. The excess liquid was again drained through the liquid outlet valve after adjusting the pressure difference to a value of zero at the beginning of the discharge and increasing to about 0.5, this pressure difference existing between the percolator upper portion and the space behind the filter **27.** The discharge lasted about 15 minutes. A reaction temperature was then established of about 165°C by introducing steam through the entire filter surface at a pressure difference of at least 0.8 atmospheres between the lower and the upper section of the percolator. This reaction temperature corresponded to the saturation steam pressure in the percolator upper part **12.** The time for heating to the desired temperature amounted to about 20 minutes.

The stated reaction temperature was then maintained for about 40 minutes by admitting controlled amounts of steam through the lower filter section **24** (this was the reaction time for the first phase of the sugar formation in the main hydrolysis). The washing of the sugar took place thereafter at a reduced temperature and acid concentration in a similar manner as in the preceding description and observing the following schedule:

TABLE II

Liquid Charge No.	Dextrose concentration wt.-%	Amount m^3	Discharge No.	Dextrose concentration wt.-%	Amount m^3
1	5.8	32	1	7.3	37
2	4.5	32	2	6.1	32
3	3.0	32	3	4.5	32
4	1.6	25	4	3.2	25
5	—	20	5	2.1	20

The first discharge and part of the second discharge totaling about 4.6 t of extract, of which 3.85 t were dextrose, and which were present in a solution of 58 t were then eliminated and used for the further processing to desired end products. The other discharges as in the previous case were employed to prepare the charges **1** to **4** for the next run and for this purpose were placed in storage tanks.

III. Third Stage — Hydrolysis of Remaining Cellulose

About 30 m^3 of 4% sulfuric acid were then pumped at a temperature of 90° through the upper filter section **22** into the percolator **10.** Simultaneously, steam was added through the lower filter section **24** as in the preceding case.

By increasing the pressure in the upper portion of the percolator to about 1 atmosphere above atmospheric, the excess liquid was drained in the same manner as previously. There then followed the heating of the charge through the entire filter to a reaction temperature of about 180°, that is, to a corresponding saturated steam pressure in the upper section **12** of the percolator **10.** This temperature was maintained for about 25 minutes by introducing controlled amounts of steam through the lower filter section **24.** This was then followed by again washing out the formed sugar as in the previous example, but following the scheme shown in Table III.

TABLE III

Liquid Charge No.	Dextrose concentration wt.-%	Amount m³	Discharge No.	Dextrose concentration wt.-%	Amount m³
1	5.4	26	1	6.1	36
2	4.0	25	2	5.3	25
3	3.6	20	3	4.6	20
4	1.65	20	4	3.4	20
5	—	20	5	1.8	20

The first discharge with an extract of 3.2 t in the form of a solution of about 38 t and containing 2.4 t dextrose was then eliminated and, together with the extracts from the preceeding extraction, was subject to the further processing to obtain the desired end products. Altogether there were obtained in this process 7.8 t extract in the form of a solution of 96 t which contained 6.25 t dextrose. This corresponded to a means concentration of about 6.5 wt.-% dextrose or 8.1% dry substance consituting a degree of purity of about 80 percent.

IV. Fourth Stage — Lignin Recovery

At the end of this entire extraction process, there were left in the percolator a lignin residue comprising about 9 to 9.5 t dry substance and containing still about 18 to 19 t water. The evacuation of this residue was effected through an evacuation outlet **58** at the bottom end of the conical portion **14** of the percolator after the temperature of the percolator was brought up by admitting steam from below (valves **52, 54** and **56** which were opened consecutively) to 150°–175°C corresponding to a steam pressure between 5 and 8 atmospheres above atmospheric. The lignin cake thus was broken up by expansion of the water under pressure and was removed mixed with steam in about 1 minute. The lignin-steam mixture was then separated in a cyclone in which the lignin residue was received at the bottom end through outlet **58** and the steam permitted to escape at the top through rapid action closure **18.** After closing of the rapid action closure **18** the percolator was then ready for another run.

EXAMPLE 2

It may be of interest in a particular case only to go through the preliminary hydrolosis and subsequently to obtain the lignin from the more or less still intact cellulose of the starting material such as beechwood. The preliminary hydrolosis in this Example therefore was the same as in Example 1 except that a shorter reaction was used in order not to damage the cellulose and also employing nitric acid instead of sulfuric acid in order to be able to work with percolator that was plated with chromium nickel steel plates of the "V 4A" type. The xylose rate thereby was reduced from 18 percent to between 10 and 16 percent of the initial beechwood dry substance.

The concentration of the xylose solution is therefore somewhat lower and in case of somewhat reduced amounts of liquid amounted to about 8.0 percent. After the preliminary hydrolosis there was obtained a contents in the percolator amounting to about 20 tons of hot dry substance which were then processed to pure cellulose as presently described.

CELLULOSE RECOVERY

A charge was formed by dissolving fresh free agents or the melt obtained from burning the waste liquor. This material was dissolved in a suitable fraction of waste liquor. The conversion could then be effected by the so-called sulfate process. In that case the melt dissolved in a fraction of the waste liquor was reacted with alkali for instance of the type $Ca(OH)_2$. There resulted an amount of fresh free agent dissolved in spent liquor of about 5.2 t consisting of about 3.5 t NaOH, 0.85 t Na_2S and 0.85 t Na_2CO_3. This presupposed that the excess from the last run which resulted after the percolator filling was included in a composition. The charge then contained: 5.2 t of fresh free reagent plus 4.6 t waste liquor-dry substance plus 30 t water.

The liquid excess then contained 1t chemicals + 1t waste liquor-dry substance + 15t water. This excess was drained through the lower portion of the percolator after admission of the charge through the upper portion of the filter and simultaneous admission of steam through the lower portion followed by a short time of standing in order to effect the equalization of the concentration. In the percolator there then remained 20 tons of hot dry substance saturated with a solution containing 4.2t chemicals, 3.6 t waste liquor and 55t water.

These contents of the percolator which was saturated with liquid were now heated to a temperature of 145° by admitting steam through the filter and establishing a pressure difference of at least 0.8 between the lower and the upper part of the percolator and then subsequently maintaining the temperature for about 30 minutes by admitting controlled additional amounts of steam.

This step was followed by the washing out of the formed alkali-lignin by means of a plurality of liquid charges which had been obtained from the waste liquor of the previous runs. The first or the first two discharges however were eliminated. These discharges contained the highest concentration. The last two charges of liquid were again water in order to obtain a well-washed cellulose. The temperature in the percolator could be kept at a decreasing level for consecutive extraction steps similar to the previously described operation. The temperature prior to the last charge should still be at least 110°C. The following table illustrates the details of the proceeding.

TABLE IV

Liquid Charge No.	Waste Liquid-Dry Substance Wt.-%	Amount m³	Discharge No.	Waste Liquid-Dry Substance Wt.-%	Amount m³
1	10	30	1	17.6	47
2	7.4	30	2	12.7	31
3	3.6	30	3	8.8	31
4	1.6	25	4	7.1	25
5	—	25	5	3.8	25
6	—	25	6	2.0	25

The first discharge containing 10 t of waste liquor-dry substance was then split as follows: 3.2 t waste liquor-dry substance and 15 t water were used together with the excess from the reaction which contained 1t fresh reagents, 1t waste liquor-dry substance and 15 t water for the purpose of dissolving 3.2 t fresh reagents containing about 0.4 t filler materials. Instead of fresh reagents, it is, of course, also possible to use the melt from the firing of the liquor which then must be subjected to an alkaline treatment. The total solution was used as a charge for the next run.

The remainder of the first run amounting to:

from the first charge	6.8 t waste liquor-dry substance in 32 t water
from the second charge	3.7 t waste liquor-dry substance in 25.5 t water
amt. altogether to:	10.5 t waste liquor-dry substance 57.5 t water

was eliminated and used for the burning of the waste liquor for the purpose of steam generation and recovery of free agents.

The remainder of the second discharge and the following discharge were used for making up in sequence the charges 1 to 4 for the next percolation run.

It is noted that it would also be possible to distribute the chemicals added to two or three charges in which case the second and even the third charge may already be simultaneously extraction charges.

Prior to these reaction-extraction steps, the mixture may be heated in a similar manner as in the preceding Examples to between 140° and 150°C and be maintained at this temperature for 15 to 30 minutes. The further extraction charges could be carried out at stepwise lower temperatures. There resulted in the percolator a cellulose (12 to 13 tons of dry substances) having a high content of α cellulose which could then be evacuated with water to a cellulose storage tank and could later be submitted to further processing.

Without further analysis, the foregoing will so fully reveal the gist of the present invention that others can by applying current knowledge readily adapt it for various applications without omitting features that, from the standpoint of prior art, fairly constitute essential characteristics of the generic or specific aspects of this invention and, therefore, such adaptations should and are intended to be comprehended within the meaning and range of equivalence of the following claims.

What is claimed as new and desired to be protected by Letters Patent is set forth in the appended claims:

1. A percolator for chemically decomposing materials, comprising an upright percolator vessel having an inner surface; annular filter means in said vessel and comprising an annular wall substantially coaxial with said inner surface and connected at the upper and lower ends to said inner surface while being spaced over a major portion thereof from said inner surface so as to form with the latter at least one closed annular space, said peripheral wall being provided with a plurality of apertures arranged at different levels of said wall and each diverging from the inner to the outer surface of said peripheral wall and providing communication between said annular space and the interior of said vessel; and valve controlled passage means communicating with said annular space for feeding a fluid into and out of the same.

2. A percolator as in claim **1,** wherein the total cross-sectional areas of the apertures in said peripheral wall is equal to between 0.5 and 1.5 percent of the interior cross-sectional area of the percolator vessel at the bottom thereof.

3. A percolator as in claim **1,** wherein said apertures have a minimum diameter at the inner surface of said wall of between 2 and 5 mm.

4. A percolator as in claim **1,** wherein said apertures have a minimum diameter at the inner surface of said wall of between 3 and 4 mm.

5. A percolator as defined in claim **1,** wherein the combined cross sectional areas of said apertures are substantially in the range corresponding to 0.2–4 percent of the interior cross sectional area of the percolator vessel at the bottom thereof.

6. A percolator as in claim **1,** wherein the apertures are arranged in a plurality of horizontal rows and wherein the distance between adjacent rows decreases in downward direction.

7. A percolator as in claim **6,** wherein the spacing between the lowermost rows is equal to between ½ and ⅓ of the spacing between the two top rows.

8. A percolator as in claim **6,** wherein the apertures of adjacent rows are disposed in a manner as to be offset, in the horizontal direction with reference to each other.

9. A percolator as in claim **6,** wherein the horizontal spacing between said apertures is equal to 50mm in the top row and decreases uniformly towards the bottom and wherein the vertical spacing between apertures is equal to 50 mm, as measured along the surface of the filter, and decreases uniformly towards the bottom to 20 mm.

10. A percolator as in claim **1,** wherein the percolator vessel has a lower conical portion and the filter means has a conical peripheral wall, and wherein the filter means is attached to the wall of said conical portion of the percolator vessel.

11. A percolator as in claim **10,** wherein the top end of said peripheral wall portion is free of perforations.

12. A percolator as in claim **10,** wherein said conical peripheral wall of said filter has an inclination of 60 percent.

13. A percolator as in claim **10,** wherein the filter means has an upper section and a lower section, the lower section having between 50 and 67 percent more apertures than the upper section.

14. A percolator as defined in claim **13,** and including a sealing ring at the joint between said upper and lower sections and sealingly engaging the inner surface of said and the outer surface of said peripheral wall so as to divide said annular space into a pair of annular spaces separated from each other, said valve controlled passage means comprising separate valve controlled ducts respectively communicating with said pair of separate annular spaces.

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