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PROCESS FOR MANUFACTURING FURFURAL

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This invention relates to improvements in processes for manufacturing furfural and particularly to improvements in processes wherein furfural is produced by steam digestion of pentosan-containing roughage materials such as corncobs, oat hulls, rice hulls, cottonseed hulls, etc.

Heretofore, furfural has been produced on commercial scale mainly by processes essentially like that described in U.S. Patent No. 1,735,084. In the process described in this patent the pentosan-containing material is dampened with a dilute, nonvolatile acid and then subjected to steam digestion in a closed vessel providing means for ingress and egress of the steam. Relatively high steam pressures of the order of 60 pounds per square inch gauge are maintained. The steam flow maintained effects both digestion of the pentosan-containing material and steam distillation of the resulting furfural to a recovery system. The apparatus employed includes a rotary steam digester or autoclave which is provided with a plurality of pipes or steam inlets in order to pass a large number of fine jets of steam through the reaction material undergoing treatment. A valve-controlled outlet for the steam is provided so as to permit maintenance of the desired steam pressure within the digester and allow steam distillation of the furfural produced in the digester to the recovery system. Improvements on this process or similar processes have been essentially only in the apparatus used or in the manner of employment or disposition of the apparatus. For example, instead of a single rotary digester a plurality of digesters may be set up so as to distill their product into a common header leading to the recovery system. By such arrangement only one of the digesters need be down at one time and the over-all operation is in effect continuous.

The prior art processes have not been entirely satisfactory since the yield of furfural which can be obtained by these processes are far removed from what is theoretically possible. Some increase in yields can be obtained by improvements in utilities or equipment employed but the cost of these improvements cannot usually be justified by the amount of increase in yields obtained. Regarding equipment, the digester facilities are the most expensive single item in the construction of a furfural plant. This large expense is a result of the severe conditions (high pressures) for which this equipment must be designed and the fact that the raw material employed is of rather low bulk density, thus limiting the size of the digester charge.

It is an object of this invention to provide improved processes for producing furfural in which increased yields are obtained economically when the processes are practiced on a commercial scale.

Another object of the invention is to provide improved processes for obtaining furfural in which a higher bulk density furfural source-material is obtained which can be charged in larger quantities to digesters of given size, thus increasing the output per digester.

Other objects of the invention will become apparent from the description hereinbelow and the hereunto appended claims.

The above objects are accomplished according to this invention by improvements on those processes in which furfural is produced by steam digestion of a pentosan-containing material in the presence of a nonvolatile acid catalyst, e.g. sulfuric acid, phosphoric acid, alkane sulfonic acids, etc. According to the invention the raw material is first subjected to the flow of steam therethrough (i.e. pre-steaming) with a steam pressure on the raw material less than that necessary to produce digestion of the raw material. Accordingly, no substantial amount of furfural is produced at this time. The pre-steaming is then followed by conventional steam digestion to produce the furfural.

In one embodiment of the invention the pre-steaming of the raw material is effected by a continuous flow of steam through the raw material under a steam pressure no greater than about 20 pounds per square inch gauge, preferably no greater than about 15 pounds per square inch gauge, and for a time period of about 15 to about 75 minutes, preferably about 30 to about 60 minutes. Where a time period of about 60 minutes is employed the steam pressure is substantially no greater than atmospheric pressure.

The raw material is pre-steamed with or without the acid catalyst and added moisture (sufficient only to wet the material) customarily employed in the subsequent conventional steam digestion.

The pre-steaming is effected in the same vessel as is employed for the subsequent steam digestion or is accomplished in a separate vessel. In the latter event, the vessel may be of light, inexpensive construction since it need only withstand the low pressures employed in the aforedescribed pre-steaming.

As a result of the pre-steaming there is a substantial increase in the bulk density of the pentosan-containing raw material. This shrinkage effect on the raw material is particularly advantageous where the pre-steaming is accomplished in a separate vessel apart from the regular digester vessel. As a result of the increase in bulk density a greater amount of the pre-steamed raw material may be charged into the digester and accordingly there will be a greater output of furfural per digester charge.

In order to further illustrate the invention the following examples are given, it being understood however, that these are not for purposes of limitation but rather of illustration. In each example the steam source employed for both pre-steaming and steam digestion was superheated steam (530° F. at 150 p.s.i.g.), the quantity of steam entering the digester being regulated by a valve in the steam inlet line. The desired steam pressure on the charge within the digester was maintained by controlling the escape of steam from the digester to the condenser recovery system.

Example 1

An apparatus essentially the same as that illustrated in the aforementioned U.S. Patent No. 1,735,084 was employed with the exception that the cylindrical digester was replaced by a spherical one. The digester was charged to its maximum operating capacity with 16,500 pounds of corncobs having 14.6% moisture (14,000 lbs. dry weight). According to the A.O.A.C. method for determining furfural the potential furfural charge would be about 23% of the dry weight of the charge or in this case 3243 pounds furfural. To the charge was added 6330 pounds of water and 212 pounds of sulfuric acid (95% strength) to adjust the moisture content and acid content to 62% and 1.5% of the dry weight of the charge respectively. Pre-steaming was then accomplished according to the invention by passing steam (about 5000

pounds/hour) through the charge for 30 minutes while maintaining the steam pressure within the digester at 15 pounds per square inch gauge. No substantial amount of furfural was found in the steam passing through the condenser recovery system.

Steam digestion of the pre-steamed corncobs was accomplished by raising the steam pressure on the charge to 40 pounds per square inch gauge and then gradually increasing the steam pressure to a final pressure of 100 pounds per square inch gauge over a time period of six hours, the amount of steam used averaging about 7000 pounds/hour. About 2205 pounds of furfural were obtained from the condenser recovery system. This amounted to 68% of the aforementioned potential furfural.

The procedure was repeated with the exception that no pre-steaming was employed. About 2009 pounds of furfural were obtained which amounted to 62% of the aforementioned potential furfural.

Example 2

About 18,000 pounds of corncobs (14.6% moisture) from the same carload as was used in Example 1 were charged into a steaming vessel similar to the digester employed in Example 1 but of lighter-weight construction and larger capacity. Water and sulfuric acid were added to give the same percentages as in Example 1. Pre-steaming was then effected by passing steam (about 5000 pounds/hour) through the charge for 60 minutes while maintaining the steam pressure on the charge at atmospheric pressure.

A steam digester identical to that used in Example 1 was then charged to its maximum operating capacity with the pre-steamed corncob mixture. The amount of corncobs in this charge was found to be 15,700 pounds (dry weight), an increase of 11.3% over the amount that could be charged in Example 1. Steam digestion of the charge was then carried out as in Example 1. The amount of furfural obtained from the condenser recovery system was 2491 pounds or 69% of the aforementioned potential furfural.

Example 1 demonstrated that higher yields of furfural are obtainable when the pentosan-containing material is pre-steamed according to the invention rather than merely steam digested. Example 2 demonstrated that pre-steaming of the pentosan-containing material would not only increase yields obtained by subsequent steam digestion but also would increase the bulk density of the raw material to be charged to the steam digesters. With the greater bulk density a larger amount of the raw material could be charged to the steam digester and accordingly increased the yield of furfural per digester charge.

By the improved processes of the invention furfural can be produced more economically and in greater yields. Furthermore, by making possible the use of low-cost light-weight pre-steaming vessels apart from the steam digesters a considerable saving can be effected in the capital investment required to produce a given amount of furfural.

Modifications of the present invention will be obvious to those skilled in the art of manufacturing furfural and such modifications are to be considered as being within the scope of the present invention.

We claim:

1. In a process whereby furfural is produced by steam digestion of a pentosan-containing raw material in the presence of a nonvolatile acid catalyst, the improvement comprising subjecting said raw material, prior to said digestion, to the continuous flow of steam therethrough under a steam pressure maintained at no greater than about 20 pounds per square inch gauge for a time period of about 15 to about 75 minutes, no substantial amount of furfural being formed during said time period.

2. In a process whereby furfural is produced by steam digestion of a pentosan-containing raw material in the presence of a nonvolatile acid catalyst, the improvement comprising subjecting said raw material, prior to said digestion, to the continuous flow of steam therethrough under a steam pressure maintained at no greater than about 15 pounds per square inch gauge for a time period of about 30 to about 60 minutes, no substantial amount of furfural being formed during said time period.

3. In a process whereby furfural is produced by steam digestion of a pentosan-containing raw material in the presence of a nonvolatile acid catalyst, the improvement comprising subjecting said raw material, prior to said digestion, to the continuous flow of steam therethrough under a steam pressure maintained at about 15 pounds per square inch gauge for a time period of about 30 minutes, no substantial amount of furfural being formed during said time period.

4. In a process whereby furfural is produced by steam digestion of a pentosan-containing raw material in the presence of a nonvolatile acid catalyst, the improvement comprising subjecting said raw material, prior to said digestion, to the continuous flow of steam therethrough under a steam pressure maintained at substantially no greater than atmospheric pressure for a time period of about 60 minutes, no substantial amount of furfural being formed during said time period.

References Cited in the file of this patent

UNITED STATES PATENTS

1,735,084 Miner et al. ---------- Nov. 12, 1929

UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 2,884,428 April 28, 1959

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It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 2, line 63, for "14,000 lbs." read -- 14,100 lbs. --.

Signed and sealed this 18th day of August 1959.

(SEAL)

Attest:

KARL H. AXLINE
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