INTRODUCTION

Agricultural residues have a great potential as a chemical resource. Cereal residues make up a large portion of these agricultural residues (Betroj, 1981). In this paper, we describe three different aspects of cereal bioutilization. The first aspect involves acid hydrolysis of corn stover. A "low temperature percolation" process is described which combines hemicellulose and cellulose hydrolysis through a sugar/acid recycle. The second part of this paper deals with the potential use of corn fiber derived from wet-milling processes. If the carbohydrate portion of the fiber is hydrolyzed under mild conditions without damaging the protein content, it may be possible to increase ethanol production together with producing a higher protein material. The last portion of the paper deals with a new development in biotechnology: the use of polysaccharides as selective sorbents. It involves the use of corn (or other biomass materials) to dehydrate ethanol to anhydrous product. This technology has the potential of being relatively more energy efficient than conventional azeotropic/extractive distillations, and it may be easier to implement in small or medium size plants.

Although throughout the paper we discuss the use of biomass to produce ethanol, ethanol is only one of the potential products obtainable from renewable
resources. Other chemicals such as ethylene, acetaldehyde, acetic acid and butanol (Jullander, 1975) can be derived from biomass, either through direct chemical or biochemical processes or through ethanol conversion. We believe that advances in the biomass research area may lead to expanded markets of agricultural commodities for chemical production.

PRODUCTION OF SUGARS FROM BIOMASS RESIDUES

Biomass materials are composed mainly of cellulose, hemicellulose and lignin. They are the structural components of the biomass and comprise up to 80% of the biomass weight. They are present in an approximate ratio of 6:4:1. Table I gives the typical composition of some crop residues.

About 500 million tons (dry wt.) of crop residues, including 150 millions tons of corn residues, are generated yearly in the U.S. (Detroy, 1961). If technical problems such as residue collection, land management and cellulose conversion can be at least partially solved, large quantities of ethanol would be available from biomasses. For example, if only 10% of the corn residue could be utilized, about 1 billion gallons of ethanol per year could be produced.

Production of ethanol can be carried out in a sequential node/hydrolysis of the carbohydrate fraction followed by fermentation, or in a simultaneous process: biomass is directly fermented to ethanol (Tadisch et al., 1983). The former approach appears to be at the most developed stage.

Lignin is a three-dimensional random polymer formed from a variety of phenolic compounds. It acts as a cement between the cellulose fibers. Lignin can be chemically removed (e.g., pulping), leaving behind the cellulose as a fiber (Goldstein, 1978).

Hemicellulose is a polysaccharide polymer of mainly 5-carbon sugars (pentoses). The major portion of the polymer consists of xylose units joined through 1-4 linkages, plus side chains of diverse units such as arabinose, mannose, galactose and glucose. Hemicellulose has a lower molecular weight.
## TABLE I. CONSTITUENTS OF SELECTED BIOMASS MATERIALS. VALUES GIVEN IN PERCENT DRY WEIGHT
(Based on Ladasch et al., 1983)

<table>
<thead>
<tr>
<th>Material</th>
<th>Xylan</th>
<th>Araban</th>
<th>Glucan</th>
<th>Galactan</th>
<th>Acetyl</th>
<th>Acid Anhydride</th>
<th>Total</th>
<th>Hemicellulose</th>
<th>Cellulose</th>
<th>Lignin</th>
<th>Ash</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wheat Straw</td>
<td>18.5</td>
<td>1.6</td>
<td>2.2</td>
<td>0.7</td>
<td>2.4</td>
<td>2.2</td>
<td>27.6</td>
<td>34.0</td>
<td>18.0</td>
<td>10.0</td>
<td>1.3</td>
</tr>
<tr>
<td>Rye-grass Straw</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>27.0</td>
<td>37.0</td>
<td>5.0</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>Cornstalk</td>
<td>18.0</td>
<td>2.2</td>
<td>3.6</td>
<td>0.8</td>
<td>3.6</td>
<td>4.4</td>
<td>32.6</td>
<td>33.5</td>
<td>11.0</td>
<td>3.0</td>
<td>---</td>
</tr>
<tr>
<td>Corncob</td>
<td>25.0</td>
<td>3.0</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>28.0</td>
<td>36.5</td>
<td>11.0</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>Bagasse</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>34.0</td>
<td>38.0</td>
<td>11.0</td>
<td>---</td>
<td>---</td>
</tr>
</tbody>
</table>
than cellulose and does not have a highly ordered crystalline structure present (Bunse, 1981; Sjostrom, 1981).

Hemicellulose can be hydrolyzed either with dilute acid (5-10% sulfuric acid) at 100°C (Tao et al., 1982), dilute acid (0.1 to 0.4% sulfuric acid) at 170°C (Lee et al., 1978), or even with strong acid (30-60%) at low temperatures (30-80°C) (Kobayashi et al., 1962). In laboratory experiments, corn residue (stalks) were hydrolyzed with 5% sulfuric acid at 100°C (Tao et al., 1982). The hydrolysis is carried out with a moisture limited environment (50-80% moisture) for 4-6 hours (Mehlberg et al., 1960). The sugars obtained were recovered by leaching the hydrolyzed material. Since this material has a moisture content below 80% and since the equilibrium between the sugars in the solid phase and in the liquid phase is linear (Vochoch et al., 1982), a 10-15% sugar solution can be obtained using an appropriate leaching scheme. A high sugar concentration is desirable in order to obtain an ethanol concentration above 5% after fermentation and hence, avoid prohibitive ethanol recovery costs (Ladisch and Dyck, 1979).

Xylose can be dehydrated using acid to form furfural (Thompson, 1981), which is the only chemical now commercially derived from hemicellulose. Another minor use of xylose could be in the production of xylitol, a low calorie sweetener (Funk, 1975). Since hemicellulose can account for about 30% of the biomass, its utilization is essential for the economics of an overall biomass conversion plant. In this context, the fermentation of xylose to ethanol, either using glucose isomerase (EC5.3.1.5) to convert xylose to xylulose which is then fermented to ethanol (Gong et al., 1981a), or direct xylose fermentation (Gong et al., 1981b; Jeffries, 1981, Schneider et al., 1981) is essential for the successful use of biomass. Improvements in yields (currently about 60%) and ethanol concentration (less than 4%) are still required before fermentation of xylose to ethanol can be commercially implemented. Arabinose is also present in significant quantity in the hydrolysate of hemicellulose from
agricultural residues. Fermentation of this sugar to ethanol has also been recently reported (Finn et al., 1983), and much research is needed before ethanol can be produced from arabinose on an industrial scale.

Cellulose is a linear polymer of glucose joined through β-1-4 linkages. The β-1-4 linkages are very resistant to both enzyme and acid hydrolysis. The easily hydrolyzable portion of the cellulose is called the "amorphous" region while the resistant portion is the "crystalline" region. On the average, cellulose is 15% amorphous and 85% crystalline (Bungay, 1981). Enzymatic hydrolysis is hindered by the crystallinity of the cellulose and its association with lignin (Tsao et al., 1978). In order to enhance enzymatic hydrolysis, a pretreatment step is required. The pretreatment changes the physical but not the chemical structure of the lignocellulosic material, making it more susceptible for hydrolysis. Examples of pretreatment include high energy radiation, grinding the swelling agents. An excellent review of the different chemicals and physical pretreatments and their effects on enzymatic hydrolysis is given by Fan et al., 1982. Even though large numbers of pretreatments have been tried in the laboratory and are technically capable of enhancing cellulose conversion, their cost, which in many cases involves reagent recycle, is a major hurdle for commercial use (Ladisch et al., 1983). (Note: If a pretreatment having a reasonable cost is developed, its major impact may be in the increased digestibility of lignocellulosic materials for ruminant feed).

Cellulose can also be hydrolyzed using mineral acids at high temperatures without a pretreatment step although yields are generally lower than 60%. Major processes were developed during World War II and thereafter. In the U.S., processes using dilute sulfuric acid (0.5-2%) at high temperatures (170°C and higher) were developed (for example, the Madison process, Saeeman, 1945; Harris, 1945; Hajny, 1981). The Madison process involved the use of percolators where the biomass and the acid flow counter-current. Recent research has updated this process with the use of extruders for continuous systems (Grethlein, 1978;
Church and Woodridge, 1981; Brenner and Rugg, 1982).
The scale-up of such systems still needs to be further researched. Alternate acid processes utilize other acids such as hydrochloric acid (e.g., Borgius-
Rheinau process) (Ladisch, 1979; Bungay 1981). A
general description of acid hydrolysis processes
together with review of the Russian literature is
given by Wenzel (1970). Due to their high cost, use
of acids other than sulfuric acid requires an acid
recovery system.

In order to reduce capital costs, sulfuric acid
hydrolysis can be carried out at lower temperatures.
The hydrolysis at lower temperatures then requires an
impregnation (pretreatment) step. One method to
obtain the impregnation of the lignocellulosic biomass
with concentrated sulfuric acid involves spraying the
dry biomass with concentrated acid (Peoria process,
Dunning and Latrop, 1945). After impregnation, the
acid is diluted to 8% followed by hydrolysis at 120-
130°C for up to 10 min. The impregnation of biomass
through acid spraying poses serious mixing problems
due to the viscosity of the acid, difficulty in
distributing a liquid evenly in biomass particles and
controlling the exothermic character of the impregna-
tion step. One way to avoid the mechanical problems
involved in acid spraying, is to dry biomass which
had been previously soaked in dilute sulfuric acid
solution. A pretreatment of the lignocellulose takes
place during drying. After the drying step, water is
added to dilute the acid to 8% and the material is
hydrolyzed at 120°C for 20 min. Effects of sulfuric
acid concentration in the soaking step, drying time
and hydrolysis conditions were studied for corn
stover (Tsao et al., 1983). Initial results indicate
that cellulose conversion of up to 90% can be obtained
when closely controlled drying conditions are used in
the pretreatment step.

The concept of impregnation upon drying can be
coupled with hemicellulose hydrolysis to create a
"low temperature percolation process" (details given
in Tsao et al., 1982). After hemicellulose hydrolysis
and leaching of the sugars, the biomass is soaked in
a dilute (10-20%) sulfuric acid tank. The soaking is
carried on at 90-100°C for 2-4 hours. After soaking, excess moisture is removed from the lignocellulose by a pressing device to bring the moisture down to about 50%, followed by careful drying. Water is then added and the biomass is hydrolyzed. The liquid portion is separated from the non-hydrolyzed solids and is recycled to the hemicellulose hydrolysis tank (Fig. 1). Based on laboratory runs, a sugar/acid ratio of 3/1 could be feasible. The sugar to acid ratio is very important since sulfuric acid is a major cost in the process. Even the dilute acid/high temperature processes give a sugar to acid ratio in the range of 1/1 to 7/1. Hydrolysis of Solka Floc and newsprint in a dilute acid process gave glucose conversions of 45-55% with a glucose to acid ratio of 1:1 to 2:1 (Grethlein, 1980). The process as shown in Fig. 1 was carried out in our laboratories for corn stover (Tsao et al., 1983). One technical problem with such a process is the formation of a product which is a mixture of sugars (mainly glucose and xylose).

Fermentation of such a complex mixture requires further studies.

We believe that the conversion of biomass to sugars is a timely idea. Recent developments in the fermentation of pentoses together with improvements in both enzymatic and acid hydrolysis have improved the economic outlook of large-scale plants. Further research and development is required in many areas including better understanding of the pretreatments. The potential amount of liquid fuel obtained from biomass residues may be small when compared to the overall needs of the country, but in absolute terms, their market value is in the billion dollar category.

HYDROLYSIS OF CORN-FIBER

Corn is a good example of a product with high-value co-products. There is no "waste" in the processing of corn, either in the wet-milling or whole-grain process. The value of co-product is very important for the overall economics in ethanol production. For example, in a fuel ethanol plant based on a wet-milling process, the credit for the
Figure 1. Low temperature “percolation” process.
co-products (feed, meal and oil) accounts for about 50-60% of the raw material cost while for a whole-grain plant, the co-product credit (DGs) accounts for 30-40% of the raw material (Keim, 1983). In these well designed processes, it is difficult to define areas for "magnitude of order" improvements. However, for a large plant size, relatively small gains in production can have a significant economic impact. One possible area of interest is the hydrolysis of the corn-fiber.

After the starch granules have been separated from the protein matrix, they are washed in order to be fractionated from the pericarps and other fibrous fragments. The washed tailings are termed "fiber". Continuous screw presses then dewater the fiber to reduce its moisture content to about 60% (Watson, 1970). The fiber is combined with other streams (cleanings, steepwater solubles and germ cake), dried and sold as Corn Gluten Feed with a minimum of 21% protein in the U.S. and 23% protein and fat for export (Keim, 1983). The corn gluten feed finds its greatest use in ruminant rations. It contains 14% starch according to Shroder and Heiman, 1970.

The amount of ethanol produced from a bushel of corn is directly related to the amount of starch recovered. Due to the complexity of the wet-milling process, 100% starch recovery is not economically feasible (Anderson, 1970). In this perspective, the addition of a corn-fiber hydrolysis step to recover both the starch and the hemicellulose fraction may be appropriate. Table II shows the potential amount of ethanol which can be obtained from fiber for a 100,000 bushel/day wet-milling operation. Additional motivation for starch recovery from fiber could also be to provide additional dextrose for HFCS in an ethanol/HFCS swing process.

Since the amount and the analysis of the corn fiber obtained in a commercial process is not widely available in the literature, the numbers given in Table II were based on data obtained in a wet-milling simulated laboratory procedure (Watson, 1967). These results (Watson, 1967) indicate that 9.5% of the original dry weight of the corn is recovered in the
<table>
<thead>
<tr>
<th>Table II: Potential Ethanol Production from Fiber (Based on 100,000 Bushel/Day)</th>
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<tbody>
<tr>
<td><strong>Amount of Fiber Produced</strong> = (100,000 bushel_day) x (56 lb corn_bushel) x (0.845 lb dry_corn_bushel) x (0.095 lb fiber_corn) x 449,540 lb fiber_day</td>
</tr>
<tr>
<td><strong>Potential Amount of Ethanol Produced from Starch in the Fiber only</strong> = (449,540 lb fiber_day) x (0.11 lb starch_fiber) x (0.90 hydrolysis_efficiency)</td>
</tr>
<tr>
<td>= (449,540 lb fiber_day) x (0.11 lb starch_fiber) x (0.90 hydrolysis_efficiency)</td>
</tr>
<tr>
<td>= (449,540 lb fiber_day) x (0.11 lb starch_fiber) x (0.90 hydrolysis_efficiency)</td>
</tr>
<tr>
<td>= (449,540 lb fiber_day) x (0.11 lb starch_fiber) x (0.90 hydrolysis_efficiency)</td>
</tr>
<tr>
<td>Potential Amount of Ethanol Produced from Xylose only = (449,540 lb fiber_day) x (0.20 lb xylose_fiber) x (0.90 hydrolysis_efficiency) x (0.51 lb ethanol_xylose) x (0.80 fermentation efficiency_xylose) x (6.5 lb ethanol_gal)</td>
</tr>
<tr>
<td>= (449,540 lb fiber_day) x (0.20 lb xylose_fiber) x (0.90 hydrolysis_efficiency) x (0.51 lb ethanol_xylose) x (0.80 fermentation efficiency_xylose) x (6.5 lb ethanol_gal)</td>
</tr>
<tr>
<td>Total potential: 2.82 x 10^6 gal/\text{year}</td>
</tr>
</tbody>
</table>

(a) From Watson, 1967
(b) 1 year = 330 production days
(c) Assumes 20\% xylose is in the fiber, only
(d) 80\% fermentation efficiency of xylose, based on laboratory results (Ladisch et al., 1983).
fiber stream and that this stream contains 11.4% starch and 11.3% protein. The hemicellulose content of the fiber was estimated as being 30% of the fiber with 65% of the hemicellulose fraction made-up of xylose and 35% of arabinose (Watson, 1967). In summary, our first estimates are based on 9.5% fiber recovery with a fiber composition of 11.4% starch and 20% xylose. Using these figures, together with selected hydrolysis/fermentation efficiencies (see Table II), we obtained a potential of 2.82 million gal ethanol/year for a 100,000 bushel/day wet milling plant. Of course, the potential ethanol produced is dependent on fiber composition. Furthermore, we speculate that small disturbances in the wet-milling process may increase (or decrease) the amount of starch left in the fiber.

We believe that the hydrolysis of the corn fiber material is feasible, at relatively mild conditions and low acid costs based on previous literature on the acid hydrolysis of cellulosic materials (Ladiach, 1979). In order for such a process to be economically feasible, the protein cannot be damaged during the hydrolysis step and the high-protein material obtained has to be marketable. A high-protein material is conceivable since mild hydrolysis should remove a large portion of the solids. Properties of the resulting material as an animal feed would have to be determined. Other technical problems which could impact fiber utilization include the fermentation of arabinose resulting from the hydrolysis of the hemicellulose. One possible use would be fermentation of arabinose and other saccharides to single-cell protein. This would moderate the effects, if any, of the unfermented sugars and sugar products on potential fouling of downstream equipment. We believe advances in these areas could open new markets for corn fiber.

CORNMEAL AS AN ADSORBENT FOR WATER-ETHANOL SEPARATION

During the early stages of the development of gasohol, the separation of ethanol from water was considered to be excessively energy intensive, consuming up to 50% of the overall energy used in a typical grain ethanol plant (Scheller, 1974; David et al.,
1978; Ghose and Tyagi, 1979). The large energy requirements are in part due to the existence of an azeotropic ethanol-water mixture (95.6 wt %, at atmospheric pressure). The azeotrope can be broken by using low pressure distillation, extractive distillation or azeotropic distillation. Both extractive and azeotropic distillation requires the addition of a third compound such as benzene, cyclohexane or heptane (Black, 1980; Honeywell, 1980). Reduction in the overall steam consumption was achieved by a multiple effect configuration and by operating the extractive and concentrating towers at high pressure (Katzen et al., 1980). The steam consumption was reported to be 15 to 20 lbs steam per gallon of anhydrous product (ca. 15,000 to 20,000 Btu/gal product). Such multiple effect evaporators require high-pressure steam which may not be readily available. This technology also appears to be subject to economies of scale.

In order to reduce the energy costs, other approaches may be used. The use of adsorbents such as lime, calcium chloride and silica gel, have been reported (Noyes, 1923; Davis and Swearingen, 1931; Friel, 1933; Smith, 1949). Recently, adsorption processes using biomass materials together with a vapor feed (Ladisch and Dyck, 1979; Voloch et al., 1980), and molecular sieves with a liquid feed (Pitt et al., 1983) have been reported.

Starch, cellulose, xylan, cornmeal and biomass residues are potential adsorbents for the dehydration of ethanol (Hong et al., 1982). Among these materials cornmeal seems to be the first choice due to its price and physical properties with respect to flow in packed columns. Following is a general description of the relevant properties and operation characteristics of cornmeal for ethanol dehydration. (More detailed studies may be found in Voloch et al., 1980 and Ladisch et al., 1983a).

Process Outlook and Background

The separation of water-ethanol vapors is carried out using a vapor feed to a column packed with cornmeal (Fig. 2). Even though we were able to obtain
Anhydrous ethanol from a feed as diluted as 40 wt% ethanol (Ladisch et al., 1983a), economics of the process indicates that the feed should be about 90 wt% since up to this concentration distillation is relatively energy efficient (Ladisch and Dyck, 1979). The column is kept above the dew-point of the feed mixture, which means that the column is operated at an initial

![Diagram](https://via.placeholder.com/150)

**Figure 2.** Conceptual scheme for a continuous adsorption system. Show column 1 adsorbing while column 2 is regenerated; dotted lines indicate reverse cycle.

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Figure 3. Temperature and concentration breakthrough for a 4" column (based on Ladisch, et al., 1983a).
temperature of 78°C or higher. The ground corn (average particle size 0.5-1 mm) is previously dried using a hot (90-120°C) gas. Laboratory experiments were carried out using air as the drying agent, but due to a possible hazard potential of ethanol/air mixtures at these conditions (Zabetakis, 1965), large commercial plants would probably use either CO₂ or N₂.

The adsorption of the water on the corn is exothermic and gives rise to a temperature wave which travels through the bed together with a water concentration wave (Fig. 3). Before the water

(a) Adsorbent Inside Column

(b) As Observed At Outlet

Figure 4. Breakthrough profile.
concentration wave breaks through, the column is switched from adsorption cycle to desorption cycle. Desorption is achieved by flowing hot (80-120°C) gas counterflow through the bed. The overall system is made continuous by using 2-4 columns such that while one column is adsorbing, the others are desorbing.

The breakthrough time is related to the length of the mass transfer zone (Fig. 4) which is dependent on a number of parameters including gas flow rate, particle size, pressure and temperature (Kovach, 1978). In general, the separation capacity of cornmeal is greater than 0.95 gal anhydrous product/100 lb corn/cycle for 190-proof (92.5 wt%) feed and 4500 lbs corn (cost of less than $500) is enough for a 1,000,000 gal/yr plant. It is feasible to have a continuous system where corn will be continuously dried and passed counter-current to ethanol vapors to dry the ethanol. The “Wet” corn could be processed further (Ladisch and Dyck, 1979). However, the mechanical aspects and the overall energy requirements of such a system make it less desirable than a fixed bed adsorber.

Energy requirements for an adsorption-desorption system include the mechanical energy (blowers) to flow the gases through the bed and the energy to preheat the drying gas. Since the adsorption cycle is switched off before the temperature wave breaks through, most of the energy released during water adsorption is contained in the bed. The drying gas has to carry only sufficient extra heat value to compensate for energy losses. Calculations based on laboratory experiments indicate that a well designed system could require as little as 1000 Btu/gal anhydrous produce obtained from a 190-proof feed as compared to 9000 Btu/gal (Black, 1980) required for a benzene column. This energy savings translates to 3.2-4.8¢/gal product if energy is priced at $4 to $6/Mcal.

Cornmeal is stable under cyclic operation (Ladisch et al., 1983a). If the temperature in the column is allowed to drop below the dew-point of the ethanol/water vapor mixture, the vapor will condense. This in turn can cause caking of the corn upon drying.
which is obviously undesirable. The initial moisture on the corn depends on the relative humidity of the drying gas, but it is estimated to be in the range of 1-3 wt% initially, and during the adsorption cycle, the moisture loading increases by as little as 1 wt%.

Under these conditions, the corn should be stable with respect to starch degradation.

The separation capability of the cornmeal is not restricted to ethanol-water mixtures. Other alcohols such as n-propanol, isopropanol and t-butanol were dehydrated under similar experimental conditions (Ladisch et al., 1983a).

The adsorption and desorption properties of water on starch and cornmeal at temperatures below 50°C is well described in the literature (e.g., Bushuk and Winkler, 1957; Rodriguez-Arias et al., 1963; and Do-Sup and Pfoert, 1967). The use of cornmeal to dehydrate alcohols at higher temperatures occurs outside the range of the known isotherms. The capability of the material to discriminate between water and the alcohol, is still not understood. Starch was shown to adsorb different amounts of ethanol and water from a liquid mixture and did not adsorb any ethanol when dry (Kurilenko and Yakovkina, 1960). Similar results were obtained for vapor mixtures of ethanol and water (Hong et al., 1982). Further research will lead to determination of isotherms for water and ethanol at temperatures above 60°C (data not currently available) which will be helpful in increasing our understanding of the operation of the system and data for the design of commercial plants. These studies together with fundamental studies on the ways biomass materials are able to discriminate between water and alcohol molecules may lead to the development of a new class of "super-adsorbents" based on biomass materials which could have applications to systems other than ethanol/water.
CONCLUSION

The outlook for renewable resources in biotechnology is encouraging. Advances in biotechnology will continue to impact the large scale utilization of these resources. Although further research is needed in many areas, the developments described in this paper can also be considered in the context of evolution of new technology in industrial practice. We speculate that in the near future, the use of polysaccharides (e.g., corn) to selectively absorb water may be implemented for the production of anhydrous ethanol in place of azeotropic distillation. The next stage could conceivably be the processing of corn fiber from wet-milling processes in order to upgrade its net value. This will ultimately result in the development of processes based on cellulosic residues to provide fermentable carbohydrates which would supplant dextrose.

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