

# UNITED STATES PATENT OFFICE

2,503,241

## METHOD OF PRODUCING LIGHT-COLORED SIRUPS

Leo M. Christensen, Lincoln, Nebr., assignor to National Agrol Company, Washington, D. C., a corporation of Michigan

No Drawing. Application May 24, 1946,  
Serial No. 672,169

11 Claims. (Cl. 195—31)

1

This invention relates to a method of producing light-colored syrups from starchy material, more particularly to a novel method of producing syrups of a highly desirable light color from starchy material by enzymatic conversion.

As is known, at the present time, there are two general methods employed for the manufacture of sugar or sugar syrups from starch or starch-containing materials. The process most generally employed comprises the separation of the starch from the other grain constituents, by suitable mechanical means, and its subsequent conversion, by cooking with dilute mineral acids, to a mixture of sugars including dextrose and maltose. The second process involves the use of saccharifying enzymes such as the amylases of malt, mold bran and certain bacteria. These amylases serve to convert the cooked starch to sugars. The products produced from the described process are generally designated "acid converted" or "enzyme converted" syrups respectively. Dextrose and maltose may be recovered from these syrups in crystalline form by methods well-known in the art.

Enzyme converted syrups possess a distinct inherent advantage as compared to acid converted syrups. Such enzyme converted syrups contain about the same amount of dextrose, about twice the amount of maltose and only about half of the amount of dextrans as are produced by acid conversion. Since the dextrans have no sweetening power, the enzyme syrups are thus sweeter than the acid converted syrups. However, this advantage, while recognized in the past, has been offset considerably by reason of the fact that the enzyme converted syrups are brown in color and have certain flavors that makes their use objectionable in many products. For these reasons the enzyme converted syrups have found but a limited commercial use even though, as noted, they are superior in sweetening power.

It has commonly been assumed or postulated that the color and much of the flavor of the enzyme converted syrups are due to some type of chemical combination of sugar and proteins. These proteins are present because of the fact that the enzymatic saccharifying agents used contain proteases as well as amylases, and thus bring into solution larger amounts of amino acids and polypeptides than are derived when acid conversion is used. This is particularly true when the enzyme conversion is applied to ground whole grain as is not infrequently done.

This color, typical of enzyme converted syrups, develops during the concentration of the prod-

2

uct. The original hydrolysate generally contains only about 15% of sugars and in commercial practice it must be evaporated to a concentration of about 60 to about 65% sugars. This concentration may be effected at atmospheric pressures or at lower temperatures under reduced pressures; in either case the color begins to develop when the sugar concentration reaches about 40%, by weight, and the color intensifies as the concentration progresses. In the past, efforts have been made to reduce this undesired color of the enzyme converted syrups. Treatment of the original hydrolysate with a good quality of decolorizing carbon reduces the color somewhat, and the concentrated or partially concentrated syrup may be somewhat decolorized by such methods. However, these methods are expensive and are performed with difficulty when applied to the viscous syrups.

As a result of intensive experimentation in this field it has been found that the color and much of the flavor of the enzyme converted syrups does not derive from proteins, as had previously been supposed, but from other constituents of the grain. Thus, when "pure" starch, such as is made by a very careful mechanical separation from other grain constituents is employed, the color and flavor, generally speaking, are just as pronounced as though large amounts of protein had been originally present. It has been ascertained as a result of intensive investigation that the impurity in the starting material which ultimately gives rise to the color in the syrup, and to some extent, the flavor, was derived from some constituent of mechanically purified starch, and not, strictly considered, a protein.

It has long been known that starches contain in their cell walls certain sterols, waxes and phospholipids. These products are water insoluble and are not solubilized by acid conversion. However, it has been determined that the common amylase preparations hydrolyze these constituents to water soluble forms, furthermore, it has been ascertained that these compounds readily lose water on heating and the dehydrated derivatives thus produced are highly colored.

It was then found that if these color-forming bodies could be removed from the starting material the undesired color of enzyme converted syrups could be substantially eliminated. To establish this fact a quantity of "pure" corn starch was extracted with a solvent for the sterols, waxes and phospholipids and the extracted starch was subsequently converted to syrup. For this purpose 100 grams of "pure starch" was ex-

tracted with petroleum ether. By reason of this extraction 0.25 gram of an oily impurity was recovered. The extracted starch was then converted by enzymatic conversion and was found to have the composition generally obtained by other methods of saccharification and was practically water white in color. Further experimentation demonstrated that to obtain such water white enzyme converted syrups it was not necessary to use starch but that ground whole grain could be employed and that if such grain is extracted with a good fat solvent it also could be converted by enzymatic agents to yield a syrup of very low color without decolorizing or bleaching. Obviously, any readily available effective fat solvent may be employed such, for example, as chlorinated hydrocarbons and the like.

It will thus be seen that the broad concept of the invention comprehends the defatting of the starchy material at any suitable stage before the development of color. For obvious reasons this defatting is carried out on the dry raw material but in view of the availability of effective water immiscible fat solvents it is apparent that defatting may be carried out at any desirable stage in the process. As will be seen hereinafter the defatting of the starchy material presents other advantages of prime economic importance in respect to the filtration of the saccharified mash.

In carrying out the invention it is preferred to start with whole ground grain or with starch rich in grain fractions although it will be appreciated that any starchy substrate may be employed. The dry starting material is extracted, as previously noted, with an effective water immiscible fat solvent, such as petroleum ether, trichlorethylene and the like, and such extraction is continued until substantially 95% of the fat content is extracted. Such extraction may require from about one-half hour to as long as 8 hours more or less, depending upon the physical character of the material, the solvent and the type of equipment used. Preferably the grain is ground to 20 mesh or finer and is treated in an efficient extraction unit. With effective separating conditions the fat-like constituents are substantially completely removed within a period of about an hour. The solvent, as will be appreciated, is passed through an evaporation and recovery system and the denuded solvent is recycled to the extractor. The recovered fats may be utilized for any desired purpose.

The defatted starchy fraction is then mixed with from 2 to 3 parts of water and cooked. If the starchy material is substantially pure starch about 3 parts of water are employed such amount being less if the starting material contains less starch. In the preferred operation the starchy material is first moistened with about one-half the amount of water and this slurry is introduced into the balance of the water, the latter being at a temperature high enough to insure substantially instantaneous gelation. When corn is the starting material employed this temperature should be above 65° C.

The aqueous starch slurry is then cooked for a suitable period of time as, for example, for a period of about 5 minutes at 150° C. or, if desired, for a longer time and at a commensurately lower temperature.

As is known and as is more particularly described in U. S. Patent 2,342,330, losses are incurred in cooling cooked mash down to the saccharification temperature when normal methods of slow cooling are employed. These losses ap-

pear to be due to an irreversible hydration of the starch during the orthodox step of slow cooling of cooked starch from a temperature of about 100° C. down to the optimum saccharification temperatures, which latter are about 60° C. for malt and about 55° C. for mold bran. In the present process therefore this quick cooling with consequent avoidance of starch retrogradation is recommended to thereby insure higher sugar yields.

The cooked starch may be preliminarily cooled by evaporation at reduced pressure after which it is added with instantaneous mixing to a volume of cold water containing approximately 10 pounds of malt or approximately 3 pounds of mold bran, or their equivalent in amylase content, per 100 pounds of the original grain. The volume and temperature of the cold water is adjusted so as to practically instantaneously reduce the temperature of the mixture to the optimum saccharification temperature which in the case of mold bran, as noted above, is substantially 55° C.

This saccharifying mash is then maintained at a temperature of from 45° C. to 55° C. for a period of from 60 to 120 minutes. Thereafter the mash is heated to 80° C. or higher, is screened to remove larger fiber particles and is thereafter filtered to remove suspended matter. The filtrate may be treated with decolorizing carbon if the malt or mold bran have yielded any appreciable color to the solution. Such colors, if present, are readily removed by the carbon. Thereafter the clarified filtrate is evaporated, preferably at reduced pressures, to a syrup of the desired concentration. The reaction throughout the process should be pH 4.8 to pH 5.5.

As noted previously, the preliminary step of defatting the grain, or other starchy substrate, results not only in the production of light-colored syrup but also insures marked economies in the filtration step. As is known, the filtration of the saccharified mash, prior to evaporation, is the most difficult operation in the whole enzymatic syrup process. It has been ascertained that when defatted starchy material is employed not only is the amount of suspended matter to be removed much lower but also the filtration rate is markedly faster than in typical methods where such preliminary defatting is not carried out.

These major benefits are apparent from the data in the following table. In the operation from which the data of Table I is derived, the saccharified mash was first screened, then centrifuged, in a solid bowl centrifuge, and filtered on a suction filter through paper.

Table I

	Corn meal	Defatted corn meal
Suspended matter in centrifugate, percent.....	2.5	0.25
Filtration rate of centrifugate, Gallons/minute/ sq. ft., without filter aid.....	0.01	0.05
Filtration rate of centrifugate, Gallons/minute/ sq. ft., with filter aid.....	0.04	0.20

From an inspection of the data in the table it will be observed that by removing the fat content of the corn there is a 90% reduction in the amount of suspended matter to be removed by the filter. This is directly reflected in a five-fold increase in the filtration rate applicable equally with and without a filter aid. This rapid filtration, as will be appreciated, is of profound economic significance since for the same thru-

put the filtration area required for processing defatted corn is but one-fifth of that necessary for the undefatted corn. Furthermore, it has been found that when defatting is invoked the filters can be operated substantially twice as long between cleanings as when undefatted grain is used.

It will be appreciated that utilization of the principles of the present invention insures not only marked economies in the production of enzyme converted syrup but also results in a novel product, i. e., a syrup having the desired sweetening power of the enzyme converted syrups coupled with the low color value characteristic of the acid converted syrups.

While a preferred method of effectuating the principles of the invention have been described it will be understood that this is given didactically as one illustrative method of achieving the advantages of the novel basic concept, namely, of the discovery of the essential color imparting factor in the source material and its removal at any suitable stage in the operation by any appropriate procedure.

I claim:

1. A method of producing syrups of high sweetening power and low color which comprises, extracting a starchy substrate with a solvent for sterols and waxes until a substantial proportion of the sterols and waxes are removed, heating an aqueous slurry of the extracted starch to effect gelation thereof, cooking the starch at elevated temperatures, then cooling the cooked mash to optimum saccharification temperatures and inoculating the cooled mash with an amylase-containing saccharifying material.
2. A method in accordance with claim 1 in which the amylase-containing material is a vegetable amylase.
3. A method in accordance with claim 1 in which the amylase-containing material is a fungal amylase.
4. A method in accordance with claim 1 in which the amylase-containing material is barley malt.
5. A method in accordance with claim 1 in which the amylase-containing material is mold bran.
6. A method in accordance with claim 1 in which the amylase-containing material is a mixture of barley malt and mold bran.

7. A method of producing syrups of high sweetening power and low color which comprises, extracting a starchy substrate with a water immiscible fat solvent, heating an aqueous suspension of the extracted starchy material to effect gelation of the material, cooking the material at cooking temperatures, rapidly cooling the cooked mash to optimum saccharification temperatures and without substantial retrogradation of starch and saccharifying the cooked mash with enzymatic saccharifying material.

8. A method of producing syrups of high sweetening power and low color which comprises, extracting a starchy substrate with a water immiscible fat solvent, heating an aqueous suspension of the extracted starchy material to effect gelation thereof, cooking the material at suitable cooking temperatures, rapidly cooling the cooked mash to optimum saccharification temperature by quickly mixing the cooked starch with the requisite amount of cold water containing an enzymatic saccharifying agent, maintaining the saccharifying mash at such optimum temperature for a period of time sufficient to insure the desired saccharification, screening the saccharified mash, filtering out suspended matter and evaporating the filtrate to the desired sugar concentration.

9. A method according to claim 8 in which the converting agent is barley malt.

10. A method according to claim 8 in which the converting agent is mold bran.

11. A method according to claim 8 in which the converting agent is a mixture of barley malt and mold bran.

LEO M. CHRISTENSEN.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
220,116	Behr et al. ....	Sept. 30, 1879
770,059	Gaff et al. ....	Aug. 12, 1902
2,280,723	Schock .....	Apr. 21, 1942
2,314,282	Levin .....	Mar. 16, 1943
2,342,330	Christensen .....	Feb. 22, 1944
2,365,788	Warburton .....	Dec. 26, 1944