

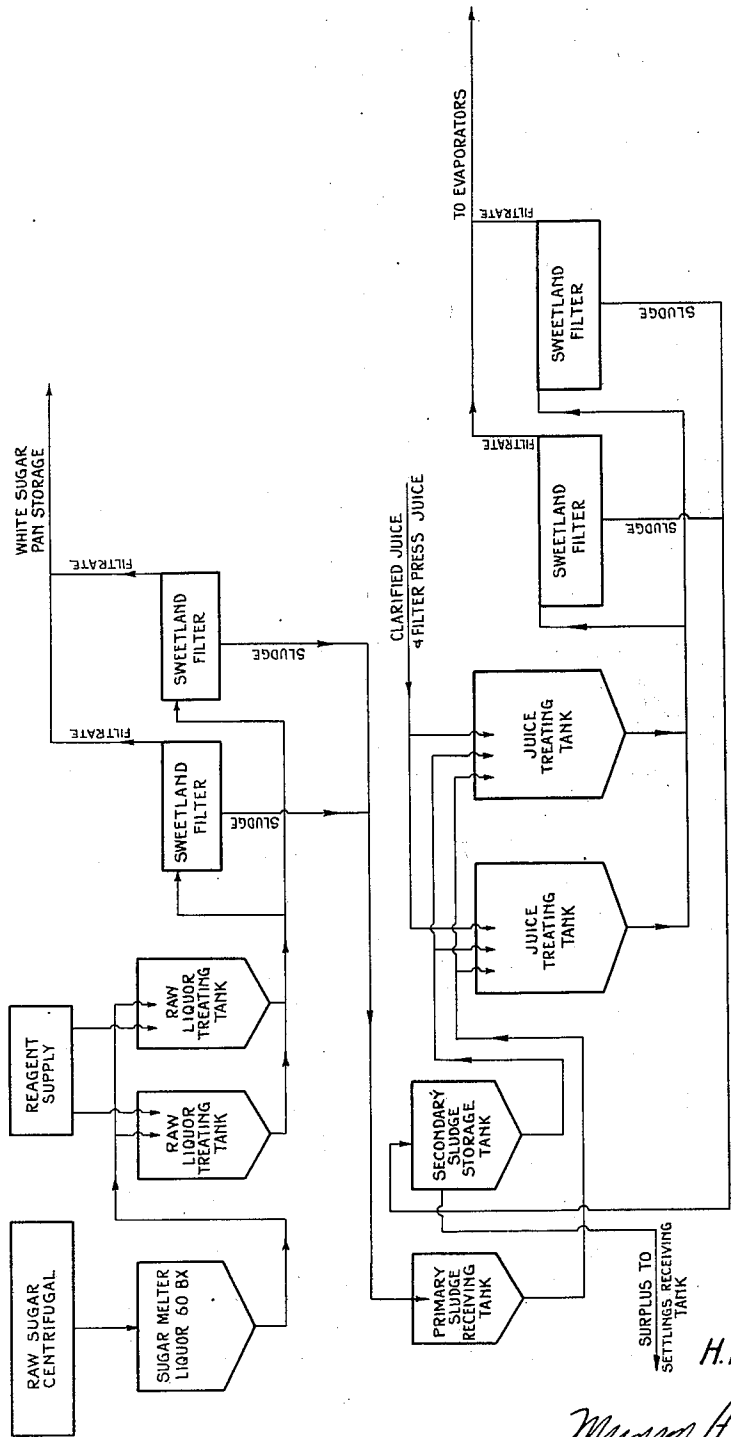
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SUGAR REFINING

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## SUGAR REFINING

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The invention relates to the manufacture, the purification of sugar, syrups and juices, and to the preparation of an improved reagent employed in connection therewith.

In the manufacture of white sugar from sugar-cane and sugar beets there are several conventional processes now in use that have been employed for many years. For example, in the manufacture of refined cane sugar or beet sugar, it is the practice to first produce a commercial sugar ranging in purity from 96% to 98.5%. To refine this sugar it is first necessary to remove most of the molasses film adhering to the crystal, and in some refineries it is the practice to dissolve a part of the crystals themselves at this stage. This part of the process is usually called the affination step.

These washed crystals are then melted into a liquor of over 60% solids, depending upon the individual practice. This raw liquor, as it is commonly termed, is filtered with some filter aid, or may be given a preliminary defecation with some clarifying agent such as phosphoric acid. The filtered or clarified liquor then is run over bone char, which has the property of removing coloring matter and inorganic salts. Its prime object is to decolorize the liquor, but where the percentage of char on melt is large, a removal of inorganic salts is also effected.

The liquor from the char is then boiled into white sugar. This process is usually referred to as the classical method of producing white sugar. While it has withstood the test of time largely because of its well developed technology, it still has many inherent disadvantages which make it a costly one, and because of these, the refiners' margin is out of all proportion generally to the price of the raw sugar.

The prime disadvantages to the bone char process are: first, a large capital investment is required; second, the over-all refining cost is high; third, a large supply of fresh water is essential, and fourth, supervision by a highly trained staff is essential to reduce inherent losses.

A newer process that has found some use depends upon the action of vegetable carbons for decolorizing of sugar liquors. This process in the United States is generally employed in conjunction with a raw sugar factory making its own raw sugar. As it is practiced, the procedure is essentially the same as in the bone char process, with the difference that vegetable carbons are used in place of bone char.

Disadvantages of this process may be listed as follows: first, a large investment cost and operat-

ing cost; second, a high manufacturing cost; and third, the revivification of the carbons is difficult, and its technology is not completely understood.

In the manufacture of beet sugar, the process most universally practiced in the United States is known as the carbonation process. This juice is heavily limed, and by carbonating in two steps, a removal of impurities is obtained, resulting in the desired purification. It is further bleached with sulphur dioxide. Variations of the operating procedure depend to some extent upon the individual preference of the operators. While this process has been in use for many years, it too presents some inherent objections. Among these may be mentioned the following: first, an inferior quality of sugar is obtained; second, the investment and operating costs are also high, and third, its operation is complicated, requiring close supervision in all the various steps.

In my process, advantage is taken of the fact that the impurities producing the color in sugar house products are in a colloidal state, and that these colloidal impurities have a negative electric charge. I further take advantage of the fact that aluminum salts can be prepared in such a form that they assume highly activated colloidal properties possessing a positive electric charge.

It is an accepted theory in colloidal chemistry that when a colloid carrying a negative charge is added to one with positive charges, a neutralization of these charges occurs, resulting in the flocculation of both. This is the fundamental basis of my process.

I have found that if aluminum hydrate is prepared from any of the common aluminum salts such as aluminum chloride and sodium aluminate, or aluminum chloride and sodium hydroxide, or aluminum sulphate and sodium aluminate, a highly activated colloid can be formed, provided there is present some salt such as sodium chloride, and provided the pH or hydrogen-ion concentration of the colloidal mixture is approximately 4.7-5.2 pH.

The fact that it is necessary to have a salt present such as sodium chloride points to the fact that the activity of this colloid is not due to the aluminum hydrate, but a product of hydrolysis such as aluminum oxychloride, and it is the presence of this product of hydrolysis that gives the positive colloid its highly activated properties. Therefore, in my process the positive colloid is not simply a form of aluminum hydrate,

but a product of hydrolysis between sodium chloride and aluminum hydroxide.

I find that this colloid can be prepared by adding a solution of sodium aluminate to a solution of aluminum chloride, stirring the mixture rapidly until the reaction is slightly alkaline, approximately 7.5 pH. The salt may be partially removed by employing a system of multiple filter pressing, or by decantation, or any of the known methods. Finally the pH is adjusted to 4.7-5.2.

For my process, I have found that a ratio of approximately 1-10 between the sodium chloride and aluminum hydrate is the preferred equilibrium to produce this activated, colloidal aluminum oxychloride. Although an excess of salt will not prevent the formation of this colloid, to be practicable in my process, the salt should be maintained at a minimum concentration and still form this activated colloid.

The reagent produced on combination of the chemicals referred to above, when prepared as herein described, is not aluminum hydroxide or hydrate, but aluminum oxychloride. This is an activated colloid and is further rendered more efficient through hydrolysis. It is at its greatest efficiency when adjusted to a pH of 5.2.

In the manufacture of the reagent aluminum oxychloride, let it be assumed that the two chemicals aluminum chloride ( $AlCl_3$ ) and sodium aluminate ( $Na_2Al_2O_4$ ) are employed. The sodium aluminate in solution at about 10° Bé. is added slowly to a dilute solution of aluminum chloride which is kept in rapid agitation by the use of a stirrer, in this case through a centrifugal pump. The sodium aluminate is added until the product produces a blue reaction to bromthymol blue and consequently a pH well above 7.0, possibly near 8.0.

The aluminum, the hydroxide and the chloride behave as follows:

$Al^{+++} \rightarrow AlOH^{++} \rightarrow Al(OH)_2^{+}$  etc., with decreasing dissociation and decreasing dispersion, a floc with the following formula will form

$[Al(OH)_3]_x \cdot [Al(OH)_2]_y^{+} + xCl^{-}$   
which expressed in simplest form, thus



is the formula of the oxychloride of aluminum carrying a positive charge and not the hydrate of aluminum which carries a negative charge and has a formula of



The action of the aluminum oxychloride on the colloids of sugar solutions is that of neutralization of the charges of those colloids which are negative in character, thereby causing the formation of a precipitate which is made up of the mentioned chemical combined with the impurities of the sugar solutions. Among these impurities are certain coloring matters and dye stuffs, colloidal in character, which likewise carry negative charges and are precipitated out by the flocs of aluminum oxychloride.

Having discovered this fact, viz. that aluminum oxychloride is the reagent formed when combining the various chemicals listed and that this reagent when correctly made decolorizes sugar solutions very efficiently, and that this decolorizing is consistent when the reagent is aluminum oxychloride, and that on the other hand, when the mixture of the same chemicals is incorrectly made, the product is not aluminum oxychloride but alu-

minum hydroxide or hydrate and has a negative charge, consequently incapable of precipitating the negatively charged colloidal impurities of sugar solution and therefore inefficient as a decolorizing agent, the claims of the present application are accordingly directed to the improved purifying reagent and to its use in the clarification of sugar solutions.

In my process, I take advantage of the fact that while a certain amount of colloid may be required to obtain the desired results, the sludge thus formed still contains a certain amount of unspent colloid which can be used again or several times. In its practical application in a sugar process, I therefore use the following procedure as it might be applied to the manufacture of white sugar from cane juice.

In the first cycle in the manufacture of white sugar from cane juice, I add sufficient (approximately .05-.10%) activated positive colloid to the juice now known as evaporator supply juice; that is, a mixture of clarified juice and filter press juice, to produce a brilliantly clear juice, resulting in a pronounced and definite removal of color, and accompanied by a removal of impurities. The sludge from the filters will be returned to what I call secondary sludge storage tanks.

From this super-clarified juice I can produce a dull grey and almost white sugar. This sugar, which I will call "raw sugar", is dissolved into a 60-Brix solution, heated to 75-80° C., and treated with approximately .15%-.25% activated colloid, producing a brilliantly clear and water-white liquor. From this liquor I produce white sugar in the usual manner.

The sludge formed in the filtration of this raw sugar liquor is sent to what I term the primary sludge storage tank for re-use in treating the evaporator supply juice. The cycle now begins and remains this: a portion of primary and secondary sludges are used in treating this juice, yielding this super-clarified juice. The sludge from these filters, that is, juice filters, is returned to the secondary sludge tank for re-use. Obviously, as the surplus of this sludge increases beyond a certain amount, it is discarded into the mud settlings receiving tank, where it is eventually removed in the filter cake.

Thus, after one complete cycle in my process, fresh reagent is used only in treating the dull grey sugar liquor, the sludge from these filters going to the primary sludge receiving tank. Here it is used to make up a part of the charge for treating the evaporator supply juice, the balance of the charge consisting of sludge from the juice filters.

In this manner, I not only produce a water-white liquor, but also remove a certain amount of impurities in the clarification of the juices, thus increasing the recovery.

The actual amount of activated colloid used will vary from .15-.25% on solids in the sugar liquor, producing a white sugar equal in quality and surpassing that of most bone char refined sugars.

The cycles of operation will be more apparent from the accompanying flow sheet illustrative of my invention.

While in the foregoing description of my process the use of sodium chloride and aluminum hydrate have been particularly referred to in connection with the preparation of the colloidal reagent, and while such materials are employed according to the preferred embodiment of my

invention, it will be apparent that the invention is not confined to the use of these particular chemicals in the preparation of the highly activated colloidal reagent.

5 Another application of my process is in the preparation of syrups for canning purposes, such as fruits. In this application of my process and reagent, I first prepare a syrup of what is usually termed washed raw sugar; that is, a high purity raw sugar polarizing approximately 98.5 to 10 99.0. This syrup is heated to 75-85° C. and added to the reagent, usually .15-.25 per cent of solids in the syrup is sufficient to effect the desired clarification. If it is desired to produce an 15 absolutely water-white liquor, I can accomplish this by the addition of a small quantity of decolorizing carbons. I have found that .25-.50 per cent of carbon in solids will produce this result. By means of suitable filtering equipment 20 this treated syrup is filtered. The filtrate which is colorless and odorless is then ready for use.

Advantage can be taken of the unspent colloid

by reusing it in the manner described in my process for the manufacture of white sugar.

Obviously, I can use this same combination of reagent and decolorizing carbon in a process of manufacturing white sugar in which the procedure would be practically the same as that 5 used for preparation of canning syrups, but instead one would boil into white sugar.

What I claim is:

1. A process for clarifying sugar solutions 10 which comprises flocculating the impurities of said solution by means of aluminum oxychloride.

2. A process as set forth in claim 1 wherein the hydrogen ion concentration is adjusted to 15 between 4.7 and 5.2 pH.

3. A process as set forth in claim 1 wherein the hydrogen ion concentration is adjusted to approximately 5.2 pH. 15

4. A reagent for use in clarifying sugar solutions comprising a colloidal solution of aluminum oxychloride. 20

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