A. VON WACHTEL & C. WOEGERER.

PROCESS OF CLARIFYING RAW SUGAR.

APPLICATION FILED MAR. 2, 1912.

1,067,890. Patented July 22, 1913.

Fig. 1.

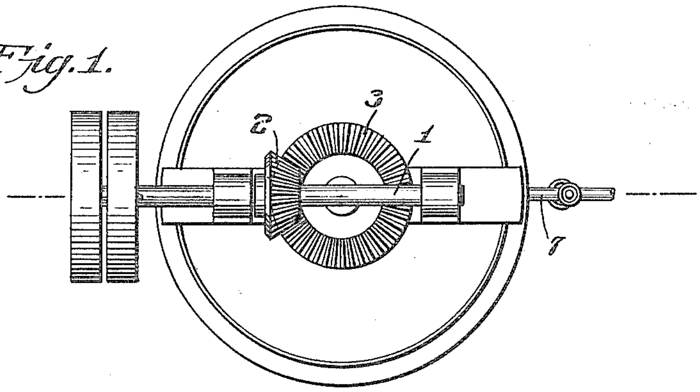


Fig. 2.

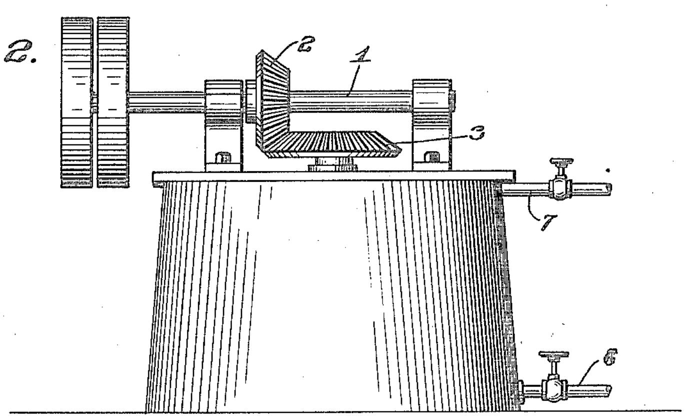
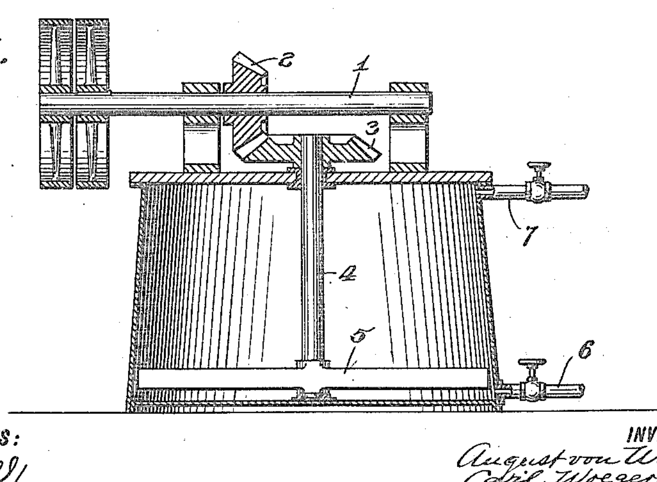


Fig. 3.



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PROCESS OF CLARIFYING RAW SUGAR.

1,067,890. Specification of Letters Patent. Patented July 22, 1913.

Application filed March 2, 1912. Serial No. 681,297.

To all whom it may concern:

Be it known that we, AUGUST VON WACHTEL, citizen of the United States, and CARL WOEGERER, subject of the Emperor of Austria-Hungary, both residing in the city of Philadelphia, State of Pennsylvania, have jointly invented certain new and useful Improvements in Processes of Clarifying Raw Sugar, of which the following is a full, clear, and exact description.

Our invention relates to the clarification of raw sugar stock, is best adapted for cane sugar, and has for its object the provision of improved methods for that purpose in full substitution for certain steps in the process as now practised, to the end that certain of those steps may be eliminated, a more speedy and complete accomplishment of clarification ensue, with consequent lessening of cost and somewhat increasing the yield of crystallizable sugar produced directly from any given quantity of raw stock operated on.

To these ends, our invention consists of the novel method or process of clarifying raw sugar stock hereinafter described, the identifying novel features being pointed out in the appended claims.

As well known in the art, the raw sugar treated contains in addition to the small crystals of pure sugar and adhering syrup, a varying quantity of deleterious impurities which are both mechanically and chemically combined with the stock, principally with the syrupy constituents, and which impurities must be completely removed, as also a waxy impurity which, while not deleterious in character, tends to cloud the liquid and delay, if not prevent to some extent, its clarification and filtration. For the removal of the deleterious impurities the present method of clarification involves as its first step a washing of the raw sugar, first with a low grade syrup, then with clear water, in a centrifugal machine, and this necessarily involves a loss to some extent of the comparatively small sugar crystals very easily soluble in water. After such washing it was customary to remove the washed sugar stock from the centrifugal machine to a melting pan, so called, which is a dissolving tank provided with a mixing arrangement wherein it is treated in hot water and by aid of steam until completely dissolved, the solution showing, when correctly manipulated, a temperature of 160–180° F., at a density of 26° Bé. This solution is then clarified either in the same tank or as a rule in another similar tank called a mixer, by adding lime and phosphoric acid or hydrofluoric acid and finally passed through filter bags to remove precipitated impurities prior to the filtering over bone-black and ultimate evaporation of the water of the solution to effect crystallization.

Our novel method or process dispenses entirely with the aforesaid preliminary step of washing in a centrifugal or other like machine and also with the employment of the usual last step of filtering out precipitated impurities by means of filter bags or the like, and this is accomplished by the employment of certain clarifying agents directly in the so-called mixing tank or mixer operated under the conditions, in the manner and with the functional effects as hereinafter stated; and these are very much more than the elimination of certain steps in the known process because of the prevailing conditions that the cost of profitably clarifying raw cane sugar cannot exceed, if it reaches two cents per hundred pounds; that no poisonous clarifying agents may be used; that sugar stock solution must be worked at a minimum density of 26° Bé.; that a quick and especially a complete precipitation of the deleterious impurities should be effected, to leave these impurities as a precipitate and the supernatant liquid clear with a slightly acid or alkaline reaction and a prevention of the growth of micro-organisms in the syrupy constituent, which tend to cloud the solution and retard and make difficult the clarification and impede the filtration of the suspended impurities. Our improved process successfully meets all these conditions and produces, in practically one operation, a more complete clarification in less time and more economically. We will describe it, as applied for example to raw sugar cane stock, containing on the average 94–97% of saccharose.

In the accompanying drawings we have illustrated, for example, a mechanical device, known in the art as a "mixer", and capable of being used to successfully practise our new process.

In said drawings Figure 1 is a plan view of a horizontal mixer; Fig. 2 an elevation; and Fig. 3 a vertical section thereof.

As these devices, both single and double paddle, are well known in the art, it will be sufficient to say that 1 is the driving shaft, suitably supported across the open top of the mixing tank, and by suitable gearing 2, 3, drives a shaft 4 which rotates a paddle 5, valvular discharge pipe 6 and overflow 7 being provided if desired.

Dispensing entirely with the centrifugal machine and with the preliminary washing with syrup and water therein now commonly employed as heretofore stated, we place the raw sugar stock directly in the mixer, either a horizontal or vertical mixer,—and to our experience a horizontal mixer provided with two helices, right and left handed, one running within the other on the same shaft,—to be the best mechanical mixing device for our purpose. Sufficient water, preferably slightly warm, is to be used, in the first instance, to make a somewhat concentrated solution of the sugar stock at a temperature not higher than approximately 140° F.; and in so doing it is preferable to put the water in the mixing device before adding the sugar stock. In the final step of the process, hereinafter described, an additional quantity of water is added, equal in volume to about that used in making the solution as aforesaid. The water is previously charged with a small quantity of finely comminuted chalk mineral, preferably Danish or English chalk evenly floated, say 2 to 15 pounds, in 50 gallons of water for 1000 pounds of sugar, the quantity of chalk dependent upon the extent of impurities apparent in the raw sugar stock. The chalk should be ground fine enough to pass through a 250-mesh sieve, and we found water-floated chalk superior in effect to air-floated material; and the finer the chalk is floated the smaller will be the quantity required. Instead of chalk an equivalent will be found, though at greater cost, in precipitated lime or magnesia carbonate.

After the contents of the mixer have been worked, only until all the original lumps of the raw sugar stock are disintegrated and all crystals evenly distributed in the syrupy carrier, in that way dissolving as little as possible the sugar crystals suspended in the liquid, there is then added to the aforesaid contents of the mixer, a cold solution of "bleach" or hypochlorite of lime (hypochlorite of magnesia can also be used) distributed in little water, in proportion of about 4 gallons for 1000 pounds of sugar, so as not to reduce the concentration of the total liquid to any extent. The best concentration is about 1 part bleach to 5 to 10 parts water.

The proportion of the bleach should not exceed $\frac{1}{10}$ of 1% of the sugar treated and frequently a quarter of that percentage will be sufficient for high grade sugar stock. After distributing the bleach by a few turns of the mixing arrangement, hot water not above 150° F. is added and in quantity equal to about 40% of water of the sugar stock treated, wherein a small quantity of phosphoric acid (H—3—PO—4) has been dissolved (best kept in a wooden tank) not exceeding $\frac{1}{10}$% of the weight of sugar stock to conform with the amount of bleach added before. The final reaction of the solution is very slightly acid.

During the addition of water, the mixing apparatus is kept in motion and continued after it until all sugar crystals are dissolved. The order in which the elements are added is not material except in degree of result, that stated being the best. The liquid which has about a temperature of 120–130° F. and a density somewhat above 30° Bé. is then let down in a settling tank provided with a heating arrangement and an overflow rim. The tank is rather higher than broad and has a slanting bottom with outlets for the discharge of the clear juice and the sediment, as generally practised.

The settling tank is filled not quite full with the liquid from the mixing tank, and heated to a temperature of 160–212° F. mostly 180–190° F. according to the quality of the sugar stock. The temperature is higher with lower grades of sugar stock. The steam for heating may be preferably admitted directly in the bottom of the liquid, by a perforated coil, in which case the amount of condensed water formed has to be taken in consideration—or indirectly by a steam jacket or any suitable means. The ultimate density is preferably between 26° and 30° Bé., but can be above or below these limits, say 20° to 32° Bé.

During the heating the level of the liquid in the tank will rise, partly by expansion, partly by water of condensation in case of direct steam, partly by forming of a scum. The increase in volume should bring the level of the liquid close to the top and close to overflow, after the desired temperature is reached, and the ultimate overflow is effected by a careful and slow addition of liquid from the mixer (or by addition of hot water) and by adding some lime water in the bottom of the tank with a quantity of lime just sufficient to change the slightly acid liquid, so as to show a desired low alkaline reaction. The addition to be continued until all scum formed is drawn off. The addition of lime water is essential in this step of the process. The liquid settles absolutely clear in $\frac{1}{2}$ to 2 hours leaving a sharply accentuated precipitate at the bottom in $\frac{1}{10}$ to $\frac{1}{6}$ of the total volume. The liquid can be siphoned or otherwise drawn off and the sediment be filtered over filter presses especially when the precipitate is diluted with some hot water. The scum is best filtered separately after some dilution with water. From analysis made there was never an increase of inversion but always an increase of purity observed of at least ⅓ of 1%. The color was vastly improved. At this last stage of the process it will be found that some of the impurities originally in the sugar stock after treatment with clarifiers, will not be precipitated but clearly separate and float to the surface as a scum in form of a distinct thin layer; and this scum being then removed, the liquid contents are left standing, though heated, when all other impurities will collect in large flakes, within a half hour to two hours, at the most—as mentioned above—and settle as a precipitate. As the liquid contains a small percentage of the free phosphoric acid employed, the precipitation will be accelerated by addiing a little lime water to neutralize the liquor or make it slightly alkaline as far as may be desired.

So treated, the usual filtering by filter bags is entirely dispensed with; the clear liquid left after removal of the scum and formation of the precipitate as stated being siphoned or otherwise drawn off and delivered at once to the bone-black filters; while the precipitated sediment at the slanting bottom of the settling tank together with the adhering liquor is then removed and delivered to usual filter presses or other convenient filtering apparatus and when diluted with some hot water readily forming cakes of sediment. It should be further stated, that the scum is distinctly separated as one result of the process because chlorin is liberated, which in turn at the higher temperature employed (above 160° F.) liberates oxygen gas; while the action of the phosphoric acid and other acids present in sugar stock on chalk liberates carbonic acid gas and both these light gases separate and float the scum. The latter may also be filtered after removal to recover any adhering sugar solution; and that such scum is best removed by an overflow—as stated—in the settling tank, as it contains all the wax, resin and lime soaps, is sticky and therefore has a tendency to adhere to the walls of the container and if removed as formed, the formation of clear liquor is speeded and more perfect.

Our improved process is further differentiated from that now in present use wherein the raw material is first dissolved at 160–180° F. at a density of 26° Bé., and the clarifying then effected. The liquid being in strongly colloidal condition, the precipitation at that density is incomplete, so much so, that even the filtered liquor will show cloudiness when diluted with water.

In our process all the clarifiers begin to act in more diluted solution before all sugar is dissolved in water. As the later dissolving sugar is very pure and the once formed precipitates are not re-dissolved when all sugar stock is gone in solution, we must have a superior clarification. In our process the preliminary water washing is not needed because the effect of the described successive steps of our process, effectually separates the impurities from the solution, even before the pure sugar crystals are formed and separated, also making the work more regular and avoiding the destructive effects on the sugar solution of the micro-organisms contained in the precipitate.

Our process is applicable also to clarification of second grade or molasses sugar, except that larger quantities of the clarifying mediums must be employed. For lowest grade molasses sugar polarizing for instance, 73.8%, we recommend 30 lbs. of chalk, 3 lbs. bleach, 2½ lbs. of phosphoric acid to 1000 lbs. of raw stock; the solution with water to a concentration of 29° Bé.; the resultant giving a clear liquid and a precipitated sediment of about ⅙ of the volume.

Aside from the improved results in the product obtained, our process effects a notable economy in the cost of clarification.

Having thus described our invention, we claim as new and desire to secure by Letters Patent:—

1. The process described of clarifying raw sugar stock which consists in first subjecting a given quantity thereof, in a suitable container, to the action of water containing in suspension an insoluble carbonate of an alkaline earth, a bleaching agent, and an acid agent adapted to separate the gaseous constituents of said elements, and combine with their insoluble bases to maintain their insolubility; then subjecting the liquid mixture to the further liquefying effect of an additional body of water sufficient when all sugar crystals are dissolved to produce a minimum liquid density of 20° Bé.; heating the liquor so prepared and agitating it, in the container, in a heated condition, until the sugar crystals are dissolved, then suspending the agitation and allowing the liquor to settle in the container until separation of impurities is effected.

2. The process described of clarifying raw sugar stock which consists in subjecting a given quantity thereof in a suitable container to the action of water containing in suspension an insoluble carbonate of an alkaline earth, a hypochlorite operating as a bleaching agent and having a destructive action on micro-organisms, adding thereto an acid agent adapted to liberate the chlorin and some carbonic acid constituents of said carbonate and hypochlorite, and combine with their bases to maintain their insolubility, then subjecting the liquor so prepared to further liquefying effect of an additional body of water, heating the liquor and agitating it until the sugar crystals are dissolved,

the water added being sufficient to produce a minimum density of 20° Bé., then suspending agitation and allowing the liquor to settle until separation of impurities is effected.

3. The process described of clarifying raw sugar stock which consists in subjecting a given quantity thereof, in a suitable container, to the action of water containing in suspension a finely ground insoluble carbonate such as chalk or precipitated lime, a hypochlorite as a bleaching agent, and sufficient phosphoric acid to liberate the chlorin and some of the carbonic acid constituents of said elements; then heating and agitating it and maintaining a minimum liquid density after all sugar crystals are dissolved, of 20° Bé. at a temperature less than 212° F., then suspending the agitation and allowing the liquor to settle until the impurities are separated from the clear sugar liquor, in part as a supernatant scum and in part as a precipitate.

4. The process described of clarifying raw sugar stock which consists in subjecting a given quantity thereof, in a mixing container, to the action of water containing in suspension a finely ground insoluble carbonate, a bleaching agent containing a chlorin constituent, and sufficient phosphoric acid to liberate the chlorin and some of the carbonic acid constituents of said elements; then heating and agitating it and maintaining a minimum liquid density after all sugar crystals are dissolved, at 20° Bé. at a temperature less than 212° F., then suspending the agitation and allowing the liquor to settle until the impurities are separated from the clear sugar liquor, in part as a supernatant scum and in part as a precipitate.

5. The process described of separating the impurities and producing a clarified sugar solution, which consists in first applying to the raw stock finely comminuted insoluble carbonate, hypochlorite of lime and phosphoric acid, in a volume of water less than that finally employed; then agitating the liquor in the container until all the sugar crystals are dissolved, and simultaneously heating it to not more than approximately 150° F. to produce a liquid density varying from 20° to 35° Bé., then arresting the agitation and raising the temperature to approximately less than 212° F., then drawing off all the lighter impurities separated from the liquor as a floating scum, and finally effecting a separation of the remainder of the impurities as a precipitate.

6. The process described of clarifying raw sugar stock which consists in subjecting a given quantity thereof in a suitable container, to the action of an insoluble carbonate having a calcium base, a hypochlorite bleaching agent and sufficient phosphoric acid to liberate in part their respective carbonic acid and chlorin constituents, in a body of hot water sufficient to produce a minimum density of 20° Bé., after agitating the liquor and dissolving all the sugar crystals, then raising the temperature to not in excess of 212° F. during settling of the liquor and separation of the impurities.

7. The process described of clarifying raw sugar stock which consists in subjecting a given quantity thereof, in a container, to the action of an insoluble carbonate such as chalk or precipitated carbonate of lime, and a bleaching agent such as a hypochlorite of lime, in the presence of an acid adapted to form an insoluble compound with lime, in a body of hot water sufficient in volume from time to time to produce, under agitation and maintained heat and after all sugar crystals are dissolved, a minimum liquid density of 20° Bé., and a maximum density of 32° Bé., then heating the liquor to impart thereto a maximum temperature of 212° F., and adding an alkaline neutralizing agent, such as thin lime water, then allowing the liquor to settle to produce a separation of impurities in two forms, namely, a floating scum and a precipitated sediment.

In testimony whereof, we have hereunto affixed our signatures this 29th day of February A. D. 1912.

AUGUST VON WACHTEL.
CARL WOEGERER.

Witnesses:
A. M. BIDDLE,
R. A. DUNLAP.