

UNITED STATES PATENT OFFICE.

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IMPROVEMENT IN EXTRACTING, REFINING, AND CRYSTALLIZING SUGAR.

Specification forming part of Letters Patent No. 90,762, dated June 1, 1869.

To all whom it may concern:

Be it known that I, LOUIS JOSEPH FRÉDÉRIC MARGUERITTE, of the city of Paris, in the French Empire, have invented a new and improved process for extracting sugar and increasing its produce in manufacture, refining, and forming it into loaves by means of alcohol; and I hereby declare that the following is a full, clear, and exact description thereof, which will enable others skilled in the art to make and use the same.

The first part of my invention refers to extracting sugar and increasing its produce.

The use of a mixture of alcohol with an energetic acid—say the sulphuric acid—for decomposing the molasses has already been indicated by me, and also two methods of extracting sugar from the residues, which retain it in an uncrystallizable state.

The first method consists in using alcohol (or wood-spirit) acidulated, comparatively diluted, in acting on common concentrating molasses, in holding thereby the sugar in dissolution, and after filtering, for the purpose of separating the sulphates, the chlorides or salts of the acid which was used in precipitating the sugar, by an addition of acetone, (pyroacetic spirit) ether, chloroform, sulphuret of carbon, essence of turpentine, either heavy or light coal-oils, &c.

The second method consists in causing the acidulated alcohol and molasses or saccharine material to react to their maximum concentration, and in obtaining the sugar in the solid state by precipitation.

A third method, which I now propose, is based likewise on the decomposition of the molasses by an energetic acid amidst the alcohol to such a diluent degree that the sugar may be held in dissolution; but instead of obtaining the precipitation through the acetone or pyroacetic spirit, ether, &c., I obtain it by mere simple direct crystallization.

I will now describe this new method of operation.

I take two hundred and twenty-five pounds (one hundred kilos) of exhausted molasses, marking in the cold state 45° Baumé, with twenty-two gallons (one hundred liters) of alcohol, marking 85°, and five per cent. of sulphuric acid—say one and one-fifth gallon, (five

liters) or twenty and three-fourths pounds, (about nine kilos, two hundred grams.)

The alcohol might be used in a more concentrated state; but then the molasses should be more diluted.

The whole is worked up in an apparatus provided with any suitable agitator, and the mixture, which is rendered homogeneous within a few moments, is filtered so as to separate it from the mineral salts formed. In this state the alcoholic liquor, which holds in dissolution the sugar on one hand, and on the other hand the acid displaced by the acids used, will not let off any sugar, on account of this remaining dissolved in the alcohol, marking 85°, which has been diluted of all the amount of water contained or inclosed in the molasses. It might be thought that by adding to the liquor twenty-two or forty-four gallons (one or two hundred litres) of alcohol, concentrating thereby the alcoholic medium, so that, theoretically, the sugar could not keep dissolved, the crystallization of the sugar would be obtained within a very short time; but such is not the case. There will be precipitated a small quantity of an impure colored sirup, which will get crystallized and yet not yield, by a great deal, the proportion of sugar that should be expected, the alcoholic liquor retaining most part thereof, and not giving it up completely after many days—nay, many weeks—rest, whatever may besides be the temperature of the ambient air. The question therefore has been to find out a means of determining the crystallization of the sugar by modifying the state of supersaturation of the liquor, which, by its permanence, has rendered the process absolutely inefficient.

I have imagined that, by throwing into the liquor some sugar-crystals, these, by the numerous surfaces or facets they present to the liquid by the attraction exerted between the particles of the same nature, would provoke and render, if not immediate, at least very rapid, the crystallization of sugar which appeared to be blasted; and my expectation proved true. In fact, if a weighed quantity of sugar-crystals is thrown in, so as to occupy the whole height of the liquid, in contact with the alcoholic dissolution proceeding from the molasses being attacked by alcohol at 85°,

first acidulated with a further addition of twenty-two gallons of alcohol at 95°, the following phenomenon will be observed: The alcoholometric standard of this attacking liquor, (16°,) formed of one part molasses, one of 85° alcohol and one 95°, may rise 38°, which indicates the crystallization of a large amount of sugar, while a portion of the same liquor, abandoned during the same time out of the contact of the sugar-crystals, does not increase in alcoholic degree, and by the same reason will deposit no sugar.

The alcoholometric degree of the liquor set in contact with the crystals has undergone the following augmentations: The initial standard being 16°, after one hour passed will be 35°; after two hours passed will be 45°; after three hours passed will be 48°; after four hours passed will be 50°; and next day, after eighteen hours, will mark 54°—say an increase of 38°. This increase of the alcoholic degree is proportionate to a deposit of sugar, which may be verified and rated unquestionably by the direct weighing of the crystals operated upon. It has risen to thirty-five per cent. of the weight of the molasses. This produce may be increased by the addition of a third volume of alcohol.

Previous to weighing the crystals upon which has settled the sugar, they must be washed in neat alcohol, and dried accurately, when the excess in weight indicates absolutely the amount of sugar extracted from the molasses.

As practically the drying of considerable amounts of sugar might meet with some difficulties, it is preferable to pass a decoloring material saturated with sugar, whereby the alcohol is carried off, which is afterward readily recovered by distilling. After the complete flow of the decoloring material, impelled by suction, it only remains to dry the crystals in the stove-room.

The presence of sugar-crystals in a saccharine alcoholic liquor does manifestly influence crystallization, which is of the utmost importance. The settling of the sugar appears to take place the more actively when the liquor contains more sugar and the crystallizing-surfaces are more multiplied. In other words, powdered sugar renders the crystallization of sugar far quicker. In fact, each crystal is increased and completed without apparent transition, and without any alteration of its initial state and constitution.

To prevent any precipitation of the sulphates with sugar, I add to the alcohol a very small quantity of chloride of calcium, or acetate of lime, which forms a sulphate of lime quite insoluble in alcohol, and which is eliminated by the filtering process. The sugar is thus extracted from molasses directly, by way of crystallization, in a state of great purity.

This last process has over the two foregoing ones the immense advantage of requiring the use of only one liquid—alcohol—and two very plain operations: first, the working up of the molasses, together with the acidulated alco-

hol, and the filtering of the liquor; second, the abandonment of this liquor to crystallization in presence of sugar-crystals. Such is my method of treating the molasses.

As regards the other cooked masses for the various jets in fabrication and refinery, I collect, by the ordinary turbinating process, all the sugar of first and second jets, which always is of very good quality. The sirups are concentrated and then treated exactly like the molasses, by adding, however, a sufficient amount of alcohol, at 85°, to dissolve the sugar contained in each of the saccharine matters.

The second part of my invention refers to refining and shaping the sugar into loaves.

Recent improvements, namely—double carbonating—have enabled sugar-manufacturers to obtain their first and second jets in such a state of purity as to be proposed directly for consumption as granulated sugar without undergoing the refining process. This sugar, of apparent good quality, was not so welcome in trade as would have been supposed, owing to various causes: first, its peculiar condition, its big granulation, its hardness, its slow dissolving, and its liability to mix with dust and extraneous bodies; second, its unpleasant odor and taste, its real impurity, the result of which frequently is a turbid dissolution. This odor results from a certain amount of fatty matter made use of in manufacturing sugar to facilitate its cooking, and which taints the crystals. I was enabled, by washing this sugar with a mixture of alcohol and ether, to extract and collect, through evaporation, rather notable quantities of nauseous-scented fatty bodies. Further, the manner in which this sugar is obtained will imply its lack of perfect purity.

It is obvious, indeed, that the turbinating and decoloring processes cannot be so nicely effected that no particle of molasses-sirup can remain on the surface of the crystals. Such a result cannot be obtained but by frequent decoloring, which would imply the inconvenience of dissolving large quantities of sugar already obtained.

It proved rather easy to remove the objection referring to the bigness of the grains. A more exiguous granulation was produced, but meeting with more difficulty than the others for the washing and decoloring, so that the sugars most in use and most easily dissolved were the most impure. In these conditions the shaping into loaves could not be attempted, and before thinking of their agglomeration it was necessary to proceed with the purifying thereof, which point I have realized; and therefore this second part of the invention has for its object, first, purifying sugar from its impurities; second, agglomerating the same, shaping it into loaves of any form, and, in a word, making directly refined sugar. The results I arrive at by the following means are, the white sugar of trade is first dried, malaxated, or worked up, then sifted and separated into samples of different sizes.

In a working-up apparatus, the powdered sugar is placed, with an alcoholic dissolution of the same, (passed previously on animal-black,) at a degree which varies according to the bigness of the grains. After agitating the mixture it is directed to a closed compressing apparatus, much like that made use of for making bricks, cakes, and other agglomerates of coal.

The walls against which the compression is made are of a filtering nature, and permit the flow of the surplus decoloring material. The sugar thus compressed gets purified and acquires a sufficient compactness to be transferred on a cloth, or by any other means, to a closed stove-room, where it is dried and the vaporized alcohol is collected.

The shaping into loaves may be also effected in the ordinary molds, which are filled up with powdered sugar, with addition of the saccharine alcoholic decoloring material, whereby the sugar is first purified, and next an aqueous saturated decoloring material, which displaces the alcohol, collected afterward through distillation. Next to the action of vacuum, which carries away the excess of liquid, the crystals shall retain a sufficient portion thereof for their proper agglomeration.

The molds, which, from the beginning of the operation, have been placed in a close stove-room, are now submitted to a current of hot air, and after the loaves have acquired a certain density they are uncovered, and their desiccation is completed. After this complete desiccation the sugar is perfectly white and pure, and may be rendered as hard as ordinary refined sugars, but much less so, too, which I consider as an advantage. In fact, according as the alcoholic dissolution applied to the agglomeration of the crystals is either more or less concentrated, it will dispose in their interstices a greater or less quantity of sugar, and yield thereby a more or less compact, solid product.

The alcoholic dissolution is intended not only to unite or gather together the crystals, but also to dissolve and carry away the fatty matters and the little amount of molasses which covers their surfaces, so that the sugar so treated may not present any of the objections opposed to the primitive product, but be possessed of all the qualities of refined sugar.

The alcoholic dissolution can serve a number of times, having care to decolor it with animal-black; but it must be distilled and renovated as soon as it no longer purifies and agglomerates the crystals properly. The residuum of this distillation is brought to the cooling-point, and will, by the ordinary processes, yield sugar, which may be refined and shaped into loaves in the manner aforesaid.

The little quantity of molasses resulting from this operation is treated by the method which has been precedently mentioned.

Instead of the alcoholic dissolution, I have

employed an aqueous saccharine decoloring material to purify and agglomerate the crystals; but the results obtained were not satisfactory with regard to the appearance and also the purity of the products, and no doubt, owing to this result, this already-proposed method has not yet been employed industrially.

One of the consequences resulting from the use of alcohol is that it allows obtaining candy-sugar very rapidly and in a very high state of purity.

The operation is disposed and conducted in the following manner: Two recipients are superposed, the upper one including the sugar to be dissolved and the lower one the crystals to be treated. These two vessels communicate through a tube. In the upper vessel alcohol is introduced, marking 50° to 60°, which, on going through a worm plunged in hot water, gets heated, can dissolve sugar and be supersaturated. In this state it passes down to the lower vessel, where it meets with the crystals, which it develops by getting cooled.

The cooling should not be too abrupt, because, as said, the desupersaturation—in other words, the crystallization of a saccharine dissolution—is taking place very rapidly at the contact of the sugar-crystals.

The liquor which has abandoned its sugar is brought back, by means of a pump, into the heating-worm, thence into the upper apparatus, whence it takes a fresh charge of sugar, to abandon it afterward on the crystals contained in the lower vessel. The operation, as seen, may be continuous.

This refining method is the natural complement of the foregoing operation, both forming a whole considerably advantageous to sugar-manufacturers, which advantages may be recapitulated thus: First, the present method of fabrication for the third jet and the treatment of molasses is substituted by the above-described process, which procures a surplus produce of about twenty per cent.; second, this sugar, collected by the old and the new systems, can be refined and lumped with the greatest facility at the works in the very place of their production; whence it results that sugar-manufacturers, who are both the producers and holders of the raw material, will be enabled to cumulate the benefits of both manufacturing and refining.

Having thus described my invention, what I claim as new, and desire to secure by Letters Patent, is—

As far as regards the first part of my invention:

1. The working up of the molasses, together with 85° alcohol, acidulated with any energetic acid, or variable proportions of alcohol at various degrees or temperatures, and the filtering of this mixture, substantially as specified.

2. Successive additions to the saccharine

dissolution of various volumes of alcohol, at 95°, or more concentrated, if possible, substantially as specified.

3. The setting in contact of the saccharine liquor with sugar-crystals, or hemp skeins, fabrics, or any supports previously covered with sugar-crystals, for the purpose of determining the crystallization of the liquor, which otherwise would remain inert, substantially as specified.

4. The crystallization of sugar amidst the alcoholic liquor, and obtaining the same in the pure state, in the presence of the eliminated acids, by sulphuric acid and all impurities contained in the molasses, substantially as specified.

As regards the second part of my invention:

5. Purifying the white or raw sugars of trade by means of alcohol more or less concentrated, substantially as specified.

6. Agglomerating this so pulverized purified sugar by a saccharine alcoholic dissolution of variable concentration, using for these two operations spirit of wood, substantially as specified.

7. Using an aqueous saccharine decoloring material for agglomerating and lumping the sugar-crystals washed by alcohol, substantially as specified.

8. Agglomerating the crystals, either with or without compression in close apparatus, substantially as specified.

9. Preparing sugar-candy by the continuous increase of the sugar-crystals amidst the solution of sugar into alcohol.

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Witnesses:

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