

# UNITED STATES PATENT OFFICE.

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## IMPROVEMENT IN OBTAINING SUGAR FROM MOLASSES.

Specification forming part of Letters Patent No. **148,375**, dated March 10, 1874; application filed February 20, 1874.

*To all whom it may concern:*

Be it known that I, LOUIS JOSEPH FREDERIC MARGUERITTE, of Paris, France, chemist, have invented Improvements in Making Sugar Crystallize in Molasses and saccharine juices in general; and I do hereby declare that the following is a full, clear, and exact description of the same.

This invention is based on the supposition that if certain salts possess the property of rendering sugar soluble others will render it less so—that is to say, will make it crystallize, which has led to practical results of great value in sugar-making.

A great number of salts, when added under certain conditions to a saturated solution of sugar, at the ordinary temperatures, will promote the more or less rapid crystallization of a portion of the sugar contained. The quantity thus eliminated from the solution varies according to the nature of the salt employed, and, in certain cases, is very considerable. The action of the salt, whether favorable or not to the crystallization of the sugar, appears to depend rather on the base of the salt than on its acid; thus, for example, nearly all the salts of potash are unfavorable in various degrees; salts of soda are either without effect or favorable; while the salts of magnesia and those of manganese all produce such a degree of crystallization as to greatly increase the product. The salts of soda, ammonia, baryta, lime, strontiane, magnesia, manganese, iron, zinc, will most of them provoke the crystallization of sugar.

The salts I propose first to use are those which, their bases being favorable, are found in abundance in nature, or which are the residue of another manufacture, and of such small value as to permit of their being entirely devoted to the purpose. The salts most suitable for use on a large scale are sulphate of soda, and among magnesian salts the sulphate and chloride of magnesium, obtainable from seawater in inexhaustible quantities. Among magnesian salts I use chloride of manganese, forming a residue of the manufacture of chlorine, and the sulphate of which may be readily produced from the chlorides; also sulphates of iron and zinc and their chlorides, which are obtainable in sufficiently large quantities. It is unnecessary to include the salts formed with

organic acids, which are rare and expensive, not excepting the acetates, nitrates, and ammonia salts which may also be mentioned in the same category, including nitrate of soda, they being all high priced, although they might be subsequently utilized as fertilizers.

The recrystallization or regeneration of the salts employed—the salts of ammonia, for example—would give rise to complications which should be avoided in the manufacture of sugar. In laying stress on the exceptions I have made in the enumeration of the salts which are capable of inducing the crystallization of sugar, it is in order to indicate clearly those only which are of practical utility.

This point being established, the following is the method of operation: It is evidently unnecessary to treat the first product of the beet or cane, as this is at once convertible into sugar on being boiled to grain, nor yet the second products, which crystallize quickly and without difficulty; but for treating third-quality sirups, and molasses especially, the process presents great advantages. Experiments made upon molasses cannot fail to be decisive when sugar is extracted from a product which has been put aside as useless for the purpose with the ordinary process of manufacture. I take, for example, sulphate of magnesia, which is very active, and is both abundant and cheap. I add to the spent molasses, (containing, say, fifty per cent. of sugar, fifteen per cent. of salts, and twenty per cent. of water,) crystallized sulphate in the proportion of twenty per cent., by weight, together with a little water, to make a solution of the sulphate marking 100° Baumé. The whole is then subjected to centrifugal action in a machine having either imperforated sides or very fine wire-cloth; the sulphates of lime and potash precipitated are retained, and the liquor is then filtered through charcoal and boiled *in vacuo*. After cooling, a certain quantity of pounded sugar is added to form nuclei, and the sirup is lastly subjected to the ordinary temperature of fillings, the heat being alternately raised and lowered, and after a few days crystallization becomes exceedingly abundant and continues to increase for some time, after which the hydro-extractor is employed. The above comprises the whole operation, which is very simple.

The proportions of sulphate of magnesia, or



other salt employed, will vary according to the nature of the molasses and the results of experience. The same conditions will apply to the use of chloride of magnesium, and the sulphates and chlorides of manganese and other salts. The chlorides possess the advantage of not forming sulphates of lime, but their action would appear to be less prompt and efficient. These various salts, when added to the molasses, react on those it originally contained, and the subsequent double decompositions and the double salts which may be produced must be borne in mind, in order to give rise as much as possible to those combinations which are most favorable to the crystallization of the sugar, for which reason it is advantageous to employ sulphate of magnesia, which precipitates the lime and potash in the form of sulphates, and forms a corresponding quantity of salts with a base of magnesia, all of which, as has been before observed, promote to a greater or less extent the crystallization of the sugar.

The above method of treatment may be applied both to molasses and to thirds, and when employed for second products, although with less advantage, it has the immediate effect of considerably increasing the yield of the second and third products, and at same time of increasing the value of the thirds, which become equal to seconds.

It has been seen that certain salts, when added to the molasses and sirups generally, will effect the crystallization of a portion of the sugar they contain, and that the nature of the base has a great influence on the quantity of sugar deposited, while other salts, on the contrary, will impede the crystallization to an extent which may be readily ascertained. It should be observed that the acid acts according to its nature in each class of salts, for which reason, in a general way, the chlorides and nitrates are less favorable than the sulphates.

Experience having shown that the solubilizing action of salts generally with a base of potash is to prevent crystallization, it is important to eliminate the same as much as possible from the molasses or sirups. The saccharine matter in which this elimination may most easily and efficiently be effected is molasses, properly so called, as it is in the latter product of the ordinary manufacture that are accumulated all the salts, among which, those of potash, which are the most prejudicial, predominate. Suppose molasses to yield fifteen per cent. of sulphuric ash, representing sulphate of potash. In order that double decomposition may take place between the salts of potash and the sulphate of magnesia, the latter salt must be added in the proportions of their respective equivalents—that is to say,  $1.089 : 1.395 :: 15 : X = 19.875$  kilograms, or, in round numbers, 20 kilograms (44 lbs.) of hydrated sulphate of magnesia for every 100 kilograms (220 lbs.) of molasses.

The sulphate of magnesia is first dissolved

in a small quantity of water, and then poured into the molasses, which is diluted to about 35° Baumé, the whole being at the ordinary temperature. In this manner an abundant precipitate is immediately formed, consisting of sulphate of lime and sulphate of potash, which are separated in a centrifugal machine or filter-press. The clear liquor is then filtered through charcoal, and finally boiled to proof in the ordinary apparatus. When cooled to about 90° or 100° Fahrenheit, a nucleus of fine sugar is next added, and the crystallization at once commences and becomes very abundant.

This crystallization of the sugar results from the elimination of the potash, the salts of which are prejudicial, its place being taken by the magnesia, whose salts are favorable thereto. The same remarks apply in the case of lime. Other sulphates besides those of magnesia will produce analogous results, such as sulphates of soda, magnesia, zinc, or iron. The chlorides, and, among others, chloride of magnesium, although favoring the crystallization of the sugar in a solution of pure sugar, will not eliminate lime and potash, but will increase the amount of salts, and, consequently, the density of the liquor at proof, and for this reason they do not possess nearly the same advantages as the sulphates.

It has been seen that the selection of the salt is an important point, and that, of all the salts which promote the crystallization of sugar, sulphate of magnesia is the one which furnishes the best results, both because its base favors crystallization and because its acid eliminates, in the form of insoluble sulphates, the lime and potash which are prejudicial.

It is evident that it is preferable to operate on molasses rather than on the anterior products, because, as has been before mentioned, the salts in molasses are at a maximum degree of concentration, and the effect of the sulphate, when added, is immediate and final. The sulphates of lime and potash are instantly precipitated, leaving but small quantities to be afterward deposited, which are soluble between 35° Baumé and the proof-point, 46° Baumé, whereas, should the first and second products be so treated, the sulphates of lime and potash would, at each boiling, and, indeed, throughout the whole process of concentrating, be deposited in the worms and on the sugar-crystals.

In all cases, the sugar thus obtained should be freed from the sulphates of lime and potash it contains. This may be effected in three ways: First, the sugar may be redissolved, filtered through bone-black, and boiled, above eighty per cent. of pure sugar being thus obtained. The sirups from the centrifugal machine, having been evaporated, are then suddenly cooled to prevent the sugar from crystallizing, and to cause the deposition of the sulphate of potash, which is afterward separated from the liquor by centrifugal action; or, secondly, the impure sugar is added to or



dinary molasses brought to the point of saturation—that is to say, in a condition in which it will not dissolve the sugar, which it therefore holds in suspension. In this manner the sulphates alone are dissolved, and, after turbinizing, the sugar will be found to be pure, the molasses thus acting as a kind of purifying-liquor, which, not being saturated with sulphates, dissolves them, leaving the sugar intact, and, this purification having been effected, it may be treated with sulphate of magnesia; thirdly, the raw sugar obtained is redissolved in pure water until saturated; it is filtered; a solution of chloride of calcium or of barium is added, until not any more sulphate of lime or of baryta is precipitated. The liquor is evaporated after filtration, and, when crystallized to grain, furnishes eight-tenths of pure white sugar, while the sirupy residue is used in the next operation.

It is evident that the sirups draining from the turbine after the redissolution of the sugar purified by the first method, and which contain sulphates, may also, like the purifying-liquor above mentioned, be returned to the general mass of molasses to be treated.

I claim—

1. The process hereinbefore set forth of crystallizing sugar in saccharine liquors by adding thereto a chemical salt such as described.

2. The process of purifying the sugar thus crystallized, as described.

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Witnesses:

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