PLANTATION
WHITE SUGAR MANUFACTURE

From A.M. Duperu. 1874-1944
Ref. Mgr. C & H Sug. Ref. Corp.
Crockett. California 1915-1932
See "Chemical Engineering" Apr 1945 P181

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WHITE SUGAR MANUFACTURE

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LONDON:
NORMAN RODGER,
2, St. Dunstan's Hill.

1913.

PREFACE.

The present work on the Manufacture of Plantation White Sugar as originally issued last year in Dutch under the title of "Handleiding voor Tropische Witsuikerfabrikatie" met with such a cordial reception in Java that a second edition was necessary within six months, and yet a third one will be produced ere long. So it was thought well to extend the use of this work to readers in other countries also, and with that object in view Mr. James P. Ogilvie, F.C.S., the technical editor of *The International Sugar Journal*, undertook to prepare an English edition of the Handbook which is herewith offered to the very widespread circle of English-speaking cane sugar producers all over the world in the hopes that it will meet a very urgent need of the day. We have only to add that in our opinion the translator has completed his task with extreme care and praiseworthy accuracy, and we fervently hope that this labour of his will be as welcome in other tropical countries as was the original Dutch work in Java.

THE AUTHORS.

August, 1913.

CONTENTS.

THE CHEMISTRY OF WHITE SUGAR MANUFACTURE.

THE MANUFACTURE OF WHITE SUGAR.

PLANTATION
WHITE SUGAR MANUFACTURE.

INTRODUCTION.

The object of this book is two-fold.

In the first place, it endeavours to give the reader a practical insight into the most generally adopted methods of preparing white sugar, describing the processes of juice purification, and the factory plant employed.

Secondly, it offers a theoretical consideration of the subject, and demonstrates the reasons just why such and such a procedure and no other must be adopted. This second object is indeed of greater importance than the first, since (as is hoped) it will form a basis for independent thought and independent research, without which all instruction remains a dead letter.

Before proceeding to discuss methods of manufacture, we propose presenting a succinct account of the chemistry of white sugar manufacture, and this must be thoroughly understood and learnt in order to study the subsequent parts with any profit.

For the sake of clearness, this account will be divided into the following sections :—

I. Influence of alkalis and alkaline earths on the constituents of cane juice.

II. Influence of acids on the constituents of cane juice.

III. Influence of heat on the constituents of cane juice.

IV. The colouring substances of the cane, and those which are produced during manufacture.

V. The different fermentations which may occur in the sugar factory.

THE CHEMISTRY OF WHITE SUGAR MANUFACTURE.

I.

INFLUENCE OF ALKALIS AND ALKALINE EARTHS ON THE CONSTITUENTS OF CANE JUICE.

As is well-known, the cane juice expressed by mills is a turbid fluid rendered frothy by entangled air. Its colour varies from light grey to dark green, and is derived from matter in the rind of the cane stalk.

It contains in solution all the soluble constituents of the cane, *i.e.*, sucrose, reducing sugars, organic and inorganic salts, organic acids, pectins and gums. Besides the air bubbles already alluded to, it further contains in suspension fine bagasse, clay, and sand, together with chlorophyll and albumin. Fresh mill juice has an acid reaction, which in the case of unripe or fully ripe cane is weak, but with over-ripe cane is greater.

The substances referred to will be discussed in their proper sequence, and in regard to their action with alkaline bodies.

Sucrose (Saccharose or Cane Sugar).

Alkaline substances, such as potassium, sodium, or calcium hydroxides (potash, soda, or lime) act upon solutions of sucrose (cane sugar) only when heated with them in a concentrated condition, the sucrose not being decomposed when boiled with only a few per cent. of potassium or sodium hydroxides (potash or soda), etc.

On boiling with concentrated solutions of alkalis, sucrose is decomposed into lactic, formic, acetic and humic acids, and humin substances, all of which of course unite with the base present to form salts.

In the case of low concentrations, the alkalis unite with sucrose, even in the cold, readily forming soluble compounds having an alkaline reaction, called *saccharates*, which require for their neutralization just as much acid as corresponds to the amount of the base present in the compound.

Calcium hydroxide, and the hydroxides of the other alkaline earth elements, barium and strontium, likewise form saccharates with varying quantities of the base; for instance in the case of the calcium compounds

Calcium monosaccharate: $C_{12}H_{22}O_{11}.\ CaO.$
Calcium disaccharate: $C_{12}H_{22}O_{11}.\ 2CaO.$
Calcium trisaccharate: $C_{12}H_{22}O_{11}.\ 3CaO.$

Of these the first two are soluble in water, but the third is practically insoluble. On boiling solutions of the first two, the saccharates are split up into insoluble trisaccharate and free sucrose. Clear solutions of mono- and di-calcium saccharate on being heated consequently become gradually turbid, the following reaction taking place:—

$$3C_{12}H_{22}O_{11}.\ CaO = C_{12}H_{22}O_{11}.\ 3CaO + 2C_{12}H_{22}O_{11}.$$

If carbon dioxide (carbonic acid) is introduced into a solution of mono- or di-saccharate, or into a suspension of calcium tri-saccharate, the gas is at first almost completely absorbed, but soon the mass becomes gelatinous and viscous, so that the carbon dioxide is only partly combined, the remainder escaping free. On, however, continuing the introduction of the carbon dioxide, the viscous liquid which copiously forms bubbles, will gradually become more fluid, the whole of the calcium carbonate being precipitated and the carbon dioxide being again almost completely absorbed until the point of neutrality is reached. The gelatinous and viscous compound formed by the introduction of carbon dioxide into the saccharate is a so-called "calcium hydro-sucrocarbonate," presumably of the following formula

$$C_{12}H_{22}O_{11}.\ 2\,Ca(OH)_2.\ 3\,CaCO_3.$$

This compound but slowly absorbs carbon dioxide, and only when it has for the most part been decomposed does the neutralization or saturation again proceed normally. Finally, all saccharates without distinction decompose on treatment with carbon dioxide into free sucrose and the carbonate of the base employed. Upon this reaction the principle of *carbonatation* is chiefly based.

Reducing Sugars ("Glucose").

The reducing sugars (often termed "glucose") are usually held to consist of glucose (dextrose or grape sugar) and fructose (levulose or fruit sugar) in varying proportions. As the action of alkalis on each of these sugars differs but little, the effect upon glucose alone will here be considered. This question is probably the most important one presenting itself in the manufacture of white sugar in the tropics.

Dilute alkalis and alkaline earths, especially at a high temperature, change glucose into a mixture of glucose, fructose, and mannose, the first two of which are finally present in about equal quantities, while the last constitutes a much smaller portion. Thus the addition of 5 grms. of lime at 70° C. (158° F.) within a few hours converts 100 grms. of glucose, dissolved in 400 grms. of water, into the forementioned mixture which is optically inactive.

In the cold the alkalis and alkaline earths unite with glucose to form *glucosates,* which are analogous to the saccharates. These compounds do not crystallize, and are easily decomposed. Calcium glucosate, for instance, is very unstable, and decomposes upon exposure to light at 50° C. (122° F.), assuming a brown colour.

Depending upon the temperature, concentration, etc., there is a *decomposing effect* of the glucose, which is of the greatest importance in sugar manufacture. In this case the glucose is converted principally into organic acids.

If a solution of glucose be heated below 70° C. (158° F.) with potassium or sodium hydroxides (potash or soda) the alkaline reaction gradually disappears, and on turning brown the salts of two acids called glucinic acid and saccharinic acid are produced. The former of these is very unstable, and decomposes immediately on heating and concentrating, but only gradually at ordinary temperature, into humic acid, acetic acid, formic acid, and carbonic acid, while some apoglucinic acid is also formed.

Saccharinic acid is more stable, and also forms well crystallizable salts. In the free state, however, it gradually decomposes, turning brown. To this spontaneous decomposition must be attributed the fact that the originally alkaline or neutral liquor may gradually lose its alkalinity or neutrality and become acid. This may be the cause of the acidification of syrups in sugar factories.

By the action of lime on glucose at temperatures below 55° C. (131° F.) the chief product is lactic acid, a very stable acid, not subject to spontaneous decomposition, though at higher temperatures the forementioned acids—glucinic acid and saccharinic acid, with their coloured decomposition products—are formed. Since the calcium (lime) salts of acetic acid, on the contrary, are colourless, during carbonatation, when much lime is present with the glucose, care must be exercised that the temperature remains below 55° C. (131° F.), so long as the liquid is not neutral.

All soluble normal salts having an alkaline reaction, without distinction, decompose glucose at boiling point, turning it brown; especially do the alkali and alkaline earth carbonates exhibit this phenomenon to a very decided degree.

By "alkaline" and "neutral" is meant here the reaction upon phenolphthalein, which for our purpose generally gives the most reliable indications, and which is almost always employed in sugar manufacture in every part of the world.

Organic Acids.

As has already been stated, the amount of these depends on the ripeness or over-ripeness of the cane. They comprise glycolic acid, oxalic acid, malic acid, succinic acid, tannic acid, etc., and in decaying cane, chiefly also acetic acid. In so far as concerns those not forming soluble calcium compounds, they are precipitated in the process of defecation by lime. The remainder, together with the decomposition products of glucose, referred to above, form the so-called "calcium organic acid" salts, which will be frequently considered in this manual.

Ash.

Generally speaking the ash content of the sugar cane is very small, and mostly, sometimes as much as 50 per cent., consists of potash. In the process of tempering by lime this potash is partly liberated as such; by treatment with sulphurous acid it is converted into sulphite, and into carbonate by carbonic acid. In this connection it must be borne in mind that these soluble compounds will, in the presence of the soluble calcium organic acid salts, be mostly or wholly decomposed with the formation of potassium organic acid salts and insoluble calcium sulphite or calcium carbonate.

Pectins and Gums.

Pectin is only partly precipitated by lime. Cane gum is precipitated but little or not at all by such small quantities of lime as are used in defecation, but it is for the most part eliminated from the juice with larger quantities of lime (as are used in carbonatation).

To this circumstance is principally to be attributed the fact that in carbonatation factories the filtration of the cane juices and syrups is so much easier a process than in those practising defecation.

II.

INFLUENCE OF ACIDS ON THE CONSTITUENTS OF THE SUGAR CANE.

As it may be considered superfluous to discuss the action of concentrated acids, since these are not employed in sugar factories, the following pages will be limited to a discussion of the influence of dilute acids.

Sucrose (Saccharose or Cane Sugar).

Dilute acids exercise upon sucrose a so-called *hydrolysing* effect by which one molecule of sucrose with one molecule of water is converted into one molecule of glucose and one molecule of fructose,

$$C_{12}H_{22}O_{11} + H_2O = C_6H_{12}O_6 + C_6H_{12}O_6.$$

The sucrose which previously deflected the plane of polarization to the right thereby loses this property and becomes levo-rotatory. Hence this phenomenon is termed *inversion*, and the sugar formed is called *invert sugar.*

The study of inversion plays an important rôle in the manufacture of white sugar, since, if it is not carefully prevented, a loss of sugar is involved.

The rate of inversion depends on the nature of the acid employed, on its concentration, and on the temperature. To the most rapidly inverting acids belong hydrochloric and nitric acids; while, generally speaking, organic acids must be classed with the weaker inverting acids.

Reducing Sugars ("Glucose").

Dilute acids exercise little action upon glucose. With fructose solutions, however, when they are heated with them for a considerable time, this is not so, the rotatory power of this sugar being thereby decidedly modified.

Organic Acids, Ash, Albumin, Pectins, and Gums, etc.

The action of dilute acids upon these constituents of cane juice is of little or no importance.

III.

INFLUENCE OF HEATING ON THE CONSTITUENTS OF CANE JUICE.

Sucrose (Saccharose or Cane Sugar).

Upon prolonged boiling of pure solutions at ordinary pressure, by taking up water, sucrose is gradually converted into a mixture of glucose and fructose in equal parts. Hydrolysis or inversion, therefore, occurs. The material of which the vessel in which the boiling takes place is made greatly influences the rate of inversion. Thus, in a copper vessel inversion will be much more rapid than in a silver one, while in a glass vessel it will be slowest.

Investigations conducted by HERZFELD have shown that the rate of inversion directly increases with the temperature and concentration.

Perfectly dry sucrose does not undergo any change on being heated, even to melting point, 160° C. (320° F.), but with the least trace of moisture the sucrose turns a dark colour, and liberating one molecule of water forms *caramel (or burnt sugar)*, which is much used in breweries and sugar factories as colouring material. By further heating the blackish brown caramel, carbon dioxide, formic acid, etc., are given off, and the result is simply a glossy carbon which, on continued heating, completely burns into carbon dioxide.

Reducing Sugars ("Glucose").

When glucose is heated above 100° C. (212° F.) it assumes a brown colour. Moist fructose decomposes at a lower temperature, when it gives off water and carbon dioxide.

IV.

COLOURING SUBSTANCES OF THE CANE AND THOSE PRODUCED DURING THE PROCESS OF MANUFACTURE.

In this chapter the investigations which have been conducted in Java in connection with the manufacture of white sugar are, to some extent, focussed. It scarcely needs to be mentioned that these investigations have not yet been concluded; they are, in fact, hardly begun. The colouring substances of raw and purified cane juice are so complex, and still so little understood, that the researches in this connection can as yet be only regarded as the first modest attempts in this direction. The colouring substances of which a slight knowledge has already been obtained may be divided into:—

a. *Vegetable Colouring Substances,* viz., those already present as such in the cane; and

b. *Chemical Colouring Matters, i.e.,* those as are formed, or may be formed, during the process of manufacture.

A schematic discussion of these colouring bodies is subjoined.

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a. VEGETABLE COLOURING SUBSTANCES.

In chemistry, these substances are in general divided into two primary groups, namely, Pigments and Chromogens. Pigments are those colouring substances which as such occur already formed in plants, and from which the latter derive their colours. In sugar cane we distinguish, for instance, *chlorophyll,* which imparts to the stems their main colour; and *anthocyan*, to which the bark of the cane owes its secondary tint. The latter, for instance, greatly preponderates in Black Cheribon cane, Black Manila, Red Ceram, G.Z.A., etc. In G.Z. No. 247, a small amount of anthocyan occurs side by side with a large quantity of chlorophyll. G.Z. No. 100, is practically without bark pigment.

In addition, the cane fibre contains *a yellow pigment* which is very intense, and must be regarded as one of the enemies of white sugar manufacture. This is the notorious pigment which turns bagasse yellow as soon as it comes into contact with lime, soda, or any other alkaline substance. We will refer again to this pigment later.

Chlorophyll.

The chlorophyll contained within the rind cells of the stalks is of little or no consequence in white sugar manufacture, for the simple reason that it remains undissolved in water and in cane juice, being therefore present only in suspension. On filtration after the juice clarification, it is consequently removed together with the other suspended particles, such as bagasse, clay, etc., without affecting the colour of the sugar subsequently produced.

Anthocyan.

In contradistinction to chlorophyll, anthocyan is readily soluble in water, so that during the process of milling it passes almost completely into the cane juice, causing the juice of the darker varieties of cane to be so much more coloured than that of the lighter kinds. By carefully scraping off the bark cells with a piece of glass and soaking them in water, a solution of the anthocyan itself is obtained, which possesses an extremely intensive colouring power, as has been proved experimentally.[1]

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[1] *Archief*, 1909, 302.

Anthocyan is completely precipitated by lime, so that if this pigment is not present in too large amount, and if sufficient lime is employed, it need not occasion any further concern in the manufacture of white sugar. Unfortunately, however, this is not always the case. As we have already said, the percentage of anthocyan differs considerably in the various types of cane, while in the different methods of clarifying the juice varying quantities of lime are utilized.

If, for instance, a variety of cane with a slight amount of pigment in its bark has to be crushed, the amount of lime used for defecation is usually sufficient for precipitating this small quantity. It is, however, quite a different matter when a purple or claret-coloured variety has to be treated. In that case the amount of lime used for defecation is quite insufficient for the elimination of the colouring body. Thus the favourable results of this method of manufacturing white sugar (saturation with sulphurous acid) are partly due to the fact that the varieties chiefly planted contain little colouring matter.

It has, however, been determined that—even in the case of the very darkest varieties—the addition of lime as milk-of-lime at 20° Bé. at the rate of 6 per cent. by volume of the cane juice is sufficient in all circumstances to precipitate the anthocyan. Now in the case of saturation with sulphurous acid, the amount of lime used is 1 per cent. by volume at the most; while, on the other hand, in carbonatation it is from 7 to 10 per cent., so that with the latter method all bark pigments are completely eliminated, and it is, therefore, independent of the variety of cane treated.

Nor is anthocyan entirely decolorized by sulphurous acid, the consequence being that sulphitation of the concentrated juice, which is invariably carried out in white sugar factories without distinction, is unable to bleach this colouring substance, even temporarily.

The Yellow Pigment of the Fibre.

The pigment, which completely pervades the cane fibre to such an extent that even after protracted boiling in water or alcohol this fibre still turns yellow when treated with alkalis, exhibits all the characters of an indicator, *i.e.*, in an alkaline solution it is an intense yellow, and in an acid solution it is colourless. This encrusting pigment which, according to the latest researches,[1] belongs to the aromatic compounds and exhibits characteristic lignin reactions, has been termed "saccharetin."

That all cane juice in consequence of the crushing process becomes mixed with saccharetin is shown most distinctly if the solutions used for polarization in the laboratory, after having been clarified by lead acetate, are rendered strongly alkaline by any base, when they will immediately turn yellow, but on the subsequent addition of an acid in excess will become decolorized.

As this proves that even lead is unable to precipitate saccharetin, it is not anticipated that defecation with lime, even in the carbonatation process, can eliminate it from the juice; and this pigment must therefore be looked for in all the products of manufacture. Since, however, it is colourless in acid solution it occasions but little trouble in white sugar manufacture. The colouring of the well-known Demerara crystals is nothing but the saccharetin mentioned. Saccharetin is insoluble in water and alcohol, but not in ether, by means of which it can be isolated.

As regards Chromogens (literally, colour-formers), these are bodies which, being colourless themselves, are only formed through the action of foreign influences. As a

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[1] *International Sugar Journal,* 1912, 53.

clear parallel in another plant—indigo—we may mention *indican*, which is absolutely colourless, and only upon oxidation yields the well-known indigo blue colour, "indigotin."

That chromogens must be present in cane juice is proved by the fact that all juices and syrups in sugar factories upon standing a long time become dark coloured from the surface downwards, even though no sulphurous acid has been employed. Their presence has likewise been established in beetroot juice. But nothing further is known concerning them.[1]

b. CHEMICAL COLOURING MATTERS.

Among these bodies, which may be formed during the manufacturing process, may be included in the first place the coloured decomposition products of reducing sugars, next the coloured iron compounds, and lastly the products of overheating the sugar, *i.e.*, caramel, etc.

Since they are of the utmost importance to the tropical manufacture of white sugar, we will consider each of them separately.

Decomposition Products of Glucose.

We have already seen that glucose and fructose, as well as the mixture of these, invert sugar, generally spoken of as *reducing sugars*, in the presence of bases or of salts with alkaline reaction, very readily split up into dark-coloured decomposition products.

If this occurs in an alkaline medium, the acids formed in the process will unite with the respective base into salts of the organic acids, the liquid gradually becoming less alkaline. In the foregoing pages, we have already seen that in sugar juices in the tropics, calcium or potassium

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[1] *Archief*, 1911, 823.

(lime or potash) salts of organic acids, which are dark coloured, are bound to occur. Now, these salts are darker in colour than the corresponding acid salts, and the latter, in their turn, are darker than the acids themselves. Glucose, however, which has given rise to these acids, is entirely colourless.

Now the organic acid salts referred to are among the most inimical substances in the manufacture of white sugar, as has been demonstrated in the clearest manner by the crystallization of these compounds with refined sugar, the originally white crystals then becoming quite brown.[1]

They can be only partially bleached by sulphurous acid, and even this decolorization, as with all reducing agents, is only temporary. The concentrated juice, in which they occur, can indeed to some extent, although in no case completely, be bleached by means of sulphurous acid; but the risk remains that the sugar after standing some time again becomes oxidized in contact with the atmosphere, and that the original colour, varying from grey to black, of the decomposition products of reducing sugars returns, these sugars darkening subsequently.

It follows from the foregoing remarks that in order sufficiently to contend with this danger it will be advisable to see that the glucose remains unattacked, since this is colourless. Of course, it will not be always possible entirely to prevent this, but the sugar manufacturer who knows his business will not for a moment lose sight of this important factor. In the course of the following explanation of the various methods of clarifying the juice this point will repeatedly be dealt with at full length.

It may, however, be observed at once that since these coloured decomposition products are formed exclusively in

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[1] *Archief*, 1909, 294, *et seq.*

an alkaline or neutral medium, the logical conclusion is that their production may most successfully be prevented by always keeping the juice slightly acid; while on the other hand, of course, the degree of acidity must not be allowed to rise so that inversion—and consequently loss of sugar—will result.

Salts of Iron.

These compounds, which were formerly considered to be the greatest enemies of white sugar manufacture, have at the present day lost their importance for the tropical industry. As a matter of fact they continue to constitute a great source of danger in the beet sugar industry, and in all refineries, but the methods of manufacture peculiar to the tropics greatly reduce this risk.

In chemistry two series of compounds of iron are recognized and are based upon (1) ferrous oxide FeO, and (2) ferric oxide Fe_2O_3. As will be seen from the formula, the second is a higher stage of oxidation than the first, *i.e.*, it contains a greater percentage of oxygen. The two series referred to are based on these two oxides, viz., ferrous oxide compounds and ferric oxide compounds, the former being converted into the latter by oxidation, while the latter reverts to the former by reduction. For the sake of brevity, they are termed *ferrous* and *ferric* compounds respectively.

The principal distinctive character of these compounds is that the ferric salts are mostly very dark in colour; while on the contrary ferrous salts in solution are nearly all as colourless as water, and in the crystalline state are at most pale green in colour. Since in the beet sugar industry, and in refineries generally, only ferric salts are met with, because no reducing agents are usually employed, the dark colour of these salts constitutes a great source of danger.

Altogether different conditions obtain in the tropical industry, where, as has already been stated, sulphurous acid or other reducing agents (sodium hydrosulphite, stannous chloride, etc.) are constantly employed for decomposing the troublesome decomposition products of reducing sugars; this reduction converts the ferric into ferrous salts, rendering them colourless.

That in this form they are really harmless to the colour of the sugars has been demonstrated, by causing both ferric and ferrous compounds to crystallize together with refined sugar, the results being very different.[1]

It has, moreover, been shown that in acid solution iron is unable to crystallize with sugar, which is also to the advantage of the cane sugar industry, because in beet sugar manufacture crystallization is effected in an alkaline or at most a neutral medium; whereas in the tropics, on the contrary, it invariably takes place in an acid solution.

This acid reaction is the necessary result of the use of sulphurous acid, but it is at the same time indispensable because, as we have already seen, the organic acids are lighter in colour than the acid salts, and these in their turn lighter than the normal salts. If, therefore, it is impossible to bleach the calcium and potassium organic acid salts by reducing agents, it is quite possible to effect this by means of some acid which decomposes them permanently.[2]

Products of Superheating.

True caramel is rarely or never formed under normal conditions in a sugar factory, since the temperature does not generally rise sufficiently, heating being effected by steam of 150° C. (302° F.) at the most. Formerly, when heating took place exclusively by means of naked fire the

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[1] *Archief*, 1909, 294.

[2] *Archief*, 1911, 14, *et seq.*

conditions were of course different, and much caramel must undoubtedly have been formed.

As a rule we have to deal with the lower products of overheating sugar, and these constitute colouring agents of little importance. Caramel can be bleached only to a slight extent by reducing agents, and might thus prove a formidable disturbing factor in the preparation of white sugar; but the heating products referred to here, which generally have only a yellow appearance, are on the contrary readily bleached by sulphurous acid and are of little or no consequence.

That means, when these products of overheating are already present in the concentrated juice they may be rendered innoxious, but not when they are produced *after* the crystal has already been formed, when, of course, sulphurous acid—or any other reducing agent—can no longer be of any avail. Yet this overheating will mostly occur in the crystalline crusts which are attached to the coils. Therefore it is quite essential to clean the vacuum pans out well with steam every time they are opened.

If, however, the pans are constructed without coils but with a heating body with a horizontal tube-plate (as in evaporating apparatus) the risk of an inferior quality of white sugar being produced becomes considerably greater. For even with a thorough cleansing by steam, crystals or sugar crusts will remain lying on the horizontal surface of the tube-plate, being unable to escape and becoming firmly fixed. If during the subsequent boiling such incrustations are not dissolved, they will mix with the white sugar producing numerous yellow crystals in the latter, which mischief may, with insufficient supervision, at times assume great proportions.

Pans with heating bodies should, therefore, only be employed in white sugar factories (except perhaps for the after-products) when the upper tube-plate has a considerable incline towards the escape pipe in the centre of the steam body.

Finally, for the sake of completeness reference may be made to the fact that at the commencement of the campaign the first sugars will assume a grey or yellow tint which, according to researches conducted with this object in view[1] owes its origin to suspended impurities in the juice derived from insufficiently cleaned apparatus or pipes, and not, as was formerly supposed, to some insoluble colouring substance or iron.

Proper filtration is, therefore, of the utmost importance under all circumstances, but especially at such early periods of the season. In most instances this first sugar is dissolved and returned to the first juice, the solution thus formed being refiltered, after which the product again becomes quite white.

V.

THE DIFFERENT FERMENTATIONS THAT MAY OCCUR IN THE SUGAR FACTORY.

Various processes of fermentation may occur in sugar factories which, especially in the preparation of a superior product, should be prevented as much as possible. Hence some consideration of this matter is desirable.

Fermentation is a term used for chemical decomposition resulting from the vital functions of micro-organisms, the best known processes of this nature in daily life being the fermentation of grape-juice into wine and of malt-wort into

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[1] *Archief*, 1911, 14, *et seq.*

beer. Both cases principally concern alcoholic fermentation, *i.e.*, alcohol (ethyl alcohol) and carbon dioxide are formed from carbohydrates—

$$C_6H_{12}O_6 = 2\ C_2H_5OH + 2\ CO_2.$$

The rate of fermentation depends chiefly on the *temperature*, the *concentration*, and the *reaction* of the liquids in which these micro-organisms develop and multiply.

The most favourable *temperature* at which these organisms increase most rapidly usually ranges between 30 and 45° C. (86 and 113° F.), most of them being killed at 80° C. (176° F.).

The most favourable *reaction* is a neutral or very slightly acid one. An alkaline reaction is generally unfavourable, with one exception, with which we shall become acquainted when dealing with dextran fermentation.

As regards *concentration*, the vital conditions for yeast cells are much more favourable in dilute than in concentrated liquids. It becomes patent from the foregoing remarks that especially in white sugar factories many conditions favouring fermentation occur side by side in unconcentrated juice. In these factories dilute liquids are employed at low temperatures and with neutral or slightly acid reaction, so that constant attention must be paid to this matter. It is fortunate, however, that adding lime in excess, as is practised for defecation in all white sugar factories, causes a fairly strong alkaline reaction, which, when accompanied by a sufficient rise in temperature, kills most organisms originally present in the cane juice.

Glucose and fructose are directly fermented by these organisms. Sucrose, however, requires first to be inverted in order to be fermented, although nearly all varieties of yeast secrete a kind of ferment (invertase) capable of converting sucrose into invert sugar.

Fermentation of sugar juice not only involves loss of sugar, but products are then formed which may have a deleterious effect on the quality and brightness of the juice and of the sugars obtained from it. Hence absolute cleanliness is essential in tropical white sugar factories in which a very fine product is really desired. Sweet liquor on the floors, for instance, may give rise under the favourable conditions of the climate to the formation of yeast growths from which infection may spread in all directions.

In cane sugar factories the following forms of fermentation may occur:—Dextran fermentation, lactic fermentation, butyric fermentation, and alcoholic fermentation. Each of these will now be briefly discussed.

1. Dextran Fermentation.

In nearly all colonial sugar factories raw juice is infected by a micro-organism called *Leuconostoc mesenterioides*, which is capable of rapidly forming from sugar a gelatinous substance, principally consisting of dextran, which process is accompanied by a strong formation of lactic and acetic acids. This is one of the few organisms which require an alkaline reaction as one of the principal conditions for their propagation.

By the production of acid the vitality of *Leuconostoc* is weakened, and if the liquid be neutral or very slightly alkaline dextran fermentation soon ceases. In a strongly alkaline solution, on the other hand, in which the acids produced can immediately be neutralized, fermentation proceeds at an incredibly rapid rate, and in a short time much sugar can be decomposed.

Dextran fermentation in cane juice needlessly increases principally those substances forming soluble compounds with lime, which are either coloured or else increase the ash content to an undesirable extent. An effective preventive against *Leuconostoc* in cane juice is the disinfection of the mills with a 1 per cent. solution of sodium fluoride.

2. Lactic Fermentation.

All the sugars present in cane juice are attacked by lactic acid bacteria, of which several kinds may be present. Reducing sugars are most readily decomposed, whereas sucrose, as already stated, requires to be inverted before fermentation can set in:

$$C_{12}H_{22}O_{11} + H_2O = 2\,C_6H_{12}O_6,$$
$$C_6H_{12}O_6 = 2\,C_3H_6O_3.$$

Lactic fermentation is one of the most frequently occurring in tropical sugar factories when using carbonatation (in the neutral lukewarm filtered juice) as well as when using defecation (in the slightly acid muddy juice, which has been allowed to cool too long). Raising the temperature to 70° C. (158° F.) and upwards is the best means of prevention.

3. Butyric Fermention.

When a liquid has been subjected to lactic fermentation and is left standing a long time, the lactic acid is usually converted, by the action of other bacteria, into butyric acid, carbon dioxide, and hydrogen.

$$2\,C_3H_6O_3 = C_4H_8O_2 + 2\,CO_2 + 2\,H_2.$$

Butyric fermentation is generally very noticeable in the vicinity of tropical sugar factories as a result of the discarded molasses, or from the rejected wash-water from the syrup tanks. It is characterized by a peculiar, unpleasant and penetrating smell, like rancid butter.

It does not occur in the juice, since time is generally insufficient to allow of this. In syrups of high concentration it is likewise unable to exist, and can only make its appearance when the latter are strongly diluted.

4. Alcoholic Fermentation.

This form of fermentation seldom occurs of its own accord in sugar factories. It is, however, produced artificially in dilute molasses for the manufacture of spirits.

Finally, reference is made sometimes to so-called "froth fermentation," a phenomenon greatly resembling that of ordinary fermentation, except that it only occurs with high concentrations. It is produced by the spontaneous decomposition of the calcium (lime) salts which, as has already been explained, arise from the decomposition of reducing sugars by lime at moderate temperatures. Micro-organisms are, therefore, not concerned in its production. Through the large quantities of carbon dioxide evolved from this decomposition, the concentrated massecuite, etc., commences to froth, and at times runs over the boiling pans, the cooling troughs, or the crystallizing tanks, the temperature at the same time rising considerably. Especially in the preparation of solidified molasses, when this has to be brought to a high state of concentration, much trouble is sometimes occasioned by froth fermentation. The chief concern is then to see that the temperature does not rise too high.

The possibility of froth fermentation setting in is an additional reason for constantly guarding against needless decomposition of reducing sugars.

THE
MANUFACTURE OF WHITE SUGAR.

From a chemical point of view the manufacture of white sugar may be divided into two principal divisions, *i.e.*, *clarification of the raw juice*, and *treatment of the concentrated juice and the syrups.* Whereas the latter process, the treatment of concentrated juice, differs but little or not at all in sugar factories, the method of clarifying the raw juice, on the other hand, varies considerably.

According to the methods employed in the latter process, factories are classed as Carbonatation and Sulphitation factories. The discussion of these two methods of clarification is the main object of this manual, and the success of the manufacture of white sugar chiefly depends on a true and clear conception of the chemistry of these two methods, which are otherwise fairly parallel. Fortunately, the mechanical installation for these processes is very simple.

Before the preparation of plantation white sugar was taken up, only one method of clarification was known. Raw cane juice which, as we have seen in the Introduction, invariably has a more or less acid reaction, was tempered in the cold with so much milk-of-lime until it showed a weak alkaline reaction to litmus paper, after which it was boiled and allowed to settle. This was the ordinary defecation process, also called "upward separation," because a large proportion of the impurities, instead of settling, was carried with the particles in suspension to the surface of the liquid.

When later some conception of the chemical nature of the processes involved was acquired, it became apparent that the evaporation and boiling of these alkaline juices frequently occasioned difficulties in the manufacturing process because the decomposition products of the "glucose," which were generated by the lime on raising the temperature, were very viscous and dark-coloured. At first an attempt was made to convert the calcium (lime) organic acid salts into sodium salts by adding cheap commercial soda (sodium carbonate) to the juice, since these sodium salts are considerably less viscous than the calcium (lime) salts of the same acids.

This method, however, had certain drawbacks: (1) Many impurities were introduced into the juice by the soda; and (2) to ascertain the amount of soda required involved accurate determinations of the quantity of lime, which in practice proved very troublesome.

At last it began to be understood that in this case, as in all similar matters, prevention was the real solution of the difficulty and promised the best results. In order as much as possible to obviate the formation of the viscous decomposition products of "glucose," it was recognized that the clarified cane juice would have to be made less alkaline, or, better still, neutral. With this object just sufficient lime was added to neutralize the acid reaction of the raw juice, so that consequently there could be no excess of lime.

This was a considerable step in advance, which was so successful that soon manufacturers went further, and the liming process was again reduced and a weak acid reaction instead of a neutral one to litmus paper was produced.

The result was the production of a juice which was more easily boiled and cured, *and especially of lighter-coloured sugars.*

The great drawbacks, however, which came to light upon the application of this improved defecation process, were twofold, *i.e.*: (1) on account of the reaction being kept acid, the juice was generally treated with too little lime for the proper elimination of all the impurities which otherwise could be precipitated; and (2) it was very difficult, if not impossible, to gauge with absolute certainty at any moment for each juice the quantity of lime to be added, so that frequently the reaction was too acid or now and again even alkaline. In short, no rule was possible.

The latter circumstance especially has been instrumental in introducing "saturation" into the cane sugar industry. Just as in analytical chemistry, in volumetrically determining minute quantities, occasionally an excess is added which is subsequently titrated back, so in tempering with lime the practice (borrowed from the beet sugar industry) has been introduced into sugar factories of first adding lime in a large excess, and afterwards removing this excess by means of some acid to any desired reaction, be it alkaline, neutral, or acid.

Of these saturation methods carbonic acid saturation, introduced by French machinery manufacturers, was the first to be applied, and has since been called "*carbonatation*" from the French term for this process.

It was not till later that sulphurous acid saturation, or briefly "*sulphitation*," became generally employed in the cane sugar industry. As, however, by "sulphitation" is understood the sulphuring of the syrups and of the molasses, it is also distinguished by the term "raw juice sulphitation." The nature of the process, however, is best indicated by the term "sulphurous acid saturation," for which reason the latter nomenclature will be used as much as possible in this manual.

After this historical introduction we will proceed to the separate discussion of the two methods.

I.

CARBONIC ACID SATURATION (CARBONATATION).

Of the two saturation methods, carbonic acid saturation is the older one. Sulphurous acid saturation has been adopted on account of the fact that the former required too much lime, and the process consequently became too expensive. In carbonatation the quantity of lime used is in fact ten times that employed in sulphurous acid saturation. If less lime were used, it would be found that sedimentation or filtration would not very well be possible—at least at the temperature attained. On the other hand in sulphurous acid saturation, settling of the calcium sulphite (sulphite of lime), even in small quantities, is effected much more readily. In carbonatation calcium carbonate (carbonate of lime) is formed.

Now the carbonic acid saturation process, which, as has been stated, is copied from the European industry, had to be entirely modified for tropical conditions. Although as a matter of fact the juice from sugar beet shows in most respects a resemblance to that obtained from the cane, an essential difference exists in one particular, which is of so material a nature that, whereas otherwise we would simply have had to copy beet sugar manufacture, we are compelled to act upon our own initiative as regards the chemical aspect of the matter. This one particular is the presence of large quantities of "glucose."

Where according to the theory of Prof. De Vries, sugar is deposited in the form of starch in the chlorophyll corpuscles of the leaves of sugar-producing plants to be carried downwards during the gradual conversion into sugar, it will be evident that the lower any part of the plant, the more complete will be the conversion of the starch into sugar.

Now in the case of sugar beet the root is harvested, while in that of sugar cane the stem is gathered; and it will be clear that, relatively speaking, this process of conversion will be less perfect in sugar cane than in sugar beet and that consequently more of the inversion product "glucose" will be present in the latter. Conformably with this view, the top of the cane stem invariably contains more "glucose" than the base. Moreover, as a result of the heat in tropical countries the point of maturity is in close proximity to that of over-ripeness or decay, and the products first produced from the transformed sucrose will again be invert sugar.

It is the presence of these reducing sugars which makes the manufacture of white sugar in the tropics or sub-tropics so infinitely more complicated than that in temperate climes, and necessitates constant and undivided attention.

While in Europe the diffusion juice is heated at once to 90° C. (194° F.) and defecated by means of large pieces of unslaked lime, this process is altogether out of the question in cane juice carbonatation. The rise of temperature above 50° C. (122° F.) must be avoided at all costs, while lime must never be used in the unslaked state in view of the heat liberated on hydration. Hence, one is entirely restricted to the use of milk-of-lime.

Whereas in Europe the juice is constantly kept alkaline during the whole process of manufacture, in cane producing countries this is carefully avoided; and, as has already been explained when dealing with defecation, the chief aim is to render it neutral or even acid as soon as possible.

If we study the chemical action of lime on cane juice, it will be seen that first the free organic acids will unite to form calcium (lime) organic-acid salts. The combined organic and inorganic acids (for instance, potassium salts) may likewise be decomposed, when if they form insoluble compounds with the lime the potassium will be liberated as potassium hydrate.

It is, therefore, of the utmost importance constantly to bear in mind that in the case of tempering with lime in excess, the resulting alkaline reaction is not only to be attributed to the free lime, but also, although of course to much less an extent, to free potassium hydroxide (potash).

The lime also partly unites with the albuminoid, gummy, and pectin substances, etc., while we have noted that during tempering with lime in the carbonatation process (using 7–10 per cent. by volume of milk-of-lime at 20° Bé.) all anthocyan and chlorophyll, that is all the bark pigments, are precipitated. The sugar further combines with the excess of lime, which at the outset occurs in suspension, dissolving it, as long as the juice is not heated, forming saccharate.

Of the utmost importance in reference to what will be discussed further on when dealing with carbonatation, is the composition of the milk-of-lime used for defecation. The slaking of the lime is dependent on time and temperature, and the longer the milk-of-lime is allowed to stand, and the higher the temperature at which hydration takes place, the more homogeneous and the more complete will be the operation.

In proportion to the care bestowed on its preparation, the milk-of-lime will contain a quantity of unhydrated particles of lime, lumps which, if not carefully removed by sieves, may give rise to complications, especially in single carbonatation, as will be seen later on.

It will be advisable, therefore, to use lime as fresh from the kiln as possible for preparing the milk-of-lime, and to protect the slaking drum against loss of heat in order that during slaking the highest temperature possible may be maintained. Afterwards the milk-of-lime should be passed through a proper system of sieves, stirred in a spacious trough as long as possible, and cooled off.[1]

For the reasons enumerated above, the lime must, therefore, *never* be applied in the form of dry powder slaked in the open air, no matter which method of clarifying the juice be adopted. As a matter of fact, in this form it can hardly, if at all, be sifted.[2]

After tempering by lime and settling in the cold have taken place, the carbonic acid saturation is started. In order, however, to promote as much as possible the proper mixing of the milk-of-lime with the juice, and also to shorten the period of defecation, in view of the decomposition of "glucose," the carbonic acid is introduced previously, or together with the milk-of-lime, into the cold juice, the gaseous acid, which is forced into the juice by a powerful pump, causing strong agitation.

By this treatment with the carbonic acid gas those compounds which are insoluble in water will first be converted into carbonates, in other words, the alkalinity of the lime will be diminished before that of the potash, for the simple reason that potassium carbonate is soluble, while calcium carbonate (carbonate of lime) is not. Certain cases may, therefore, be conceived when, although the liquid remains alkaline, the carbonic acid can no longer induce further precipitation. This latter alkalinity is termed "potash alkalinity."

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[1] *Archief*, 1911, 1410.

[2] Limestone used for preparing the milk-of-lime must *always* be carefully analysed, and should never contain more than 0·5 to 1 per cent. of magnesium oxide.

The union of lime with carbonic acid in cold solutions containing sugar (for the juice still remains unheated) is not so simple as might be imagined in the following equation:—

$$\underset{\text{Lime}}{Ca(OH)_2} + \underset{\text{carbon dioxide}}{CO_2} = \underset{\text{calcium carbonate}}{CaCO_3} + \underset{\text{water.}}{H_2O.}$$

It must not, in fact, be forgotten that lime is present as saccharate, and that on neutralization by means of carbonic acid at about the middle of the process a so-called double compound of calcium saccharate and calcium carbonate is formed, as has already been mentioned in the introduction. This insoluble "calcium hydrosucrocarbonate," which has the following formula:—

$$C_{12}H_{22}O_{11}.\ 2\,Ca(OH)_2.\ 3\,CaCO_3,$$

renders the juice extremely viscous and dense, causing the carbonic acid to pass through with difficulty, and the carbonatated juice to begin to froth in such a manner that it would run over the sides of the tank if no precautionary measures were taken.[1]

The saturation gas employed, which should contain about 30 per cent. or more of carbonic acid (CO_2), at the outset yields nearly all its content, so that it escapes with at most 4 to 5 per cent. of carbonic acid from the flues of the saturation tanks.

The percentage of carbonic acid in the escaping exhausted gases gradually commences to increase, in exact proportion to the formation of the viscous calcium hydrosucrocarbonate which absorbs carbonic acid with such difficulty, that the carbonic acid content reaches 10 to 15 per cent. This is the moment when the viscosity is at its height and

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[1] This phase of carbonic acid saturation may be clearly demonstrated in the laboratory by treating a concentrated solution of sugar (25 to 30 per cent.) with lime and carbonic acid. When sufficient lime has been added, the viscosity of the liquid may be observed at the exact moment to increase in such a manner that it gradually stiffens completely, and can no longer be forced to absorb carbonic acid.

frothing is worst. The saturation tank should then be closed in order to prevent a possible overflow, and also to avoid the injurious effect of the now freely escaping vapours laden with the gas which otherwise might easily occasion suffocation.

The measures which ought to be adopted in general practice for restricting the loss of carbonic acid and troublesome frothing over, are: (1) Decreasing the supply of carbonic acid at the right moment by opening the valve of the next saturation tank, so that the carbonic acid, which otherwise would escape, becomes available for the juice contained therein; this juice is in an earlier stage of neutralization, and, therefore, readily absorbs the carbonic acid. (2) The beating down of the froth by a jet of steam. The latter measure, however, is not always without risk, because the still strongly alkaline juice may be overheated locally, and thus produce dark coloured decomposition products of "glucose."[1]

Saturators of modern construction make overflowing impossible by their height, which for tropical juice is certainly preferable. The same result may, however, be obtained by beating down the froth, not by steam, but by carbonic acid, the effect being obtained by the bursting of the carbonic acid bubbles.[2]

The moment of violent frothing having arrived, the heating of the juice may be proceeded with, because the alkalinity is now very much reduced. Not only has half of the free lime formed calcium carbonate (carbonate of lime), but a portion of it is present in the insoluble form, as "calcium hydrosucrocarbonate," phenolphthalein paper showing that the original blood-red reaction has changed to a very slight red.

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[1] *Archief*, 1909, 119. [2] *Archief*, 1909, 219.

By the heating now in process, and the constant motion of the carbonic acid bubbles, the troublesome double salt will gradually decompose, the reaction on phenolphthalein paper becoming gradually deeper red, and the percentage of carbonic acid contained in the escaping exhausted gas again decreasing until finally the thin liquid juice again absorbs nearly all carbonic acid. Finally, the calcium hydrosucrocarbonate splits up into sugar and calcium carbonate (carbonate of lime) and the process is completed.

Having arrived at this stage, we may state the various views which have given rise to the two different methods of carbonatation, depending on the number of stages occupied in the process. Thus in Java at least it is differentiated into single and double carbonatation, each of which will be treated separately below.

Since, however, the double process is almost exclusively applied, this should be considered first. With an alkalinity of about 400 mgrms. of calcium oxide (CaO) per litre of juice—therefore when its reaction to phenolphthalein is still strongly alkaline—the supply of carbonic acid is stopped and the crude juice is filtered through filter-presses, the resulting clear juice, termed "first carbonatation juice," being next further neutralized likewise by carbonic acid in a separate apparatus. This is the so-called "second carbonatation" which is succeeded by the second filtration. The juice has, consequently, been neutralized in two stages. In the single process, however, the crude juice is fully saturated in one stage to the neutral reaction to phenolphthalein, and subsequently filtered only once.

The result in either case is a neutral clear juice which, moreover, is of practically the same composition, and can forthwith be evaporated.

a. Double Carbonatation.

This method has been copied entirely from the European industry. It is based on the observation that the juice saturated in two stages is lighter in colour, and also that some salts of lime, such as calcium oxalate (oxalate of lime), are more readily precipitated in an alkaline medium and can, therefore, be more easily filtered. If this is not done, and filtering does not take place until the liquid is neutral, or only slightly alkaline, there is a risk that a portion of the calcium oxalate precipitated originally will again be dissolved.

Other acids likewise possess this property, as for instance, glucinic acid, with which we become acquainted in the introduction as one of the decomposition products of "glucose." For calcium glucinate has the property of forming a so-called "basic glucinate" in alkaline solution, which, contrary to the neutral salt, is insoluble. We will find, however, that in cane juices saturated below 50°C. (122°F.) this acid will hardly be produced.

When double carbonatation, therefore, is applied in the cane sugar industry it is not in order to obtain a lighter coloured juice—for this is not the case—nor in order to produce less salts of lime or to effect better clarification—since rather the contrary happens—but only and solely because the double process is the *safer* one.

Possible errors in the reaction of the first carbonatation juice may be rectified on second carbonatation, and any turbid juice of the first filtration can easily be clarified on second filtration. It is thus simply a matter of attention and convenience. As against this, however, the greater cost of installation and labour is to be taken into consideration.

As we have already mentioned, in this double method saturation is arrested as soon as the juice shows an alkalinity of about 400 mgrms. of calcium oxide (CaO) per litre, experience having shown that at this stage of alkalinity the raw juice can be readily filtered. If filtration were attempted at an earlier stage, it would be found that this was scarcely possible on account of the great viscosity of the still incompletely decomposed calcium hydrosucrocarbonate. Moreover, since this double carbonate is insoluble, some sugar would be lost in filter-press cakes.

Nor is filtration at a later stage advisable, because in the case of an alkalinity less than 400 mgrms. per litre, too little calcium carbonate would be formed on second saturation, causing the second carbonatation juice to filter less readily. It would, in fact, pass through in a turbid state, and milk-of-lime would have to be added at the second carbonatation in order to increase the quantity of the precipitate. This alkalinity of 400 mgrms. of calcium oxide (CaO) per litre is probably not always exclusively due to lime, but is partly also derived from the potassium hydroxide (potash) originally present in the cane juice and liberated on tempering with lime.

Since the potassium carbonate is soluble it cannot assist in increasing the quantity of precipitate. If the potassium hydroxide liberated, when the cane is rich in that constituent, equals or exceeds the 400 mgrms. of calcium oxide (CaO) per litre, it is evident that even on second carbonatation no precipitate will be formed at all, and the juice will be as clear on leaving as on entering. Fortunately, however, this condition is of extremely rare occurrence. But, as we shall find later on, it is attended by risk as regards a good quality of white sugar.

Now as to determining an alkalinity of about 400 mgrms. of calcium oxide (CaO) per litre, we possess various means which we will proceed to enumerate :—

(1) The surest means, of course, is direct titration with N/56 or N/28 sulphuric acid. For this purpose as much of the raw juice is filtered as will allow 10 c.c. of the clear juice to be measured with a pipette. After the addition of a few drops of phenolphthalein solution, the standard acid is added drop by drop until the indicator becomes decolorized. If about 8 c.c. of the N/56 or 4 c.c. of the N/28 acid have been used, the alkalinity of the juice will be about right.

(2) As this titration requires skill, which cannot be expected from most native workmen, and also involves considerable expenditure of time, manufacturers as a rule restrict themselves to the use of the so-called "Dupont paper," which is ordinary filter-paper soaked in a mixed solution of phenolphthalein and oxalic acid. This mixture must be of such strength that the paper after having been dried in the air only assumes just a very faint rose tint on being dipped in a liquid having an alkalinity of 400 mgrms. of calcium oxide (CaO) per litre.

(3) Finally, a very approximative method exists of judging the reaction of the fluid from its appearance. As we have already noted, the carbonatated liquid only filters or settles readily on reaching an alkalinity of about 500 or 400 mgrms. of calcium oxide (CaO) per litre. Previous to this stage the mass is much too viscous. Now, if from time to time during saturation, a sample of the raw juice be examined in a test glass, the moment when it is precipitable will distinctly be observable. As soon as the required alkalinity begins to be reached, the precipitate sinks rapidly. It is evident that only after some routine will a certain amount of aptitude be acquired. The best and safest way, therefore, is to combine the second and third methods in such a manner that as soon as precipitation becomes posssible resort is had to the exclusive use of Dupont paper.

When the correct alkalinity of the crude juice has been attained, its temperature must also be correct. The sources of heat met with in carbonatation are the following :—

(1) *Chemical Heat*:—The union of lime with carbonic acid liberates a slight amount of heat, which, however, is of little importance, amounting only to a few degrees.

(2) *The Temperature of the Saturation Gas*:—As the carbonic acid is obtained from a lime-kiln, which burns at very high temperature, it will, notwithstanding the most careful cooling, invariably maintain a temperature of 45 to 50° C. (104 to 122° F.) On account of the great volume of gas forced through the liquid, it is unavoidable that this should be more or less heated by it. A rise of about 5° C. must be expected.

(3) *Combating Froth by Steam*:—In factories where the height of the saturation tanks necessitates the beating down of the froth by steam, this causes a considerable rise in temperature, which, as we have already seen, is not entirely without risk as regards local superheating of the alkaline liquid containing "glucose," giving rise to dark coloration. The rise in temperature will, of course, depend upon the length of time steam is used for this purpose, but an average rise of 10 to 15° C. may safely be looked for.

(4) *Coils*:—Heating by means of coils is in fact the only method available. It is, moreover, harmless, because it can never cause overheating, the violently agitated juice not allowing time for this. Carbonatation installations should, therefore, be designed in such a manner that heating is effected only in this way.

The moment for the admission of steam into the coils should not be chosen too soon. Fixed rules in this connection do not exist; but, empirically, it has been found that in double carbonatation presumably the right moment is on violent frothing.

We have to bear in mind in this connection that a high temperature will cause the "glucose" to decompose in the presence of lime, and that there is a risk of the occurrence of dark coloured compounds, or in any case of an unnecessary increase of the ash content; but on the other hand, it should by no means be overlooked that we are unable entirely to dispense with these increased temperatures in order to obtain a thoroughly energetic action of the lime upon the other impurities of the juice.

It will, therefore, be necessary to take a mean course, as has already been pointed out. If carbonatation were carried out entirely without heating, it would be seen that the resulting clarified juice was generally darker in colour than that obtained at an increased temperature; in other words, the impurities would have been insufficiently removed from the juice. Which of these impurities are the principal ones requiring heat in order to be more easily precipitated, has not yet quite been established; but we may at any rate mention the vegetable colouring agents, both pigments and chromogens.[1]

The most advantageous temperature to which the crude juice should be raised is a minimum of 45° C. and a maximum of 55° C. (113° F. and 131° F.). It is advisable to keep the juice as much as possible between these two extremes, *i.e.*, at 50° C. (122° F.). At this temperature there is little risk of dark coloration by the decomposition products of "glucose," as principally lactic acid, the salts of which are also colourless, is formed. If the temperature were eventually kept too high, the readily decomposable acids mentioned in the Introduction, glucinic and saccharinic

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[1] *Archief*, 1911, 805.

acids, would be produced, as is the case in defecation, and their lime salts would occasion much trouble.

Apart from the fact that they are coloured, they give rise on concentration to the so-called "froth fermentation" which is not real fermentation but is similar in its characteristics. The carbonic acid formed by the decomposition of the acids may cause violent frothing of the molasses, massecuites, etc., which would ultimately flow over the pans.

As soon as the contents of a saturation tank have been sufficiently carbonatated, having therefore acquired the correct temperature and alkalinity, they are run off and conducted under pressure by means of a so-called "scum pump" to the filter-presses, where the crude juice is separated into press scum and first clarified juice.

The amount of lime for tempering the raw juice is without exception regulated during carbonatation by the running of the filter-presses. If this is not normal, *i.e.*, if the jets of juice issuing from the taps are not ample, or if a filter-press just started ceases running, a deficiency of lime is thereby indicated. If in such circumstances the chambers of the filtering apparatus are opened, it will be seen that no hard dry filter-cake has been obtained, which is essential, but on the contrary only a thin, moist, slimy layer of scum, which is with difficulty removed from the cloth.

As in consequence of a defective flow of the presses a complete cessation of the process may soon ensue, no time must in such a case be lost to increase the quantity of lime. Moreover, the press piping should be provided with a return valve to enable the insufficiently limed juice already run off to be pumped back, in order to supply it with the requisite amount of lime, and again neutralize it by means of carbonic acid to an alkalinity of 0·04 per cent.

The point in question is, of course, that the great excess of lime used in carbonatation is quite superfluous for the proper clarification of the juice. For this purpose good results may be obtained by using less milk-of-lime. The principal reason for employing this excess of lime is to render the crude juice capable of being readily filtered. The more calcium carbonate is contained in it the more granular will be the precipitate and the more readily will the juice settle or be filtered.

If in the course of the carbonatation process it is observed by the forementioned precipitation tests, in test glasses, that the precipitate sinks less readily, this should be taken as a warning to add more lime without loss of time.

Proper neatness in connection with the filter-presses is of great importance for both methods of carbonatation. In fitting the presses with the filter cloths, which are generally used double, special attention must be paid that they are stretched as carefully as possible so as to avoid any folds, as on subsequently starting the presses small apertures may then occur in them, through which, during filtering under pressure, the juice would squirt out with great force.

On starting the presses the joint bolt should first be examined to see that it is properly tightened, after which the whole should be thoroughly steamed. The latter process is necessary for closing the pores of the cloths, as otherwise the first juice to pass through would be turbid for a considerable time. The steaming is also necessary for killing and rendering innocuous any colonies of bacteria which may be present, and only too readily form growths on the cloth. Not until the steam can be seen to escape from the nozzles, in the form of vapour, should the steaming be stopped and the crude juice be admitted.

Although the mechanical arrangement of the presses will not be dealt with in this work, it should nevertheless be expressly urged that careful attention must be paid to see that the small pipes conducting the raw juice from the chief supply pipes to the chambers are always kept quite clean and are continually pierced. For if this should be omitted for a time, these pipes might easily become obstructed by the indurated impurities retained. The consequence would be that the corresponding chambers on subsequent filtering remain empty, while the adjoining chambers are under full pressure. Caving in of the frames, and repeated breakdowns of the clear juice chambers, would result from this.

Of the greatest importance in carbonatation in general is the *washing or "sweetening-off" of the press-cakes.* These still contain a large quantity of sugar, *i.e.*, from 7 to 8 per cent., naturally in the form of juice, which is left behind in the filtration scum. In order entirely or partly to prevent this loss of sugar, two methods may be adopted, *viz.*, washing *inside* or washing *outside* the press. The former method is to be preferred; and in properly conducted factories the percentage of sugar in filter scums may by this means be reduced to 1 per cent. or less without the use of much water.

(1) *Washing Inside the Press.*—This should be effected by means of water of not too high a temperature, so as not to dissolve too much of the already precipitated scum, and also in order not to introduce coloured decomposition products of "glucose" into the clear juice. This water is allowed to pass through the filter-cakes for such a period that the issuing sweet-water possesses a density equal to 1° or 2° Brix, according to how far it is desired to continue the washing process. This determination of Brix is done by means of a sweet-water hydrometer, specially constructed for that

purpose, the temperature at the same time being taken into account.

The most convenient method of "sweetening off" is the so-called "fractional" process, in which the last third of the washing water, which consequently is most diluted, is again utilized for the next press. The first two-thirds are at once mixed with the clarified juice. This can be done with safety, since it has been demonstrated experimentally that the purity of this washing water is but a few degrees less than that of the clarified juice.

The volume of washing water can be regulated at will, and measured in the juice gutters.

(2) *Washing Outside the Press.*—In this method, which, as has been stated, is more complicated, less clean, and less economical as regards the use of water, the press cakes are first removed from the press, and stirred in a malaxeur or revolving drum with moderately warm water, say at 50° C. (122° F.), till a thin paste is formed, which is then forced by means of a pump into a second set of filter-presses.

In this case the filtrate is generally utilized for maceration, in contradistinction to the former method, in which it is mixed directly with the clarified juice. This is, in fact, the only reason why this method is employed, for although more water is required for reaching the same degree of exhaustion, it does not entail increased evaporation of water in the factory.

This method of washing requires the utmost cleanliness, as otherwise acidification speedily sets in.

The first carbonatation and filtration are succeeded by the second process of a similar nature. Two methods can here again be employed in this process, *i.e.*, *intermittent* and *continuous* carbonatation.

(1) By the *intermittent* method the first clarified juice is pumped into separate tanks to a certain level, as on first saturation; and these volumes of juice neutralized, each independently, by means of carbonic acid. This method thus requires a larger number of saturation tanks.

(2) By the *continuous* method, on the other hand, the juice passes in a continuous stream through a single tank. It is pumped into this from below, and runs out above through an overflow pipe of its own accord. During its passage it is neutralized by a stream of carbonic acid, which has to be carefully regulated accordingly.

There is not the slightest doubt that the latter method is by far the better of the two.

It is cheaper, since it requires only one tank, which, on account of the small amount of precipitate, requires no cleaning; it is also quicker and simpler. No satisfactory reason can be assigned for not adopting the continuous process.

It should be distinctly urged here that, generally speaking, in the manufacture of white sugar in cane-producing countries the time occupied in clarifying juice, by any process, should be *as short as possible*, as the decomposition of "glucose," and, consequently, the formation of salts of lime, stands in direct ratio to it. Although with a low temperature no great amount of dark colouring will result, the unnecessary increase of the ash content should be avoided.

In order to make this period as short as possible, the number of saturators used in carbonatation should be as few as possible, while care should be taken—by a sufficient capacity of the lime-kiln on the one hand, and of the presses and evaporators on the other—that the juice does not remain unnecessarily in the saturation tanks, the number of these being reduced as much as possible.

Theoretically, the first carbonatation process does not require more than three saturators, *i.e.*, one filling, one saturating, and one emptying. As in practice, however, it is necessary from time to time to clean one, and repairs and stoppages must also be taken into account, four tanks should be provided in every factory, this being the maximum. If all four tanks are in use it will be possible to saturate two at the same time, which, as has previously been stated, is essential for an economical use of carbonic acid. For the second carbonatation, as already indicated, only one saturator is required, making a total of five tanks for any factory of any size.

The capacity of each tank should depend on the quantity of cane to be crushed, and may vary from 3,000 to 10,000 litres (765 to 2550 gallons).

The reaction of the second carbonatated unfiltered juice on not too sensitive phenolphthalein paper should be faint pink. Although for chemical reasons phenolphthalein does not invariably give accurate results in the case of sugar juices, it possesses, nevertheless, the great advantage that the change of colour from red to pink or from pink to white may easily be observed even by a native workman, for which reason it is the only really useful indicator in a sugar factory.

If, however, neutralization were attempted *with the solution*, the process would go too far and over-carbonatation would occur, hence the less sensitive paper reaction, just referred to, is recommended.[1] By over-carbonatation is meant the process in which by excess of carbonic acid the already precipitated

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[1] *Archief*, 1909, 721; and 1911, 1415.

calcium carbonate is redissolved with the formation of soluble calcium bicarbonate :

$$\underset{\substack{\text{Calcium carbonate}\\ \text{(insoluble)}}}{CaCO_3} + \underset{\text{carbon dioxide}}{CO_2} + \underset{\text{water}}{H_2O} = \underset{\substack{\text{calcium bicarbonate.}\\ \text{(soluble)}}}{Ca(HCO_3)_2}$$

Now this soluble calcium bicarbonate on being boiled has the objectionable property of splitting up into carbonic acid, water, and insoluble carbonate, a process which consequently occurs in the evaporating apparatus. This insoluble calcium carbonate deposits around the heating pipes of the evaporators, forming an encrustation which considerably reduces the transmission of heat from steam to juice. Another great disadvantage of over-carbonatation arises from the fact that not only will the originally precipitated calcium carbonate be partly redissolved, but organic salts and colouring matter precipitated during carbonatation will also again be taken into solution. Calcium carbonate during precipitation has the capacity of mechanically carrying down and depositing other salts of lime and potash in solution, and these also are redissolved on over-saturation.

For these reasons, during carbonatation it is advisable not to carry the process of saturation too far, but rather to remove any trace af alkalinity *after filtration.*[1]

It will, therefore, be prudent on second carbonatation to keep the test paper saturated with phenolphthalein and dried in the open air, at a somewhat slight pink reaction, because, as will be explained further on, we are concerned not only with lime still present in the free state, but probably also with potassium hydroxide (potash). Attention has already been drawn in these pages to this circumstance as an important factor in the manufacture of white sugar.[2]

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[1] *Archief,* 1912, 668.

[2] *Archief,* 1909, 721 ; 1910, 409.

We have already stated that the potash alkalinity should only be removed when all the free lime has been converted into carbonate. Now the union of potassium hydroxide (potash) into carbonate proceeds exactly in the same manner as that of the lime salts. The resulting salts differ, however, in that calcium carbonate is insoluble and has a neutral reaction to phenolphthalein ; while potassium carbonate is soluble, and has a distinctly alkaline reaction on that indicator, colouring it red. In practice it will, therefore, be found that the paper turns more or less pink, even though all the free alkali, both of lime and potash, has already been combined.

If the process be still continued, the said pink colour will disappear because, like calcium carbonate, where there is excess of carbonic acid potassium carbonate will form an acid salt, potassium bicarbonate, which has no alkaline reaction :

$$2\,KOH + CO_2 = K_2CO_3 + H_2O.$$

Potassium hydroxide + carbonic acid = potassium carbonate + water.

$$K_2CO_3 + CO_2 + H_2O = 2\,KHCO_3.$$

Potassium carbonate + carbonic acid + water = potassium bicarbonate.

As, however, the formation of calcium bicarbonate and potassium bicarbonate will take place concurrently during second carbonatation, one can be certain that when the paper no longer turns pink, the previously precipitated calcium carbonate has been re-dissolved and that encrustation will be greater than is necessary.[1]

If on the other hand the liquid is kept too red, there is a danger that the free lime remains unneutralized in the juice, so that the matter requires much attention and experience. In order to meet this, some factories are provided with heaters in which the second unfiltered juice is boiled before

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[1] A similar result will be obtained when the lime used contains too much magnesia.

being submitted to the second filtration. This procedure is based upon the principle just elucidated, that acid salts are split up at boiling heat; but in this connection it should be noted that this decomposition requires a certain amount of time, so that in order completely to decompose them these salts would have to be boiled a long time in the heaters, this, of course, being impossible. Moreover, as we shall see later on, for other reasons this method is not to be recommended.

After this digression, which is indispensable for obtaining an accurate idea of the process, we return to the second carbonatation.

Without further heating, the juice will of course have acquired a temperature lower than that of the first carbonatation, therefore below 50° C. (122° F.), and it can readily be filtered at this low temperature. It may happen, however, that at this temperature the filtered juice is somewhat turbid, or, which is worse, has soured. In that case the juice should be heated by means of the coil always present in the second saturation tank, provided that the temperature does not exceed 70° C. (158° F.)

Souring of the filtered juice may occur, because at 50° C. (122° F.) all fermentation organisms in the juice will not have been killed, namely, certain lactic acid bacteria, which particularly at this temperature sometimes exhibit a distinctly noticeable vitality.

In the juice itself, which is quickly brought to evaporation, and soon heated to 100° C. (212° F.) in the first heating vessel, the risk of fermentation is slight; but in the filters used for the second carbonatated juice, these organisms may be deposited on the filter-cloths and form colonies, causing fermentation in a remarkably short time if not prevented by raising the temperature to about 70° C. (158° F.).

The bacteria form lactic acid from the "glucose," and presumably also from the sucrose, causing the juice gradually to acidify, as can readily be detected by the colour of the second clarified juice which is completely bleached. Loss of sugar must consequently be expected.[1] The concentrated juice subsequently obtained, moreover, remains dull in that case.

Filtration of the second carbonatation juice is effected in quite different ways in different factories. In some it is passed through low-pressure filters with a drop of a few feet, or it is pumped into filter-presses by means of a scum pump. There is no doubt that the latter method involves waste of power and steam. Sometimes the two methods are combined, and the cloudy juice is passed through the filter-presses by its own pressure, which of course is preferable.

In using low-pressure filters, such as those of Danek, Dehne, etc., the filters as a rule cannot be steamed, whereas in filter-presses this can easily be managed, and should in fact never be neglected in order to kill any bacterial colonies which may be present. In such filtration the greatest attention should be paid to the production of the clearest possible juice, to proper washing, and to the cleanliest possible treatment.

b. Single Carbonatation.

Formerly, none but the single carbonatation process was practised. Manufacturers started from the assumption—not altogether inaccurate—that most cane juices in general were so easily purified, *i.e.*, that they contained so few impurities (except the troublesome "glucose") that fractional carbonatation was altogether superfluous.

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[1] *Archief*, 1911, 1418.

When subsequently instead of muscovado and grades below 20 D.S., superior sugars came to be demanded in the market, a gradual change in this respect took place and manufacturers, without distinction, by degrees adopted the double process, especially since it was supposed that by this process certain specified decomposition products of "glucose" could be removed from the juice. The products in question were the lime salts of some organic acids—such as glucinic acid—which could form basic salts and which, being insoluble, would be retained by the filter-cloths on first filtration.

It was proved only quite lately[1] that this contention was based on an error since it could be shown that the percentage of calcium organic acid salts was no higher in single carbonatation than in double, quite the opposite. It is indeed well known that (contrary to what obtains in defecation) at the low temperature applied in carbonatation at the present day most probably no glucinic acid is formed from the "glucose," but that almost exclusively calcium lactate is produced, which has not a basic salt.

As has already been stated when dealing with double carbonatation, the only advantage of that method over the single process is that it is safer; in other words, it requires less attention, since carbonatation and filtration both take place in two stages. What through negligence or carelessness might be spoiled during first carbonatation or first filtration can be remedied during the second respective processes. In this connection it should, however, be borne in mind that where during first carbonatation and filtration mistakes are possible, this certainly also holds good with the second processes.

As regards the technical installation and the carrying out of the process, it is evident that these are cheaper both

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[1] *Archief*, 1911, 808.

as regards cost of material and erection, as well as in respect, of the ultimate cost of labour. For a saving is effected in filters and cloths as well as in wages for the second carbonatation, besides special supervision, so that a little more care and attention during single carbonatation and filtration will be quite justified, while the mechanical loss of sugar, which is an unavoidable result of second filtration, is thereby obviated.

Single carbonatation, moreover, has a great advantage from a purely chemical point of view, namely, it is much quicker. It is more rapid, because saturation is completed in one stage, so that the "glucose" does not remain in contact with the lime as long as in the double process. For in the latter, carbonatation is intermittent, the alkaline juice being drawn off into large tanks and stirred, after which it is somewhat slowly filtered, while finally the still alkaline juice has to be pumped up once more for the second carbonatation. If, moreover, this second saturation is not effected at once the juice remains standing a needlessly long time.

In exact proportion to the longer duration of this process, the lime will form organic acids, and will thus needlessly increase the ash content. This is one of the reasons why the single method will invariably give a lower percentage of lime in the clarified juice than the double process.

One disadvantage of single carbonatation which, however, in its turn has partly favourable results, is the great influence exercised on the regular course of the process by the preparation of the milk-of-lime, to which a brief allusion has already been made. For if the milk-of-lime has been more or less imperfectly slaked and sifted—the latter being, of course, the principal thing—small lumps of lime will find their way into the cane juice; these cannot form saccharate with the sugar present, since they are very speedily surrounded by an insoluble layer, whereby they escape all combination. This insoluble layer may be formed by the carbonic acid concurrently pumped into the juice, or it may arise from the formation of insoluble lime salts with the organic acids in the juice.

Many of these small lumps, however, gradually disintegrate through the violent agitation and the rise in temperature occasioned during carbonatation, thus combining with the sugar. As soon, however, as the heating steam and the saturation gas are turned off, this so-called "after-slaking" ceases, taking nevertheless invariably a few minutes to do so.

If the reaction of the quiescent muddy juice be tested after a few minutes with phenolphthalein it will be seen that the paper, originally quite colourless, has become slightly red owing to absorption of lime.

The process just described, indeed, greatly influences the practical working of the method, for contrary to what obtains in double carbonatation—where the reaction on phenolphthalein paper has to be kept faintly pink—complete neutralization must be attained, so that a slight amount of over-carbonatation should purposely be brought about in order that the lime subsequently liberated shall be combined.

If this is insufficient, and a distinctly pink colour is obtained after standing some minutes, it will be necessary to turn on the supply of carbonic acid for a few moments, and thus get rid of these last traces of alkalinity. In practice, the carbonatation tank filled with crude juice is allowed to stand for the space of exactly three minutes (measured by a sand-glass) with closed carbonic acid and steam valves, before finally saturating.

Were the unfiltered juice now simply allowed to settle nothing further would happen; but the pressure exerted by the scum pump during subsequent filtration must be taken into account. Again, some of the particles of lime will be unable to withstand the fairly strong pressure in the presses, and at the last moment be subject to "after-slaking," *i.e.*, they will disintegrate and yield lime.

We will indeed observe that the filtered clarified juice invariably shows a faint pink reaction to phenolphthalein, however carefully it may previously have been neutralized. Yet the percentage of lime in the clear juice has not increased. In fact, as a rule it decreases. The reason is probably to be attributed to the calcium bicarbonate (bicarbonate of lime) occurring in most carbonatation juice.

It has already been explained how the presence of free potassium hydroxide (potash) during saturation to a neutral reaction to phenolphthalein will nearly always give rise to overcarbonatation, and consequently to re-solution of already precipitated calcium carbonate, forming calcium bicarbonate. Even without the presence of free potassium hydroxide (potash), oversaturation may easily occur as the result of the unreliability of phenolphthalein in the presence of carbonic acid. Now it is just this calcium bicarbonate, present in nearly every juice, which readily unites with the lime liberated on "after-slaking" in the presses, with the formation of insoluble calcium carbonate:

$$Ca(HCO_3)_2 + CaO = 2\,CaCO_3 + H_2O.$$

Calcium bicarbonate + calcium hydroxide = calcium carbonate + water.

This is, therefore a second reason why in single carbonatation the percentage of soluble lime salts must be less than in the double process. But it is clear that in most cases more lime will be liberated in the presses than is required to precipitate this small quantity of calcium bicarbonate.

This surplus will in an entirely analogous manner attack any potassium bicarbonate present, and decompose it also :

$$2\,KHCO_3 + CaO = K_2CO_3 + CaCO_3 + H_2O.$$

Potassium bicarbonate + calcium hydroxide = potassium carbonate + calcium carbonate + water.

By the presence of bicarbonate, therefore, all lime finding its way into the juice by the slaking of particles of lime must invariably be precipitated as insoluble carbonate, so that it cannot assist in raising the percentage of lime itself.

It will nevertheless be advisable to continue the control of the slightly alkaline, filtered, clarified juice and from time to time to titrate the same, because, as is shown by these equations, from its alkalinity is given a direct clue as to the more or less successful preparation of the milk-of-lime, *i.e.*, its straining through sieves. If the juice is too alkaline, say more than 60 mgrms. of calcium hydroxide (CaO) per litre, the sieves should be examined for repairs.

The logical inference to be drawn from the preceding statements is that in the single process over-carbonatation is nearly always rectified in the filter-presses, so that the evaporators will keep clean much longer than in double carbonatation, which in fact is clearly proved to be the case in practice. This avoidance of over-carbonatation, which in practice is effected with difficulty by neutralization, is one of the greatest advantages of the single process. For in the case of such over-saturation not only calcium carbonate will be dissolved; but previously deposited calcium and potassium organic acid salts mechanically carried down with the carbonatation precipitate are re-dissolved likewise. Colouring matter originally removed as insoluble is also taken up in solution.

It stands to reason that any over-saturation which may occur will, therefore, be much more detrimental in the single than in the double process. For in the latter, the major portion of the impurities are already removed during the first alkaline filtration, whereas in single carbonatation all the scum is still present in the juice.

A weakly alkaline final reaction of the filtered clarified juice is thus absolutely essential in this case. This red reaction to phenolphthalein must be removed by a couple of carbonic acid bubbles, since it is by no means immaterial at what reaction the juice starts evaporating. With a few carbonic acid bubbles, the juice returns to exactly the same condition as in double carbonatation.[1]

Since in single carbonatation only one filtration takes place, the latter should result in juice as clear as crystal without the slightest dimness, the same in fact as is required of the second filtration in double carbonatation. For it should be distinctly understood that turbidity in carbonatation, whether single or double, invariably results from the presence of calcium carbonate which is able, even in suspension, owing to its alkaline property—not a negligible quantity—to decompose "glucose" and, therefore, to colour the juice, as has been proved by experiment.[2] Every reason for the proper supervision and handling of the presses is, therefore, of double weight in this case, namely, extremely careful arrangement of the cloths and adequate steaming. The latter is essential not only for closing the pores in the cloth, but also for the destruction of any possible colonies of bacteria, the juice in course of filtration having an almost neutral reaction, and being an easy prey to lactic fermentation if the presses are not sterilized periodically.

It follows, as a matter of course, that in this case a souring might be especially detrimental, since the quantity of the precipitate is so much greater and more heterogeneous.

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[1] *Archief*, 1911, 670.

[2] *Archief*, 1912, 732.

Supplement.

With regard to the working of the lime-kiln—an indispensable adjunct in carbonatation—we cannot enter into details here, because (1) this is of a purely technical nature, and does not therefore find its proper place in a chemical manual, like the present work; but also because (2) a sufficient number of treatises dealing with the subject exist. Whereas in the domain of chemical manufacture considerable evolution has occurred during the last few years, making a detailed discussion of white sugar manufacture desirable, little or no change has taken place on the technical side of the subject.

On the other hand, only the completely normal processes having thus far been outlined, it will not be out of place to give some directions in case of trouble arising in the process of carbonatation. In the preceding pages, it has already been pointed out that one of the first conditions for obtaining a good quality of white sugar is to make the process of liming and of saturation as short as possible. Since the colouring of the juice, as well the ash content, are so intimately connected with the duration of the process, this requires no further comment. The disturbing factors in question may be of various natures :—

1. Deficiency of Carbonic Acid.

The faulty action of the lime-kilns is one of the most frequent factors in preventing rapid and proper saturation. The causes may be various. Those most frequently occurring in practice are for instance :—

(*a*) An undesirable displacement of the dissociation zone in the kiln. When, for instance, the draught is irregular, or the pumps do not run regularly, it may happen that through insufficient attention the space for the decomposition of the limestone is either too high up or too low down.

If the displacement is downwards, this will generally become apparent from the fact of red-hot limestone issuing from the kiln. Upward displacement is more dangerous, because this may sometimes give rise to caking of the limestone involving the risk of "hanging" in the locally constricted space. In either case, deficiency of carbonic acid (CO_2) will generally result from the increase of the carbon monoxide (CO) content.

To delay or accelerate drawing the lime is the respective remedy in either case.

(*b*) A very frequent cause of deficiency of carbonic acid is the moisture of the coke. This is especially the case where coke from gas factories is used, because it is the custom in these factories to quench the red-hot contents of the retorts by means of water instead of allowing it to cool of its own accord.

Acting thus is more profitable because it prevents loss by further combustion, and, moreover, causes increased weight. This matter should receive special attention. In factories drawing their coke supply from elsewhere, this will of course occur less frequently, although the risk of getting it soaked by rain remains.

During combustion the water vapour formed will be reduced by a portion of the carbon to hydrogen and to carbon monoxide, again entailing a loss of carbonic acid (carbon dioxide). This condition may be recognized on opening the sight-holes about the level of the dissociation space or above it. If carbon monoxide and hydrogen be present these inflammable gases will betray their presence by jets of a bluish flame issuing from the sight-holes in unison with the strokes of the pump. This may best be remedied by keeping all the sight-holes in the kiln open for a time, whereby the air supply, and consequently the percentage of carbonic acid (carbon dioxide) is generally increased at once:

$$C + H_2O = CO + H_2$$

Carbon + water = carbon monoxide + hydrogen.

$$2\,CO + O_2 = 2\,CO_2$$

Carbon monoxide + oxygen = carbon dioxide.

It may be generally stated that every irregular action of the lime-kiln immediately results in a deficiency of carbonic acid. This may be met *temporarily* by increasing the number of strokes of the carbonic acid pump, but the real nature of the mischief should be traced as quickly as possible, so as to reduce the pump to its normal course as soon as possible.

Since, moreover, it will generally be some time before a lime-kiln, whose action has been interrupted, can be brought back to its normal function, it will be advisable meanwhile to increase the proportion of coke to limestone for the time being. Whereas the proportion in weight of coke to limestone may as a rule be reckoned as 1 : 10, in these circumstances it may be reduced to 1 : 9, or in extreme cases to 1 : 8, which must not, however, be maintained too long.

Since the absorption of the carbonic acid is *proportionately* higher from a *mixture rich in that gas*, it is evident that a deficiency of carbonic acid will cause retardation of saturation for two reasons.

2. Distribution of the Carbonic Acid.

A second cause of retarded carbonatation may be defective distribution of this gas throughout the juice. Where, for instance, so-called "spider webs" are employed in the process it may happen that these become defective or obstructed, so that the saturation gas is either insufficiently distributed, or else is forced only on one side of the juice. In either case retardation results. The tanks of the first saturation should therefore always be kept as clean as possible, and be examined continually. For this purpose a spare tank should always be kept.

3. Inaccurate Liming.

A third cause leading to too protracted saturation is insufficient attention to the milk-of-lime. If, for instance, the milk should weigh more than 20° Bé., without this fact being suspected, a very undesirable stagnation may result. It is also possible that on proceeding to handle cane from another field, or of another variety, one is suddenly confronted by the fact that the liming is greater than is necessary or desirable. The different kinds of cane must therefore be well mixed in crushing in order to obtain an even sample of juice.

4. Abnormal Juice.

This condition, which may occur practically every year in any factory, is extremely troublesome, and arises from the crushing of cane which from various causes may be too unripe. This is especially of frequent and continued occurrence in the case of fallen crops. The albuminous or gummy substances may then occasion too great a viscosity of the juice, whereby the absorption of carbonic acid may considerably diminish, and saturation last extra long, in fact, twice as long as usual, being in consequence very uneconomical. Here, again, the only remedy is a thorough mixing if crushing the cane of different fields; while the manufacturing conditions should be taken into account when harvesting troublesome crops. Earlier heating of the juice might be of avail in this case, but would yield too dark a juice.

Another source of trouble in the clarification of the juice may arise in the *filter-presses.* The least stagnation in the presses may recoil upon the immediately preceding process, *i.e.*, carbonatation. In other words, the juice ready for the next process will have to be kept in the saturation tanks, frequently even after having been already limed. Especially in double carbonatation is this a very serious nuisance.

Juice rich in glucose may thus occasionally stand for a considerable time exposed to a strong alkaline reaction, during which re-solution of lime and the formation of decomposition products of glucose continues, especially with a rise in temperature. In case of stagnation in the presses, the most prudent course invariably to be adopted is to keep the juice cold and unlimed. If any of the juice has inadvertently already been limed, carbonatation will have to proceed, but it will be best not to heat this until it can be filtered.

This stoppage of the presses, which is of such frequent occurrence in tropical factories, requires special attention. Every stage at which juice may have to be kept standing, and especially that of clarification, must be so nicely gauged that, in case of stagnation of the presses, the mills may be stopped as soon as possible. This is in fact the only really proper solution of the problem. The greater the number of saturators or storage tanks, etc., in a factory, the longer the stopping of the mills—which is regarded, unjustly so, as the last remedy—will be delayed; and the longer in such case the still alkaline, warm, glucose-containing juice remains standing, the greater will be the mischief. The same applies to the other stations: the greater the number of saturation tanks, the greater the risk of souring if the juice has already been neutralized, or of increasing the ash content if the juice is allowed to remain alkaline. Large sized storage tanks for thin or concentrated juice also occasion risk of needless acidification, and all these circumstances will unavoidably and unfavourably affect the quality of the sugar, both as to colour and lustre.

In this connection it may be particularly mentioned that these unfavourable influences will as a rule not be traceable in the first product, but on the other hand they will be all the more noticeable in the after-products. In factories where all the sugar is obtained in one stage, these apparently too subtle questions become of the greatest importance.

In view of all this, it has already been premised in these pages that the number of saturation tanks in each factory should not exceed five, even for single carbonatation, where altogether about an equal volume of lime per unit of juice has to be neutralized. The crushing capacity of a factory, within practical limits of course, should affect the capacity of the saturators, but not their number.

Stagnation in the filter-presses may be caused by :—

1. Deficiency of Lime.

It has already been stated above how deficient liming may be detected. It happens sometimes that through insufficient attention the milk-of-lime may be too thin, weighing less than 20° Bé. The presses will not be able to elaborate the viscous mass quickly enough, giving rise to stagnation.

2. Drawing off "Underdone" Juice.

Juice is said to be "underdone" when it has not acquired the requisite alkalinity of 400 mgrms. of calcium oxide (CaO) per litre. If by mistake the contents of a tank are drawn off before this limit has been attained, or if one of the saturation tanks happens to leak, the so-called "calcium hydrosucrocarbonate," which is extremely viscous, reaches the cloths of the filter-presses. The issuing juice immediately betrays its presence in the gutters by its violent frothing, and soon the cloths prevent all further egress.

An accident of this nature generally entails a complete cleaning of the cloths, and may occasion a delay of hours. In either case, the juice already drawn off has to be pumped back to the saturators in order to be carbonatated anew.

3. Quality of the Cloths.

In every factory the greatest care ought to be exercised in the selection of cloths. If once a suitable quality has been obtained, it is advisable to keep to it as much as possible. If for various reasons, *e.g.*, of a financial nature, a new kind has to be tested, one should be very careful, and at first procure only a small quantity, say sufficient for one press, and compare this with another press which at the same time has been provided with a new set of the original cloth.

It frequently occurs that some filter-cloths which answer the purpose very well in European carbonatation factories occasion the greatest disappointment in colonial factories, because after a few filterings no further juice passes through them. Even after soaking in hydrochloric acid no change in this respect occurs. It appears, therefore, that the peculiar fibre of these cloths is saturated with silicic acid to such an extent that all porosity is lost.

4. Soft Cakes.

Apart from a deficient supply of lime, or a too alkaline reaction, these may be produced as a consequence of the low pressure of the scum-pump. The pressure piping of this pump should, therefore, always be furnished with a manometer, which will immediately indicate when the pump leaks, or for any other reason does not act properly. Generally a pressure of 30 to 45 lbs. per sq. in. is the most desirable.

II.

THE ACID THIN-JUICE PROCESS.

In discussing this modification of the European process of carbonic acid saturation, which makes it more suitable for tropical sugar juice so rich in glucose, the next chapter of this Handbook, namely, that dealing with the sulphitation of the crude juice, or sulphurous acid saturation, must of necessity be forestalled to some extent.

The term *sulphurous acid saturation* already indicates that this saturation method differs from the one hitherto dealt with, in so far that the gaseous carbonic acid is replaced by the gaseous sulphurous acid. Now when cane juice, limed in excess, is neutralized by means of this sulphurous acid which, as we shall see presently, should proceed until exactly the same reaction sets in, *i.e.*, about neutrality to the same indicator, phenolphthalein, it is evident that the composition of the resulting clear juice must differ from that obtained by carbonic acid saturation.

When dealing with the latter method it was carefully explained in what manner liming will liberate a portion of the potash (as potassium hydroxide), so that the filtered clarified juice contains potassium carbonate. In a completely analogous manner the clarified juice obtained by sulphurous acid saturation will contain potassium sulphite. The latter salt has an alkaline reaction to litmus (not to phenolphthalein), so that in this case also we can here speak of a potash alkalinity of the clarified juice after neutralization. In an analogous manner the carbonatation juice will invariably contain a small quantity of calcium carbonate ($CaCO_3$), and the sulphitation juice some calcium sulphite ($CaSO_3$), as both salts are in a slight measure soluble, especially in liquids containing sugar.

Now it is found in practice that the clarified juice, that produced by carbonic acid as well as by sulphurous acid saturation, increases in colour during concentration, this increase having no relation to the concentration, *i.e.*, if the concentrated juice resulting from either saturation method is reduced by means of water to the original density of the thin-juice it will be seen that this diluted juice is considerably darker than the original thin-juice before concentration.

While the two methods of clarifying juice are absolutely parallel up to this point, a difference, therefore, now manifests itself by the fact that in carbonatation the increase of coloration is very considerably greater than in sulphitation. It is evident that the cause of this difference is to be looked for in the respective behaviour of sulphites and carbonates, because the presence of these salts is in fact the only point of material difference between the two kinds of juice.

As we have seen, moreover, that all dark coloration in the clarified juice can be traced principally to the presence of glucose in the cane juice, the logical course to take is to observe the action of the salts in question: soluble carbonates and sulphites, with reference to solutions of glucose.[1]

Now on boiling a dilute glucose solution separately with potassium sulphite and potassium carbonate (or potassium bicarbonate, which amounts to the same thing), it will very soon become manifest that this solution, at ordinary reaction, becomes dark coloured by the carbonate, but in no wise by the sulphite. This unmistakably points in a definite direction.

Before, however, going into the matter, it should be pointed out that besides the soluble potassium carbonates and sulphites, cane juice also contains soluble lime salts of organic acids, so that it must be expected that insoluble calcium carbonate or sulphite will be produced side by side with organic potassium salts:—

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[1] *Archief*, 1910, 409.

Calcium (lime) organic acid salt + potassium carbonate = potassium organic acid salt + calcium carbonate.

Calcium (lime) organic acid salt + potassium sulphite = potassium organic acid salt + calcium sulphite.

It is, therefore, manifest that for this reason only slight traces of potassium carbonate or potassium sulphite can be present in the sugar juice except in those cases where the amount of potash liberated by the lime is equivalently greater than the lime dissolved. These cases are rare in tropical juices.

Besides the soluble sulphites and carbonates of potassium, those of lime (which occur in an equally slight degree) must also influence the accumulation of coloration during evaporation, but it stands to reason that the much more alkaline alkali carbonates will produce more colour than calcium carbonate.[1]

The influences thus exercised upon the colour of the juice by the carbonates and sulphites alluded to are in exactly opposite directions. The carbonate compounds, on account of their strong alkalinity, have a distinctly darkening effect; whereas the sulphurous acid salts on the contrary have a decided bleaching action. Hence the increase of coloration in concentrated carbonatation juice will invariably be greater than in concentrated sulphitation juice.

The other soluble salts present in cane juice also exercise a colouring influence on the glucose, namely, those which exhibit an alkaline reaction, be it ever so weak. Thus the organic acid lime salts will invariably display this tendency, while the more basic potassium salts possess it in a still higher degree. In this connection we speak of the "hydrolysing alkalinity" of these salts. Neutral compounds, such as potassium chloride, etc., or acid compounds, do not produce any coloration.

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[1] *Archief*, 1912, 559 and 652.

When the source of the increased coloration in carbonatation juice was detected, the means of correcting this was readily established, namely, by slightly sulphuring the dilute carbonatation juice, *i.e.*, just sufficiently to remove its alkalinity to litmus, a portion of the troublesome carbonates being converted into sulphite according to the following formula :—

$$\underset{\text{Potassium carbonate}}{K_2CO_3} + \underset{\text{sulphurous acid}}{SO_2} = \underset{\text{potassium sulphite}}{K_2SO_3} + \underset{\text{carbonic acid.}}{CO_2}$$

$$\underset{\text{Calcium carbonate}}{CaCO_3} + \underset{\text{sulphurous acid}}{SO_2} = \underset{\text{calcium sulphite}}{CaSO_3} + \underset{\text{carbonic acid.}}{CO_2}$$

For various reasons,[1] the whole of the carbonates will not be decomposed in this reaction, and it requires a greater acidity to make the conversion complete. Unfortunately, for practical reasons, a neutral reaction to litmus must not be exceeded. This requires some explanation.

The action of sulphurous acid on cane juice is very complex. In addition to the forementioned conversion, some of the calcium organic acid salts will also be decomposed :—

Calcium organic acid salt + sulphurous acid (SO_2) = calcium sulphite ($CaSO_3$) + the organic acid.

Now since this neutral action to litmus means that the liquid is in reality—that is, from the chemical point of view—already acid in consequence of the organic acid liberated, this sulphuring of the thin-juice has been termed the *acid thin-juice* process.

The organic acids liberated, according to the above formula, are in part volatile, such as lactic acid, formic acid, acetic acid, etc., and will, therefore, escape with the effect condensed water during evaporation of the sulphured

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[1] *Archief*, 1909, 43 and 64.

juice. If, however, the juice is rendered *acid* to litmus paper by means of sulphurous acid, some sulphurous acid in addition to the organic acids will escape, which must be prevented at all cost, as otherwise the boilers and the heating tubes of the evaporators will be attacked, thereby causing premature deterioration.

In view also of possible inversion, the sulphuring of the thin-juice must not of course be continued until too acid a reaction sets in, since, especially during subsequent evaporation at a higher temperature, this might cause loss of sugar. Still, the organic acids liberated with an acid reaction have but a slight inverting power, and even sulphurous acid as such will not occasion great risk in dilute solution. It is only upon oxidation to sulphuric acid that the danger of inversion increases.

Thus practice teaches that the risk of inversion is considerably less than that of the corrosion of the apparatus. If it were possible to neutralize the vapours in the course of distillation from the thin-juice, in other words, to deprive them of the sulphurous acid carried off, one could without fear continue the sulphuring as far as a distinct acid reaction to litmus sets in, *but at present this is altogether out of the question.*

It may, however, be observed that the corrosive action of the acid juice itself is considerably less than that of the vapours, so that there is always much greater risk of the brass tubes of the evaporators corroding on the outside than on the inside. This phenomenon is probably due to the fact that vapours mix more readily with the air than does juice, a fact standing in direct relation to the oxidation of sulphurous acid into sulphuric acid.

After these theoretical explanations, we will proceed to discuss the practical operation of the acid thin-juice process.

1. Controlling the Sulphuring of the Thin-Juice.

The sulphuring of thin-juice is best carried out in an ordinary saturation tank, as used for carbonatation, but provided with a wooden chimney. Care is to be taken that, besides the ordinary carbonic acid conduit, a sulphur pipe is added, likewise reaching as far as the bottom of the tank. Moreover, this tank, like that used for the second saturation, should be adapted for continuous use, the juice being pumped in below and run out above in a constant stream.

Just below the highest level a test tap is fixed in the front side of the tank from which the sulphured juice should issue drop by drop, to be caught in a funnel underneath and be conducted to a container.

In applying the acid thin-juice process, therefore, the number of saturation tanks must be increased to six.

The control of sulphuring, in other words, the correct reaction of the sulphured juice, is of the utmost importance for the subsequent processes of manufacture as well as for the preservation of the apparatus and accessories. If litmus paper were employed, which, in view of the directions already stated, would appear to be the easiest and simplest, one would be confronted with practical difficulties, *i.e.*, the unreliability of this paper in an atmosphere more or less saturated with acids (carbonic and sulphurous acids).

Although this might be obviated by using a solution of the indicator, it being preserved in securely-closed bottles, yet great difficulty would be experienced in detecting with certainty the exact change from blue to purple, which, especially with artificial light, would lead to unavoidable mistakes. The best way, therefore, is in this case also to avail one's self of the only indicator of practical utility in sugar factories, *i.e.*, phenolphthalein.

The solution is of course employed, and an ordinary titration is made with N/100 alkali in a Vivien tube.

This standard alkali before being used is mixed in the laboratory with phenolphthalein solution and, in order to obtain indications as regular as possible, this is always done in exactly the same proportions. The resulting red liquid is carried to the factory in securely closed bottles.

Titration is thereupon effected in such a manner that the Vivien tube is filled as far as the zero mark with N/100 potassium hydroxide, and so much of the sulphured juice poured in till the red colour of the liquid is just completely discharged.

The exact number of cubic centimetres (c.c.) of the juice which should be added (read from the Vivien tube itself) is determined a few times each day, using for titration juice exactly neutral to litmus, prepared by daylight.

If the requisite number of cubic centimetres of the juice, just sufficient to decolorize the titrating liquid, is 10·0, and on titrating the juice it is found to be 9·0, this means that the juice is too acid, and that the sulphurous acid supply valve must be slightly closed. If, on the contrary, it is found to be 11·0, the juice is insufficiently acid, and the valve has to be opened somewhat further.

After some practice these directions are fairly easy to follow, and are soon executed with promptness and accuracy, so that this work may well be left to natives.

2. Controlling the Effect Condensed Waters and their Neutralization.

As has already been explained, the effect condensed waters, when applying the acid thin-juice process, invariably exhibit a more or less acid reaction in consequence of the partly volatile organic acids liberated by the sulphurous acid.

If too acid a reaction has been guarded against, in other words if the juice always has remained neutral to litmus, little or no trouble of corrosion will be experienced with regard to the brass tubing of the heating bodies. When steel tubing is in use no sulphuring, however weak, of the thin-juice should take place, because these volatile acids decidedly corrode iron.

Since, moreover, effect condensed waters are invariably utilized for feeding the boilers the risk of corrosion remains, for which reason it is essential that these waters where possible should be carefully neutralized, or if necessary rendered slightly alkaline, so as to obviate all risk.

If, however, the reaction of the juice is kept too acid, a decidedly corrosive effect on the brass tubing would in the long run become manifest; as this tubing is very expensive, the detrimental effect of corrosion is greatest here, more especially because neutralization can with difficulty be effected in the evaporators.

One has therefore continually to guard, by means of titration as referred to above, against the neutrality to litmus being surpassed, in which case—as has been determined experimentally—no sulphurous acid can be evolved. In order to control the results some of the tubes may, after the termination of the campaign, be removed from the heating chamber for examination of their outer surface. It may here be stated that the condensed waters from the first vessel possess the greatest degree of acidity, and those from the succeeding vessels a proportionately less amount.

Yet, in case of *too acid* a reaction, corrosion will most probably be equally manifest in all the vessels, because, although organic acids are first evolved at the beginning of evaporation, various experiments have established the fact that sulphurous acid is liberated uniformly during the whole of the concentrating process.

It is evident that when the sulphured juice is kept neutral to litmus the condensed waters distilled from it will not show an acid reaction to this indicator. Therefore the principal means of control is to dip a piece of litmus paper from time to time in the first condensed water, when its reaction can readily be ascertained. If this reaction is not acid, the other waters cannot be acid either. We will mention another means of control later on.

As regards neutralizing the condensed waters, the simplest way is by means of the Werkhoven dropping apparatus which can be fixed to the feeding pipe of the boiler, a dilute soda solution continuously trickling into the water feeding the boiler, which is in this way neutralized. The degree of the neutralization is determined separately for each boiler, for which purpose samples of the boiler water are drawn daily from the gauge-glass cocks, after well blowing through the latter.

The number of titrations to be made daily, therefore, corresponds to the number of boilers, and is effected by means of N/100 potassium hydroxide and phenolphthalein. It will be best to keep the boiler water slightly alkaline, about 10 mgrms. of calcium oxide (CaO) per litre. A stronger alkalinity may give rise to priming in the boilers.

It is probably better, although not simpler, to effect the neutralization of the condensed waters earlier. For not only the body of the boiler will be attacked by the organic acids, but also other iron portions, such as those of the evaporators, the effect pipes, pumps, ammonia tubes, etc. For this reason it is advisable to commence the neutralizing process as soon as possible after the vapours are condensed to water.

It is evident that from the standpoint of present day technique it must be considered impracticable to neutralize the juice vapours themselves, which would otherwise, on account of the brass tubes of the evaporators, undoubtedly be most desirable. The soda solution can, however, be conducted into the steam tubes connecting between the vessels, so that it can of its own accord flow down to the bottom of the vessels where it will neutralize the condensed water collected. The effect pumps and pipes will also benefit from this.

For this method of neutralizing a quadruple evaporating apparatus will require three dropping apparatus, which may be fixed between the first and second, the second and third, and between the third and fourth vessels respectively. By providing the gauge glass attached to the soda vessel of this apparatus with a graduated scale, the soda supply can readily be regulated, and the amount indicated on this scale by the hour or the shift. With regular sulphuring of the thin-juice, neutralization will practically be automatic without requiring much supervision.

The evaporating vessels themselves, and the steam tubes connecting them, as well as the ammonia tubes can, however, be protected against corrosion even without the application of soda (as will be explained further on when dealing with vacuum pans). For this purpose, after carefully cleansing, they are painted a few times in succession with Venetian red or ferric oxide, which is acid proof.

If on titration of the water in the boilers it should be found that its alkalinity is gradually increasing too much, the supply of soda should be stopped during 24 hours or longer, when the reaction will of its own accord decrease. If, on the other hand, the reaction in general is correct, but only a single boiler shows too alkaline a reaction, the boiler in question should be blown out an extra time.

It stands to reason, of course, that when chemical admixtures are conveyed into the boilers, it is necessary to blow off oftener than otherwise would be the case, and to exercise circumspection during this process, so that any possible deposits may have a chance of first properly settling downwards before being discharged. The amount of soda, however, conveyed into the boilers in the course of the acid thin-juice process is so small that an increase of the density of the water in the boilers has never been detected. About 122 lbs. of the crude soda of commerce to 10,000 tons of cane are employed.

3. The Risk of Inversion during the Sulphuring of Thin-Juice.

Although a pure solution of sugar upon being heated in the open air with sulphurous acid already exhibits incipient inversion at about 50° C., there is little risk of this in cane juice. If this pure solution were heated *in vacuo* it would at once be found that decomposition only commences at 80° C., proving, in this case also, that sulphurous acid in itself possesses little inverting power, but upon oxidation in the air to sulphuric acid it exerts a very strong action in this respect.

Quite a different result is obtained when, instead of pure solutions of sugar, we have to deal with juices as obtained from cane and beet. Upon acidifying these with sulphurous acid, organic acids, as stated previously, are liberated, while the sulphurous acid principally unites with the bases present in the juice, and these organic acids have but slight inverting power. Thus, Ostwald states that if the inverting power of hydrochloric acid is taken as 100, that of formic acid is only 1·5, of lactic acid 1, while in the cases of acetic and succinic acids it is only 0·5. It will thus be seen that in such instances the chances of inversion at once decrease enormously.

If, in addition, these raw juices are evaporated *in vacuo* instead of in open evaporators, so that the last traces of free sulphurous acid cannot oxidize, such juices can, if the reaction is not too acid, be heated if necessary up to 90 or 95° C. without practically any trace of inversion being noticeable.

Before the acid thin-juice process was introduced into Java, it had been the custom for years to sulphur the concentrated juice to a distinct, and sometimes to even a strongly acid reaction (about 0·1 per cent.), after which it was concentrated in vacuum pans at 65 and 70° C., without there ever being any question of inversion. It was therefore firmly anticipated that in sulphuring thin-juice as far as a neutral reaction to litmus this risk would also be nil, especially because dilute liquids are absolutely in no wise more subject to inversion than concentrated liquids. This surmise was amply confirmed in practice, for in no case was any increase of glucose ever observed.

4. Volatility of Sulphurous Acid.

The question has presented itself whether it would not be possible during the acid thin-juice process to replace the very volatile and therefore extremely troublesome sulphurous acid by another inorganic non-volatile acid. If the alkalinity causing the decomposition of glucose were removed by another acid there might, perhaps, be a chance that the same result might be attained, but this expectation has proved erroneous.

Such an acid would have to satisfy the three following conditions:—(1) it must have the same bleaching power as sulphurous acid; (2) it must decrease corrosion; and (3) it must decrease the risk of inversion. Now it has been established by experiment that carbonatation juices which had been neutralized by means of another acid (for instance sulphuric or phosphoric acids), actually exhibited a slight improvement in colour, but not to the same extent as that to be obtained by means of sulphurous acid.

This, of course, is the result of the nature of the salts formed. Only sulphites possess the property of not colouring glucose solutions on being heated, a property due to their reducing character, *i.e.*, they are easily oxidized to sulphates, in which form they are invariably found subsequently in the syrups and molasses:

$$\underset{\text{Calcium sulphite.}}{2\,CaSO_3} + \underset{\text{Oxygen.}}{O_2} = \underset{\text{Calcium sulphate.}}{2\,CaSO_4}.$$

The chances of inversion, moreover, will increase rather than diminish, because, as we have observed, the liberated organic acids (which in any case will be the same) exert little or no inverting action, and any possible excess of sulphuric or phosphoric acids is of course much more injurious.

Finally, the risk of corrosion will not be diminished, for even though a non-volatile acid be employed, the water in the boilers will nevertheless become acid, since here again, not the sulphurous acid, but the liberated organic acids will volatilize.

It will be desirable to consider the latter phenomenon for a moment. It establishes the fact that there can never be any question of a permanent acidity of cane juice. No matter in what way the juice acidifies, whether by the addition of volatile or non-volatile acids, by fermentation or by the melting-in of after-products of an acid reaction, a proportionate amount of free organic acid will invariably upon evaporation pass into the juice vapours, the reaction of concentrated juice in consequence being constantly comparatively less acid than that of thin-juice.

Apart from the fact that in all these circumstances the boiler water must not be lost sight of, it should also constantly be borne in mind that if this acid reaction—such as occurs in sulphuring thin-juice—is essential for obtaining a good colour, the juices which become neutralized of their own accord should be rendered acid anew, so that sulphuring concentrated juice must invariably be recommended. It is, however, evident that in such case the concentrated juice will require less sulphuring than when no sulphuring of the thin-juice has taken place.

5. Brightness of the Thin-Juice.

One of the first conditions for the complete success of the acid thin-juice process is that the filtered carbonatation thin-juice should be as bright as possible, not the slightest trace of turbidity remaining behind. In single as well as in double carbonatation this turbidity invariably consists of calcium carbonate (carbonate of lime), and its presence will jeopardize the success of the process, as will be understood from the following explanation.

We have seen that a portion of the organic acids will be liberated during the sulphitation of the thin-juice, and these, when calcium carbonate is present in suspension, will gradually decompose the latter liberating the carbonic acid. It will thus happen that a turbid filtered juice at the outset is distinctly bleached by sulphuring, but very gradually resumes after some minutes its original yellow colour, in other words, it again becomes slightly alkaline in its reaction to litmus.

All trouble has consequently been in vain, and the liquid is consequently again able, on being heated, to decompose glucose, a brown coloration resulting.[1] This is the reason

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[1] As will be remembered, even without sulphuring, a completely clear thin-juice is essential, since calcium carbonate in suspension is able to bring about a strong decomposition of glucose.

why at one time the method, followed in many sugar factories, of sulphuring the carbonatated muddy juice as far as neutral reaction to litmus had to be abandoned. Lime was indeed dissolved but no improvement of coloration resulted.

6. Practical Results of the Acid Thin-Juice Process.

A peculiarity of sugar juices is that they are darkest in colour when they show an exactly neutral reaction to phenolphthalein. Thus if three equal volumes of neutral juice are taken, and some alkali is added to one, and some acid to another, three different colorations will be obtained. The alkaline juice will be yellow, the neutral grey, and the acid pale yellow.

When, therefore, the clarified juice of carbonatation factories is sulphured, it will become greyer and lighter according to the degree of acidity. It retains this pale colour even during evaporation. During this process the difference between ordinary carbonatation and the acid thin-juice process is greatest. Thus, if with the former method the juice in the four gauge glasses of the evaporating vessels is observed gradually to become distinctly darker and darker in colour, so that at times the concentrated juice is brown or almost black,[1] this changes instantly if sulphuring of the thin-juice takes place.

In that case there is comparatively little or no difference in the colour of the juice in the first and the last gauge glass, while even if an experiment were made using more sulphur than would be desirable for avoiding corrosion, it would be possible to obtain quite a golden yellow concentrated juice in the fourth vessel, which would scarcely

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[1] Besides being the result of a trace of alkalinity, the dark coloration in the evaporating vessels is principally a question of the time during which the juice remains in the apparatus. The shorter this time, the better.

require any subsequent sulphuring. It is very unfortunate, for this reason especially, that hitherto no solution has been found of the purely technical problem as to how the juice vapours are to be effectively neutralized directly after being generated. With such a strong acid reaction there is indeed no risk of inversion but certainly a danger of corrosion of the brass heating tubes of the evaporators.

The sulphured concentrated juice which in ordinary carbonatation is of an orange colour exhibits only a light yellow tint if the thin-juice has been sulphured, while the first massecuite is also bleached from brown to yellow. The formation of dark-coloured decomposition products of glucose has been reduced to a minimum by the acid reaction of the juice, and since carbonatation further eliminates the bark pigments, principally only saccharetin will remain in the mass, it being the cause of the characteristic yellow colour. By protracted boiling the massecuite has been rendered less acid than the concentrated juice, and can even be almost neutral in its reaction to litmus if but slight sulphuring has taken place, at which reaction saccharetin is yellow in colour.

The sugar obtained in the first instance from this first massecuite distinctly shows the characteristic yellow colour. As has already been stated in the Introduction, all products of a cane sugar factory are impregnated with saccharetin. In proof of this a centrifugal containing white sugar may be moistened with some lime water and machined dry, when the sugar will have assumed the colour and appearance of Demerara crystals.

In consequence of the lighter massecuites, but especially as the result of an extremely diminished viscosity (owing to the absence of an excess of organic acid lime salts), the centrifugals work much better and more rapidly. The run-off thus obtained is very light in colour, and may through filtration by means of ordinary low pressure filters be readily separated from its turbid matter. The result is a sparkling, clear, port-wine-coloured syrup.

The acid thin-juice process may be applied in double as well as in single carbonatation. It is especially applicable to the latter because, as we have seen, the clarified juice in single carbonatation is always, in a varying degree, more alkaline than in double. This stronger alkalinity is quite readily eliminated by the sulphuring of the thin-juice.

In double carbonatation, moreover, lactic fermentation will sooner appear in the filtered juice, which may indeed entail loss of sugar, but will in any case produce clearer concentrated juice because the liberated lactic acid will in a slight degree have the same effect as sulphuring As in single carbonatation this is practically debarred, a definite acidification is particularly desirable.

III.

RAW JUICE SULPHITATION OR SULPHUROUS ACID SATURATION.

This method of clarifying juice is used much more frequently than is carbonatation, and would undoubtedly deserve to be discussed in the first place in this Handbook, were it not that carbonatation is an older process, and has consequently been more thoroughly investigated and studied, while its chemistry may be considered to be of a more complex nature.

As, nevertheless, the chemical processes enumerated under carbonatation with but slight exceptions are fairly analogous to those occurring in sulphurous acid saturation, a sound knowledge of these chemical problems may be regarded as entirely indispensable in connection with this method of clarification of the juice.

Carbonic acid and sulphurous acid saturation are in their essence so completely parallel, that in no case would it be wise or advisable to study the two methods independently.

The principal differences between the two, from a chemical standpoint, arise from the lesser energy with which the smaller quantities of lime employed act upon the impurities of the juice, and from the replacement of the carbonic acid salts by those of sulphurous acid.

The technical installation is, nevertheless, totally different. Sulphurous acid saturation is principally chosen on account of its greater cheapness, which is naturally the result of the lesser use of lime, which amounts to only about one-tenth to one-fifteenth part of that used in carbonatation.

Although against the greater cost of the carbonatation method must be placed most probably a more energetic clarification, therefore a greater yield of sugar, this is a matter of so subtle a nature, and so difficult of demonstration by correct figures, that the latter consideration has hitherto exercised little influence in the choice of methods. The economic side of the subject does not, however, come within the scope of this Handbook.

On considering the advantages and disadvantages of the two methods of clarification from a technical point of view, it may be assumed in principle that carbonic acid saturation is misplaced on chemical grounds in cane juices which are rich in glucose. It is accompanied by the formation of soluble carbonates which, perhaps, of all normal salts possess the greatest capacity of decomposing glucose, with the result that, in order to accommodate itself to the conditions, this method of saturation requires modification. On the other hand sulphurous acid saturation is only suitable where cane varieties which are not too much coloured have to be dealt with.

If it should chance that a valuable variety of cane were cultivated which unfortunately had a dark-coloured bark, or if speaking in general the kinds of cane at present coming to the front should be lost to cultivation, the quality of white sugar produced by the factories employing the raw juice sulphuring process would undoubtedly deteriorate. For the present, however, we have no occasion to indulge in such speculations as to possible future contingencies.

The greatest difference, from a technical point of view, between the two methods lies in the readiness with which their juices are filtered. Whereas in sulphurous acid saturation juices are filtered with difficulty, in carbonic acid saturation not only dilute and concentrated juices, but also the much more viscous syrups, may be filtered readily. This difference is due to the more energetic action of lime, previously alluded to, in carbonatation whereby not only gummy and pectic substances are readily precipitated, but likewise suspended impurities, which would hinder filtration by clogging the pores of the filtering cloths.

The colour of the clarified juice, naturally for the same reason, benefits by the greater amount of lime used, being, in the absence of bark pigments, in the ratio of 2 : 3, or in other words, carbonatation filtered juice has only two-thirds the coloration compared with sulphitation raw juice. It is evident, however, that this proportion may vary continually in the course of manufacture, although the advantage of thin-juice sulphuring will be on the side of carbonatation as far as the production of syrups.

It does not necessarily follow from this that the sugar obtained by means of the cheaper method will be inferior in colour to that produced by the dearer and more intensive process. This might be so where a dark variety of cane had to be worked, but otherwise this need by no means be the case.

Crystallization of the first-products from the primary massecuite is effected from a colour mixture not yet sufficiently concentrated to impart to the sugars, carefully cured by means of steam, any striking differences in colour. A change, however, occurs in this respect as soon as the colouring constituents of the massecuite commence to accumulate in the mother-liquors or syrups. Differences of colour in the after-products can generally be avoided only in a complicated manner.

It should here be once more pointed out that in the manufacture of white sugar the greater or smaller amount of success obtained in the clarification of juice in general, and from a technical or chemical point of view in particular, must never be judged from the first-product but always from the after-products, unless, of course, all the sugar is produced in a single process.

As regards the execution of the two saturation methods, against the repeated and sometimes complicated settling stages of raw juice sulphitation, must be placed the more troublesome and intricate lime-kiln system of carbonatation. Respecting simplicity, therefore, the former method carries the day, from which, however, it must not be inferred that supervision will be less in a sulphitation factory than in a carbonatation one. Far from it, for the former also requires extremely strict and continuous control by an expert chemist.

Sulphurous acid saturation agrees most in its chemical aspect, which will require no further explanation, with single carbonation. Those advocating raw juice sulphitation cannot sufficiently be impressed, therefore, with the necessity for seriously studying this carbonatation method. In both methods of defecation excess of lime is at first applied, which is subsequently removed at one stage by saturation, so that reactions and the preparation of milk-of-lime here also play an important rôle. A difference, however, arises on account of the much higher temperatures which are usual and essential in raw juice sulphitation.

The principle of the latter method resides in a thorough heating-up of the unfiltered juices after the lime has been neutralized, which heating-up should later be repeated both with the clarified juice and the concentrated juice. This will more completely precipitate calcium sulphite, and probably organic acid and lime compounds (colouring matter, etc.), coagulate albuminous matter, and prevent acidification with its deleterious effect on the quality of the white sugar. The influence exercised by this rise of temperature upon sugar is as follows:—

1. Sucrose.

By heating the various juices to boiling point, that is, to a temperature above 100° C., and maintaining this heat, some sucrose will be lost by decomposition into invert sugar, according to HERZFELD's investigations referred to in the Introduction (page 10). The amount of sugar thus decomposed has, however, not yet been determined with certainty.

2. Reducing Sugars.

Owing to the high temperatures of defecation, glucose upon coming into contact with alkalis is decomposed, with a strong darkening, which with a view to the quality of the white sugar must of course always be carefully avoided. While, therefore, in carbonatation only a needless increase of the ash content was to be apprehended without risk of darkening, in sulphurous acid saturation the case is entirely different.

The Method of Operating.

The method of operating this clarifying process is now as follows :—The quantity of lime added ranges from about 6 to 10—in some exceptional cases to 12 or more—litres of milk-of-lime of 15° Bé. to 1000 litres of raw juice. On account of the slight tempering, the milk-of-lime is never made thicker than 15° Bé., which enables it to be more readily weighed.

It is manifest that the greater the tempering, the more energetic will be the action of the lime upon the impurities in the juice. Although it is a fact that the slighter tempering is chosen specially on account of the reduced cost as compared with carbonatation, and that this is, therefore, the chief concern, it will not be wise to go to an extreme in this economizing attitude. The increase of lime exercises an ameliorating influence on the colour as well as on the settling character of the juice, and consequently on the quality of the sugars, but the economical maximum of tempering has not thus far been determined.

The sulphurous acid, with which the existing excess of lime is entirely removed in the cold, is generated in a sulphur oven provided with an air compressor, a description of which is outside the scope of the present work.

Of the first importance here, as in carbonatation, is the reaction at which saturation must be discontinued in order to obtain a juice which, from a chemical point of view, satisfies every condition. The possession of an indicator which is both reliable and of practical utility is here also an important factor, for which purpose phenolphthalein is used exclusively.

Now whereas in carbonatation a close watch has to be kept that the indicator does not show too sharp a reaction, because in the presence of carbonic acid it is unreliable, *i.e.*, normal carbonates are indicated as being still alkaline, the case is quite different when sulphurous acid is applied. The indications are much more accurate in the latter method, so that this test can be utilized in its most sensitive form. Instead of air-dried paper, a solution itself is employed which, after careful preparation, is preserved in well-closed bottles.

If the various proportions of lime contained in sulphured raw juice be determined by the different colour reactions upon phenolphthalein, it will be observed that the lowest percentage of lime, *i.e.*, the most successful saturation, occurs at such reaction when the solution indicates practically no further decoloration. It will, of course, be unsafe to continue saturating until the indicator remains white, because in that case there would be absolutely no certainty that the liquid is not too acid.

The detection of the very last, very slightly pink reaction is not always equally simple and requires some routine. The best way of conducting this test is to place the juice and the indicator together on a white porcelain plate, taking care that this does not take place in an atmosphere charged with sulphurous acid, and always in the same proportion, the decoloration of the solution in these extremely sensitive tests being to a certain extent dependent upon the quantity of the indicator solution employed, which at times may lead the observer astray.

It is, therefore, desirable always to bring together the same number of drops of the juice and the same number of drops of the indicator solution; for instance, three drops of juice and one drop of phenolphthalein, or two drops of juice and two drops of phenolphthalein. Other methods favoured in factories consist in placing a small quantity of juice at the bottom of a porcelain dish when a few drops of neutral phenolphthalein solution are added; or in placing a single drop of juice by means of a glass rod on paper freshly moistened with the solution.

Just as in single carbonatation, so in sulphurous acid saturation a retrogressive reaction may sometimes be observed when the neutralized juice has been allowed to stand for some time before proceeding with the boiling. In this case also it will depend on a more or less successful preparation of the milk-of-lime, *i.e.*, on the presence in it of unslaked particles of lime some of which, during the mixing with the juice and the agitation caused in it by saturation, will disintegrate. Other particles, however, will be enveloped with a thin insoluble layer of organic or sulphurous acid lime salts and escape the reaction of the cold juice. Although until recently little attention was paid to this fact in raw juice sulphitation factories, it is nevertheless of still greater consequence for this method of clarification than for the forementioned single carbonatation, and deserves exhaustive treatment in this place.

It would be of comparatively little use to combat the evil of retrograde reaction from neutrality to weak alkalinity in the present method by following the directions recommended for single carbonatation, *i.e.*, to allow the juice to stand for a few minutes and to complete the saturation after this, because the mischief is so much more deeply seated in the present case. In carbonatation we have noted that a portion of the lime particles will disintegrate through the pressure in the filter-presses, rendering the filtered juice alkaline, so that subsequent thin-juice sulphitation or carbonic acid neutralization is advisable.

This pressure, it is true, need not be apprehended in sulphurous acid saturation, but against this must be placed the dangerous rise of temperature to boiling point, which in single carbonatation may be avoided. For it is highly presumable that this increased heat will cause the remaining particles of unslaked lime to disintegrate, whereby quite imperceptibly an increase of alkalinity will occur in the juice-heaters which will, however, soon disappear of its own accord, because at this temperature the lime liberated will attack the glucose which it will decompose, accompanied by the usual darkening of the juice.

In raw juice sulphitation this difficulty can of course never assume such great dimensions as in carbonatation where ten to fifteen times the quantity of lime is used, but it is undoubtedly of sufficient importance to pay great attention to the *preparation of the milk of lime* in these factories, which for the reasons just mentioned is so much easier.

The best means of controlling the proper preparation of the milk-of-lime for the present saturation process is from time to time to let the juice stand cold for a few minutes and to see whether after this interval the original neutral reaction to phenolphthalein has become modified. If this is the case, the milk-of-lime is useless. A more protracted slaking at a higher temperature, a prolonged standing, but especially very careful straining of the milk, will bring about a great if not a total change in this respect. For these reasons dry lime must never be employed.

If the retrograde reaction continues, even after all preventive measures have been adopted, it may frequently be accounted for by too great a percentage of magnesia in the lime used, seeing that magnesium oxide, which is insoluble, and is, therefore, not immediately neutralized by the sulphurous acid, exercises a powerful dissociating action.

Whether the last traces of alkalinity be due to lime or to the excessive percentage of magnesia, in either case a proper settling or filtration will be jeopardized and entail risk of darkening.

It has already been stated that it is useless to attempt controlling the reaction of juice already boiled, since any possible alkalinity present will already have disappeared.

As we have observed, the properly neutralized juice is brought to boiling point about 102°-104° C. (215-219° F.) in juice-heaters, but practice has shown in most cases that in order to obtain a proper settling and a good quality of juice it is advisable after this increased temperature to effect *after-defecation, i.e.*, once more thoroughly to boil the juice for a prolonged period in open juice-heaters or eliminators. It would appear that the impurities in consequence coagulate more readily and subside more rapidly and completely.

Moreover, air and vapour bubbles attached to the impurities, which prevent their settling, will be driven off.

Latterly, it has been pointed out that boiling after the completion of the neutralizing process may occasion the loss of certain advantages which might be obtained if the unsulphured but already limed juice could be heated to not too high a temperature before neutralization is commenced.[1] This point requires a brief examination.

The excess of lime supplied to the juice not only exercises a more energetic action on the impurities than a quantity which—as in the earlier defecation—is just sufficient for neutralizing the acid reaction; but it has besides, to a certain extent, an antiseptic action. In the Introduction, the chapter on fermentation processes (page 21) has shown us that with a few exceptions the yeast cells which may occur in juice do not meet with the requisite vital conditions in a strongly alkaline medium.

Carbonatated juice will, therefore, in general, contain fewer organisms than sulphurous acid saturation juice, which is still further enhanced by the fact that this alkaline reaction in carbonatation is assisted by a rise of temperature to 50° or 55° C. (122-131°F.). It may, therefore, be advisable to adopt this increase of temperature as practised in carbonatation.

There are, however, other reasons why this increase of temperature should be imitated, *i.e.*, the increase in energy of the liming action from a chemical standpoint. With these changed conditions, pectic and gummy matters will in general be more readily precipitated, but the vegetable substances—and, perhaps also, other unknown colouring ones, both pigments and chromogens especially—will be more readily decomposed by lime at a high temperature than in the cold.

The only objection which may be raised is the fear of greater decomposition of glucose and re-solution of lime

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[1] *Archief*, 1911, 819.

or darkening of the juice, which has in fact been the reason why boiling has been deferred until after neutralization. Carbonatation has, however, proved that for proper clarification and bleaching of the juice a rise of temperature to 50° C. (122°F.) or higher is desirable, while it has been shown this will not increase the percentage of lime salts in the clarified juice. It may consequently be safely assumed that what the great excess of lime used in carbonatation is unable to effect, the small quantity employed in sulphurous acid saturation will likewise be powerless to accomplish.

Experiments carried out on these lines have indeed shown that with an *initial temperature* of 60° C. (140°F.), and not too liberal liming, the percentage of lime does not increase in the sulphured juice; that its colour frequently improves; but that above all souring of the cooling juices does not set in as soon or so readily as in the process hitherto adopted.[1]

Following the after-defecation the heated juice is led to the settling tanks, where it remains undisturbed until all the impurities have subsided. These tanks must be protected against heat losses as much as possible, since a fall of temperature with this method of clarifying, which only slightly sterilizes, will entail risk of fermentation or souring. It may briefly be stated here that, as long as the juice has not been concentrated, this will undoubtedly be the principal concern of the white sugar manufacturer employing the raw juice sulphitation process.

If those applying this method have to be content with settling instead of filtering the juice, it needs scarcely to be mentioned that this purely mechanical process requires the greatest possible supervision, and the closest study, since a good sample of white sugar depends not only on the colour of the juice, but likewise on the suspended impurities which may occur in it.

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[1] *Archief*, 1911, 827.

In this connection it is very irrational to carry on this extremely important process in iron tanks, thus screening it from all observation, and we would, therefore, recommend supplying each tank with a wide gauge-glass having a graduated scale behind it for reading off the degree of settling at any hour of the day or night. In this way one will receive timely warning when, through unforeseen circumstances, the amount of lime requires to be modified, whereas otherwise this will only become manifest when much juice has already been treated in an injudicious manner.

An increase of lime required may, for instance, be occasioned by crushing badly frosted, burnt, or dying cane, or by large quantities of clay being carried away with the roots, etc., owing to rain. In short, the liming is regulated as in carbonatation, according to the good or bad separation of the filtered juice from the impurities originally contained in it.

The influence just referred to of the impurities in suspension in the juice of sulphurous acid saturation, need not *always* be unfavourable. Calcium sulphite in suspension, for example, opposes darkening of colour during evaporation.[1]

On no account, however, should the settling process be in the least neglected for this reason.

Sulphitation.

Sulphitation is carried out in the tanks for measuring the raw juice or in sulphur boxes specially constructed for the purpose. Opinions in this respect are divided ; some consider that sulphitation in measuring tanks has the practical objection that the subsequent syrup and the scums from the concentrated juice cannot be returned to the raw juice. The return of these impurities to defecation, however, would probably meet with no obstacle.

A chemical condition is, however, that, as in carbonatation, clarification should proceed as rapidly as possible, that

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[1] *Archief*, 1912, 732.

therefore the lime shall remain in contact with the juice as short a time as possible, since otherwise, as we have observed, even in the cold the percentage of ash will needlessly increase, be it ever so little. In view of this it may, perhaps, be inadvisable to pass the limed juice, as sometimes happens, through a mixer before being led to the sulphitation tanks. A thorough mixing of the lime and the juice may be obtained without this. Just as in carbonatation, it is recommendable in this case also to admit the lime into the apparatus used at about the same time as the saturation gas (whether it be a measuring tank or a sulphur box), and also to commence heating about the same time to 60° C. (140° F.) by means of coils.

Probably for the same reason the heating of the limed unsulphured juice by means of juice-heaters is not to be advised, seeing that this again requires time, which will cause the lime percentage to increase.

It may be considered questionable, however, whether it be desirable for a measuring tank to be fitted with coils and heated each time, with consequent expansion.

The best method of preliminary heating, therefore, will be with the raw unlimed juice. In this way a saving is effected in the coils, and the juice is pumped directly through a portion of the juice-heater, after which it is limed and sulphured, and finally heated in the remainder.[1]

Just as in the acid thin-juice process, where the ultimate neutrality is effected by means of sulphurous acid, it has to be borne in mind in raw juice sulphuring that likewise the condensed waters may become acid. For it is impossible, especially in the cold, to unite all the admitted sulphurous acid with the base so that none will remain in the solution for volatilization. In other words, the reaction is incomplete,

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[1] *Archief*, 1912, 1006.

and the condensed water runs more or less risk of containing sulphurous acid, which has to be guarded against.

Heating to a certain preliminary temperature, moreover, will probably assist in making the reaction more complete, which will also affect the troublesome deposit of calcium sulphite in the juice-heaters, frequently leading to stagnation in the process. Iron pipes in the juice-heaters will sooner be obstructed than copper, and are besides more difficult to clean.

This calcium sulphite, which is only formed with a rise of temperature, because this salt is less soluble in hot than in cold solutions, is one of the most troublesome bodies in the sulphurous acid saturation process.

With heavy liming more trouble is experienced in this matter than with light liming. It is, moreover, evident that a preliminary heating of the raw juice will considerably diminish this incrustation, since in the juice-heaters no lime as yet is present. The transition from the amorphous to the crystalline form of the insoluble calcium sulphite is one of the principal reasons of the incrustation.

Practically, the muddy juice should never be heated above 90° C. (194 F.) in the juice-heaters, and should only be raised to boiling point in open after-defecation pans. The considerable incrustations will thereby be avoided. Where much trouble with incrustation is experienced, the juice-heaters must be cleaned in rotation and at stated intervals, say regularly every 12 to 24 hours. Longer intervals render cleaning more difficult out of all proportion.[1]

Continuous sulphitation, no more than continuous carbonatation, may not take place where the single method is in use. It is impossible by this means to ensure a proper and regular process, while the final reaction especially, which is of so much consequence in this case, is too difficult of control.

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[1] *Archief*, 1912, 1008.

The Treatment of Muddy Juice.

As regards the treatment of muddy juice the following may be stated in this connection: The juice which has subsided in the settling tanks is carefully tapped off by means of floaters. By this means it is possible to let the juice flow slowly from the surface downward without agitating the easily disturbed precipitate. As the pipe leading from the floater will after the settling process probably contain some of the sediment, this is first carefully drawn off by a separate stopcock into the muddy juice pipes.

The clear juice thus obtained can immediately be evaporated, but in many factories it is first re-filtered in filter-presses or through bronze gauze.

After a thorough second boiling, the muddy juice is carried to a second set of settling tanks, having about one-fifth the capacity of the first tanks.

The second settling should be arranged and controlled exactly as the first, except that the gauge-glasses referred to are of course not required, since they are only necessary for correct liming, and effect their function during the first settling. If the juice properly subsides in the first instance, this will undoubtedly be likewise the case in the second.

As has already been observed, the chief concern in working the muddy juice is *that no souring or fermentation shall take place.* The bacteria and yeast cells, which occur so frequently in tropical white sugar factories, will not fail to attack this dilute neutral or slightly acid juice as soon as the temperature falls sufficiently to establish the required vital conditions. Hence the necessity of repeatedly boiling the juice where this method is in use, and of working up the muddy juice as quickly and as cleanly as possible.

The second settling tanks should always be well washed after being emptied and if the muddy thick-juice is added to the first muddy juice, all the stated precautionary measures need extra attention.

The second sediment is boiled a third time and filtered either in filter-presses or in Taylor filters. It is evident that the longer the whole process of clarification lasts, the greater will be the risk of souring, that is to say, the chance of acidification increases as the muddy juice nears its final treatment, so that with the final filtering this danger will undoubtedly be greatest. Hence, the filter-presses should, as in carbonatation, before use be thoroughly steamed for a considerable time in order to kill any possible bacterial colonies, and also in order that cooling of the juice by the iron may be reduced to a minimum. With the Taylor filters too the temperature should be kept as high as possible.

Operation by means of filter-presses is cleanest, but frequently somewhat darker juice is obtained thereby, because iron is dissolved from the filter-frames and absorbed. Seeing that in consequence of the subsequent acidification and reduction of the concentrated juice this iron has little or no injurious effect on the sugars, this need not worry us much. On the other hand, souring is probably more easily prevented in these apparatus than in bag filters.

Apart from the loss of sugar, the result of souring is generally a juice more difficult to treat (especially as regards filtration), usually producing a less fine sugar, as has already been pointed out when dealing with carbonatation. The viscosity is increased, which is especially noticeable later when elaborating the syrups, and which *will invariably happen in the case of any defect in the clarifying process.*

The scums of sulphurous acid saturation will contain from 8 to 11 per cent. of sugar, which is often partly recovered by malaxation outside the filters, as is usual in carbonatation. The liquid thus obtained is generally somewhat darker in colour than the original thin-juice, for which reason it is used for imbibition or returned to the raw juice. No lime must on any account be added during malaxation. It has, however, not yet been determined with certainty whether this lixiviating process is without risk for a good quality of white sugar.

Sometimes, when the juice has been heavily limed, the scums are exhausted in the press, and this is effected by admitting the water by the same course as the raw juice. Generally speaking, the question of washing in raw juice sulphitation has so far been but little studied.

As a rule, the first consideration with this method of clarifying is to arrange matters in such a manner that after the original cold liming no more lime need be added. This rule is subject to no exception. The least addition of lime will involve darkening of the juice, not to mention the needless increase of the ash content. It is a mistake to suppose that any possible souring can be checked by the addition of lime to a neutral or a slightly alkaline reaction. The only result to be obtained by it is the formation of lime salts, which at the temperature of fermentation are of such a nature that they will split up spontaneously into acid decomposition products, so that souring will rather be promoted. The only check against souring is re-boiling.

As we have already noted with carbonatation, the stoppage of the presses, of so frequent occurrence, occasions great risk as to the quality and composition of the juice, and this is also the case with this method of clarifying, indeed to a greater extent.

It may be taken for granted that the least stagnation during the successive settling and filtering processes will have certain detrimental effects. Once the raw juice has been obtained, care must be taken that, as in carbonatation, the entire process as far as the last filtering is carried out without interruption. If stagnation should occur, it is imperative directly to stop the process, and *to let the juice stand cold and limed.* There is no other way out of the difficulty.

If once liming, sulphuring, and boiling have been effected, and for one or other unavoidable reason the necessity arises for deferring the settling process or the elaboration of the muddy juice, the greatest care should be taken that the temperature falls as little as possible; but such an irregularity can never be entirely made good.

Settling.

Finally, we must for a moment return to the subject of settling. It is evident that, apart from a liberal liming, the regular process of settling must also depend upon the density of the juice. If this is high, the suspended particles it contains will not subside as quickly and as readily as when it is low, so that this must be taken into account when the muddy thick-juice is returned.

Moreover, it has been determined experimentally that in saturation to neutral reaction to litmus instead of to phenolphthalein, settling is generally much more rapid and complete. If juice has been saturated to neutral reaction to phenolphthalein, it will still turn litmus distinctly blue, because the sulphites formed have an alkaline reaction to litmus. If, however, saturation is continued until a neutral reaction to litmus sets in, the sulphites will have been converted into bi-sulphites (partly at least), and the juice is therefore acid.

$$\begin{array}{ccccccc} CaSO_3 & + & SO_2 & + & H_2O & = & Ca(HSO_3)_2. \\ \text{Calcium sulphite} & + & \text{sulphurous acid} & + & \text{water} & = & \text{calcium bi-sulphite.} \end{array}$$

$$\begin{array}{ccccccc} K_2SO_3 & + & SO_2 & + & H_2O & = & 2\,KHSO_3. \\ \text{Potassium sulphite} & + & \text{sulphurous acid} & + & \text{water} & = & \text{potassium bi-sulphite.} \end{array}$$

It would appear that the impurities of sulphurous acid saturation settle more readily, and to a greater extent in an acid than in an alkaline or neutral medium. It is, however, patent that manufacturers must never avail themselves of this circumstance, because during the evaporation of such an acid juice much sulphurous acid, the result of dissociation of the bi-sulphites, would pass into the effect condensed water, and occasion great risk of rapid corrosion in the brass heating tubes of the evaporators.

It does not by any means follow from this that the acid method would not result in a better quality of white sugar if it were possible to neutralize the vapours of the juice soon after their formation, as has already been mentioned under thin-juice sulphitation. The brighter juice and the more rapid process would probably outweigh the slight increase of lime salts.

As regards the content of these lime salts, the remarkable fact presents itself that, notwithstanding the much weaker liming, this will invariably be higher in the filtered juice obtained by the sulphitation process than in that produced by carbonatation. If, for instance, the amount of combined lime in single carbonatation, which mostly resembles raw juice sulphitation, averages from 100–200 mgrms. of calcium oxide (CaO) per litre, in the latter it ranges from 200–300 mgrms. Although this question, like many others in this saturation process, has been little investigated, it may nevertheless be assumed that this phenomenon is based on a different proportion between the organic and inorganic lime salts.

While, for instance, the lime constituents of the filtered juice in carbonatation, apart from a slight amount of inorganic lime salts (sulphate, carbonate, etc.), consist by far the largest portion of colourless calcium organic acid compounds (especially calcium lactate, or, in the case of over-ripe cane, calcium acetate), in sulphurous acid saturation on the contrary the greater part of the lime salts will consist of calcium sulphite which, especially at lower temperatures, is more soluble in solutions containing sugar than is calcium carbonate.

It has, moreover, been determined that with heavier liming the percentage of soluble lime in juice clarified by means of raw juice sulphuring is lower than in the case of lighter tempering. This appears to be based partly upon mechanical precipitation and partly upon chemical clarification.

Although, therefore, in sulphurous acid saturation care must also be taken that the lime content of the filtered juice does not increase unduly, which, just as in carbonic acid saturation, should constitute the *chief control in a proper and judicious clarification,* on the other hand not too much anxiety need be entertained about the greater percentage of lime in the sulphitation than in the carbonatation process, providing all the operations have been properly conducted. For the calcium sulphite is, as we have already noted, from a colour point of view one of the most valuable constituents of juices containing glucose, which in carbonatation is but too deficient, care being taken to remedy this deficiency as much as possible by sulphuring the filtered juice.

For the sulphites, like sulphurous acid itself, possess, although of course to a considerably less extent, a certain reducing power in that they eagerly oxidize to sulphates with any available oxygen.

Apart from the fact, therefore, that they are able to prevent a great portion of the coloration unavoidable otherwise in practice, they can, after completion of their function, readily be eliminated from the juice, calcium sulphate being less soluble and being readily separated from suspension in the thick-juices and syrups, when they can no longer exercise any chemical influence upon the formation of molasses.

IV.

TREATMENT OF THICK-JUICE.

The treatment of thick-juice in a sugar factory is and should be nothing but a corrective of the clarifying process. That is to say, the quality of the thick-juice is always a test of the quality of the thin-juice. This statement should continually be kept in view in white sugar manufacture.

The more or less technical perfection of a method of clarifying juice may indeed be judged from the resulting untreated thick-juice; in other words, the more involved and complicated will be the treatment of the thick-juice, the less perfect the clarifying process has been.

An ideal white sugar thick-juice should be of such a nature that it can be boiled to a light coloured non-viscous massecuite entirely without correction or further treatment, *i.e.*, without filtration, or sulphuring, or additional chemical purification.

The great difference between a treated thick-juice and one which requires no treatment whatever for meeting the most exacting claims, does not betray itself so much by the nature of the first-product (although it may rather by the quality of the after-product), as by its keeping properties.

The tropical white sugar industry and its technique are, as has already been once observed, still in their infancy. Thus far the chief product is judged, as regards its colour and lustre, only at the time of production, and little attention is paid to its ultimate behaviour.

So long as this article of consumption finds a ready sale abroad, matters will proceed smoothly, but it is advisable to be prepared against times of over-production and shifting of markets, in which case the sugar must not only at first *be* of a good white colour and of a pure lustre, but must *remain* so.

The following directions will demonstrate in what measure practice and experience have succeeded in realizing this ideal condition for the concentrated juice by various methods of clarification.

The concentrated juice may require additional treatment in two different ways, *i.e.*, mechanical or chemical. When evaporation has proceeded to a fairly considerable degree, the substances which at this stage of concentration have not sufficient water to remain in solution will gradually commence to separate. In the case of over-saturation, the bicarbonates or bi-sulphites of lime formed will have been decomposed in the evaporating apparatus by boiling, so that the insoluble calcium carbonate or calcium sulphite will have separated in a suspended state.

When dealing with clarification, we have already noted that in consequence of our defective technical installations the thick-juice cannot be prepared in such a manner that during the concentrating process no additional colouring substances are formed, these having to be bleached somehow or other. Now, the mechanical impurities will have to be removed either by means of filtration, settling, or elimination, while the chemical decolorization must be entrusted to the powerful reducing agent—sulphurous acid.

These are the principal processes constituting the treatment of thick-juice. In the following pages they will be discussed in further detail in connection with the two methods of clarifying.

a. Carbonatation.

A properly carbonatated juice will invariably yield a thick-juice containing so few particles in suspension that filtration may safely be omitted. If it is found, therefore, that the thick-juice obtained by this method is turbid, the first requisite will be to trace the causes thereof in the clarifying process. They may be manifold.

Thus a turbid concentrated juice will be produced if the thin-juice has already been turbid before evaporation. In order to prevent this, close attention must be paid to the second filtration in double carbonatation and to the filtration in single carbonatation. Where the expense of clarification is incurred exclusively in order, in the first place, to render the juice as readily filtered as possible, the manufacturer is bound to make the filtering process as successful as can be from the very beginning. It should especially be remembered where this method of clarification is employed that it is the more expensive process of the two, and that every superfluous manipulation and every needless increase of plant should be avoided.

A second cause of a turbid carbonatation thick-juice has already been mentioned *i.e.*, over-carbonatation.

A third cause may be the possible re-melting of later products in the filtered juice. In factories with modern plant this re-melting should not occur except in the case of small quantities of caked sugar, and the two former determining causes should always be avoided so that the concentrated juice will not require to be filtered.

Of course the thick-juices obtained during the early stages of the campaign, and those after stopping for some time during the crushing process, form an exception to this. They always contain great quantities of mechanical impurities consisting of particles of rust from the pipes, etc., and scrapings from the evaporators, which polluting and insoluble matters should most certainly be removed. Even after an eventual filtration of the concentrated juice, a sufficient amount of impurities will find its way into the juice obtained from the additional apparatus beyond that stage, so as to produce a considerably less valuable sugar, which first sugars should always be re-melted.

For these various reasons a temporary thick-juice filtering station for the concentrated juice will be requisite in carbonatation factories which, from its nature, will be required to be used only for a few days in the campaign, but which for that reason may very well be arranged to do double service, and thus be utilized for filtering syrups (first jet). This filtration, to which we will return later on, is naturally of equal influence on the quality of the molasses sugar, as is the filtration of the thick-juice on the quality of the first-product.

The filtration of the carbonatation concentrated juice proceeds very readily and rapidly at all stages of concentration and at all temperatures, so that all kinds of filters can be used for this. Low-pressure filters, with a difference of level from 6 to 9 ft., as well as filter-presses, are used for this stage, although of course it requires no further mention that presses will occasion needless use of power and steam.

The scum retained by the thick-juice filters in the course of these last-mentioned stages is generally of a purely mechanical nature, and is therefore easily washed, which washing must, of course, never be omitted. Of souring or fermentation there is little or no question in the case of these thick-juices, which have a density of about 60° Brix; but this does not, of course, by any means preclude the necessity of the utmost cleanliness.

N.B.

After this mechanical treatment which, as we have observed, is only a temporary necessity, follows the chemical treatment, which at the present standpoint of technique must never be omitted in carbonatation. We refer to sulphitation.

Where ordinary carbonatation is applied without the acid thin-juice process, the concentrated juice will generally have assumed a very dark colour during concentration, being proportionally darker in fact, as we have previously explained, than that obtained by means of the ordinary raw juice sulphuring, which is attributed to a deficiency of sulphites in the carbonatation thin-juice.

N.B.

If the acid thin-juice process is applied, an immediate change will be observed in the colour of the thick-juice, and the sulphuring or bleaching of the untreated concentrated juice can be considerably diminished. If the corrosion of the evaporation tubes did not bar the way, that is to say, if we had at our disposal an apparatus for neutralizing the juice vapours before their entry into the next evaporating chamber, thin-juice sulphuring in carbonatation might, without risk of inversion, be carried so far that a thick-juice would be produced, which in most cases could dispense altogether with any additional treatment whatsoever.

This is, of course, capable of practical demonstration by continuing the thin-juice sulphuring to its maximum for a very short period, when a light yellow concentrated juice, as clear as crystal, having its viscosity reduced to a minimum, will be obtained after evaporation. Such a thick-juice could be boiled directly, without any modification, to first massecuite, and we would have attained our forementioned ideal; but for purely technical reasons we are as yet far from this attainment.

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Sulphitation of the concentrated juice must never be entered upon before filtration is finished for two reasons :—

(1) Because filtration requires a certain time, and it is of course undesirable to allow the concentrated juice to stand in the open air with a strong sulphurous acid reaction for a longer period than is absolutely necessary. Probably the risk of oxidation of the volatile sulphurous acid to sulphuric acid, and consequently the danger of inversion, would hereby increase.

(2) Because in case impurities should have entered the concentrated juice as a result of over-carbonatation or deficient filtration, these would be dissolved by sulphitation and disappear before they could have been filtered. It is evident that neither of these two reasons will have much weight in carbonatation as against the other method of clarification, but for the sake of correctness and completeness both factors should be taken into account and the plant installation based upon them.

An important matter in thick-juice sulphuring is its control, and the extent to which it may be continued. Just as in the sulphitation of the thin-juice, it is of the utmost importance to ascertain the acidity of the thick-juice, since this is in fact the only safe index to any possible risk of inversion or corrosion. The colour of the concentrated juice, however, precludes direct titration by means of alkali and phenolphthalein indicator, as recommended for thin-juice. The exact colour change can no longer be observed.

For this reason the ordinary iodometric determination of sulphurous acid and its compounds has hitherto been applied in all factories, which in general will furnish figures about parallel with the acidimetric titrations, which it can replace without much inconvenience.

The said iodometric determination is based on the fact that sulphurous acid and its salts are oxidized by free iodine to sulphuric acid and sulphates. Therefore, as soon as all the sulphurous acid has been converted, the iodine solution dropping into the liquid will remain unchanged. If now this liquid contains a substance which will act as a sensitive indicator of any free iodine present, the termination of oxidation may be determined with fair accuracy. The iodine indicator usually employed is a solution of starch, which will immediately turn blue with the formation of the so-called "starch iodide," especially when fresh.

This process is carried out in such a way that always the same quantity of the sample is mixed with the same quantity of starch solution in a graduated tube. To this mixture is added, drop by drop, a solution of iodine in potassium iodide, of the strength of 2 grms. of iodine per 1000 c.c. At first the blue coloration which is formed disappears immediately, but becomes gradually more permanent. As soon as the blue colour of the starch iodide is finally fixed, the titration is finished, and the number of c.c. of the iodide solution used is read off directly on the graduated tube. Here, as in thin-juice sulphitation, a Vivien tube is used. These tubes can, of course, be modified according to one's liking, thus making them answer their purpose more conveniently.[1]

The more iodine solution is used, the greater the amount of sulphitation. Naturally this method is not quite exact, because the concentrated juice always contains substances which will also be oxidized by the iodine, but it is quite sufficient for practical purposes. In order to ascertain the

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[1] According to VOLHARD'S modification of the Bunsen method, it is more accurate (at least with these fairly concentrated solutions) to reverse the process, *i.e.*, to add the sample of juice to a definite quantity of the iodine solution, as otherwise there is a risk of an inaccurate reading, through reduction of sulphurous acid to sulphur; but it is to be noted, firstly, that in a factory with a native *personnel* no very correct analyses can be expected; and, secondly, that the unavoidable last drop in titration probably causes less error when it consists of a dilute solution of iodine than when it is a concentrated solution of sulphurous acid. This point requires, however, considerable attention.

extent of the error, the unsulphured concentrated juice is generally also examined iodometrically. The volume of iodine solution required to produce the blue colouring may eventually be deducted from the volume necessary for the sulphured concentrated juice. It will be found, however, in the course of ordinary carbonatation that the percentage of oxidizing substances in the original unsulphured juice scarcely varies, so that, in fact, a fixed figure may be adopted.

The case is of course immediately altered when the acid thin-juice process is applied. This introduces sulphurous acid into the thin-juice, and the percentage of substances oxidized by iodine in the original concentrated juice will rise and fall in proportion to this sulphuring. The determination of the substances capable of being oxidized is here of the greatest importance. In the first place it enables us to determine accurately the extent of the concentrated juice sulphuring, and in the second place it is a means of control of a regular and proper thin-juice sulphuring, for if the latter has proceeded uniformly and accurately the variation of the iodine figures in the original concentrated juice will be slight.

In applying the acid thin-juice process, therefore, the degree of concentrated juice sulphuring is judged from the difference in the iodine figures of the original and of the sulphured concentrated juice.

The decoloration caused by sulphuring is twofold. In the first place it proceeds from the acid reaction imparted to the concentrated juice, a decoloration engendered not only by sulphurous acid, but by any acid more powerful than the organic acids occurring in juices. In the second place decoloration arises from the reducing and consequently bleaching action of the sulphurous acid itself, but it is neither as great in extent nor as lasting as the former, although it is nevertheless indispensable, *inter alia* for converting the ferric salts into ferrous salts, as we have already noted.

As regards the degree of sulphuring, it follows from the foregoing that this can never be subject to fixed rules. For various reasons—density, more or less successful clarification, decomposition of glucose, etc.—one concentrated juice may be darker than another, and consequently will require heavier sulphuring. The sulphuring of thick-juice should always be carried to the maximum. This may be determined by adding a drop of acetic acid to the sulphured juice. If this bleaches the juice still further, it will indicate that sulphitation has not proceeded sufficiently.

The point of maximum bleaching in carbonatation will generally be effected with about 0·08 - 0·10 per cent. of sulphur dioxide (SO_2), while in applying the acid thin-juice process the acidity will as a rule not exceed 0·06–0·08 per cent.

N B

There is no reason why sulphuring of the concentrated juice in white sugar factories in general should not be carried out by the continuous process. The sensitiveness and the accuracy of the iodometric control, as we have seen, is not very great, so that concentrated juice sulphuring will only yield approximate figures. There is thus no reason why for the sake of its accuracy a much simpler installation should be rejected.

The continuous sulphuring process of concentrated juice is effected by means of two saturation tanks. In the first tank the filtered concentrated juice is roughly sulphured to just below the required point for the maximum bleaching, while but little sulphurous acid is admitted to the second tank in order clearly and easily to observe the final reactions. The supply of sulphurous acid to the first tank may remain unaltered, while the supply stop-cock of the second is turned on or off according to requirement. In both tanks of course the juice should be admitted from below and run off at about the same level. From the second tank it simply runs to the syrup supply tanks.

Regarding the risk of inversion, this is not very great, as has already been pointed out when dealing with the acid thin-juice process, especially in carbonatation, because there the resulting concentrated juice never requires heating, but invariably possesses a temperature of 60° C. (140° F.) or less. Much has been written on this subject mostly to the effect that inversion does not occur.[1]

For proper control it is, however, advisable in white sugar factories daily to determine the amount of glucose in the unsulphured concentrated juice and in the first massecuite. Under normal conditions a slight decrease of the glucose factor will be observed; if, however, an increase takes place one has to exercise care.

Thin-juice sulphuring can also be properly controlled if, besides the forementioned determinations of the amount of glucose in the thick-juice, the percentage in the thin-juice is determined daily. During the evaporation from thin to thick-juice the glucose ratio should remain about stationary. This method of control, provided it is carried out carefully, will be sufficient.

b. Sulphurous Acid Saturation.

Since raw juice sulphitation as a method of clarification can never be as intense as carbonatation, the treatment of concentrated juice after the former process must consequently be somewhat more thorough; whereas in carbonatation no mechanical purification is, in fact, required, in sulphurous acid saturation on the contrary this is the chief factor.

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[1] *Archief*, 1907, 601; and *ibid.*, 1901, 64.

The thin-juice of the sulphitation process obtained through settling cannot from its nature be as clear as that yielded by carbonatation, and however slight the remaining turbidity may be, it will become more and more evident on concentration. There are, besides, other reasons why this thick-juice will in this case be always more turbid, for the percentage of inorganic salts in the thin-juice of raw juice sulphuring is always considerably higher than in carbonic acid saturation.

In discussing the former method of clarifying, we have already pointed out that the percentage of organic salts is different from that which obtains in carbonatation, because in raw juice sulphitation inorganic lime salts (especially calcium sulphite) form the chief factors, and in carbonatation organic lime salts preponderate. Now, the latter are extremely soluble and will not, even at the strongest concentration, be easily separated, while on the contrary all inorganic lime salts of cane juice (sulphites, phosphates, silicates, etc.) separate readily because, in general, they are but little soluble.

Especially when a portion of the calcium sulphite, originally occurring in solution during the evaporation process is oxidized to calcium sulphate, which is still less soluble than the sulphite, a very distinct sediment will occur in the concentrated juice.

It is of course essential for obtaining a first-class sugar that the product should be crystallized from a liquid as pure and as clear as possible, a reason why in sulphitation factories every possible attention is paid to the settling and filtering of the concentrated juice. It is unfortunate that just with this method of clarifying, where a thorough filtration of the concentrated juice is such a desideratum, this juice is so much less susceptible to filtration than that of the other method which requires no filtration. The juice, already so viscous in its dilute form, will not of course improve in this respect by concentration, so that the filtration will meet with almost unsurmountable obstacles. It is only by means of special plant that it can be effected.

Many manufacturers, indeed, confine themselves to simple settling, which proceeds in about the same way as that of thin-juice, both as regards the manner of drawing off the liquid, and the separation into clear juice and muddy juice ("bottoms"). It should here, however, be borne in mind that a proper settling is proportionate to the difference which exists between the density of the concentrated juice and that of the subsiding impurities. If this difference is considerable, in other words, if the juice is less concentrated, settling will proceed much more readily, and *vice versâ.*

If measures have been taken for settling, it should to some extent be borne in mind that the concentrated juice must not be allowed to become too thick. In general the impurities subside very readily and rapidly at 26° Bé, but above 27·5° Bé. the process is less successful. In the case of very concentrated juices for instance at 30° Bé. and upwards, it will be well not to expect too good results of the settling process. Even the addition of chemicals before the commencement of settling, such as baryta, potassium ferrocyanide, or sodium phosphate, will be unable at this increased density to promote settling. Often the contrary will be the case, the heavy baryta, for instance, giving an unusually difficult settling.

The best results as regards clearness are really to be obtained by means of filtration. This may be effected either by Dehne filters (low-pressure filters with a difference of level of about 15–18 ft.) or by palm-fibre filters, which consist of ordinary cylinders filled with the fibre, the juice entering from below and flowing away from the top. Contrary to what occurs in settling, the addition of chemicals may here have much beneficial effect. In would appear that, as has already been observed under the heading of Second Carbonatation, proper filtration may be promoted by artificially increasing the amount of sediment in the juice.

A drawback of filtration is the amount of water, generally very considerable, required for washing the filters. By proceeding methodically, and effecting the sugar exhaustion process, not only by water, but also by the mere efflux of time, this may to a great extent be obviated. When water is thus sparingly used, but is allowed to stand in the filters for a considerable period before being drawn off, a very satisfactory result may be anticipated. We must nevertheless not lose sight of the fact that the inorganic impurities, which thus are filtered out of the concentrated juice, were originally in solution in the thin-juice, so that in washing there will be risk of their being in part re-dissolved and carried back to the thick-juice.

We learn from these data that both methods have their advantages and drawbacks, although the better results are to be expected from filtration. In either case it is necessary to raise the concentrated juice to boiling point, in the first place for diminishing the viscosity, and in the second place for temporarily lowering the density.

This boiling will, therefore, facilitate both filtration and settling. Moreover, practice has shown that not only is it advisable thoroughly to boil the concentrated juice, but that it should be especially accompanied by proper elimination. As already has been pointed out when dealing with thin-juice (which likewise greatly benefits by careful elimination), the insoluble matter appears to separate and cohere more readily in consequence, while many mechanical impurities may be skimmed off with the scum on the surface. It follows as a matter of course that when the clarification of the juice has been conducted properly, and the drawing off of the clear juice in both thin-juice settling processes really is properly controlled, little or no trouble will be experienced with regard to mechanical impurities, which may rise to the surface of the concentrated juice. For this reason in most factories this clear thin-juice is once more filtered through fixed or rotating sieves of bronze gauze, from which, however, not too much must be expected as regards the very finely divided suspended particles.

What has already been observed previously may here be repeated, namely, that the more complicated the treatment of the concentrated juice, the more defective generally will be the clarification of the juice and its control. If it is therefore observed that on elimination of the concentrated juice too many impurities have to be skimmed off, the settling processes of the thin-juice will have to be carefully examined.

This increase of temperature, to which the concentrated juice is unavoidably subjected, is a disadvantage not to be underrated. In the first place, loss of sugar—even though slight in extent—is bound to occur in these concentrated liquids as a consequence. But in the second place, this higher temperature requires to be lowered by means of cooling, which has to be effected in view of the next process, *i.e.*, sulphitation, which of course must not be carried out at too high a temperature, especially while the concentrated juice is exposed to the open air and is not *in vacuo*. We will return to this matter when dealing with sulphitation.

It has already been briefly observed that in raw juice sulphitation factories chemicals are frequently added to the concentrated juice. Originally the object was to improve the filtration. By the introduction of baryta or potassium ferrocyanide, the elimination of iron was at first aimed at, but the excellent effect on the resulting filtration also began to be appreciated.

X

Nevertheless, these chemicals are at present hardly employed anywhere; first, because they are no longer required for the elimination of iron; and secondly, because sodium acid phosphate forms an excellent medium for filtering concentrated juice, not only causing calcium phosphate to be precipitated, but at the same time exercising a decolorizing influence on the concentrated juice, a property of which the two first-mentioned chemicals are devoid. The lime salts of the organic acids are converted by sodium phosphate into the less coloured and less viscous sodium salts, while on heating free phosphoric acid will also be generated, which, while attended with risk of inversion, has yet a bleaching effect.

N.B.

It is clear that the application of the acid or, if necessary, of the normal sodium phosphate in carbonatation, will produce exactly the same bleaching and rectifying effects, at least when a filtration of the concentrated juice is left over for this purpose. It may, however, with equal advantage be applied to the syrups which in carbonatation are invariably filtered and heated, if it is desired to reap all the advantages of the more expensive method.

The application of the phosphate to the defecation concentrated juice must of course precede elimination since, just as in the case of baryta, successful precipitation is promoted by boiling. It is, moreover, believed that the addition of baryta bleaches the juice, because this salt decomposes the lime organic acid salts with the precipitation of insoluble barium organic acid salts. In this manner the dark lime salts will be eliminated from the juice.

In reality, however, every concentrated juice contains so much soluble sulphate and sulphite, which immediately combines with the baryta, that even with large applications of this salt decomposition of the lime salts or bleaching action is never capable of demonstration. It has, indeed, at times been assumed that this is the case because the concentrated juice appears lighter in colour during the subsequent boiling. But this condition is observed just the same in the ordinary elimination of the concentrated juices without any chemicals being employed. This is simply accounted for by optical reasons, since the supposed bleaching effect again disappears of its own accord after the hot juice has cooled down. It thus appears that owing to the decreased cohesion of the molecules in consequence of the higher temperature, the juice will appear to be lighter in colour than it actually is. In judging the colour of juices and syrups this phenomenon should always be carefully borne in mind.

As in carbonatation, in the treatment of the thick-juice the greatest possible cleanliness is of the first importance. It is true that here also in consequence of the greater concentration, there is less chance of fermentation or souring, but it must not be forgotten that on settling the thick-juice, part of it is returned to the raw juice, and will thus increase the risk of souring in the subsequent thin-juices.

The settling tanks of the thick-juice should therefore, after completion of the subsidation process, be washed as carefully as possible, when care should be taken that any scum adhering to the sides of the tanks is carefully removed. It is in these particularly that the bacteria congregate.

Without the requisite precaution the muddy thick-juice may sometimes occasion the less satisfactory settling of the thin-juice. For, being so much more strongly concentrated than the raw juice there is a possibility, when too much of it is introduced at one and the same time, of its density increasing to such an extent that a proper settling is practically impossible. In that case it is of importance that the muddy thick-juice should be returned to the mill, slowly and regularly, so that it may be distributed as carefully as possible over large volumes of the raw juice. This will ensure a slight, but above all a gradual increase of the density of the thin-juice, which is indispensable for a properly controlled settling.

After the mechanical rectifying process, described in detail in the foregoing pages, has been completed, the further chemical correction comes next, *i.e.*, decolorization by means of sulphitation. We have already noted that the concentrated juice is generally too warm to proceed with this at once. Factories in which the concentrated juice is allowed to subside first will have little or no trouble on this head, since during this settling cooling will take place of its own accord. It is, however, a different matter in factories with a filtering stage where the concentrated juice reaches a temperature of nearly 100° C. (212° F.) after filtration. In this case cooling to 65° (149° F.), or below it, is absolutely necessary.

Now this cooling of the thick-juice may take place in different ways. Generally old juice-heaters, or tanks with coils, in which the juice is cooled by means of water, are employed. Coolers, in which the juice flows slowly over a cooled surface in the open air, are also used, but then a dark colour may form owing to the oxidation of certain substances present. This phenomenon has already been dealt with in this work under methods of juice purification. As a result, sulphitation must be carried a little further.

Sulphuring takes place at this stage of juice purification just as in carbonatation, and is also similarly controlled. Although so far it has almost never been done, there is no real reason for not continuously sulphuring the thick-juice, which is rapidly and simply accomplished without the sulphured juice remaining needlessly long exposed to the air. The degree of sulphitation generally amounts to 0·6–0·1 per cent., though dependence must be wholly placed on circumstances, as by alterations in density, etc., sulphuring now and again requires considerable variation.

It is self-evident that in one and the same factory when grinding the same cane, and with regular work in evaporation, such a uniform thick-juice may be expected that the degree of sulphitation can be kept within certain limits. In abnormal cases, however, such as the grinding of dead or burnt cane, or difficult evaporation, and consequently an extra low density, etc., the man-in-charge must have freedom of working without being bound to any fixed rule. The only requirement is that by means of carefully made glucose determinations, he shall always guard against any possible inversion.

If in a factory it is necessary, either from time to time or continuously, so far to sulphur the juices that the limits of inversion are dangerously approached, resource is then had to the expensive sodium hydrosulphite ($N_2S_2O_4$), which possesses reducing properties in the neutral state, and which may then be added to the thick-juice in place of excessive sulphuring. It requires no argument that in such cases it is preferable to trace the cause of the abnormal sulphitation, which almost without exception lies in the use of too little lime in saturation. In factories sulphiting their raw juice, this is generally the most frequent fault, yet one which however very easily finds explanation in the fact that just in the saving which is the result of the diminished amount of lime the greatest advantage of the juice clarification should lie, a saving for which everything in this stage of manufacture is sacrificed.

It is clear that in sulphurous acid saturation it is an imperative condition first carefully to either subside or filter, and only then to sulphur. In the first place one should not commence by heating up the sulphured juice, for thereby inversion is inevitable. In the second place, in sulphuring thin- or thick-juice a precipitate never forms, so that the necessity of filtering after sulphuring is altogether absent.[1] But in the third place, as already pointed out, some of the inorganic sediment dissolves from the thick-juice in the sulphurous acid, one of the chief reasons for filtration being then annulled.

These inorganic constituents, consisting principally of calcium phosphate, sulphate, sulphite, and silicate, in part dissolve in an excess of sulphurous acid to their acid salts. If now in factories which sulphur before filtration, and in which by means of low-pressure filters a very satisfactory thick-juice is obtained, it must not be forgotten that the re-solution has a greater share in the brightness than the filtration which conveniently could have been omitted.

Especially in factories, in which from time to time after-products always giving much inorganic sediment are remelted, there is great danger of deteriorating rather than of improving the composition of the juice in this way.[2]

In this process of juice purification the sulphitation of the thick-juice should give the maximum decolorization, which can be ascertained in the simple manner described under carbonatation, viz., by adding a drop of acetic acid

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[1] *Archief*, 1909, 64. [2] *Archief*, 1909, 733.

to the sulphured thick-juice, and observing if any further decrease of colour takes place.

Regarding the working and construction of the sulphur ovens giving the sulphurous acid for raw, thin- and thick-juices, little can be said here, these being matters of a purely technical nature. The principal thing is always to take care that there should be no sulphuric acid present in the sulphurous acid, which may be the case if the gas is wet. The ovens, which possess a cooling mantle, should be regularly tested for leakages.

c. Corrosion.

Finally, it must here be pointed out that the danger of corrosion in thick-juice sulphitation exists just as in thin-juice sulphitation. The vapours, which during the boiling of thick-juice escape to the condenser, are in this case also strongly contaminated with volatile organic acids, but especially with free sulphurous acid (sulphuring having been carried to a distinctly acid reaction to litmus). In the long run the steam piping must suffer.

It is remarkable that the *juice* itself with its strongly acid reaction has comparatively little corrosive effect on the iron piping and the coils, so that these can remain in use many years without having to be replaced, which fact has already been pointed out in connection with the concentration of the thin-juice. On the contrary, the corrosion which is effected by the acid *vapour* is distinctly noticeable, particularly in the tubes of the evaporating apparatus.

The best thing to do is to smear well internally all the steam piping, and also the domes of the pans, with ferric oxide, all the juice-catchers being extra carefully treated in this way. Naturally it is of the greatest importance to treat the still new and unattacked parts directly, for as soon as the iron surface has lost its original brightness, corrosion proceeds much more readily.

In the vapour from the pans there are present sulphurous acid, steam, and small traces of oxygen. Consequently, the possibility of the formation of sulphuric acid by oxidation is very great. In the juices, under conditions of reduced pressure, there is no oxygen, or, at any rate, it is considerably less—hence the great difference in corrosive power between acid juice and acid vapour.

Yet more marked is the danger if the factory is installed with a central condenser. If, above, we spoke of traces of oxygen in the ordinary condensation apparatus, this is wholly changed in central condensation, for every time that a vacuum pan is disconnected, large quantities of air pass through the central steam piping.

Although under normal circumstances this only lasts a few minutes, the situation from the standpoint of corrosion has been rendered yet worse by the addition of the so-called "circulation valves" by which the vacuum does not every time sink suddenly as a pan is disconnected, but declines slowly and regularly. In this way the duration of the entrance of air is so much longer, and the danger of corrosion becomes proportionately greater.

Nevertheless, from the technical standpoint, these "circulation valves" are indispensable, so that for all these reasons one must pay attention to the coating of the steam piping, etc. If the opportunity has not been seized to make a good protective layer while the iron is still new and bright, then it is necessary before putting on the ferric oxide, first to remove from the surface all rust and other impurities as well as possible by means of steel brushes and the like.

The slightest unevenness on the surface of the iron may give rise to the condensation of small amounts of vapour, which may there remain a very long time. In this moisture sufficient sulphuric acid forms slowly to cause a strong local pitting. But if there is no collection of moisture no trouble in this respect (worth considering) will be experienced.

For these reasons the juice-catchers always suffer the most heavily in the factory. It is their task to hold back moisture, in fact, to catch it, and on examining their interior after the termination of the campaign, it will then always happen that they are the most attacked, particularly when no care has been taken to regularly draw off the juice. By drawing off the juice, a liquid is obtained which always reacts strongly acid, and contains large quantities of iron. Sheet iron should never be used, but only cast iron.

The painting of the iron parts of the condensation apparatus must be repeated each year, and without waiting from campaign to campaign before proceeding to the treatment with fresh ferric oxide, but carrying it out immediately after the conclusion of the season. To the juice-catchers double care must be devoted.[1]

The control of the corrosion of the pans, as well as of the tubes of the evaporating apparatus, boilers, effect pumps, etc., etc., is one of the most important duties of the technical staff of the white sugar factory.

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[1] *Archief*, 1909, 91.

V.

CENTRIFUGAL SYRUP TREATMENT.

As we have called the treatment of the thick-juice a "correction," so we may designate the treatment of the centrifugal syrup as an "after-correction." It has already been seen that in most cases such an after-correction will not be necessary if the juice purification and the thick-juice sulphitation have properly fulfilled their task.

Similarly in syrup treatment, we may speak of a mechanical purification or filtration, and of a chemical clarification by means of sulphurous acid or other chemicals. The "run-off" of the first massecuite is always very turbid, all the matter depositing during the process of boiling accumulating in this syrup. Subsidence is naturally of no avail in the case of these dense liquids, since days would then be required, at least if one did not dilute to a very great extent, an operation which would cause the re-solution of some of the sediment, and anyway would necessitate a very lengthy settling.

Only in carbonatation run-offs is filtration possible, for in sulphurous acid saturation this is precluded on account of the very great viscosity. It is, however, self-evident that this filtration cannot be neglected in carbonatation factories.

Carbonatation.

As was remarked under the treatment of the thick-juice, the installation for its filtration can be combined with that of the centrifugal syrups, thus serving a double purpose. Without extra cost of plant, it is possible in this manner to obtain brilliantly clear centrifugal syrups.

Filtration may be effected by any apparatus, but probably most conveniently by means of Danek filters, as ordinarily used in most carbonatation factories, a pressure not greater than 9–12 ft., and a temperature not exceeding 80° C. (176° F.) being applied.

An important point is that in the first runnings some sugar crystals may be present, being either fine grain escaped from the centrifugals, or else being formed by after-crystallization. Naturally, by heating to 80°C. (176°F.) a part will re-dissolve, especially if open steam is blown into the syrup. By returning the wash-waters from the syrup-filters to the unfiltered centrifugal syrup the necessary dilution is also obtained.

The filtration of these carbonatation centrifugal runnings generally takes place most conveniently at 80–75° Brix. Under some circumstances, as the grinding of special cane, further dilution may become desirable, but then it should not proceed lower than 70° Brix.

The syrup scum, which is very viscous and is greyish-black in colour, hardens well on the cloths of the filters, and after sweetening-off can readily be removed.

Since the filter scum of this concentrated liquor naturally contains a high sugar content, sweetening-off must not be neglected. This must be done with as little water as possible, otherwise there is a danger that the inorganic constituents in the dilute sugar solution may re-dissolve. Fortunately, this does not happen as easily as one might at first be inclined to think, probably because these impurities have passed over to the crystalline form.

As in the sweetening-off of the thick-juice filters in sulphurous acid saturation, this exhaustion of the scum must depend more upon time than upon a large quantity of water.[1]

Principally by this filtration much calcium sulphate and organic impurities, the latter making the syrups needlessly viscous, are removed. The filtered liquid is then brilliantly clear, and gives a very pure molasses sugar.

As to sulphiting the carbonatation centrifugal syrups, this need never take place, more especially if the acid thin-juice process, which checks the formation of dark-coloured decomposition products, has been applied.

If in the laboratory one endeavours to decolorize a small quantity of carbonatation centrifugal syrup with an excess of sulphurous acid, it will be seen that there is no lessening of the colour to speak of, which is a sufficient guarantee that sulphitation can be omitted. Should it happen sometimes that a decrease of colour does take place, then this is an indication that some mistake has been made during manufacture, as, for example, an insufficient sulphitation of the thick-juice.

Sulphurous Acid Saturation.

As has been remarked above, a filtration or subsidence of the syrup obtained by means of this method of purification is entirely excluded, which is a pity, because the turbidity is considerably greater than in the carbonatation centrifugal syrups. After what has been said on the same subject in dealing with the deposits in thick-juice, there is no need further to elucidate this. The syrups may, however, very well be eliminated and skimmed.

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[1] *Archief*, 1911, 1426.

There remains only decolorization as a possible means of improving these run-offs, although it should be remarked that generally in a well-conducted factory additional sulphitation has no further effect. This point can best be decided by now and then sulphiting a sample in the laboratory and examining the result. In factories, in which the syrups are either sulphured, or else the costly hydrosulphite is applied, it would be seen in this way that in most cases sulphuring gives no further bleaching. When there is any decrease in colour, this would probably not have happened had more lime been added in defecation, or had the sulphitation of the thick-juice been better.

In those cases, however, in which a decolorization by sulphurous acid is possible and necessary, the syrup should always be diluted before the operation to approximately the concentration of the thick-juice, *i.e.*, to 60°–65° Brix. The control of the operation then should take place in just the same way as with thick-juice, namely, iodometrically.

VI.

CURING.

While the differences between the two methods of juice purification originally were very marked, we have clearly seen that they become less the nearer the end of the factory process approaches its completion. If the differences between the treatment of thick-juice and centrifugal syrup respectively become smaller and smaller, in curing they almost cease to exist, and indeed in Java for white sugar manufacture the two processes in use are quite parallel. We may therefore in the remaining pages entirely abandon the former division into carbonic acid and sulphurous acid saturation.

Moreover, in this work, which treats specifically of white sugar manufacture, it is naturally our purpose to deal with nothing else in the section of curing than that which concerns the preparation of superior sugars, and then only in a summary way, the subject having been exhaustively discussed in detail by others more competent.[1]

In Javanese white sugar factories at the present time working is generally carried on in two different ways. Either only superior first sugar and superior molasses sugar are made, or else the whole product is obtained in one "jet." If the first-product is separated from the after-product, it is self-evident that it is possible to obtain a better quality sugar, and this in the long run benefits the reputation of the factory. As to the inferior sugar, the so-called "molasses sugar," this goes to quite another market.

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[1] See PRINSEN GEERLIGS' "Cane Sugar and its Manufacture" (NORMAN RODGER, London). Pages 244-276.

If all the sugar be obtained together, it is inevitable that the quality should suffer more or less according to the special circumstances in which the factory is placed. Although this latter method is thus simpler and more inviting, the majority of factories till now have not pronounced in its favour, but have confined themselves to the former procedure.

Naturally, when after-products for some cause or other fall considerably in price, or are no longer popular, a change accordingly is made. In such cases *force majeure* may compel all the sugars to be obtained in one jet. Undoubtedly it is best if a factory is installed according to this latest method, but does not apply it except when the conditions of the market allow.

As to the operation of the methods designated here, this is as follows :—In obtaining sugar in two jets, all the thick-juice is entirely boiled to a primary massecuite in such a manner that the "run-offs" from it shall have a purity of about 65°. These run-off syrups are now worked up by slow boiling and lengthy cooling to a molasses of about 33° quotient of purity.

When the initial purity is high, the sugar is separated in three stages, viz.: (1) First sugar, with a run-off of 70°; (2) superior molasses sugar, with a run-off of 55°; and (3) molasses sugar. This last sugar is spun white simultaneously with the superior molasses sugar, and mixed with it.

In this way one obtains a very sharp separation of first and second products. The covering syrup of the first-product, in carbonatation as well as in sulphitation, is best returned to the thick-juice, to be subsided or filtered with it. This filtration is indeed of considerable importance, since first-product crystallizes from the covering syrup, and one is thus under the same obligation as with ordinary thick-juice. Although now and then the covering syrup may be sulphured, it should be recognized that in many cases this is needless trouble, because in a well-conducted factory this syrup, even less than the first run-off, shows little effect, but also because so long as these covering syrups remain unfiltered one runs the danger of re-dissolving inorganic deposits, the sulphuring then achieving an "apparent" success. The covering syrup of the second product goes back to the first run-off.

In the method of making white sugar and molasses, the purity of the thick-juice is continually lowered by mixing in the first run-off. Generally, it is best to work with a mixture having a purity of 78°–80° when the resulting massecuite after machining gives a run-off of about 58°–60° purity. As above, the run-off is worked in one jet to molasses, obtaining so-called "molasses sugar," which, however is not machined white as before, but is pugged and drawn into the first sugar pans. The pugged grain is diluted in the pans with some mixed thick-juice, and thus forms a *pied-de-cuite* for the first sugar massecuite.

Such a first massecuite sugar consists of molasses sugar as the primary grain, and is boiled with a mixture of syrup and thick-juice having a purity of about 78°–80°. The remaining run-off once again gives molasses sugar, etc.

According to this method of operating, in which a very good quality of product has been abandoned for the benefit of the rendement in first-sugar, the blank syrup is not returned to the factory, but is simply boiled directly.

The Quality of the Sugars.

In what concerns the quality, by obtaining sugar in two jets (first and molasses sugar) often but little difference can be seen in the appearance of the first-product from carbonatation or from sulphitation, so long as cane that is not too dark has been ground, which is a *conditio sine quâ non.* As to the molasses sugar, that from carbonatation factories can frequently be made quite undried and with a polarization of 96·5° without the least difficulty, but if the molasses sugars from sulphurous acid saturation be thus taken wet from the centrifugals, trouble is then experienced, the product being too moist, too viscous, and too grey. Hence we see confirmed what has already once been previously remarked, viz., that the difference in the juice purification is principally evident in the after-products.

Molasses sugar from sulphitation factories is therefore generally dried and afterwards sent to a disintegrator, which so subdivides the grain that a distinct decrease in colour is to be observed. The difference with the carbonatation sugar is only made evident in the rendement, which is naturally lower as the consequence of its higher polarization, and the losses that are unavoidable in the drying of so fine a substance.

In the method of preparing white sugar and molasses the quality naturally depends in the first place on the colour of the cane variety ground, and then on the initial purity of the raw juice. The lower this is, the greater will be the syrup per unit of cane, and therefore the greater will be the run-off and molasses sugar to be worked up in the massecuite of the first-product; or in other words, the more inferior will be the colour. In the third place the glucose content in the original juice is a factor which is not entirely without weight.

Since all sugar is obtained at once, a mother-liquor containing much glucose always betrays its presence by the inferior lustre of the product. Although there may have been the greatest success in its manufacture, and the colour be not unserviceable, a defective lustre will often be clearly apparent.

As a rule, to the question of the initial purity, which is directly connected with the glucose content, too little attention is given. In addition to accidental circumstances, as, for example, weather conditions, the nature of the soil, or the cane diseases, that is, circumstances which can scarcely be altered, the glucose content is, to a large extent, also dependent upon factors which can certainly be controlled, namely, sufficient ripeness and the choice of the cane.

The Choice of the Cane Variety.

In the choice of the cane variety, as a rule one goes to work "commercially," but not always economically, at least not in white sugar manufacture, which is naturally here discussed, to the exclusion of other matters.

As a principal factor among many subsidiary ones, account is taken only of the total rendement of the varieties, and of the juice composition, so far as it concerns the density. One should, however, not forget that in white sugar manufacture, besides other factors, an especially important one should be considered, and this is the purity of the raw juice.

If, *e.g.*, a juice has a purity of 90°, this means that on 100 parts of dry substance, 10 parts of non-sugar are present, and if the purity amounts to 85°, on 100 parts of dry substance there will be 15 parts of non-sugar. It is thus seen that within quite narrow limits the purity can increase the percentage of non-sugar (which is what governs the quantity of sugar) by 50 per cent. With a purity of 80°, for example, the quantity of non-sugar is even 100 per cent. greater than with one of 90°.

As a matter of fact, some modification is always caused by the juice clarification, especially in carbonatation; but though this may be so apparently, in actuality generally the non-sugar removed from the raw juice by lime is not absolutely proportional to the original purity, the non-sugar coming principally from unmodified glucose and other specific organic compounds.

If, thus, a variety of cane generally producing inferior juices (and perhaps giving a high yield per acre) be chosen, in grinding this a very considerable increase in the centrifugal syrups will be noticed, in other words, under like conditions, such juice gives a greater percentage of after-products than juice of high purity.

This should naturally be nothing, if one adhered strongly to the only good white sugar standpoint, namely, to give no other first-product than that obtained exclusively from unmixed primary massecuite, all after-products being obtained as such. As a rule, this by no means happens. So soon as the proportion between first and after-product is unfavourable, one endeavours on the contrary (as above elucidated) to improve it in numerous ways.

The cause on the one side is that the factory is too often controlled according to this proportion; but on the other side, and it is the more important, that the market for the after-products is smaller than that for the first-products.

Now it is wholly impossible to improve the above-mentioned ratio between first and after-products without injuring the quality of the sugar. However this may be realized, either by re-melting, by drawing-in syrup, or by grafting grain into the pans, it always is reflected in the colour or the lustre of the first sugar. In time this is economically dangerous. The idea of obtaining the whole or almost the whole of the yield of sugar from the cane at once, which originated in Java, and which unfortunately the manufacturers of high-class white sugar do not abandon, must principally be ascribed to the low purity of the Javanese raw juice, in which no careful selection of material, or of seed, can bring about any betterment.

From the above, the conclusion must be drawn that for white sugar manufacture cane varieties having pure juices are more economical in the long run than those with impure juices. In the usual calculation of the productiveness of different varieties of cane, when the financial values do not diverge too far, that variety should certainly be chosen which promises the purest juices.

Practical Hints.

The following hints may be found of service in working up white sugar:—

Boiling.—It is not sufficient to prepare sugar with a good colour and lustre, but one must always take care, in the first-product at any rate, that the grain is not irregular and especially not too small. This is a fault to which much so-called "superior sugar" is liable.

For this reason endeavours must be made in the first place not to make the thick-juice too dense, since this renders difficult the boiling of large and regular grain. A density of 27·5 Bé. is sufficient. It should, moreover, not be forgotten that an irregularly grained massecuite machines difficultly, and demands more water and steam for covering than coarse regular grain, thus finally giving more covering syrup, so that the fuel advantage of the more concentrated thick-juice most likely disappears. Again, as we have already said above, for a good subsidence the thick-juice should not be too concentrated.

In the second place a coarse grain is attained by connecting all pans together by means of suction pipes. Thus generally more profit is drawn from the available warming surface of the vacua. This drawing-over of portions of massecuite to form a *pied-de-cuite* for the following massecuite is almost indispensable for a white sugar factory.

Centrifugal syrup.—In continuous returning of covering syrups difficulties have never been experienced. In case one also returns the run-offs themselves to the thick-juice (in anyway whatever) it is essential to liquify the syrup now and then. In time the power of centrifugalling the massecuite suffers thereby.

Machining.—Without doubt it is desirable, especially in white sugar manufacture, in centrifugalling to well fractionate the first as well as the molasses sugar massecuite, so that a complete separation of syrup is obtained. The first fraction should always be machined as dry as possible, while the second is centrifugalled so long as the run-off covering syrup shows a complete decolorization, as controlled by means of a test tube.

The first-product is covered by means of well-dried steam, and then does not require to be dried further. Indeed, as it comes from the machines the sugar feels still quite wet, although the moisture disappears spontaneously on cooling in the air. This cooling is brought about by shaking gutters, sieves, etc. But in no case may the sugar while still warm be placed in the bags, otherwise it will cake together.

The after-product is usually covered with water alone, for which an appliance capable of easy regulation is necessary. In some factories it is sent off directly while still wet; others dry the molasses sugar in the machines with some dry steam; while finally there are many which are compelled by the quality of the product to pass it through a disintegrator.

Blueing.—In what concerns the blueing of the sugar, this must always take place in the centrifugals, since there the colour is much more plain than in the pans. The use of an acid-fast blue colouring substance, indanthrene for example, in the thick-juice or massecuite has now been abandoned for some time past.

INDEX.

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