

THE PRESERVATION OF CITRUS FRUIT JUICES*

THE following memorandum has been compiled on the basis of data supplied by the following sources: The Department of Science and Industrial Research, The Imperial Institute, The Research Station, Long Ashton, and Dr. L. Lampitt, Chief Chemist of Messrs. J. Lyons and Company. Grateful acknowledgment is made of the assistance afforded in this connection.

In the United Kingdom there is an appreciable trade in the so-called Citrus Squashes and Cordials, which, it may be pointed out, are not simply natural fruit juices but juices which have undergone some form of manufacturing or preservative treatment. In the United States of America, and to a lesser extent in certain European countries, there is a considerable trade in fruit juices which are preserved in such a way as to retain unaltered the characteristics of a fresh fruit juice.

In the United States a great impetus has been given to this trade by the medical profession who have advocated the consumption of fresh fruit juice as part of a campaign for health on account of the contents of vitamins. In consequence much attention has been devoted to the preservation of citrus juices by methods which will retain in unaltered form the vitamin contents.

This campaign has as yet not found its parallel in England; it is thought that if similar propaganda was undertaken by the medical profession in this country it would greatly stimulate a trade in fresh citrus fruit juice, and might be the means of extending considerably the consumption of citrus fruit or its equivalent in juice. At present the public has not learned to differentiate between the orange and lemon squashes which are currently sold and fresh fruit juice, and it is believed that the majority of people consume these beverages in the belief that they are actually consuming fresh fruit juice.

THE EXISTING TRADE IN CITRUS SQUASHES AND CORDIALS IN GREAT BRITAIN

Orange, lemon, grapefruit and lime squashes and cordials are fairly extensively consumed in this country and are manufactured by a number of firms. The basis of these preparations is the juice of the fruit which is imported into this country from the country of origin, usually in chestnut-wood casks which are not infrequently specially lined.

Orange juices are imported from the United States, Spain, South Africa and Jamaica. Lemon juices come from Italy, Sicily and the U.S.A., and lime juices from the Gold Coast and the West Indies.

*By H. A. Tempany, C.B.E., D.Sc., F.I.C., Assistant Agricultural Adviser to the Colonial Office, in *Bulletin of the Imperial Institute*, Vol. XXXVI., No 3, July-September, 1938.

Lime and Lemon juices containing relatively high percentages of citric acid are frequently imported without addition, but orange juice with a lower citric acid and higher sugar content is liable to undergo fermentation during transit and has accordingly to be treated with a preservative; sulphur dioxide is usually employed for the purpose and is added to the juice in the form of potassium metabisulphite. The amount added is in the region of 700 to 800 parts of sulphur dioxide per million. Orange juices imported in this way are usually of a pale yellow colour, thick with pectinous matter and smelling and tasting strongly of sulphur dioxide.

To bring the sulphur dioxide content of orange juices, as imported, within the limits prescribed by the Foods and Drugs Act they require to be diluted to about one-third their original concentration; the diluent employed is usually syrup containing 45 per cent. by weight of sugar; to increase the acidity citric acid may be added, whilst sometimes flavouring is added also. The characteristic deep orange colour of orange squashes is obtained by adding colouring matter—usually carotene is used. In the squashes the pectin remains in the juice.

In the clear cordials the pectin is allowed to settle and the clear supernatant juice is racked off, though sometimes filtration is resorted to.

PRESERVATION OF CITRUS JUICE IN A FRESH CONDITION WITHOUT THE ADDITION OF PRESERVATIVES

The preservation of citrus fruit juice in a natural condition is by no means a simple problem. Such juices, and particularly orange juices, are liable to undergo changes on storage which include not only ordinary fermentation, but also oxidation processes which result in loss of flavour and of colour. Moreover it is not possible to treat them by sterilization or even normal pasteurisation as this results in imparting to them a cooked flavour which is unpleasant and also destroys the vitamins. Changes are also liable to occur in the pectinous material; on long keeping it tends to flocculate and precipitate in an undesirable manner; while in any case it is essential that in orange juices the pectinous material should be retained as its removal also causes a certain loss of flavour.

Five methods have become evolved for the preservation of fruit juices under conditions which fulfil in a greater or less degree the above requirements. They are as follows:—

- (a) Preservation of fresh fruit juices by simple storage at low temperature.
- (b) Preservation of fruit juices by "flash pasteurisation" and canning of the product.
- (c) Concentration of fruit juices by film evaporation under reduced pressure.
- (d) Concentration of fruit juices by freezing.
- (e) Treatment of fruit juices by the "Matzka" process.

In the following pages some details are given concerning each of these.

PRESERVATION OF ORANGE JUICE BY STORAGE AT LOW TEMPERATURES

The preservation of orange juice by freezing was first attempted on a commercial scale in Florida in 1931. Early operations were not very successful. Part of the trouble lay in the development of an "off taste" in the frozen juices. Subsequent experience has, however, made it possible to overcome these difficulties and the process is now extensively practised.

The essential features of the process are as follows :—

The juice is extracted either from the whole fruit by means of a high speed reamer or by a cup type press or a whirl type press, using peeled fruit. After extraction the juice is run through a strainer and thence to an evacuating apparatus which removes contained air. Thereafter it is transferred direct to the containers and is stored in refrigerated rooms at temperatures which range from 32° to 42°F. It has been found that flavour and appearance are considerably affected by the methods of extraction and it is necessary to avoid the inclusion of too much essential oil and also bitter principles from the skin. The de-aeration treatment is also very important as this affects the keeping qualities of the juice. If de-aeration is omitted juices are liable to darken in colour and go off in flavour as the result of oxidation.

Orange juice treated in this way can be preserved for several weeks in a fresh condition. A very large trade has sprung up in the United States of America in this type of juice during the last few years ; it is largely in the hands of cold storage companies and dairies. The juice is commonly stored and delivered in cardboard containers and it is normal in American cities for a carton of orange juice to be delivered at the doors of households with the morning milk.

PRESERVATION OF FRUIT JUICES BY FLASH PASTEURISATION AND CANNING

The flash pasteurisation method for preserving fruit juices consists essentially in raising the temperature to 185° to 190° F. and maintaining it at that for about ten seconds ; treatment in this way gives results comparable to heating to 160° F. for 30 minutes. The advantage of this method as opposed to ordinary pasteurisation is that as the juice is exposed to the high temperature for a very short period the development of the cooked flavour, which is an undesirable concomitant of heat treatment, is avoided.

As in the case of the preservation of fresh juices by cold storage the pasteurisation process must also be accompanied by de-aeration if successful results are to be obtained. It is also usual to combine with them treatment of the juice with a pectin destroying enzyme which enables the opacity of the juice to be controlled without affecting the flavour to the extent that would occur if the pectinous constituents were removed by filtration. Various enzymes of this nature are now marketed by a number of firms under different names. The enzyme is added to the juice after extraction and before de-aeration and pasteurisation, which have the effect of destroying the enzyme, and sufficient interval is allowed to enable the enzyme to perform its functions before the subsequent processes are proceeded with.

In the production of pasteurised citrus juices very careful selection of the fruit is essential and all damaged and partially decayed fruit must be rigorously excluded. The methods of extraction employed are also important, for unsuitable methods of extraction are liable to affect, as with cold stored juice, the flavour unfavourably. A method extensively employed is to halve the fruit mechanically and to hand-spindle the half sections over revolving burrs. Attempts have been made to crush the whole fruit in its natural state, but this method has been discontinued on account of the strong flavour imparted to the extract by the oil contained in the skin. This flavour is regarded as objectionable.

An outline is given below of a method of preparing the juice employed at an American factory which embodies the Stero-Vac process of flash pasteurisation which is claimed to be one of the most efficient for producing this type of product.

The fruit is first grated to remove the oil and then pressed whole, the juice being strained to remove seeds and pulp. The grater consists of two horizontal revolving discs about 4 feet in diameter, covered with a stainless steel fillet, which rotate at a speed of about 100 r.p.m. and revolve in opposite directions. The fruit is thrown by centrifugal action against the fillet, which punctures the oil cells. The fruit leaves the outside of the first revolving disc and is transferred to the second disc where the grating is continued. At the centre it drops into a continuous press consisting of two discs of stainless steel about three feet in diameter, which revolve in the same direction and come together for a short distance on one side. The whole fruit rolls from the grater into one side of this, is crushed and the juice extracted. After pressing, the crushed peel is lifted off the disc and the juice flows into a stainless steel trough which surrounds the lower disc. It then flows to a finisher which is a mechanical strainer of stainless steel. From the finisher the juice flows to the de-aerating unit which consists of a steam chest with a separating chamber and a condenser, all constructed from stainless steel. Juice flows through the pipes of the steam chest—which are surrounded with hot vapour—under a high vacuum of about 28 inches. Juice flows continuously from the extractors through the finisher and the de-aerating unit and is pumped out by a stainless steel vacuum pump to the can filler, which is so constructed that the juice may be broken to atmospheric pressure in an inert gas such as nitrogen.

For packing grape fruit juice plain tin cans are used, but with orange juice lacquered cans are employed in order to avoid flavour changes. After filling the juice in the cans, it is flash pasteurised by the Stero-Vac process. This process involves heating by steam injection through a patented valve in the end of each can and is performed on a specially designed machine. As the can is removed from the machine the disc in the valve is snapped into place as the result of the change in pressure; it is subsequently sealed by clinching and the cans rapidly cooled. The essential features of the process are quick heating and quick cooling combined with the removal of dissolved air.

Another method of flash pasteurisation has been devised at Long Ashton, where a series of experiments have been carried out on apple juice. In this method flash pasteurisation is accomplished by causing juice to flow as a thin film

between two metal sides of a container, which are raised to the temperature of boiling water. The juice is de-aerated subsequently to this and then canned. The Long Ashton authorities have kindly undertaken to carry out tests with this apparatus on samples of citrus fruit juice from Palestine.

CONCENTRATION OF FRUIT JUICES IN VACUO

There has been a not inconsiderable development of the concentration of fruit juices in vacuo in recent years. The principle employed is that of film evaporation which has been commercially developed in a variety of industries in the Kestner type of evaporators. The advantage of the process is that as evaporation takes place from the surface of a thin film it proceeds very rapidly while the high vacuum under which it is performed permits of the employment of a relatively low temperature.

In this way, as in the flash pasteurisation process, it becomes possible to avoid the occurrence in fruit juices concentrated by means of it of the cooked flavour, which is objectionable.

In the process as applied to fruit juices it has been found necessary to make special provision for retaining certain volatile substances which affect the flavour of the finished product and which are removed during the course of evaporation. This is accomplished by the incorporation in the plant of a special device whereby these substances are trapped and condensed, thus permitting of their readdition to the finished product.

A number of plants are engaged on the commercial operation of this process in the United States and a number of brands of concentrated orange juice prepared by the process are on the market. One of the best known is called "Califorange". There is already a small import of this type of product into the United Kingdom, where it is finding increasing favour with manufacturers of orange squashes and cordials.

In operation it is understood that after extraction of the juice some of the pulp may be removed by filtration or by treatment with enzyme in order to obtain a product that is not too viscous and difficult to handle. Such concentrated juices contain about 60 to 70 per cent. total solid matter and it is stated that they can be kept without change at ordinary atmospheric temperature indefinitely. It is understood that the preparation of pure concentrated orange juice by the process is sometimes difficult by reason of the low acidity which affects the keeping properties and that a trade is in consequence springing up in mixtures of concentrated orange and lemon juices, the higher acidity of the lemon juice enhancing the keeping powers of the mixture.

It is understood that one small factory in Palestine has experimented with the concentration of fruit juice in vacuo.

THE CONCENTRATION OF FRUIT JUICES BY THE METHOD OF FREEZING

Possibly the most interesting of all the various processes for concentrating fruit juice is that of concentration by freezing. The process depends on the application of the well-known principle that when a solution is cooled below

the freezing point of water separation into two phases occurs, a solid phase consisting of pure ice crystals and a liquid phase consisting of the original solution in a more concentrated condition. Theoretically, therefore, it is possible to effect concentration to any desired degree by freezing the solution and then separating out the ice crystals. The process has seen its most important commercial development in Germany under the title of the Krause Process, from the name of its inventor, G. A. Krause, of Munich. There are six or seven examples of the plant at work in Germany and Switzerland. A freezing process has also been developed in France with special reference to the concentration of wines and grape juices. A certain amount of experimental work on the method has been performed by the Department of Scientific and Industrial Research at the Low Temperature Research Station at Cambridge and by the Daniel Sieff Research Institute in Palestine.

It is understood that the process has not so far obtained any commercial application in the United States of America, but the operations of the Krause concern are being watched very closely with a view to possible industrial development.

In its original form the Krause process consisted essentially in freezing the juice in some form of suitable vessel and then transferring the frozen mass to a centrifugal in which the concentrate was separated from the ice crystals by centrifuging. Worked in this way the process has certain obvious disadvantages; in the first place it is discontinuous; a further and more serious objection is that by freezing alone it is only possible to effect concentration up to a total solid content of 55 to 60 per cent. Concentrated to this extent fruit juices are incapable of being preserved unchanged at ordinary atmospheric temperatures and require to be stored under refrigerated conditions if they are not to develop undesirable characteristics, such as darkening in colour, change of flavour, and alterations in the pectinous constituents which cause them to flocculate and precipitate out very rapidly.

Samples of orange juice concentrated in this way and stored at -20° C. for over a year were seen at Cambridge and were found on dilution to have preserved their flavour very well indeed and to correspond exactly with fresh orange juice. It is obvious that the necessity for storage at low temperatures is a serious drawback, and it has more recently been stated that if higher concentrations of total solids can be obtained this drawback is removed and juices will keep unchanged at air temperatures. It is considered by Krause and others that concentrations of between 60 and 65 per cent. are reasonably safe, but the susceptibility of concentrates to change is a function of the acidity and juices of low acidity require a higher degree of concentration than do juices with higher acidity if they are to maintain their condition at air temperatures.

The acidity of citrus fruit juices, is, however, in their favour in this respect.

One of the difficulties in the way of securing higher concentrations than 50 per cent. is that at greater concentrations the concentrates become viscous and do not part readily from the ice crystals. One method of getting over

this difficulty would appear to be pretreatment of the juices with enzyme to dissolve some of the suspended pectinous material, thereby lowering the viscosity of the finished product.

It is stated that Krause has now perfected a modification of his original process whereby all these difficulties have been surmounted and fruit juices concentrated up to 80 per cent. total solids content, while the process has in addition been made continuous. Through the courtesy of Dr. Lampitt of Messrs. J. Lyons and Company, samples of raspberry and apple juice concentrates stored at air temperature were seen and it was stated that they had been concentrated up to 80 per cent. total solids content. They appeared to have retained unchanged the aroma and flavour of the original juices. A factory incorporating all the latest innovations is stated to be operating in the Rhineland, but details of the modified Krause process are at present jealously guarded. For what it is worth, I may say that it is believed that the modified Krause process incorporates the principles of pretreatment with a pectin destroying enzyme, preliminary concentration by freezing, followed by final concentration by heat under reduced pressure, but no certainty can attach to this.

It is believed by a number of authorities that ultimately the freezing method or a modification thereof will supersede all other methods of concentration of fruit juices.

It is understood that representatives of the Krause concern have been appointed in the majority of countries.

THE MATZKA PROCESS

The Matzka process is so called after its original inventor Dr. Wincenty Matzka.

The process seems to depend upon a combination of low temperature flash pasteurisation and metallic silver sterilisation, the liquid being passed in thin layers between two heated metal surfaces. The temperatures attained by the juices are, however, lower than those usually considered necessary for pasteurisation, actually temperatures of from 130–140° F. are employed, and the sterilising action is claimed to depend on the so-called oligodynamic action of the metal with which it is in contact. Moreover, the two metal surfaces are different and electrically insulated from one another so that some electrolytic action is supposed to take place. A certain amount of the metals goes into solution, and the possible effect of this on human health has been questioned.

The process has been tested experimentally by the Ontario Research Foundation and a report on these investigations has been published under the signature of Dr. A. Douglas Barbour, Head of the Biochemistry Department.

In its simplest form the apparatus consists of two concentric tubes, the inner one being of silver and the outer of stainless steel; heat can be applied to the inner surface of the inner tube and the outer surface of the outer tube while the juice under treatment flows through the space between the two tubes. Juice intended for treatment requires to be de-aerated as in ordinary

flash pasteurisation, and may be treated with an enzyme or filtered to clarify it. After treatment, the juice is filled direct into bottles with suitable arrangements for sterilising them so as to prevent after-infection.

Various juices were treated experimentally in the Ontario trials and uniformly satisfactory results were reported. Among the juices treated were orange juice and grapefruit juice, and it is claimed that the products, as well as keeping satisfactorily, compared very favourably with commercial samples of similar juices which had been prepared by the ordinary flash pasteurisation process. Commercial plants are stated to be operating the process at Tiel in Holland, at Tremestieri in Sicily, at Carcagente near Valencia in Spain, at Cheswold, Delaware, in the U.S.A., and at Whitby, Ontario, in Canada.

It is further stated that samples of Matzka processed juice have been examined by officers of The National Research Council of Canada, who were satisfied that it had a superior and more natural flavour than any other processed fruit juice sampled. It is further stated that the concern at Whitby, Ontario, which was inaugurated in November, 1937, has had difficulty in keeping up with the demand for its products.

CONCLUSION

To sum the matter up, the present trade in citrus juices in this country is mainly concerned with the orange, lemon, and grapefruit squashes and cordials which are extensively sold. These are, however, far removed from natural fruit juices, and it is believed that if a regular supply of fruit juices preserved by more modern methods became available they would rapidly replace the existing types.

There seems little doubt that the existing demand by the trade in this country is for raw juices rather than for concentrated juices. This is doubtless because the unconcentrated juices at present suit methods of manufacture. If, however, by freezing or other method of concentration the trade could obtain a concentrated juice offering technical or commercial advantage over the present raw juice they would doubtless resort to this material. That there is increasing interest in concentrated juices is shown by the fact that small quantities of juice concentrated by the vacuum process are already coming in.

There also seems reason to believe that interest in fruit juices is being fostered by the milk bars, which are appearing in increasing numbers, at most of which there is a growing sale for fruit juices, especially in the summer.

There also seems little doubt that consumption of citrus juices would receive a considerable impetus if it was as extensively advocated by the medical profession in the United Kingdom as it has been in the United States of America. The great extension of the consumption in the latter country is largely attributed to this factor.

It is not altogether clear which of the processes of preserving citrus juices described offers the greatest prospect of success, *i.e.*, (a) extraction of fresh fruit juice, its de-aeration and preservation by cold storage for short periods, (b) the preservation of fruit juice by flash pasteurisation and de-aeration followed

by subsequent canning, (c) concentration of juice in vacuo by a film evaporation process, (d) concentration by freezing on the lines of the Krause process, or (e) preservation by the Matzka process.

The Krause process of concentration by freezing is thought by some to offer the best prospects in the long run. On the other hand, if the Matzka process fulfils its present promise it also seems to hold out possibilities, but the objection to this process by reason of the presence in the treated juice of small quantities of silver must not be overlooked.

The Department of Scientific and Industrial Research have pointed out that there is no possibility of patenting the actual process of concentration by freezing *per se*. The only points over which patent rights can extend are the details of the apparatus employed.

In conclusion it may be pointed out that while the foregoing information applies in the first instance to the preservation of citrus fruit juices it also has a direct bearing on the preservation of juices of other kinds of fruit. In the Colonial Dependencies interest in this connection at present attaches particularly to the preservation of pineapple juice in Malaya and certain other Dependencies and to passion fruit juice in Kenya; consequently although the application of the data given lies in the first place in those Dependencies in which citrus fruit is grown, it also has an interest for a number of others.

ADDENDUM

FURTHER INFORMATION CONCERNING THE KRAUSE PROCESS

Since the foregoing was written further information concerning the Krause process has been made available in a paper by P. Bilham published in *Chemistry and Industry*, Volume 57, No. 25, pages 589-593, of June 18, 1938, and from this the following additional notes have been abstracted:—

According to Bilham the process in its present form consists in double or treble freezing in a stationary condition, transferring the ice block to a centrifugal, removing the mother liquor by whizzing, and subsequently concentrating the mother liquor by the same means.

The juice is frozen in a special vessel of such a shape and size that the moulded block of frozen material exactly fits the centrifuge employed, the general shape of the juice space is an annular ring which tapers slightly from top to bottom. The vessel is immersed in a brine tank and the brine circulates outside and inside the ring of the container thus ensuring that the temperature gradient is horizontal, which causes the ice crystals to grow along this gradient and so facilitates the separation of the concentrate.

A framework is immersed in the liquid to be concentrated, which provides a means of handling the frozen block and helps to prevent the frozen mass from disintegrating. The remainder of the plant consists of the brine tanks in which the brine is specially circulated for freezing, a second brine tank in which after a certain period of freezing the cell is allowed to "temper" (whereby

the temperature of the frozen mass becomes even throughout) and the centrifuge which is provided with a central pipe entering the bowl for the introduction of liquids to wash the ice mass. The whole plant is made of corrosion resistant alloys.

Two brine tanks are provided for freezing and one for tempering, there is one centrifuge, and auxiliary apparatus comprises refrigerating plant and thermostats with storage tanks.

In operation a cell is filled with juice and placed in the first freezing tank for an hour, it is then removed and placed in the tempering tank for an hour, which levels out the temperature throughout the mass. The cell is then plunged into warm water and immediately the content is free it is lifted on its frame, placed in the centrifuge and spun until concentrate ceases to issue from the discharge pipe. The mass is then washed with original juice and then with ice water from previously discarded ice. The washings are used again and again until the solid content has been raised nearly to the level of the first concentrate. They are then added to the bulk.

The process on the second and third concentrations is similar, save that lower temperatures are employed, while the ice from the last concentration is not washed but is added as it is to the original juice.

A cell is filled every twelve minutes and put to freeze ; during the interval cells due for centrifuging have to be handled ; ice removed from the bowl at the end of washing, transfers made from freezing to tempering tanks, and filling of second and third concentration cells made. The work is stated to be capable of being carried out by four men. The plant has a capacity of 1,250 litres of original juice per hour for continuous night and day work, the latter is essential for successful operation. With wages at 1s. 3d. per hour, electricity at 1d. per unit, and cooling water at 3d. per 1,000 gallons the prime cost of concentration is stated to be 1d. per gallon of finished product using three stages.

It is stated that the juices produced are excellent in flavour, but to ensure their keeping it is required that the soluble solid-contents be raised to 60 to 65 per cent. by the addition of sugar. With this they will keep well for six months at a temperature of 5° C. This is not in complete accord with earlier claims reported for juices concentrated by this process. It is also stated that the removal of pectin from concentrated juice by the use of pectin destroying enzymes has been proposed, but it is not clear whether this is as yet actually incorporated in the process.