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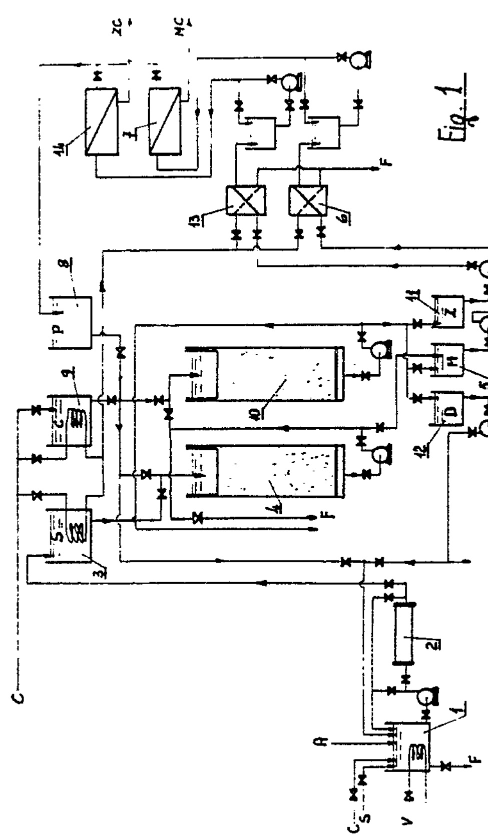
(54) Procedure and equipment for differentiated juice defecation in two resin packed ion exchange columns.

(57) The process and the related equipment are based on two resin packed ion exchange columns (4, 10) through which the juice (S) to be defecated is flowing.

In the first column (4) the juice is partially defecated through ion exclusion thus producing molasses (M) with a satisfactory commercial value, while the defecation process is completed in the second column (10) with separation of the non-sugar residue and recovery of various sugars and non-sugars having a high market value.

Treatment on two columns reduces processing time and/or the quantity of resin necessary as compared with one column processes. Polluting waste water is thus completely eliminated.



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This invention covers the separation of sacharose from juice impurities (whether non-sugars or sugars other than sacharose) with the aid of resin cation and/or anion exchangers and or the separation of different sugars (such as for instance glucose and fructose) in the mixed solution.

It is commonly known that the conventional method to separate sugar from molasses is based on vacuum boiling, followed by cooling of the massecuite and its centrifugation.

Subsequently, impurities are separated by resin packed ion exchangers.

Separation takes place through percolation of the juice through these resins based upon two main actions, i.e. ion exclusion and a physical action. Defecation by means of ion exclusion takes advantage of the properties of ion exchanging resins, which, immersed in a polar solvent, will not permit the ions contained in the solution to permeate the resin granules, while the latter will be easily permeated by non-ionic solutions such as non dissociated organic sugars and non-sugars.

The physical action is achieved by taking advantage of the resin property differentially to retain the various non-ionic molecules which are present in the juice flowing through the resin.

The technique called "chromatography" utilizes these above described two phenomena at the same time thus purifying the juice at a differentiated velocity at which the various substances in the juice to be defecated pass through the resin packing.

These techniques are, for example, described in the Italian Patents n° 560.795 and 601 035 as well as in the U.S.A.Patent n° 889.949.

This procedure, hitherto performed by one single column, has however some considerable drawbacks. First of all, the operation performed in one single column does not permit differentiation between the ion excluding and the physical action, so that defecation is not quite satisfactory.

Secondly, the molasses in which most non-sugars will be found, will have a low sugar content and will be strongly diluted. If a sufficiently pure juice is to be obtained, such molasses will be difficult to utilize and will have a very low or no commercial value, with an average 3% - 4% content of dry substance and 20% - 30% purity.

A further drawback lies in the fact that the single large sized column containing the resin requires a large amount of sparge water which, when recovered and recycled, will dilute the juice to the detriment of the concentration rate, or else water discharge will cause serious pollution problems and involve considerable purifying costs.

Finally, the utilization of only one large sized column packed to great height with resin and the possibility that this layer may be packed too tightly by the juice flow, thus leading to the formation of preferential paths, would negatively affect separation efficiency.

This patent has the aim to eliminate or minimize the above mentioned drawbacks.

The first objective is to keep the two actions, i.e. ion exclusion and the physical actions of the resins separated, so that juice defecation will yield a high purity juice and a commercial molasses having an SS ≧ 10 value and a purity ranging between 45% and 50%.

Furthermore, this invention has the aim to reduce the amount of sparge water necessary to eliminate polluting waste, to ensure a uniform flow rate of the juice through the resin, to homogenize the defecation process over the entire column section and, more generally, to cut the cost of the equipment and to reduce the quantity of resin required for purifying.

The above mentioned aims are achieved by this invention through the utilization of two sequenced, resin packed ion exchangers (or by two series of columns) of limited dimensions and/or containing a limited amount of resins.

The utilization of two successive columns will permit to differentiate their action. The first column through which the juice flows will provide for an initial but partial defecation. The juice is essentially defecated by ion exclusion and scanty physical action. The resulting molasses has a fair standard quality and a considerable Bx>10 concentration rate, 45% - 50 % purity, i.e. which much better characterics than molasses obtained from the single column system (Bx = 3-4; Qz = 20% - 30%).

When leaving the first column, the juice passes through the second column having an essentially physical purifying action to ensure further separation of non-sugars and/or of other sugars contained in the juice which will thus have a high purity.

At the outlet of the second column the molasses may be mixed with the molasses obtained at the outlet of the first column, or else it may be recycled for preliminary dilution of the entering juice, whereas other sugars and organic substances may be either regenerated and/or recycled.

When adopting two columns of limited dimensions and/or containing a limited quantity of ion exchanging resins, the cost of the equipment can be considerably reduced; the same holds also true for the operating cost since less sparge water and hence less dilution will be required, in relation to the processed juice. Polluting effluents are virtually nihil. As compared to the capacity of known single column installations, the adoption of two smaller sized columns having a smaller aggregate resin volume will not reduce the overall capacity because the juice and water require less permeation time.

According to this invention, the resin columns are fitted at their upper end with a removable loading or filling chamber featuring numerous juice distributors, whereas the columns are fitted at their lower end with properly perforated trays, the holes of which are vertically matching the distributors.

In this way, the juice uniformely passes through the resins, while preventing the resins from shifting and ensuring elutration of the resins.

The invention is illustrated in its practical and exemplifying implementation in the enclosed drawings, in which:

Fig. 1 shows the functional diagram of the system;

Fig. 2 shows the central vertical section of a resin column;

Fig. 3 Shows the separation diagram between sugars and non-sugars and/or other sugars in a single column system, according to the known methodology;

Fig. 4 shows the separation diagram with the first column according to the invention;

Fig. 5 shows the separation with the second diagram, according to the invention.

With reference to these figures, the collecting vessel S receives the juice to be defecated for instance to a re finer's syrup having 72% - 75% purity and 80 Bx. Condenser or vacuum pan water may C be added in this vessel to dilute the juice (for instance from 80 to 70 Bx) together with the additives A (fossil meal, etc.). The juice may be added steam heated V to a suitable temperature.

From the vessel 1, the juice is filtered on filter 2 and then reaches the vessel 3 fitted with a heating system for condenser water C. The latter vessel also provides for loading of the first ion exchanging resin column (4). The juice is defecated in the first column (4) prevailingly by ion exclusion. As a first step, the molasses is discharged in a sugar and non-sugar mixture having an SS ≥ 10 and 45% - 50% purity value. The partially defecated juice leaves the first column with an 83% - 88% purity. The molasses M is collected in vessel 5 and is then conveyed to the heat exchanger 6 and to the concentration device 7 for hyperfiltering and/or reverse osmosis on membranes. The concentrated molasses MC thus obtained can either directly be utilized for further processing or for further concentration and storage. The permeated water P reaches the vessel 8 and can then be used as sparge water in the columns or for dilution of the juice to be defecated, as an alternative or in addition to the condenser water C in tank 9.

At the outlet of column 4, the partially defecated (limed) juice is let into the second column 10 which is acting mainly physically.

A small quantity of molasses trickles initially from column 10 to be mixed with the molasses obtained from column 4 or else to be recycled.

Subsequently, the juice Z from column 10 will have a high purity (93%-95% at an average) which is let into the tank 11. Finally, various sugars and non-sugars will be discharged from column 10 to be collected in the fresh water tank 12 because of their fair commercial value, or else recycled.

The defecated limed juice Z is collected in tank 11 and conveyed through a heat exchanger 13, to a hyperfiltering and/or reverse osmosis membrane device 14, from which defecated concentrated juice ZC is obtained . The permeate is collected in tank 8.

The above described installation includes the pumps, valves, control and monitoring systems and whatever else is necessary for its completely automatic operation.

The possibility is envisaged to use two columns having different volumes, different resins having different grain size to optimize differentiated operation of the two columns based upon the composition of non-sugars.

Defecation of the juice as resulting in this plant is reported for exemplification purpose in fig.4 and fig.5 as compared with fig.3 which shows the result of defecation in a single column system.

In these graphs, the sugar concentration rate Z (continuous line), prevailingly ionic non-sugars (dashed line), various non-ionic sugars and non-sugars D (dotted line) are plotted on the y-coordinate, whereas the time at the column outlet is plotted on the x-coordinate.

As can be noted on fig.3, showing separation in one single column, the molasses M leaves the column after an initial time t', followed by the high-purity juice Z together with various sugars and non-sugars D.

The the low-purity molasses is diluted and therefore not easily marketable. In addition, defecation through one single column does not allow for efficient separation between saccharose and other sugars.

Conversely, according to the invention, the molasses M obtained during the initial period t' has a fair commercial value and it is followed by the juice Z conveyed to the second column.

In the second column, molasses separation occurs after a 't period, whereas the high-purity juice Z is separated between t' and t'', followed by various sugars and non-sugars D which may at least partially regenerated.

In resin-based chromatographic separation processes, the optium linear percolation speed of the liquids strained through the resin layers has a well defined value. It follows that the cycle time is univocally determined by this preset velocity and volume of the liquid to be percolated (which, in our case is the juice first and the water later), provided however that the liquid is percolated in one single column.

But if the column iis split into two columns having the same cross section but different height (for instance half of the initial column) and if not all liquid is percolated in the first column, the cycle time will be reduced proportionally to the liquid volume bypassing the first column and directly entering the second column and this in turn will proportionally increase the capacity of the plant (i.e. a greater number of cycles per day).

The above clearly shows that the adoption of two series-parallel connected columns makes it possible to increase the plant capacity, at equal installed resin volume, as compared with the capacity of single column facilities. This will considerably cut the installation costs and will also reduce the amount of sparge water to be recycled and evaporated.

According to this invention, their will be no waste water since the soft water (tank 12) as well as the permeated water coming from the hyperfiltering system (tank 8) will be recovered and used for preliminary dilution of the juice (tank 1) and for displacement in the column, thus reducing vacuum pan or condenser water consumption. Countercurrent discharge in the sewer will be occasional and in negligible quantities.

As explained above, a uniform resin level in the column and of the juice flow rate along the resin bed are essential for this process.

According to this invention, the columns 4 and 10 are fitted with a removable loading device 15, the bottom of which has distributor nozzles with levelled mouthpieces (16) so as to ensure uniform distribution of the liquid on the resins (17).

Two perforated sheets 18-19 are loacted on the bottom of the columns 4, 10, the borings of which are vertically matching the position of the nozzles 16, whilst a net 20 supporting the resins separates the latter from the perforated plate.

These devices ensure a uniform liquid flow as indicated by the arrows in fig. 2.

The tank 20 keeps the resins 17 flat on its bottom, preventing local accumulation and differentiated resin thickness.

All this permits a uniform permeation of the juice through the resin packing and a beter final defecation.

Furthermore, when the vessel is lifted, this configuration permits elutration of the resin, thus facilitating the operations while reducing idle time. The smaller grainsized resins are placed at the top where they can keep back most of the suspended impurities.

Recapitulating, the method and equipment subject matter of this invention offer the following advantages as compared with single column plants:

- separation of molasses having a fair commercial value;
- separation of a concentrated and high purity juice
- separation of various sugars and non-sugars having a high commercial value;
- reduced amount of elutration water
- integral recycling of soft and permeated water
- elimination of pollutants discharged in the sewer system
- greater capacity at equal installed resin quantities
- perfect and uniform permeation of the resin bed by the percolated liquids ;
- hence, considerable cutting of the installation and operating costs .

The process and equipment here described are uilized, as exhaustively described, for defecation of juice, for separation between sugars and non-sugars, although it may also be conveniently used to separate various sugars (such as glucose and fructose etc.) in mixed solutions.

Claims

1) Defecation processing of juice by means of ion exchange resins, **characterized by** the fact that the juice (S) passes through two series connected columns (4, 10) filled with ion exchange resins, so that the first column (4) prevailing provides for defecation by ion exclusion, whereas defecation in the second column (10) is prevailingly obtained by physical action, thus obtaining at the outlet of the first column (4) fair quality molasses (M) and juice conveyed to the second column (10) at the outlet of which subsequently low value non-sugar residues, high purity juice (Z) and recoverable sugars and non-sugars are obtained, and also including two concentration lines for hyperstraining of the juice on membranes, one of which for concentration of the molasses (MC) and the other (16, 14) for thickening of the juice (ZC), from which lines the permeated water (P) used for dilution of the juice (S) or for shifting the juice from the columns together with or in alternative of the condenser water (C) are discharged, so that the molasses (MC) thus obtained will have the desired concentration and marketable qualities, while the juice will have the required concentration and high purity and the various sugars and non-sugars will be separately recovered.

2) Procedure as described in claim 1, **characterized** by the fact that this process is not only used to separate sugars from non-sugars, but also to separate different sugars from each other, as for instance glucose from fructose in mixed solutions.

3) Procedure as described in Claim 1, **characterized** by the fact that the juice to be defecated

passes at the same time and in succession through the two columns (4, 10) thus reducing the defecation time and the required resin quantity.

4) Procedure as described in Claim 1, **characterzed** by the fact that resin washing water and the water permeated during hyperstraining are recycled without any polluting waste water .

5) Procedure as described in Claim 1, **characterzed** by the fact that the columns (4, 10) are backwashed.

6) Procedure as described in Claim 1, **characterzed** by the fact that the two columns (4, 10) may contain different resin types having differentiated grain size, because of their deferentiated purifying action by ion exclusion and physical defecation.

7) Equipment for the implementation of the procedure described in Claim 1, **characterized** by the fact that this equipment includes :

- a tank (1) containing the juice (S) to be defecated, possibly diluted with condenser water (C) and/or soft (D) and permeated water (P) corrected by additives (A);
- a filter (2) for straining of the juice (S) to be defecated coming from the tank (1),
- an other tank (3) containing the filtered juice (S) to be charged in the first resin column (4),
- a first column (4) packed with ion exchange resins to be permeated by the juice (S) for its defecation by ion exclusion;
- a tank (5) in which the molasses (M) coming from the first column (4) during an initial period (t');
- a second column (10) packed with ion exchange resins, receiving the juice coming from the first column (4) after elapsing of the (t') period for subsequent processing of this juice by a prevailingly physical action, while separating non-sugar residues from other sugars,
- a tank (11) in which the high purity juice coming from the second column (10) is collected;
- equipment (6, 7) for hyperstraining of the molasses (MC) on a membrane for its concentration,
- equipment (13, 14) for hyperstraining of the juice (ZC) on a membrane to obtain its further concentration;
- a tank (8) in which to recover the permeated water (P) if this water is utilized, together with or instead of the condenser water (C) for dilution of the juice (S) to be defecated and for transfer of the juice into the resin columns ,
- sundry equipment, such as pumps, valves and whatever else is necessary for automatic operation of the plant;

8) Equipment as described in Claim 7, **characterized** by the fact that the resin columns (4, 10) are fitted at the top with a removable vessel (15) the bottom of which is provided with distribution nozzles (16) with levelled mouthpiece to ensure uniform distribution of the juice on the resins (17), while two perforated plates (18, 19) are mounted at its lower end in such a way that the holes in the plates are vertically matching the nozzles (18), a net (20) supporting the resin being inserted between the perforated plate and the resin packing, thus ensuring that the resin will be uniformely permeated by the juice ;

9) Installation as described in Claim 8, **characterized** by the fact that the removable container (15) keeps the resins (17) levelled on the bottom, thus eliminating any preferential permeation paths.

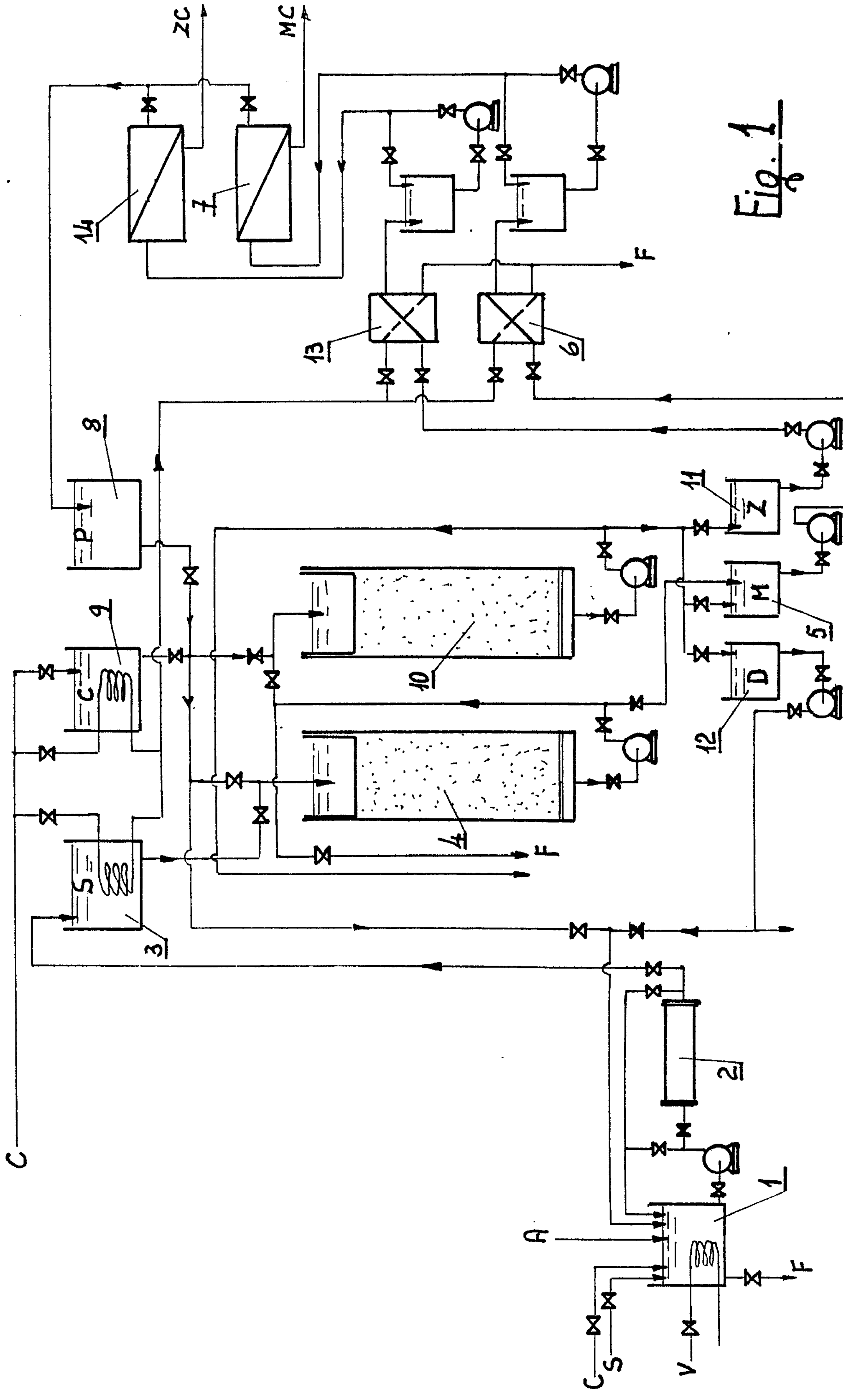

Fig. 1

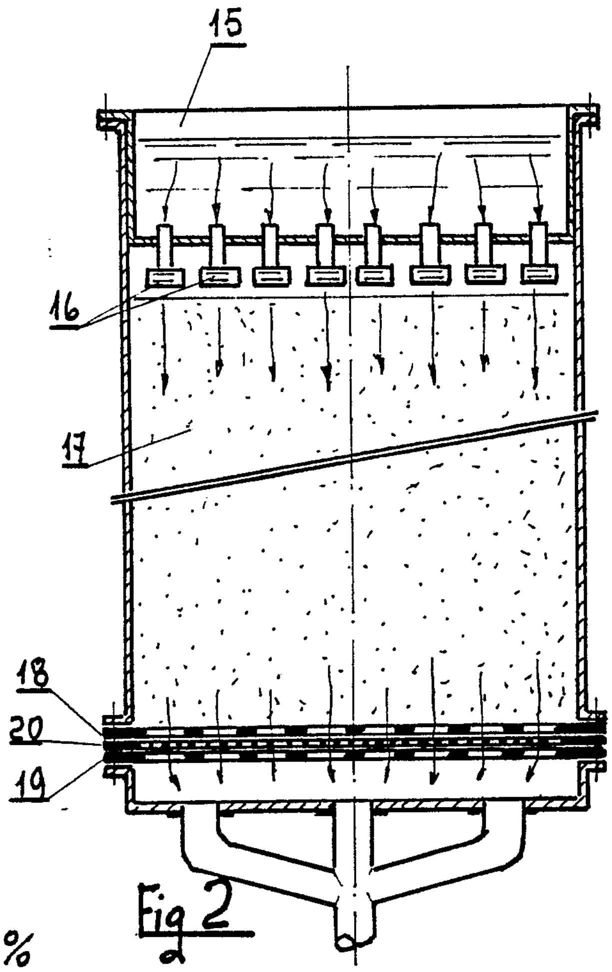

Fig 2

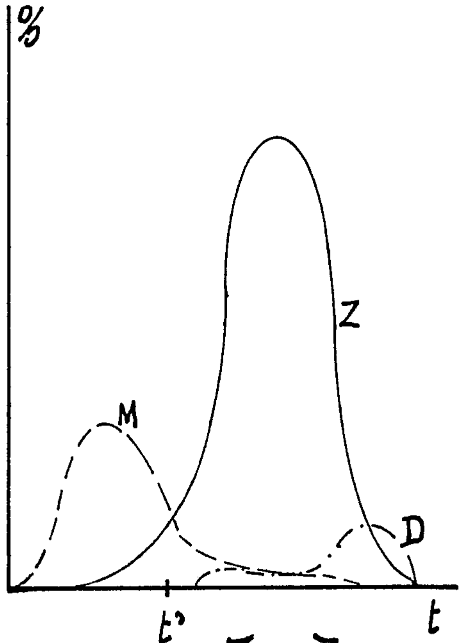

Fig. 3

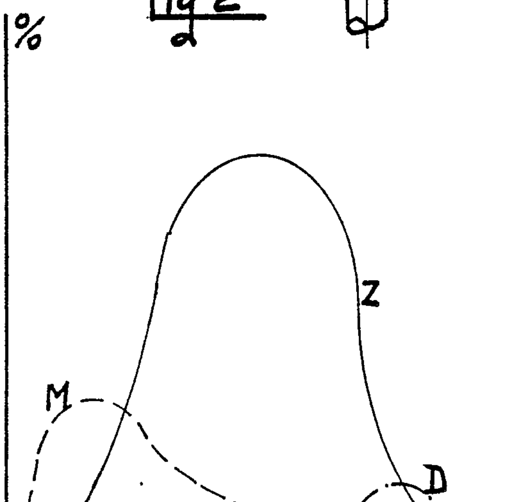

Fig. 4

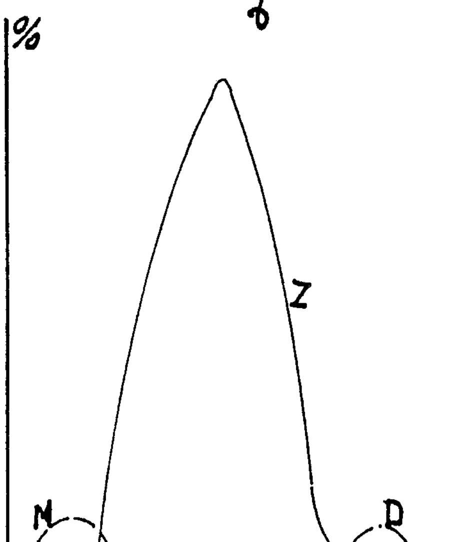

Fig. 5