UNITED STATES PATENT OFFICE.

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PROCESS OF EXTRACTING OILS FROM THE PEEL OF CITRUS FRUITS.

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To all whom it may concern:

Be it known that I, FRANK ALEXANDER McDERMOTT, a citizen of the United States, residing at Washington, in the District of Columbia, have invented certain new and useful Improvements in Processes of Extracting Oils from the Peel of Citrus Fruits, of which the following is a specification.

This invention relates to extracting the oily materials from the peels of citrus fruits, particularly such fruits as oranges and lemons.

The object of the invention is to recover a larger proportion of the oils from a given mass of peels than that which has been obtained in following any of the processes or using any of the mechanisms for this work with which I am acquainted.

In following my process, the peels are first carefully removed from the pulp of the fruit as completely as possible. This work has been largely practised heretofore where this industry has been carried on by hand. But where the work is so carried on, the peels are subjected to harsh treatment of such character that more or less of the oil is lost. For example, immediately after the peels are removed from the pulp, they are subjected to a reversal of curvature; that is to say, they are turned outside in. The outer convex side is pressed inward and the inner, normally concave, surface becomes convex. This squeezes or condenses the surface parts of the oil-carrying skin, and during the operation numerous minute globules of the oil are lost. Then while they are being, or after they have been subjected to such treatment, the newly turned surfaces are placed against absorbents with the expectation that the oil will be absorbed from the, now, concave surfaces which were, initially, on the outside. The peels are frequently subjected to crude, rough pressure and are severely lacerated, torn or comminuted, with an accompanying great loss of oil.

I, on the contrary, after cutting the peel, as an entirety, into suitably large sections, and then carefully removing it from the pulp without lacerating or marring it, maintain the relative conformations of the surfaces of the peels as they are naturally formed during growth, they being cut into as few sections as possible, say, into thirds or quarters.

After being thus carefully cut and removed, the peel sections are partially dried at a low temperature. For this they can be exposed on wire racks for several hours while relatively cool, and under such conditions that there will be no violent movements of the air around them to induce rapid desiccation. The moisture of the water contained in them is allowed to escape gently. The oils are so volatile that even air will, under its hygroscopic action, tend to carry the oils off with the evaporating water if the water vapor is carried away by rapidly moving air currents. It is desirable, however, to withdraw as much of the water as possible, but its evaporation should be stopped before the air tends to also absorb or carry off the oil vapors.

After being thus dried, more or less completely, the peels are subjected to a cutting action, such as results from the use of choppers with knives, adapted to effect a fine comminuting into cut particles. In some cases, the particles so derived can be still further reduced by gently grinding or macerating them. But these actions should be performed at a low temperature in a quiescent atmosphere to avoid volatilizing of the oils.

A mass of the finely ground peel is then put into a tightly closed vessel adapted to hold its gaseous contents in such way that their pressure can be reduced to a point where it can be delicately controlled. For example, a receiver having a supply orifice for vapor or gas, and a delivery orifice can be used but which should be adapted to be otherwise tightly closed after the introduction of the mass of peels. The delivery orifice can be connected with a condenser and with a pressure reducer.

Then, if a charge of the peels prepared in the manner aforesaid be introduced and inclosed, and if a current of vapor of water at a relatively low pressure be introduced and passed through the vessel, the oil will be extracted from the peel and carried over to the condenser, accompanied by more or less water vapor initially contained in the peel. The pressure is lowered by the action of the vacuum apparatus and can be regulated as required. It should be considerably below the pressure of the atmosphere. I have found that a pressure reducer adapted to create a partial vacuum equivalent to twenty-seven (27) or twenty-eight (28) inches (sixty-nine to seventy-one centi-

meters) of mercury, assists in obtaining excellent results. The vaporous materials from the peels, will, under these circumstances of extraction, contain not only the terpenes and other constituents of an oily nature, but also the aldehydes and other flavoring materials.

To obtain the best results, I arrange the mass of peel in a column which is relatively long in vertical dimension, and relatively narrow, horizontally, as this assists in insuring that all of the oil particles shall be extracted and carried away by the water vapor of the upwardly rising current which has a correspondingly prolonged line of travel, along which it can exert its capacity for lifting and carrying away the oil vapor.

In the condenser, the oily bodies which are there collected, commence immediately to rise to the top of the water, the two liquids being essentially immiscible although temporarily commingled. Well differentiated strata result after the liquids have remained in quiescence for a short period. And if they are allowed to stand quiescently at a low temperature for, say, twenty-four hours, practically all of the materials of one sort are effectually stratified in relation to those of the other. Then they can be finally separated by any suitable method of decanting; for instance, by withdrawing the water through an outlet duct at the bottom of the condenser receptacle, and then withdrawing the oily mass.

The resulting oils, as concerns their invisible qualities, that is, their flavors and odors, are superior to the oils obtained in the manually practised methods, and are much sweeter and better flavored than those which are obtained by the agency of extracting solvents, such as chloroform. In fact, they are so pure as oily masses, that they are pratically without color. And they can be used and marketed in this condition where pure oils of this class are desired for immediate use, even though clear and colorless.

But there is much demand in the market for oils obtained from these materials which are more or less delicately colored or tinted with the colors of the initial fruits. In order to impart a color of any desired strength, I proceed as follows:

I remove from a number of the native fruit articles, the extreme outer layers of their skins, separating them from the inner, pithy, lighter-colored layers of the peels. The outer skins contain the pigment materials which give the characteristic colors to the fruits.

This can be rapidly and economically accomplished by a suitable paring device, such as is commonly used for fruit paring, if its operative parts be properly adjusted for taking off only the extreme outer skin. The colored parings so removed are steeped at a low temperature in the distilled oil, and the pigments contained in them are dissolved therein, the oil probably also absorbing or dissolving more or less of certain waxy constituents. A liquid mass of the desired color results. The color can be varied in its strength by employing a greater or less proportion of the thin colored skins. Ordinarily, I find that the skins from fruit articles, which, in number, are from two to five per cent. of the number taken for the recovery of the oil (in the way above described), are sufficient for obtaining the standard or desired color.

The oil products described can be utilized in the conditions set forth if they are used shortly after recovery. But if they are to be kept in good condition for a considerable length of time, I further treat them as follows:

It is well known that oils of this sort, because of their containing ingredients of the terpene class, tend to become rancid and acquire the odors characteristic of the turpentines if they are allowed to oxidize; and oxidizing occurs when they are exposed to conditions favorable therefor; such as high temperature, strong light, free access of air, indeed, unless they be specifically protected against oxidation.

To guard against this, I add a suitable quantum of absolute ethyl alcohol, ordinarily introducing a volume approximately equal to ten per cent. of the volume of the oil. If oil of a cheaper grade is satisfactory, as it is for many uses, I obtain a preservative effect by the addition of ordinary alcohol. This is satisfactory where relatively large quantities of the product are desired for purposes which do not demand that it shall be perfectly clear in appearance, or that it shall have a high flavoring capacity. If it is desired to only preserve it for use within a relatively short time after recovery, and yet preserve it without loss of flavor during that short time, or materially affecting its clearness in appearance, I add a small quantity of a suitable vegetable oil, such as olive oil, in the proportion of, say, from one to two per cent., which is sufficient to protect it against oxidation for such a period. Or it may be thoroughly protected in vessels which are capable of permanently retaining a seal of a suitable non-oxidizing gas, such as carbon dioxid.

I am aware of the fact that, heretofore, oils have been more or less crudely obtained by taking the peels in their raw, green form, immediately after removing them from the fruit pulp, and adding a large quantity of water thereto after maceration to the pulpy condition. In some instances, steam at full atmospheric pressure was blown through the pulp with the expectation of its carry-

ing off the citrus oils. In other cases, a still higher precentage of water was added to produce a thin fluid paste, and heat was directly applied to the mass with the expectation of vaporizing both the water and the oil content and collecting them in an ordinary still. But the results attained by any of these processes were inferior to those which I reach. I have found that instead of increasing the water of the mass, even that which is naturally present should be reduced in proportion, and that the water vapor, if that is the oil abstracting agent relied on, should be delivered at a low pressure. Where there is a large proportion of water, the quantity of vapor carried to the still is large and the separation of the oil particles proceeds with difficulty and to an incomplete degree. Moreover, many of the bodies which I desire to retain with the oil mass are held in solution in the water. In my case, the vapor merely serves as a vehicle to carry a small quantity of heat to the oil particles and as a vehicle to carry them off when they vaporize at the relatively low temperature.

In referring to absolute ethyl alcohol, on the one hand, and ordinary alcohol, on the other, I mean to be understood as differentiating between them because of certain radical differences which I have found them to manifest in respect to oils of the present class. The ordinary alcohol (from ninety to ninety-five per cent.) manifests the tendency to produce water in the mass and cloudiness results. I have found that the absolute ethyl alcohol (ninety-nine or more per cent.) will thoroughly mix with the oil without any tendency toward cloudiness or discoloration.

What I claim is:

1. The herein described method of recovering the oily substances from the peels of citrus and other fruits, it consisting in abstracting from the said peels the native water therein at a temperature below that at which the oily substances therein are vaporized, then supplying from an external source a vapor capable of withdrawing oil and causing it to intimately commingle with and pass among and through the particles of the peels while in masses, collecting the vapors that pass away from the said masses, condensing said vapors into liquid forms, stratifying separately the oily liquids and the other liquids of condensation, and then separating the liquids.

2. The herein-described method of recovering the oily substances from the peels of citrus and other fruits, it consisting in removing the peels from the pulpy bodies of the fruit articles, evaporating the native water from the said peels while in a quiet atmosphere and at a temperature below that required for the vaporizing of the oil therein, then supplying from an external source a vapor capable of withdrawing oil, as vapor of water, and causing it to intimately commingle with and pass among and through the desiccated particles of the peels while in a mass condition, collecting the vapors of water and oil that pass away from the solid mass, condensing them into liquid forms, stratifying separately the oily liquids and the other liquids of condensation, and then separating the liquids.

3. The herein described method of recovering the oily substances from the peels of citrus and other fruits, it consisting in reducing the peels to a comminuted mass of fine particles inclosing the said mass in a gas-tight receptacle, passing streams of oil-extracting vapor introduced from an external source through the mass while subjected to a pressure below that of the atmosphere, condensing the vapors that are taken from the mass, stratifying separately the oily liquids and the other liquids of condensation, and then separating the liquids.

4. The herein described method of recovering the oily substances from the peels of citrus and other fruits, it consisting in removing said peels from the pulpy bodies of the fruit articles, vaporizing the native water from the said peels in a quiet atmosphere and at a temperature below that of oil vaporization, then reducing the peels to a comminuted mass of fine particles, then causing a vapor supplied from an external source and capable of withdrawing oils to intimately commingle with and pass through said mass of desiccated particles, collecting the vapors that pass away from the mass, condensing said vapors into liquid forms, stratifying separately the oily liquids and the other liquids of condensation, and then separating the liquids.

5. The herein described method of recovering the oily substances from the peels of citrus and other fruits, it consisting in removing the peels in relatively large sections from the pulp bodies of the fruit articles, then subjecting the said peel sections to water evaporating conditions at a temperature below that of oil vaporization, then reducing the peel sections to a comminuted mass of fine desiccated particles, then extracting the oily materials by means of currents of oil-extracting vapor supplemental to the normal vaporizable components of said mass and while under a pressure below that of the atmosphere, then condensing the vapors into liquid form, stratifying separately the oily liquids and the other liquids of condensation, and then separating the liquids.

6. The herein described method of recovering the oily substances from the peels of citrus and other fruits, it consisting in forming a mass of the peel materials in a

containing vessel in the form of a column which is vertically elongated and relatively narrow horizontally, supplying externally generated oil-extracting vapors and pressing them into and through the said vertically elongated column while said vapors and the mass are under a pressure lower than that of the atmosphere, collecting the vapors that pass from the mass, condensing said vapors in a region of condensation of similar low pressure, stratifying separately the liquids condensed from the vapors, respectively, and separating the oily liquids from those resulting from the condensation of other vapors.

7. The herein described method of recovering and preparing the oily substances from the peels of citrus and other fruits, it consisting in causing an oil-withdrawing vapor supplemental to the normal vaporizable contents of said peels to intimately commingle with and pass among and through the particles of the peels while subjected to a pressure less than that of the atmosphere, then condensing the vapors that pass from the said mass into the liquid forms, separating the oily substances in colorless condition from the other liquids of condensation, then introducing into the oils the natural pigment materials carried in the skin portion of supplemental fruit peels, and causing them to color the oil.

8. The herein described method of recovering and preparing the oily substances from the peels of citrus and other fruits, it consisting in causing an oil-withdrawing vapor supplemental to the normal vaporizable contents of said peels to intimately commingle with and pass among and through the particles of the peels while subjected to a pressure less than that of the atmosphere, then condensing the vapors that pass from the said mass into liquid forms, separating the oily substances from the other liquids of condensation, and then adding to the oil absolute ethyl alcohol, substantially as described.

In testimony whereof, I affix my signature, in presence of two witnesses.

FRANK ALEXANDER McDERMOTT.

Witnesses:
GEORGE E. EDELIN,
A. E. SPROESSER.