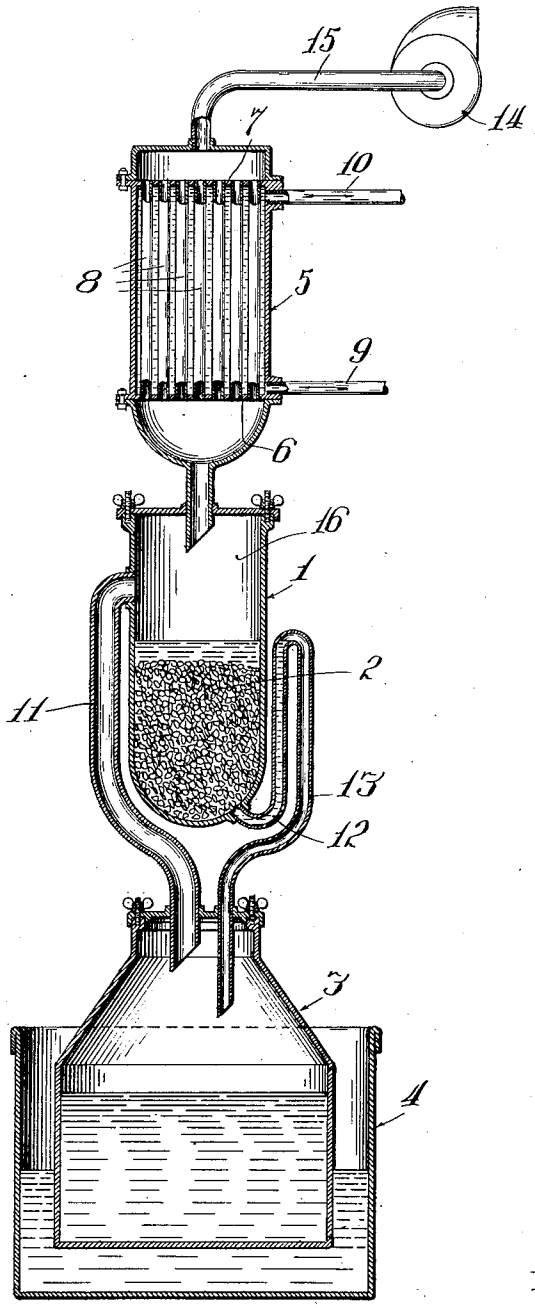
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H. BUEL

EXTRACTION METHOD AND APPARATUS

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Inventor

Hambden Buel

By Barnett & Truman

Attorneys

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HAMBDEN BUEL, OF FAIRVIEW, NEW JERSEY, ASSIGNOR TO INTERNATIONAL PATENTS DEVELOPMENT COMPANY, OF WILMINGTON, DELAWARE, A CORPORATION OF DELAWARE.

EXTRACTION METHOD AND APPARATUS.

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This invention relates to the extraction of essential oils or like substances from the raw materials containing them by use of a volatile solvent; and one of the principal objects of the invention is to provide a novel and improved method of extraction which may be carried on without loss of the volatile content of the materials treated, with economy of time and with the use and consumption of a minimum amount of solvent. This is accomplished by carrying out the entire extracting process under a reduced pressure, whereby the solvent may be kept at a relatively low temperature to prevent loss of the volatile, aroma giving substances that might be vaporized at the normal boiling point of the solvent.

The method has applicability especially to the manufacture of flavoring and extracts from the vanilla bean, but may be employed, advantageously, for other kindred operations, such as the manufacturing of other flavoring extracts, perfumery products, and other extractives containing elements which volatilize or are split or are otherwise injuriously affected at high temperatures.

A further object is to provide a novel and improved form of apparatus for practicing the aforesaid method.

The invention is illustrated in the accompanying drawing which shows a preferred form of apparatus. It will be understood, however, that the method is not to be considered as confined to the use of the particular apparatus shown in the drawing. So far as the apparatus itself is concerned, modifications thereof may be made without departure from the principle of the invention.

The drawing shows the extracting apparatus in vertical section.

The numeral 1 designates a holder for the material treated, which is shown at 2 in the drawing.

Arranged below the holder is a vaporizing apparatus for the solvent, which apparatus consists preferably of a closed container 3 arranged in a water-bath vessel 4. Heat may be applied to vessel 4, either by gas-flame, steam coils located in the vessel or any other suitable means.

Connected with the holder 1 is a condenser 5. The condenser may be of any suitable construction. It is shown as consisting of a vessel provided with two interior headers 6 and 7 for a plurality of condensing tubes 8. The space between the headers is supplied with a cooling fluid, by means, for example, of a cold water inlet pipe 9. 10 is an outlet pipe for cooling fluid, which fluid may, if desired, be artificially cooled before being introduced into the condenser to the extent necessary to completely condense the vaporized solvent to a temperature of 10° C. for example, although this temperature will necessarily vary with the pressure in the holder.

11 is a pipe for conducting the vaporized solvent from vessel 3 into the upper portion of the holder 1. 12 is a pipe leading from the lower portion of the holder to the vessel 3, preferably. It is desirable to form pipe 12 with an upward bend 13 whereby the solvent will be accumulated in the holder to a certain level, that is to say above the body of material treated, before being discharged to vessel 3. When the outflow from the holder is started, it is continued siphonically until the holder is emptied of liquid so that the action is intermittent.

Evacuating mechanism is provided for maintaining a constant sub-atmospheric pressure in the holder, condenser and vaporizing vessel 3. For example, a vacuum pump 14 may be connected by pipe 15 to the top of the condenser 5.

The method of extraction applied to the treatment of vanilla beans and using the apparatus as above described is as follows:

The vanilla beans are comminuted and placed in the holder 1 and allowed to stand under ethyl alcohol for 72 hours more or less. The alcohol may be used in an amount approximating 55% by volume of the chopped beans.

After the beans have been macerated, in this manner, as long as necessary, a volume of ethyl alcohol preferably equal at least to

the volumetric contents of the holder 1, is placed in vessel 3 and the water in vessel 4 is heated, say to 40° or 45° centigrade, to bring about vaporization of the alcohol. At the same time the vacuum pump 14 is started. The pump is operated so as to maintain a constant vacuum in the apparatus of approximately 29 inches of mercury.

The vaporized solvent passes from vessel 3 through pipe 11 into the space 16 above the material 2 in the holder and into the condenser 5. Coming in contact with the water cooled tubes 8 the vapor is condensed and is refluxed upon the material treated. As soon as the level of the liquid in the holder rises above the upper bend of siphon 13, the solvent, with extractive matters from the vanilla beans, or other material, is drawn from the bottom of the holder and discharged into vessel 3 by the siphoning action described. The vaporization of the solvent and its condensation and precipitation on the material treated is continuous so that the extracting operation may be carried on as long as may be necessary in order to remove the extractives from the material to the extent desired. Ordinarily the vaporization and condensation of the solvent will not keep pace with its discharge through the siphon so that the operation of the apparatus so far as withdrawal of the solvent and extraction is concerned, will be intermittent. That is, a certain amount of the solvent will collect and remain in contact for a time with the beans and then will be discharged, the holder being practically emptied of liquid before the siphoning action is stopped.

If desired the siphoning feature may be omitted, in which case the solvent, as condensed, will trickle continuously through the beans. As the operation is carried on in vacuo the solvent is kept at a relatively low temperature so that there is no loss of such volatile, aroma giving substances as might be vaporized at the boiling point of the solvent under atmospheric pressure.

The extracted matter may be separated from the solvent in any desired manner, for example, by distillation of the solvent. The solvent may be re-used continuously with little or no loss provided the condenser is of sufficient capacity to completely condense and reflux the vapors.

It is important to maintain a constant vacuum in order to secure even operation. There should also be no sudden changes in temperature of the solvent between the vaporizer and condenser. If need be, to prevent such changes, the apparatus should be covered with suitable insulating material.

This method of extraction is applicable to other substances than vanilla beans, and other suitable solvents, for example, acetone, benzine, carbon tetrachloride, or ether, might be used. The temperature and vacuum specified in the above vanilla extraction process are merely preferential, and may be varied in the case, for example, of the treatment of other materials or the use of solvents other than alcohol. Variations in the form, construction and arrangement of the parts of the apparatus will readily suggest themselves to those skilled in this art. It is my intention to cover all such modifications, both as to structure of the apparatus and details of the method, within the scope of the appended claims.

I claim:

1. Extraction method for materials containing extractives injuriously affected by high temperatures which comprises passing a heated solvent through the material, reducing the boiling point of the solvent to a temperature below that injuriously affecting the extractive by maintaining the space holding the material at a pressure below atmosphere, and cooling vapors from the operation to substantially completely condense the same.

2. Extraction method for materials containing extractives injuriously affected by high temperatures which comprises vaporizing a solvent and introducing the same into the space holding the material, condensing the vaporized solvent and applying the same to the material, and reducing the boiling point of the solvent to a temperature below that at which the extractive will volatilize by maintaining the space holding the material at a pressure below atmosphere.

3. Method of extracting essential substances from vanilla beans, which consists in soaking the beans in comminuted condition in ethyl alcohol, vaporizing the alcohol and introducing it into the space holding the beans, condensing the vaporized alcohol and refluxing it on the beans and reducing the boiling point of the alcohol to a temperature below that at which the extractive vaporizes by maintaining a constant pressure below atmosphere in the aforesaid space.

4. Apparatus for extraction of essential substances injuriously affected by high temperature comprising a holder for the material treated, means for vaporizing a solvent and introducing it into the holder, a condenser for condensing and returning to the holder said vaporized solvent and means for maintaining the space within the holder at a constant pressure below atmosphere whereby the boiling point of the solvent is below that at which the extractive is injuriously affected.

5. Apparatus for extraction of essential substances injuriously affected by high temperature comprising a holder for the mate-

rial treated, a vessel to contain a solvent, means for vaporizing the solvent in said vessel, a vapor duct leading from the vessel to the holder, a return duct from the holder to the vessel for the solvent and extractive, a condenser connected with the holder to receive the vaporized solvent and reflux the same upon the material in the holder and an exhauster connected with the condenser to maintain a pressure therein and in the holder and vessel at such point below atmospheric pressure that the solvent will evaporate at a temperature below that at which the extractive volatilizes.

HAMBDEN BUEL.