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PROCESS FOR PRODUCING CONFECTIONERS' HARD BUTTER

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This invention relates to confectioners' hard butter, and to the preparation of fats intended for use as confectioners' hard butter.

Confectioners' hard butter is employed as an ingredient in the manufacture of sweetstuffs, icings, coatings and fillings for biscuits, cakes and other confectionery. Confectioners' hard butter is judged largely on its ability to melt sharply and completely at body temperature and to break sharply and suddenly (snap) at room temperature.

It has now been found that certain fractions derived from palm kernel oil by hardening, interesterifying and fractionating are useful as confectioners' hard butter.

According to the present invention there is provided a process for the preparation of confectioners' hard butter from palm kernel oil, which process comprises the steps of hardening the oil to an iodine value of less than 10, interesterifying the oil, hardening being carried out either before or after interesterification, and then fractionating the hardened, interesterified oil by means of a solvent, to obtain a fraction having a dilatation at 30° C. of not less than 1000 and a dilatation at 37° C. of not more than 300.

Preferably, the fraction should have a dilatation at 30° C. of not less than 1200, and it is also preferred that the dilatation at 37° C. of the fraction should not be more than 200, more preferably not more than 100.

Mixtures of palm kernel oil with coconut oil may be used as starting materials, provided that the coconut oil constitutes not more than 25%, by weight, of the mixture.

Alternatively, the invention provides a confectioners' hard butter which comprises a fraction derived from palm kernel oil which oil has been hardened to an iodine value of less than 10 and interesterified, which fraction has a dilatation at 30° C. of not less than 1000 and a dilatation at 37° C. of not more than 300.

Hardening may be carried out under conditions of time, temperature and pressure usual in the hardening of oils and fats. Any conventional catalyst can be used and frequently it will be convenient to use a reduced nickel catalyst. It is preferred to use oils which have been hardened to an iodine value of less than 5 while, more preferably, fully hardened oils having an iodine value of less than 1 are used. Generally, it is convenient to carry out the hardening step prior to interesterification, but it is also possible to harden after interesterifying.

Interesterification may be carried out under conventional conditions and will cause a high degree of randomisation of the fatty radicals; preferably substantially complete randomisation is achieved. Suitable catalysts for interesterifying are the metal alkoxides, such as sodium methoxide, which may be used in alcoholic, preferably methanolic, solution. Generally, about 0.2% of catalyst, by weight of the oil, is sufficient to ensure complete interesterification.

Refining, bleaching and deodorising of the oil may be carried out at any suitable stage. Preferably, bleaching and deodorising are carried out after solvent fractionation, whilst the oil should preferably be refined after hardening and after interesterifying.

The fractionation of the hardened, interesterified oil may be carried out by fractional crystallisation from a solvent. Generally, it is advantageous to carry out the fractionation so as to remove a low melting glyceride fraction which may amount, for example in the case of palm kernel oil, to as much as 25%, by weight of the hardened, interesterified oil, the residual fraction having the characteristics specified. Substantially anhydrous acetone is the preferred solvent for fractionation but other substantially anhydrous solvents, for example ether and light petroleum, may be used.

To remove the low melting glyceride fraction, it is preferred to carry out the crystallisation using from 3 to 10 mls. of solvent per gram of oil. The temperature of crystallisation depends on the conditions used, in particular the ratio of solvent to oil. As a general guide it is usually convenient to use a ratio of solvent to oil of about 4:1 (volume/weight) at 20° C. For higher temperatures a lower ratio of solvent to oil will usually be necessary. Use of too high a ratio of solvent to oil may be disadvantageous as too high a proportion of glycerides may be removed, which may lead to a product having an undesirably high dilatation at 37° C. However, a fraction according to the invention may be obtained from a product having such an undesirably high dilatation at 37° C., by adding oil obtained from the mother liquors of the crystallisation, for instance, by evaporation, to correct the dilatation and yield a fraction according to the invention. Crystallisations should preferably be left at the crystallisation temperature until precipitation appears to be complete. The precipitate is then filtered off, preferably with application of vacuum or pressure and then washed with solvent at a temperature slightly lower than the crystallisation temperature.

For the determination of dilatations for the purpose of this specification, there are used a method and apparatus given in Section C–IV, 3e (52) of the "D.G.F. Einheitsmethoden" published by the Deutsche Gesellschaft für Fettwissenschaft e.V.

In the melting of fats a characteristic change of volume is observed which, especially in the case of fats solid at the normal temperature, manifests itself in a sudden increase in the volume.

The dilatation or isothermal melting expansion of a fat is the volume increase, which is expressed in $mm.^3$, determined under the conditions of the following procedure and referred to 25 g., the reference temperature being given.

The dilatometer is of glass and consists of a vertical graduated capillary tube joined at its lower end by a U-shaped capillary tube to a glass bulb surmounted by a neck which is internally ground to take a hollow ground glass stopper. The height (above the lowest point of the U-shaped capillary) of the top of the graduated tube and the top of the mouth of the bulb are 350 mm. and 70 mm., respectively. The graduations extend over a length of 250 to 290 mm., and start 1 cm. from the upper end of the tube. The graduations are marked in intervals of 5 $mm.^3$ (accurately calibrated), and cover a total volume of 900 $mm.^3$. The internal diameter of the bulb is 20 mm. and it has a volume of 7 ml. (tolerance ± 0.5 ml.). The internally ground neck of the bulb tapers downwards from an internal diameter of 15 mm. to an internal diameter of 12 mm. and is 26 mm. long. The bulb of the instrument is thus below the level of the graduations on the capillary tubing. The stopper to be inserted in the mouth of the bulb is about 95 mm. in length (including the ground portion), and is hollow

and is partly filled with lead shot to hold it firmly in position while a dilatation is being determined.

1.5 ml. of well boiled distilled water containing a little blue ink is pipetted into the bulb of the dilatometer. The dilatometer is then weighed. A sample of the fat to be examined is thoroughly de-gassed by heating at 100° C. under vacuum. The fat (at about 60° C.) is then poured into the bulb of the dilatometer and the ground glass stopper is inserted, care being taken not to include any air. The amount of fat added is such that, during the determination, the level of water never falls below the lowest of the graduations and never rises above the top of the graduations. If the initial water level on filling is about two-thirds of the height of the graduated capillary, these conditions are usually fulfilled. The dilatometer is re-weighed to obtain the weight of fat added. The hollow stopper is then partly filled with lead shot. It is then placed in a water bath maintained at 60° C. (±0.1°) and a reading of the level of the water in the capillary is made. This is the "base reading," R_{60}.

The filled dilatometer is chilled in an ice water bath for 1½ hours. It is then allowed to warm in an air bath to 26° C. at which temperature it is maintained for 40 hours. It is then again chilled in an ice water bath for 1½ hours, and then placed in a water bath at 20° C. (±0.1° C.), the dilatometer being immersed to such a depth that the water level is above the middle of the ground glass stopper.

The position of the water meniscus in the capillary is read every half hour until two successive readings differ by no more than 2 mm.³. The final reading (R_t) is used in the calculations.

A similar procedure is adopted for each temperature t at which the dilatation is required. Thus, R_{20}, R_{25}, R_{30} and R_{35} are successively determined.

Finally the dilatometer is heated again to 60° C. and the "base reading" is re-determined. If the initial and final "base reading" differ by more than 2 mm.³, the whole operation must be repeated.

THE CALCULATION

The value of the dilatation is calculated from the following formula:

$$D_t = \frac{25(R_{60} - R_t)}{W} - A$$

where:

D_t = dilatation at t° C.
W = weight of fat taken
R_{60} = base reading (mm.³)
R_t = reading of the capillary at t° C. (mm.³) and A is given in the table below

t° C.:	A
20	880
25	770
30	665
35	555

The following examples illustrates the invention.

Example 1

250 g. of a sample of refined palm kernel oil of iodine value 16.0 was hardened at 180° C. with a conventional nickel/kieselguhr catalyst equivalent to 0.5% Ni; the iodine value of the hardened oil was 4.3. This oil was twice washed at 90–95° C. with 0.1 N NaOH solution, each wash being approximately 25% of the volume of the oil. The oil was then transferred to a 1 l. roundbottomed flask and the extraneous water removed by heating for 60 minutes at 90–95° C. under a vacuum of 15–20 mm. Hg. Subsequently the oil was placed in the interesterification vessel, which is a 500 ml. roundbottomed flask equipped with means for stirring and passage of gas through the oil. The actual interesterification was preceded by a more intensive drying treatment in which dry nitrogen was passed through the oil with stirring at a temperature of 105° C. for 30 minutes; sodium methoxide catalyst, equivalent to 0.2% by weight of the oil, prepared as described below, was then added in the form of a 25% solution in methanol. The conditions were maintained for 5 minutes after which the interesterified oil was transferred to a beaker, cooled to 90–95° C. and successive water washes applied until it was free of catalyst; after drying, the oil was bleached with an activated bleaching earth and filtered.

100 g. of the interesterified oil was dissolved in 400 ml. of dry acetone and the resulting solution cooled and allowed to stand for 16 hours at 0° C. The solid fraction was filtered off and washed with 100 ml. of solvent at 0° C.; the solvent-free product (74 g.) had a dilatation of 1170 at 30° C. and 95 at 37° C. Subsequently, the final product was deodorised at 180° C. for 3 hours at a pressure of 1–2 mm. Hg.

PREPARATION OF SODIUM METHOXIDE CATALYST

Substantially dry methanol was prepared by the following procedure.

24 g. of magnesium and 300 ml. of A.R. methanol together with a crystal of iodine were placed in a 1 l. roundbottomed flask fitted with a reflux condenser. The contents of the flask were gently heated to initiate reaction but once under way it was necessary to cool with ice in order to keep it under control.

On completion of the reaction the resulting suspension of magnesium methoxide in methanol was refluxed for 2 hours after which the dry methanol was distilled off and used as follows:

220 ml. of the dried methanol were added dropwise via a reflux condenser into a 1 l. roundbottomed flask containing 23 g. of sodium (cut into small pieces about ¼" square). After complete dissolution of the sodium, the solution was ready for use as an interesterification catalyst.

(The special drying treatment of the methanol may be omitted but the resulting catalyst will be less active.)

Example 2

100 g. of the interesterified oil of Example 1 was dissolved in 400 ml. of dry petroleum ether (B.P. 40–60° C.) and the resulting solution cooled and allowed to stand for 16 hours at 0° C. The solid fraction was filtered off and washed with 100 ml. of solvent at 0° C.; the solvent-free product (66 g.) had a dilatation of 1285 at 30° C. and 250 at 37° C. Subsequently, the final product was deodorised at 180° C. for 3 hours at a pressure of 1–2 mm. Hg.

I claim:

1. A process for the preparation of confectioners' hard butter from palm kernel oil, which process comprises the steps of hardening the oil to an iodine value of less than 10, randomly interesterifying the oil, hardening and interesterification being carried out in indifferent order, and then fractionating the hardened, interesterified oil by means of a solvent to obtain a fraction having a dilatation at 30° C. of not less than 1000 and a dilatation at 37° C. of not more than 300.

2. A process according to claim 1 in which the fraction obtained has a dilatation at 30° C. of not less than 1200.

3. A process according to claim 1 in which the fraction obtained has a dilatation at 37° C. of not more than 200.

4. A process according to claim 1 in which the fraction obtained has a dilatation at 37° C. of not more than 100.

5. A process according to claim 1 in which the fraction obtained has a dilatation at 30° C. of not less than 1200 and a dilatation at 37° C. of not more than 200.

6. A process according to claim 1 in which the oil is hardened to an iodine value of less than 5.

7. A process according to claim 1 in which the oil is hardened to an iodine value of less than 1.

8. A process for the preparation of confectioners' hard butter from palm kernel oil, which process comprises the steps of hardening the oil to an iodine value of less than 5, randomly interesterifying the oil, hardening and interesterification being carried out in indifferent order, and then fractionating the hardened, interesterified oil by means of a solvent to obtain a fraction having a dilatation at 30° C. of not less than 1200 and a dilatation at 37° C. of not more than 200.

9. A process according to claim 8 in which the oil is hardened to an iodine value of less than 1.

10. A process according to claim 1 in which the solvent used is substantially anhydrous acetone.

11. A process according to claim 8 in which the solvent used is substantially anhydrous acetone.

12. A process according to claim 1 in which the interesterification step is carried out so as to ensure substantially complete randomisation of the fatty radicals.

13. A process according to claim 1 in which the fractionation from a solvent is carried out so as to remove a low melting glyceride fraction of up to 25% by weight of the hardened, interesterified oil.

14. A process according to claim 1 in which the ratio of solvent to hardened, interesterified oil in the fractionation step is about 4:1 volume/weight.

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