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## MIXED CAPRIC-CAPRYLIC ESTERS AND METHOD OF MAKING SAME

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14 Claims. (Cl. 260—411)

This invention relates to vegetable oils and the like and more particularly to the utilization of what has heretofore been considered as waste material and the production therefrom of highly valuable substances. The present application is a continuation-in-part of my copending application Serial No. 93,879, filed August 1, 1936, entitled "Mixed lauric-myristic esters and method of making same."

It is customary in the treatment of vegetable oils, for example, such as cocoanut oil for edible purposes, to refine the crude material in order to purify it and render it palatable. In the course of the refining operation, the oil is treated with an alkali solution in order to remove free fatty acids present in the oils. By this treatment, the alkali forms a soap with the free fatty acids and this in turn forms an emulsion carrying with it a certain proportion of the oil itself. This emulsion is generally removed from the oil being purified and difficulty has been encountered in finding a sufficiently valuable use therefor to render it worth while saving. Up to the present, it was common to mix this emulsion with the raw materials for soap making, but this use hardly paid for the cost of the extraction of the fatty acids from the oil.

It is among the objects of the present invention to provide a means for utilizing this waste product and to give the same a high value.

In practicing this invention, I may take the emulsion of oil and soap and saponify the same as by means of a mineral acid and Twitchell's reagent in order to break down the soap and the oil into free fatty acids. The fatty acids are then subjected to a distillation operation whereby there is recovered a fraction boiling at a low temperature and a fraction boiling at a high temperature. The low boiling fraction is then treated with glycerine in order to esterify the same and to provide a synthetic oil mixture having highly desirable properties. This type of product has considerable usefulness in that it is edible and has a low congealing point. It has a very light straw color and a pleasant taste and may be used successfully for edible purposes, such as salad oil, as shortening, and the like.

The natural cocoanut oil contains in combined form fatty acids such as caproic, caprylic and capric, having from 6, 8 and 10 carbon atoms in addition to the higher fatty acids such as lauric and myristic, having 12 and 14 carbon atoms and minor amounts of palmitic, oleic, stearic and linoleic having from 16 and 18 carbon atoms. In the natural oil, the combinations with glycerine may be such that the lower fatty acids are combined in mixed esters with the higher fatty acids and this contributes to the low and indefinite softening point of the oil.

The present invention by the distillation process has eliminated substantially completely all the acids except those having from 6 to 10 carbon atoms, so that a smaller number of esters are formed therefrom having a limited range of softening points and thus more desirable properties.

For example, I take the soap obtained in alkali refining of cocoanut oil acidulated with sulphuric acid, and split in the usual fashion with Twitchell reagent. I separate the fatty acids and fractionate by distillation into two fractions having the following characteristics:

| Constants | Low boiling | High boiling fractions |
|---|---|---|
| Color lovibond, 5¼ in. column | 5.0Y/1.0R | 6.0Y/1.1R |
| Free fatty acids as percent oleic acid | 151.4 | 126.0 |
| Acid number | 302.0 | 251.0 |
| Saponification number | 303.0 | 251.9 |
| Iodone number | 1.5 | 14.1 |
| Titer °C | 23.8 | 28.7 |

The low boiling fraction, having a free fatty acid content of about 151, consists essentially of a mixture of caprylic, capric, and lauric acids with minor amounts of caproic and of higher fatty acids, principally myristic acid. This mixture may be directly esterified with glycerine as described below to form mixed, substantially neutral esters. However, I prefer to fractionate the same to obtain a lower boiling fraction consisting essentially of caprylic, capric and lauric acids and a very minor amount of caproic and myristic acids having a free fatty acid content of about 165% calculated as oleic acid. The caprylic, capric and lauric acids are present in roughly approximately equal proportions, and they contain usually 1 to 5 parts of myristic and caproic acids.

1500 grams of this fraction is mixed with 284 grams of glycerine in a suitable vessel. The mixture is heated over a period of about two hours from a temperature of 144° C. to a maximum of 255° C. at the end of the operation. The reaction mass is agitated vigorously throughout, the pressure is maintained at atmospheric or higher, and carbon dioxide is maintained in the mixture. The temperature is then allowed to drop to a sufficiently low point, and the reaction product is subjected to steam distillation to remove excess fatty acids. The oil is refined by treatment with lye, washed, and bleached with ful-

ler's earth and darco. It it deodorized with steam at 175° C. for five hours at 6 mm. vacuum.

The product thus obtained has the following characteristics:

| | |
|---|---|
| Saponification number | 299.7 |
| Iodine number | 0.5 |
| Free fatty acids as per cent oleic acid | 0.06 |
| Odor | None |
| Flavor | Good |

The oil is water white or a very light straw color, has a pleasant taste and is quite stable. It does not become rancid even under long exposure to air.

In the above described fractionation, a higher boiling fraction is obtained having a free fatty acid content of about 135 and containing about 70% of lauric acid, the remainder being principally capric and myristic acids. This may be esterified with glycerine to give an intermediate grade of hard butter which is ordinarily called a winter grade. Or it may be otherwise used, as by addition thereof to a mixture containing considerable lauric and other fatty acids and esterified with glycerine to give a neutral product.

Although I have described my invention setting forth but a single embodiment thereof, showing the treatment of a waste by-product from cocoanut oil refining, it is, of course, apparent that my invention is not limited thereto. Other oils or fats may be used as starting materials, as, for example, palm kernel oil and the like, which may be termed lauric-myristic type of oil, it being essential that substantial quantities of caprylic and like acids be present. The invention is not confined to a waste portion of the oil but the entire oil may be treated to give the fatty acids which are distilled and treated as provided in the present invention. In fact, most fatty acids from any source may be used in accordance with the present invention. Instead of a single oil, mixtures of oils or of oils and fatty acids may be used. The products are capable of various uses, being adaptable, for instance, in ice cream coatings and the like. They may be used as salad oils, being advantageous in that the oils do not become cloudy when chilled.

These and other variations may be made in my invention within the scope thereof, and the invention is to be broadly construed and to be limited only by the character of the claims appended hereto.

What I claim is:

1. As an article of manufacture, the neutral mixed glyceride esters of fatty acids consisting essentially of caprylic, capric and lauric acids, being a light straw colored liquid, having substantially no odor and a pleasant flavor, and being stable.

2. As an article of manufacture, the neutral mixed glyceride esters of fatty acids consisting essentially of caprylic, capric and lauric acids in approximately equal proportions, being a light straw colored liquid, having substantially no odor and a pleasant flavor, and being stable.

3. A new chemical product consisting essentially of glycerides of a mixture of caprylic and capric acids, the principal constituents of said product being such that the aforesaid fatty acids are combined with the same glycerine molecules, said product being a mutually soluble mixture of neutral mixed glycerides, being a light straw colored liquid, having substantially no odor, having a pleasant flavor, being stable, and not becoming rancid upon long exposure to air.

4. A new chemical product consisting essentially of glycerides of a mixture of caprylic, capric and lauric acids, the principal constituents of said product being such that at least two of the aforesaid fatty acids are combined with the same glycerine molecules, said product being a mutually soluble mixture of neutral mixed glycerides, being a light straw colored liquid, having substantially no odor, having a pleasant flavor, being stable, and not becoming rancid upon long exposure to air.

5. A new chemical product consisting essentially of glycerides of a mixture of caprylic, capric and lauric acids, said mixture of fatty acids being derived from the fractional distillation of the mixed fatty acids of cocoanut and palm kernel oils, the principal constituents of said product being such that at least two of the aforesaid fatty acids are combined with the same glycerine molecules, said product being a mutually soluble mixture of neutral mixed glycerides, being a light straw colored liquid, having substantially no odor, having a pleasant flavor, being stable, and not becoming rancid upon long exposure to air.

6. A new chemical product consisting essentially of glycerides of a mixture of caprylic, capric and lauric acids, the said fatty acids being in approximately equal relative amounts, the principal constituents of said product being such that at least two of the aforesaid fatty acids are combined with the same glycerine molecules, said product being a mutually soluble mixture of neutral mixed glycerides, being a light straw colored liquid, having substantially no odor, having a pleasant flavor, being stable, and not becoming rancid upon long exposure to air.

7. A new chemical product consisting essentially of glycerides of a mixture of caprylic, capric and lauric acids, the said fatty acids being in approximately equal relative amounts, the principal constituents of said product being such that at least two of the aforesaid fatty acids are combined with the same glycerine molecules, said product being a mutually soluble mixture of neutral mixed glycerides, being a light straw colored liquid, having substantially no odor, having a pleasant flavor, being stable, and not becoming rancid upon long exposure to air, said product containing also a small amount of mixed glyceride esters of caproic and myristic acids having in the ester molecules at least one of the first mentioned group of acids.

8. A new chemical product consisting essentially of glycerides of a mixture of caprylic, capric and lauric acids, the said fatty acids being in approximately equal relative amounts, the principal constituents of said product being such that at least two of the aforesaid fatty acids are combined with the same glycerine molecules, said product being a mutually soluble mixture of neutral mixed glycerides, being a light straw colored liquid, having substantially no odor, having a pleasant flavor, being stable, and not becoming rancid upon long exposure to air, said product containing also a small amount of mixed glyceride esters of caproic and myristic acids having in the ester molecules at least one of the first mentioned group of acids, the total amount of caproic and myristic acids present being less than about 5% of the total acids in the product.

9. A new chemical product consisting essentially of glycerides of a mixture of caprylic, capric and lauric acids, the said fatty acids being in approximately equal relative amounts, the principal constituents of said product being such that

at least two of the aforesaid fatty acids are combined with the same glycerine molecules, said product being a mutually soluble mixture of neutral mixed glycerides, being a light straw colored liquid, having substantially no odor, having a pleasant flavor, being stable, and not becoming rancid upon long exposure to air, said product containing also a small amount of mixed glyceride esters of caproic and myristic acids having in the ester molecules at least one of the first mentioned group of acids, the total amount of caproic and myristic acids present being less than about 5% of the total acids in the product, the saponification number of the product being about 300, the iodine number being negligible, the acid content being about 165% calculated as oleic acid, and the free fatty acids in the product being negligible.

10. A method of forming neutral mixed glyceride esters of fatty acids which consists essentially in providing an oil taken from the class consisting of cocoanut and palm kernel oils, hydrolyzing said oil to free substantially all the fatty acids present, subjecting said acids to fractional distillation to eliminate both highest and lowest boiling acids and to provide a mixture consisting essentially of caprylic, capric and lauric acids, and re-esterifying said mixture with glycerine to form neutral mixed glycerides thereof.

11. A method of forming neutral mixed glyceride esters of fatty acids which consists essentially in providing an oil taken from the class consisting of cocoanut and palm kernel oils, hydrolyzing said oil to free substantially all the fatty acids present, subjecting said acids to fractional distillation to eliminate both highest and lowest boiling acids and to provide a mixture consisting essentially of caprylic, capric and lauric acids containing not over a total of about 5% of caproic and myristic acids, and re-esterfying said mixture with glycerine to form neutral mixed glycerides thereof.

12. A composition of matter comprising the mixed glyceride esters of fatty acids having principally from 6 to 10 carbon atoms.

13. A composition of matter comprising the mixed glyceride esters of fatty acids having principally from 6 to 10 carbon atoms, said fatty acids consisting essentially of caproic, caprylic and capric.

14. A method of forming neutral mixed glyceride esters of fatty acids which consists essentially in providing an oil taken from the class consisting of cocoanut and palm kernel oils, hydrolyzing said oil to free substantially all the fatty acids present, subjecting said acids to fractional distillation to eliminate both highest and lowest boiling acids and to provide a mixture consisting of acids having not over 10 carbon atoms, and re-esterfying said mixture with glycerine to form neutral mixed glycerides thereof.

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