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## PROCESS FOR THE RECOVERY OF THE UNSAPONIFIABLE FRACTIONS OF OILS

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My invention relates to a process of recovering unsaponifiable fractions of fats, and is particularly adaptable for use in extracting such fractions of cod liver oil, shark liver oil, and the like.

The invention, which has among its objects a quickly performed process requiring inexpensive apparatus, will be best understood from the following description of several examples of the practice of the process, while the scope of the invention will be more particularly pointed out in the appended claims.

As is well known, the unsaponifiable portions of fats, such as cod liver and shark liver oils, contain the valuable vitamins A and D. As the improved process lends itself to performance with a minimum of exposure of the materials to air, light and moisture, it is particularly adaptable for extracting these vitamins, the latter, particularly vitamin A, being susceptible to destruction when the material is so exposed.

As one manner of practising the present invention, but without limitation thereto, the fatty material, such as cod liver oil, may be placed in a container, after which is added dry powdered sodium hydroxid in the form of commercial lye, for example, one containing about 98% sodium hydroxid, the lye being added preferably as a mass as distinguished from gradually adding it. Preferably, the amount of lye added is such as to present an amount of sodium hydroxid in excess of that necessary to saponify the saponifiable fats in the oil, so as to cause the moisture to be absorbed and the vitamins to be thus protected from this moisture. In practice, equal portions by weight of the oil and sodium hydroxid in the form of commercial lye give satisfactory results. Immediately upon introducing the lye the contents of the container are intimately mixed as, for example, by use of a mechanical mixer. Practically immediately, saponification begins, the temperature of the contents of the container rising rapidly and saponification proceeding at a rapid rate, so that in a few minutes the mass becomes thick and pasty. At this point the material is preferably poured into pans to permit the saponification reaction to become complete and the material to cool and harden into a block of soap. If desired, the oil may be slightly heated before adding the lye to initiate the saponification reaction, but ordinarily this will be unnecessary. Preferably, care should be taken not to give such an initial heating as will cause the temperature of the mass, when the reaction occurs, to rise above from 85° to 100° C. This excess temperature may be prevented by pouring the contents into the pans as soon as the mass becomes thick. Ordinarily a cool solidified soap mass will be formed in not over twenty minutes from the time the lye is added to the container.

The soap mass formed as above described contains mixed with it the unsaponifiable portions containing the vitamins A and D. The soap mass may now be treated to remove these portions. Preferably this is done by first breaking up the soap mass, and then treating it with a volatile organic solvent for the unsaponifiable matter. As suitable solvents, ether, carbon tetrachlorid, chloroform, or the like, in which the soap and alkali are only minutely soluble, may be employed. Conveniently, the pans employed for solidifying the soap mass may be in the form of troughs to cause the solidified mass to be in the form of elongated sticks say, for example, sticks about one inch square, these being readily presented to a grinding machine which will break up the soap into a coarse powder.

The extraction of the unsaponifiable portions from the powdered or broken up soap mass may be performed by placing the soap powder in a container and adding the ether or other volatile solvent and stirring the contents of the container. Upon being allowed to stand the soap will settle at the bottom of the container, after which the liquid contents may be decanted, or prior to settling the soap may be separated from the liquid by use of a centrifugal separator or by use of a filter. It has been found that roughly 10 parts solvent to one part soap will give satisfactory results, but these proportions are by no means critical.

The ether or other solution of the unsaponifiable fractions, separated as above described, may be then treated by a distilling operation to remove the solvent, distillation preferably being performed under reduced pressure and at a temperature not exceeding 100° C. By this means a concentrate rich in the vitamins A and D is recovered. Any small fraction of soap present in the concentrate may be removed, if desired, by treating the concentrate with a small amount of ether, or other of the above mentioned solvents in which the soap is only very slightly soluble, to dissolve off the unsaponifiable matter and leave the soap behind, or the concentrate, which ordinarily is in the form of a rather thick paste, may be diluted with cod liver oil, corn oil, cotton seed oil, peanut oil, or the like, in which the soap is not soluble, and the solution filtered or subjected to centrifugal action to remove the soap. Commonly the concentrate is used to enrich the

vitamin content of cod liver or other oils, and may be added thereto, and any soap present may be removed by filtering or centrifugal action.

In the above examples of the practice of the invention, any residual amount of ether or other solvent present in the separated soap may be recovered for reuse, preferably by a distilling operation.

It will be observed in the above examples of the practice of my improved process, that the excess of lye or other hydroxid added to the oil not only saponifies the fatty fractions, but being in amount in excess of that necessary to form the soap, also takes up the moisture so that the unsaponifiable fractions are not subjected to moisture during the process. This materially aids in the recovery of a high percentage of the vitamin A which tends to be destroyed, at least in part, when subjected to moisture, particularly in the presence of heat.

It will be understood that wide deviations may be made from the above examples of the practice of my invention without departing from the spirit thereof.

I claim:

1. The process of recovering the unsaponifiable fractions of fish liver oil which comprises adding to and immediately mixing with the oil dry powdered sodium hydroxide, the latter being added to the oil substantially as a mass in large excess over that necessary to saponify the saponifiable fractions of the oil, so as to absorb moisture for protecting the vitamins and cause to be quickly formed a solidified mixture of soap and sodium hydroxide of requisite hardness to be reduced to a powder by a grinding operation; operating upon said solidified mixture to grind it to a powder; treating the resulting powder with ether for dissolving the unsaponified fractions of the oil; separating the powder from the ether solution; and distilling the ether from the powder free ether solution to obtain a concentration of the unsaponifiable fractions of the oil.

2. The process of recovering the unsaponifiable fractions of fish liver oil which comprises adding to and immediately mixing with the oil dry powdered sodium hydroxide, the latter being added to the oil substantially as a mass in large excess over that necessary to saponify the saponifiable fractions of the oil, so as to absorb moisture for protecting the vitamins and cause to be quickly formed a solidified mixture of soap and sodium hydroxide of requisite hardness to be reduced to a powder by a grinding operation; operating upon said solidified mixture to grind it to a powder; treating the resulting powder with a volatile liquid in which both the unsaponifiable and saponifiable fractions of the oil are soluble, but in which both the soap and sodium hydroxide are insoluble, for dissolving the unsaponified fractions of the oil from the powder; separating the powder from the resulting solution of the unsaponified fractions of the oil; and treating the powder free solution for vaporizing the volatile liquid solvent therefrom to obtain a concentration of the unsaponifiable fractions of the oil.

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