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## REFINED PHOSPHATIDES AND PROCESS OF MAKING SAME

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This invention relates to refined phosphatides and to processes of producing them. More particularly it relates to processes of preparing a refined vegetable phosphatide from vegetable oil phosphatidic residues without necessitating the use of alcohol or organic solvents, without precise control of acidities, and without the cautious additions of electrolyte as required by previously employed processes.

This invention relates also to a process whereby phosphatidic oil residues may be simply and economically processed to produce a refined, solid, somewhat waxy, light-colored, dry phosphatide flake or various forms of purified phosphatides containing water, these products ranging from a free-flowing viscous fluid to a heavy, gel-like semi-solid.

The present application is a continuation-in-part of our previously filed application Serial No. 494,116, filed July 9, 1943, now abandoned.

Commercial phosphatides, now available usually in the form of heavy oily liquids or thick oily pastes, have a relatively high oil content. These products present considerable difficulties in handling and transportation due to their physical natures. In addition, this high oil content inhibits their direct dispersibility in water. An oil carrier in many instances is detrimental to processes in which the phosphatide may be used. Any oil present also acts as a diluent, reducing the actual content of phosphatide in the final lecithin product.

Methods for the production of dry or pulverulent forms of phosphatides have previously been suggested. In manufacturing these products, however, there are involved the use of organic solvents or chemical treatment, or both, for the purification of the material. Some oil-absorbing material is also usually employed to take up the excess oil in order to obtain the desired stable pulverulent form. By prior processes, refined phosphatides or phosphatides having a low oil content tended to become rancid within a few weeks. In contrast therewith, our product, as herein described, has proven stable and would remain fresh for extended periods under a broad range of storage conditions.

The present invention yields, aside from others, a product which is unique and novel in that it has an unusually high phosphatide content, containing only some 15 per cent oil, containing no added extraneous materials, and one which in its final finished form is a solid light-colored flake. Since its color is light, the operation of bleaching as now ordinarily applied to commercial phosphatide pastes may be omitted for this product. Due to the low oil content of the material, it is very readily dispersible in water. It is, however, easily soluble in oil and may therefore be employed in either medium.

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The processes of this invention are applicable to the preparation of refined phosphatides from all types of phosphatidic residues which are commonly referred to as "foots," such as may be separated from vegetable oils either by settling or by the treatment of such oils with steam, or with water, followed by centrifuging. Several types of raw materials are particularly adapted for use in accordance with the process of this invention. One is the slimy residue which settles from crude vegetable oils on long standing. These settlings or foots from hydraulic or expeller-expressed crude vegetable oils, such as soy bean oils, vary in consistency from a rather hard, waxy lower layer to a thick, slimy, oil-like upper layer. In general it is preferred to use the lower layer of such oil settlings or foots, since it has been found to have a higher phosphatide content. However, the upper slimy-like layer may be used to advantage by separating the surplus oil therefrom in a centrifuge. Another source of foots, which are of slightly different character, is the residue obtained from a crude expressed vegetable oil which has been washed with water or steamed. The foots from oils so treated may be separated from the oil by centrifuging. Still another source of foots, which may be of slightly different character, is the residue obtained from steam or water-treated solvent-extracted oils. It will be recognized, however, that in accordance with this invention, phosphatidic residue from vegetable oils obtained by any process may be used. In the following description and in the appended claims, the crude phosphatidic raw material which may be treated in accordance with this invention will be termed "foots," and this term as used herein is intended to cover any phosphatidic raw material indicated briefly in the foregoing, or commonly designated as such in the trade.

The use of organic solvents, such as acetone, hydrocarbons, chlorinated hydrocarbons, esters, alcohols, etc., has generally characterized most previous processes for obtaining refined vegetable phosphatides from the crude products obtained by water-washing vegetable oils or from tank settlings of stored crude vegetable oil. The use of such solvents involves not only extra expense for material and equipment, but also may introduce serious fire, explosion, or health hazards in industrial operations. In accordance with the pres-

ent invention, a refined vegetable phosphatide may be prepared from oil foots without the use of organic solvents and without the precise control of acidities or the cautious additions of electrolyte as required by previously employed processes. In accordance with this present invention, a refined phosphatide may be produced from oil foots by first emulsifying the foots with a quantity of water, thereafter causing the emulsion to break and mechanically separating a refined phosphatide from the thus broken emulsion. The vegetable oil foots that may be used in accordance with the processes of this invention may be any of those indicated in the foregoing and are preferably obtained from soy bean oil which contains substantial quantities of phosphatides, principally cephalin and lecithin. Other phosphatide-containing oil foots may be obtained from corn oil, cottonseed oil, peanut oil, etc.

In carrying out the process of this invention, particular attention should be given to the temperature and concentration relationships both in emulsifying and in breaking the emulsions so that the process may be easily carried out, and so that an improved product will be formed. In accordance with one embodiment of this invention, a vegetable oil foots containing a substantial quantity of vegetable phosphatide is emulsified with a definite quantity of water and thereafter heated in order to produce a mass having a stiff, dough-like consistency. Thereafter the dough-like mass is cooled, allowed to stand until it has separated into three distinct layers, namely, an upper oil-containing layer, an intermediate water-containing layer, and a lower phosphatide-containing layer. This lower phosphatide-containing layer may then be separated from the remaining layers as the desired product of the process. In this particular process it is important that the amount of water used for emulsifying the phosphatidic residue be closely regulated so that a dough-like mass is produced which will separate in the manner indicated in the foregoing. A mass having the proper consistency will be efficiently formed when the water in the emulsion falls within the range of between about 4.5 and 5.5 times the weight of the phosphatide initially present in the foots to be treated. The amount of phosphatide present in the foots may be ascertained by chemical analysis as a guide to the proper amount of water to be employed.

In accordance with a further embodiment of this invention, the oil foots may be emulsified with water, and the phosphatide-containing material may be coagulated or precipitated by the addition of a water-soluble electrolyte to the emulsion. The water-soluble electrolyte may be sodium chloride, potassium chloride, acetic acid, hydrochloric acid, sulfuric acid, phosphoric acid, or the like. However, a noncontaminating divalent metallic salt, such as calcium chloride, or trivalent metallic salt, such as aluminum chloride or aluminum sulfate, is preferred for this step of the process. When the phosphatidic sol or gel is coagulated or precipitated with an electrolyte, the amount of water present in the phosphatide emulsion may fall considerably outside of the range indicated above for the process when water alone is used to purify or refine the phosphatide present in the foots.

One object of the present invention is the provision of a process for producing a refined vegetable phosphatide, such as lecithin, from vegetable oil phosphatidic residues without necessitating the use of organic solvents, without precise control of acidities, and without cautious additions of electrolyte as required by previously employed processes.

An additional object of the present invention is to provide a vegetable phosphatide which is extremely active for reducing the viscosity of plastic chocolate compositions.

A further object of the present invention is the provision of a water-containing phosphatide gel preparation which may be used directly for reducing the viscosity of such materials as plastic chocolate compositions or which may be dried and mixed with any selected oil for the same purpose.

A further and additional object of the present invention is the provision of a refined phosphatide gel composition containing substantial quantities of water, but which may be admixed with a previously dried phosphatide preparation to produce a product having any desired moisture content.

A further and additional object of the present invention is the provision of a process for preparing a light-colored vegetable phosphatide product from vegetable oil foots.

A further and additional object of the present invention is the preparation of a light-colored lecithin product in flake form, high in phosphatide content, and having marked emulsifying and thinning properties as well as high antioxidant activity.

Further and additional objects will appear from the following description and the accompanying claims.

For a more complete understanding of the present invention, reference will now be had to the following examples wherein several specific processes are disclosed showing how this invention may be used. It will be understood, however, that these designated processes are given only by way of example and it is not intended that the invention be in any way limited thereto.

*Example 1*

One hundred pounds of soy expeller oil foots containing approximately 40 per cent of phosphatides are first emulsified with 192 pounds of water. Emulsification may be hastened by vigorous agitation and sometimes assisted by warming. In this particular example, the amount of water used for emulsifying the foots is about 4.8 times the weight of the phosphatide contained in the foots as determined by chemical analysis. After emulsifying, the mixture is heated in a steam-jacketed tank or by other suitable means to the boiling point. Apparently a series of complex physical (and possibly chemical) changes occur during the heating process, resulting, at or soon after the boiling point has been reached, in the emulsion becoming a thick homogeneous mass having peculiar doughy properties. This doughy product is allowed to boil or is maintained at near boiling temperatures for 30 to 35 minutes with mild agitation and with the addition of water to replace any that may have been lost by evaporation. The emulsion has been found to maintain during this period the physical characteristics of a dough. The time of heating this dough-like mass does not appear to be critical, and experiments have shown that heating may vary from 5 or 10 minutes to over one hour with satisfactory results.

After the homogeneous mass has been boiled as described above, it is allowed to cool. During

the initial cooling stage, slight stirring may prove advantageous, but agitation should not be continued after the first few minutes or after the mix has been cooled to about 80° C. After allowing the dough to cool to about 50° C., the mix is held at this temperature until the doughy emulsion slowly breaks, that is, separates into three distinct layers. This separation will usually be completed after four to 48 hours. Although in this process doughs may vary somewhat in such respects as appearance, rate of separation, and composition, depending upon their source and manner of emulsification, it is the unique property of separating cleanly into these three phases or layers which characterizes the proper type of dough produced as described, by proper dilution and temperature control in the process.

Under various conditions the volume of the top layer finally separating may vary from 5 to 25 per cent of the total mass, depending on the purity of the raw material, the amount of water employed, and the amount of oil in the original foots, i. e., in the type of foots involved. This top layer contains oils, gums, and other impurities. It also may contain very small amounts of phosphatides. The middle or intermediate layer may amount to about 5 to 25 per cent of the total mass and is usually a clear watery solution containing very small amounts of phosphatides and some soluble carbohydrates. The third or lowest layer usually amounts to about 50 to 75 per cent of the total mass and may contain 85 per cent or more of the total phosphatides of the raw material. This lower layer may also contain oil, usually to the extent of not more than one-fourth of the amount of phosphatide present, and varying amounts of water. This lower layer is known as the phosphatide gel layer, or sometimes as the phosphatide layer.

If in the above indicated process an emulsion is prepared using too little water, or if water, evaporated during emulsification and subsequent heating, is not replenished, two disadvantageous effects will be encountered. First, the proper separation of the doughy emulsion, which appears to depend on the collodial condition or state of dispersion to produce ultimately a clean rapid separation of the oil from the hydrated phosphatide, will not properly occur and the line of separation between the top oil layer and the bottom hydrated phosphatide layer will be indistinct if the intermediate water layer is lacking. Consequently, separations of oils from phosphatides under these conditions are incomplete and difficult. Accordingly, when the emulsion is heated as above described, additions of water should be made from time to time so as approximately to maintain the original water-phosphatide ratio of the emulsion as stated above. The production of the proper dough phase is an important step in this modification of this process wherein only water is used as a separating agent. The production of the dough under centain controlled conditions, or what may be termed the "dough point," may be used as a method of determining the ratio of water to foots suitable for use in practical manufacturing operations. If to the foots to be tested an excess of water be added and the resulting emulsion be heated to boiling with stirring, upon evaporation of the excess water a point will be reached at which the mass will suddenly coagulate, that is, change from a free-flowing liquid mass to a doughy or plastic mass. This point is readily detectable and is easily reproducible. If the amount of water present is now determined, it will be found to be quite close to a ratio of 5.4 of water to 1 of phosphatide, which is a convenient ratio for practical purposes. For optimum phosphatide recovery, the optimum water ratios are from between 4.7 to 5.0 parts of water to one part of phosphatide for foots containing 35 to 45 per cent phosphatides. These ratios furnish the amount of water which will produce a suitable emulsion resulting in an efficient and facile separation within a reasonable time into the several component layers, the bottom layer of which is a refined hydrated phosphatide. For foots of higher or lower phosphatide content corresponding variations from these ratios may be advisable. However, the water ratios may be varied between about 4.5 and 5.5 or slightly higher, in which latter case decreased efficiency will be encountered. Below 4.5 the dough is usually too stiff to permit of clean separation of the components into layers. When the ratio is much above 5.5, it ordinarily takes too long to obtain the separation, the separation is quite often incomplete, and the resulting product contains excessive quantities of oil. However, in some cases, because of such practical considerations as ease or speed of working, slightly higher water ratios than the optimums 4.7 to 5.0 suggested may prove preferable despite their lower yields. Where the water ratio is increased to 5.5, the recovery of phosphatide from soy expeller oil foots will be found to decrease by one to 4 per cent, depending upon the exact nature of the foots.

The foregoing example typifies optimum conditions for the separation of phosphatides from soy expeller foots containing usually 30 to 50 per cent phosphatide, 40 to 50 per cent oil, some carbohydrate, and some free fatty acid, sterols, fiber, grit, etc.

The following examples set forth other procedures that may also be useful under certain conditions.

*Example 2*

One hundred pounds of soy expeller oil foots containing 40 per cent crude phosphatide are emulsified by strong agitation with 600 pounds of water. In this modification of the process the ratio of water to phosphatides of about 15 to 1 was found to produce the most satisfactory isolation of phosphatides from other constituents of the foots, particularly from water-soluble carbohydrates. The resulting emulsion is then heated to about 77° C. with stirring and maintained at this temperature for 30 minutes with continued stirred. One and one-half pounds of calcium chloride in a 20 per cent water solution are then added to the mixture with continued stirring, and the temperature is gradually increased to about 82° C. The calcium chloride electrolyte breaks the emulsion, the crude lecithin or vegetable phosphatide precipitating out in a doughy mass from which most of the water is easily poured away after kneading the mass while still warm. In this step most of the soluble carbohydrates are dissolved out. It will be noted that in this example, when an electrolyte is used for precipitating or coagulating the vegetable phosphatide, much higher water ratios in the emulsion are found to be desirable.

The dough precipitate is then made up al-

most to the original volume by the addition of 500 pounds of water and is re-emulsified by stirring at about 50° C. The emulsion formed is then heated to about 75° to 80° C. and is thereafter allowed to cool slowly to about 20° C. without agitation. It is then allowed to stand for 24 to 48 hours at about 20° C. This mass then separates into three layers as described in connection with Example 1. The lowermost layer here is the desired purified phosphatide gel. Sufficient calcium chloride remains in the first precipitate again to induce coagulation at the elevated temperatures whereby separation at the lower temperature is made possible. However, the second emulsification and re-precipitation reduces the calcium chloride concentration below the taste threshold and the traces of calcium chloride present in the final product have substantially no effect on the emulsifying power of the phosphatide prepared by this method.

*Example 3*

This is a modification of the process of Example 2 supra. One hundred pounds of foots are emulsified by strong agitation with 600 pounds of water at 30° C. This mixture is heated to about 77° C. and held at that temperature with mild agitation for about one-half hour. This emulsion is then treated with two pounds of calcium chloride dissolved in water, and forms a precipitate or coagulum as described in the preceding example. This coagulum is re-emulsified in the same solution by stirring and cooling rapidly to 20° C. The resulting doughy mass is then allowed to stand without agitation for 24 to 48 hours, during which time it separates into the three layers previously described. The lower layer is then separated from the others and is the refined phosphatide gel prepared in accordance with one modification of this invention.

*Example 4*

In accordance with one embodiment of this invention, the emulsion of foots and water in the absence of a coagulating electrolyte may be heated to above the atmospheric boiling point. In accordance with this example, 100 pounds of foots are emulsified with 250 pounds of water and then heated to about 60° C. and allowed to remain at this temperature with mild agitation for about 60 minutes. The resulting emulsion is then heated under about 25 pounds pressure to about 130° C. with agitation, the heat being so adjusted as to bring about this rise in temperature in about 20 minutes. The pressure is maintained for about five minutes and the resulting doughy mass is allowed to cool in a quiescent state to about 50° C. and to remain at this temperature for about 24 hours. This mass separates into three layers as described above.

In this example the water phosphatide ratio is somewhat greater than that specified as being desirable in Example 1. However, this is due to the fact that a certain amount of water is required to provide water vapor to bring the pressure to 25 pounds as designated in the particular pressure vessel used. When the steam pressure upon the treated mass is suddenly released to the atmosphere, this surplus water is vaporized as flash steam, bringing the water-foots ratio back to that cited in Example 1.

*Example 5*

As another variation of the process of this invention it has been found possible to apply electro-phoresis or electro-sedimentation in accelerating the precipitation of the lower phosphatide layer from suspension as demonstrated in the following procedure: Ten pounds of foots were emulsified by strong agitation with 30 pounds of water and immediately subjected to 100 volts potential. A gel separation occurred within one hour. In common with other electro-phoretic or electro-sedimentation processes, the process of this example depends upon many conditions which may be difficult to control, such as the state of dispersion of the phosphatides and the conductivity of the emulsion.

The preceding examples are merely illustrative and should not be considered as in any way restricting the process of this invention. It will become apparent from experiments that time, temperature, water-foots ratio, and perhaps acids or inorganic salt concentration (when used in the process) are determining factors in establishing the proper condition for the formation of the gel or sol which separates in the manner indicated above. It is obvious that results of a kind may be attained for any foots-water ratio through treatment with electrolytic and proper processing. Electrolytes do not form an essential part of the process. Also it is obvious that for any given temperature of operation a suitable water concentration and treatment may be developed in order to obtain results.

In the three layers which separate from the water emulsion of foots as described in the foregoing, the lower layer has been found to be a heavy, gel-like, brown liquid which may be separated easily by any convenient means from the top layers. This may preferably be done as above described by gravity separation. However, centrifuging may also be employed if desired. This product, when dried at 100° C. in air or at a lower temperature in vacuo, yields a product containing approximately 34 per cent phosphatide.

Oil susceptible to further refinement may be recovered from the top layer and constitutes a valuable by-product of the process of this invention. Another useful by-product is the water layer which has been found to contain substances having anti-oxidant properties.

We have found that the "phosphatide gel" described in each of the above examples may be dried to produce a novel form of phosphatide having unique chemical and physical properties. The dried material, a product of this invention, is readily dispersible in water, soluble in oil, and has very marked emulsifying and thinning properties, and through its effects on surface tension modifies the colloidal nature of suspensions and of foams to improve their stability. It is a solid wax-like product in flake form, very stable at ordinary temperatures and practically non-hygroscopic. This product is, in addition, a strong anti-oxidant and is valuable as a stabilizer for fats and oils, vitamin preparations or other materials subject to deterioration by oxidation.

The phosphatide content of the material is approximately 85 per cent, and because of its ready dispersibility in water, together with its high purity, it is especially advantageous in biscuit, cracker and confectionary formulas and in all cases where aqueous media are desirable. Its ready solubility in oil makes it applicable in all instances where commercial phosphatides are now used, and the oil used for the solution of the flake may be selected for its adaptability to any product or process in which it is to be used. This eliminates the necessity for using oils which may

be undesirable or detrimental to any given process or product.

The primary advantage of this dry, solid, waxy, flaked product is that it is readily dispersible in water and readily soluble in oil, thus adapting it for use in either medium or in any combination or formula in which both oil and water are employed. The phosphatide may be added according to convenience or practicability directly to a formula before being mixed or during mixing, in many cases lending its thinning value to reduce the power required for mixing, as in the preparation of an ointment or pigmented oil, or it may be added to a finished product and readily dispersed therein as in molten chocolate liquor to produce a controlled reduction in viscosity. It may be dissolved in water or other liquid component and thus be added in solution as in the preparation of emulsions, or it may be added with solid constituents as, for example, in so-called lecithinized flours used in preparing doughs. The flake may be added advantageously to cocoa beans before grinding them for the preparation of chocolate in order to reduce grinding power requirements and to increase yield.

Another advantage of this product is its physical state, a stable, solid, waxy, practically nonhygroscopic flake. The product in this form greatly facilitates handling, packaging and transportation. The flake may be poured, shoveled or otherwise more conveniently handled than the pastes. Because of its dry form the flake can be quickly, easily and completely emptied from its containers, thus effecting savings in time and materials; pastes cannot be removed completely from their containers except by laborious scraping, and even after scraping almost invariably some paste is lost by being retained on the walls or in the corners of the containers. For similar reasons weighing portions of flake becomes a simpler operation since the dry form phosphatide can be poured into and out of weighing vessels with utmost convenience and speed and allows quantitative delivery without careful, laborious scraping. Lightweight, nonmetallic, cheap containers may be used for the flake instead of heavy, costly metallic or glass vessels as used presently for the oils and pastes. Still another advantage is its low oil content which adapts the material for use where oil carriers are detrimental or prohibitive. The low oil content and its ready solubility in oil also enables the user of the product to use any selected oil as a carrier if an oil solution is desirable.

Another further advantage is the high phosphatide concentration in the product which enables the manufacturer to use less of the material in his formulas to give the desired effect.

A still further advantage is the unusual efficiency of the product as an emulsifying and thinning agent.

Still another advantage is the stability of the material which enables it to be stored or transported for long periods of time without danger of spoilage or detrimental change in the physical character of the product.

For the preparation of this product, the phosphatide gel may be introduced without further treatment onto a standard atmospheric drum drier. Such a drum drier consists of two parallel, hollow metal cylinders mounted horizontally adjacent to each other, coupled and driven to rotate toward each other when viewed from above, and internally heated with steam at a pressure of usually not more than 80 pounds, preferably 45–50 pounds, The space between the drums may be varied. The phosphatide gel, separated as a bottom layer in the above-described refining process for phosphatides, may be fed directly into the trough formed by the adjacent upper quadrants of the drier drums. Upon contacting the heated drums, a layer of the gel adheres to the surface of each drum and is carried through the space between the drums, the thickness of the emerging adherent layer or film being regulated by adjusting the distance between the drums. At proper rotational speed, film thickness and drying temperature, the adherent phosphatide film becomes dried in less than one revolution of the drum to a sheet which can be readily removed by a scraper. The resulting dry phosphatide sheet as it is disengaged from the rolls is warm enough to have a tensile strength sufficient to carry it from the rolls without difficulty. On cooling, the sheet becomes brittle and may be easily broken into flakes of any desired size by an agitating device such as a high speed beater which will break up the sheets without exerting any considerable friction on the material.



If desired, the bottom layer of phosphatide gel described in each of the examples cited may be further purified and partially dehydrated by treatment with calcium chloride or other inorganic salts and acids. In accordance with one embodiment of this re-purification process, the phosphatide gel is diluted with two volumes of water and stirred and heated to about 65° to 75° C. At this point a 20 per cent calcium chloride solution is added slowly with mild stirring. Violent agitation is to be avoided, since air may be introduced into the mixture which causes the coagulum to rise to the surface, making separation difficult. The mild stirring is continued until the phosphatides have precipitated out of the solution, the amount of dry calcium chloride required being roughly 0.2 per cent by weight of the diluted gel. The phosphatides then settle to the bottom of the tank or other container and the separated liquor may be drawn off. If desired, the phosphatides may be separated more rapidly by centrifuging. Other electrolytes which may be substituted for the calcium chloride in this re-purification step may be inorganic salts, such as ferric chloride, and certain bleaching agents, and also acids, such as sulfuric acid. Any of the separated lower layers of partially refined phosphatides after being kneaded to remove excess water, becomes a gel-like mass containing about 60 per cent of water, about 10 per cent of oil or free fatty acids, and about 30 per cent of phosphatides. One important and valuable characteristic of this process is that the gel-like phosphatidic product, whatever its water content, has a substantially uniform composition with respect to its phosphatide and oil content when calculated on a moisture-free basis.

Further dehydration of the phosphatidic gel may be accomplished by heating and kneading out additional quantities of water so that the resulting mass after cooling contains 40 to 50 per cent moisture and is physically quite stable. The product thus obtained dissolves easily in water or in oil and has extremely high emulsifying properties and chocolate-thinning action. Attention is directed to the fact that this product, containing 40 per cent or more of moisture, has been found to have a marked thinning effect on molten chocolate, equal to or higher than other commercial vegetable phosphatides, all of which

are substantially moisture-free but contain an equivalent amount of oil.

This product may also, if desired, be dried on an atmospheric double drum drier in a manner similar to that described above with minor adjustments as to the proximity of the rolls.

For many purposes a light-colored product is desirable. It has been found that the bleaching action of such bleaching agents as sodium chlorite or sodium hydrosulfite (dithionite) are particularly successfully applicable for producing light-colored phosphatides obtained by this process, since they may be added immediately to the water used to dilute the gel after the gel layer is drawn off. Also, such electrolytes as sodium chlorite or sodium hydrosulfite have been found suitable to cause coagulation of the phosphatide gel. The bleaching agent and any substances that may be produced by its action are largely removed by extraction in the water which separates from the phosphatides when they are precipitated by calcium chloride or other suitable agents in a further purification step such as described above. For the bleaching process, about 2.5 per cent of sodium chlorite or about 5 per cent of sodium hydrosulfite based upon the weight of the gel has been found to be adequate for most purposes. The actual addition, for example, may be made by dissolving the sodium chlorite or sodium hydrosulfite in the water used to dilute the phosphatide gel in the further purification step. After the bleaching agent and water have been added, the mixture is well stirred and the whole mass becomes a homogeneous gel. This is allowed to stand for twelve hours after which the solution is heated and phosphatides are thereafter precipitated by calcium chloride or other electrolyte and the re-purification process may be carried out exactly as described above. The resulting waxy mass is now light in color, bland in flavor, odorless, and has a smooth, even texture.

It will be understood that improvement in purity and texture of the product, whether bleached or unbleached, may be readily obtained by single or multiple washing of the coagulum or precipitate with water either with or without electrolytes. Such washing processes consist in re-emulsification and re-coagulation.

In the foots and in any of the three forms described as a "gel" (the untreated, separated lower layer), as a "wax" (the precipitated and partially dehydrated product), or as "dehydrated," the phosphatide content may be determined by extracting with copious amounts of acetone. The resulting purified acetone-insoluble residue contains proportions of phosphorus, nitrogen, and choline commonly found in lecithins, as that word is used in this application, from soy or from other sources. Its solubilities and physical properties further substantiate its identity.

While several embodiments of the invention described in the claims are shown above, it will be understood, of course, that this invention is not to be limited to these particular embodiments, since many modifications may be made in them, and it is contemplated, therefore, to cover by the appended claims any such modifications as fall within their true spirit and scope.

We claim:

1. A process of recovering a refined phosphatide from oil foots containing substantial quantities of phosphatide which comprises emulsifying said foots with water in an amount between about 4.5 and 5.5 times the total weight of the phosphatide present in the foots, heating the resulting emulsion to a temperature and for a time sufficient to produce a product having a dough-like consistency, cooling the resulting mass, gravity-separating said mass whereby three layers are formed, and separating the lowermost layer from the others as the desired product.

2. A process of recovering a refined phosphatide from oil foots containing substantial quantities of phosphatide which comprises emulsifying said foots with water in an amount between about 4.5 and 5.5 times the total weight of the phosphatide present in the foots, heating the resulting emulsion to the boiling temperature, continuing boiling while replacing the water being boiled away so that the water-phosphatide ratio will be held substantially within the above-indicated range, cooling the boiled liquid mass somewhat, gravity-separating the mass into three separate layers, and separating the lowermost layer from the others as the desired phosphatide.

3. A process of recovering a refined lecithin product from soy bean oil foots containing about 35 to 45 per cent of phosphatides which comprises emulsifying said oil foots with water in an amount between about 4.7 and 5.0 times the total weight of the phosphatides present in said foots, heating the mixture to boiling, continuing the boiling until a homogenous mass having doughy properties is formed, maintaining the water content of the boiling mixture substantially within the above-indicated range, cooling the boiled mixture to about 50° C., maintaining said mixture quiescent at said last-mentioned temperature for 4 to 48 hours during which time the mixture gravity-separates to an upper oil-containing layer, an intermediate water-containing layer, and a lower lecithin-containing layer, and separating said lower layer from the other as the desired refined lecithin.

4. A process of recovering a vegetable phosphatide from oil foots containing substantial quantities of phosphatide which comprises emulsifying said foot with about two and one half times their weight of water, heating the resulting emulsion under pressure to a temperature above 100° C., thereafter cooling the resulting doughy mass to a temperature somewhat above room temperature, gravity-separating the mass into an upper oil-containing layer, an intermediate water-containing layer and a lower phosphatide-containing layer, and finally separating said lower layer from the remaining layers.

5. Process of producing a phosphatide characterized by stability against rancidity and in the form of dry flakes which comprises subjecting oil foots containing substantial quantities of such phosphatide with a sufficient quantity of water within the range of from about 4.5 to 15 times the total weight of phosphatide present in said foots to produce an emulsion, heating the resulting emulsion to an elevated temperature for a time sufficient to convert it to a doughy consistency, thereafter allowing the doughy material thus formed to stand until it breaks to form three layers, the lower of which contains the desired phosphatide, an intermediate layer consisting substantially of water, and an upper layer consisting substantially of oil, separating the lower phosphatide-containing layer, and submitting it to a drying operation to produce a dried flaky product.

6. The process as defined in claim 5 in which

sodium chlorite is added to the doughy mass to bleach the phosphatide contained therein.

7. Process of recovering a refined vegetable phosphatide from vegetable oil foots containing substantial quantities of such phosphatides which comprises emulsifying said foots with an amount of water equivalent to about fifteen times the total weight of the phosphatides present in said foots, heating the resulting emulsion to about 77° C. while stirring for about 30 minutes, thereupon adding a sufficient quantity of an electrolyte to break said emulsion to effect the separation therefrom of a doughy mass, kneading the latter and then decanting separated aqueous solution of electrolyte therefrom; diluting the residual material to a volume about equal to that of the orginal emulsion and heating it to about 75° to 80° C. and then slowly cooling it to about 20° C. and allowing it, in the course of from about 24 to 48 hours to stratify into three layers; separating the lowermost layer which contains the desired phosphatide, and recovering the latter.

8. A process of recovering a refined vegetable phosphatide from vegetable oil foots containing substantial quantities of phosphatide which comprises emulsifying said foots with an amount of water equivalent to about 15 times the total weight of the phosphatide present in said foots to form an emulsion, heating said emulsion with mold agitation and with the addition of a small amount of an electrolyte to below the boiling point of the mixture whereby to break the emulsion and to precipitate the phosphatide; re-emulsifying said precipitated phosphatide in the original solution by again heating and then cooling the re-formed emulsion to about 20° C. and gravity-separating it to form three layers; and separating the lowermost layer which contains the desired phosphatide, and recovering the latter.

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