

DETAILED EXPLANATION OF THE INVENTION

Technical field of the invention application

- 5 The invention is an appropriate method to be applied in the field of treatment of oil and fat derived from vegetable, animal, fish and mineral sources and also to treatment of extracted substance from such oil and fat in separation of similar or different compounds that are composite of such oil and fat. For example, the field of separating complex chemical structures of different melting points in
- 10 vegetable oil like palm oil and palm kernel oil or the field of separating and refining several chemical structures or similar chemical structures that are contained in mineral oil (for example, solidified tar) after liquefying it.

Existing technology and the problem that the invention is to solve.

- 15 Since oil and fat applied to food industry are almost all of natural sources, there hardly exist a simple physical property attributable to a single composition. Therefore several problems arise out of treating natural oil and fat comprising of complex chemical structures. Among others, there is the problem due to
- 20 different climate in origin and consuming countries. Homogeneity of oil or fat in a place of origin can hardly be maintained in place of consumption because the chemical structure of high melting point solidifies due to change of the climate (to lower temperature).
- 25 Presently so called natural fractionation method prevails in separation of target components by physical filtration of liquid oil containing solidified components under controlled temperature to be fixed by separation ratio depending on the composite of the original oil or fat. Another known method of separation is the process in which takes place firstly cooling oil or fat down to induces
- 30 solidification, cleansing the solid comprising of chemical structures of high melting point with dodecyl magnesium sulphate and magnesium sulphate to dissolve out components of lower melting point that are attached to the solids

and lastly cleansing the liquid portion.

The oil separated by the above described methods is generally a composite of structures of low melting point but particles of higher melting point still remain in it and they solidify in the autumn climate in Japan. Such oil has to be heated to 35 C or above to get homogeneity. There exists no technical process of separation to have homogeneity of oil in room temperature in Japanese winter (10 C sometimes 7C or lower), although molecular distillation process is generally found to have higher efficiency of fractionation.

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In separation of wax or ester compounds with high melting point and enclosing saturated fat of high carbon number from oil or fat of animal and fish source, it is necessary to chill oil or fat for a fairly long time and to filter sediments similarly as described above. The appearance of oil thus separated is not appreciable if particles of higher melting oil remain in it even in a very small fraction.

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Mineral oil containing tar, water particles and other tiny foreign matters spread out thoroughly, if stored for a long time, gradually solidifies into a very hard substance like a rock. The method of separating different components of such a hard substance is to heat its whole to liquefy. But there is a limit in heat penetration and work load and is available no practical way of dissolving the solid.

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To prevent occurrence of solidification in oil of homogeneity due to change of climate, there is a process to lower clouding point of the oil. Clouding point is the temperature at which solidification starts in oil normally below room temperature.

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A simple method of reducing the clouding point of oil is to mix the target oil with oil of clouding point lower by 5~10 C (for example sesame oil or groundnut oil) than the predetermined cloud point of the oil at a volume ratio of 1: 0.3~1:0.4. This mixing will make the clouding point of the target oil by 2~3 C

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lower due to positive ionization. However, the result is not satisfactory. The mixing of oil with different compound will spoil some of the features of the original oil. Therefore, it is required to develop a method to selectively and precisely separate solid structures from oil that is exposed to lower temperature.

5 Filtration by itself is proved to have a limitation in selectiveness and preciseness of separation.

This invention is made in this circumstance to separate in a simpler process a specific component of oil or fat which is apparently homogeneous in a certain
10 temperature range.

The method to solve the problem and its application and effect

As described before, separation is generally done by chilling process which
15 solidifies components of high melting point in the oil. This invention, however, uses chilling process as a part of enhancing separation efficiency but does not require it as a necessary preparation.

This invention utilises difference of chemical and physical properties attributable to difference of chemical structures of compounds in oil or fat. A separation
20 agent (for example a surface active substance with a peculiar structure) is configured absorbing several components of oil so that separation of similar compounds from the others becomes possible by making use of difference of behavior of the compounds that does not exhibit in the original oil. This invention applies the above findings to separate a specific component of oil or
25 fat.

Therefore, the process essential in this invention is to set up a feature or a stimulus to induce separation of the target compounds in oil or fat. The feature exhibits only when the separation agent is added.

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In summary, the features of this invention to separate a specific components of oil or fat is that; hydrophile groups have several independent chemical structures

like aminoacids, mono-saccharides and poly-saccharides and the chemical structures in the hydrophile groups interactingly form annular compound or linear compound.

- The method is to separate a specific component of oil or fat which has a characteristic of forming layers of fractions comprising of each different structures depending on difference of reaction to a separation agent when oil or fat is mixed with solution consisting of compounds that have a peculiar surface activity and given physical stimuli from outside to separate the solution.
- 10 The separation agent to be mixed with oil or fat is (1) compounds that have a feature of surface activity consisting of hydrophobic or hydrophile groups of several chemical structures (like phosphorus with hydrophile groups of mono-saccharide and poly-saccharide, fatty acids with hydrophile groups of carboxyl groups, lipo-peptides with hydrophile groups of oligo-peptide etc.). Among those
- 15 compounds described in above (1) that have hydrophobic or hydrophile groups as surfactant, several aminoacid compounds (for example, oligo-peptide compounds) form circular structural oligo-peptide compounds taking oxygen (O) or ammonium (NH) in its organic functioning unit to stimulate separation of different chemical compounds and to intensify difference among several different compounds.
- 20 Below written are compounds of circular peptides to be applied.

(Chemical Formula 1)

- The inventors of this invention have found to separate oil or fat by the difference of hydrophobic or hydrophile capacity of the components. After paying attention to structures of organic groups that exhibit a peculiar hydrophile capacity, they found that the difference of the number of hydrophile groups attached to the surface of oil or fat particles attributable to different chemical structures means the difference of emulsifying fluidity of oil or fat that shows behavior difference,
- 30 amplified difference of apparent affinity by emulsification of a particular surface active agent.

Besides, this surface active agent is supposed to have a completely different

configuration behavior on surface of components of oil or fat, as the ratio (HM)/(WM) is totally reversed between molecular weight of chemical structure with hydrophobic groups(HM) and molecular weight of chemical structure that is supposed to induce hydrophile groups of heavier molecular weight (WM) and
 5 between hydrophobic groups of heavier molecular weight (HM) and hydrophile groups of lighter molecular weight(HM).

It is preferable to process, selecting controlled temperature from mixing operation to stationary separation or centrifugal separation of the mixture, after maintaining
 10 for a fixed time homogeneity of separation agent as mentioned earlier and oil or fat mixed at a volume ratio to be selected in the range of 1 : 0.2 ~ 1 : 10.
 Oil or fat, when applied the separation method of this invention, is divided into tiny particles. The contact area is so increased in both the particles and the separation agent that apparent metamorphosis of the particles enclosed by the
 15 separation agent or the particles of specific gravity apparently altered can be separated more efficiently and precisely by combination of rough separation using stationary and centrifugal separations as lubricating resistance decreases among the particles due to the presence of the separation agent. Repetition of this simple process improves separation efficiency remarkably.

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Implementation of the Invention

The core of this invention is to make use of the important function of separation agent that amplifies difference of similar properties, which enables to separate
 25 components of different chemical and physical properties (hydrophobic degree, melting point, specific gravity etc.) attributable to difference of chemical structure from components of similar properties in oil or fat. No documents have ever discussed advantage of applying this kind of separation agent for separation of oil or fat components from the view points of structure of separation agent and
 30 difference of its separability.

Many of microorganisms living on petroleum, a representative of hydrophobic

materials, secrete a substance of surface activity to enhance their metabolism and to protect their cells from toxic components in the oil . The surface active substance that this invention deals with is a so called bio-surfactant (such as saccharides with saccharide as hydrophile group; trehalose-lipid, rhamnos-lipid, soholose-lipid and lipo-peptides with oligo-peptide as hydrophile group; arthrofactin, surfactin). They are classified separately from the ordinary surface active substances that are synthetically provided with surface active capacity, while the former has a special feature of dissolving itself completely through organic dissolution.

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This invention applies the surface active substance of which peculiar molecules form hydrophile groups by their wide spread and aggregate and the hydrophile groups thus formed exhibit a surface character drastically different from the one that the original material exhibits, depending on the difference of absorption behavior (number of hydrophile groups and their density) reflecting surface condition of the absorption target. Therefore, selection of surface active substance will determine efficiency of proposed operation.

The surface active substances of lipo-peptide group, circular structural compounds containing aminoacids and oligo-peptides, in particular, are capable of stabilizing their molecular structures and easily applied removal operation of co-existing foreign substances so that fairly high purity can be obtained. Besides, their easy handling will enable to put the whole process in a very clean environment and also make it possible to prevent foreign substances mixed in the oil or fat in the process. Therefore, they are the peculiar substances that meet the conditions of selection of the separation agent. On the other hand, as regards solidified layer substances of mineral oil group, they were originally of liquid form but moisture, solid sands and fine particles of oxidized polymerization of petroleum quite often reacts each other in complex to form a substance which surface is as solid as a rock and has high viscoelasticity.

The separation agents as described above, if selected rightly by its hydrophobic

and hydrophile structures, permeate into this solid material and restore fluidity of structural surface and separation of water, sand, stone and oxidized particles becomes possible.

- 5 This is a tactful utilization of feature of the substance that exhibits surface activity which is generated by peculiar chemical structures in performing a function by collecting several organic functions of heavy molecules of contradictory features each other.

10 Examples of Implementation

Explanation is given hereunder of the concrete implementation of this invention in separating particular components of oil or fat.

15 (Example 1)

- 1) Preparation of separation agent that exhibits a peculiar surface active property

From lipo-peptide group of surface active agents of annular structure containing D-aminoacid residues and acidic aminoacid residues (classification is done by the
20 difference of chemical structure composing hydrophile groups), selecting pure powder of arthrofacterin (molecular weight: 1354) and mixing it with pure water to have a solution of concentration at discretion to suit the purpose.

The production method of arthrofacterin is publicized in J.P.A. Publication No.
25 Hei 6-184194.

The arthrofacterin is a bio-surfactant made of compounds generated in culturing bacteria of arthrobacter strain and is confirmed to have distinctive features requisite for classification.

The strain, arthrobacter species No. 38, is recognized in Bikouken Bonded Strain
30 No. 13198.

- 2) Demonstration of operating method to prevent such phenomenon as oil of

homogeneity at a certain temperature loses fluidity by solidification of some components when the temperature is reduced; Structure of oil used for test separation:

- 5 Average quality of Malaysian crude palm oil is used for the test as the material which components of high melting point are to be removed in the separation process described later to get a merchandise which has no clouding and less transformation.
- 10 Composition of oil, weight ratio of components and melting points of each component are given in Table 1.

Table 1.

Composition of ordinary palm oil and melting point of components

15	Composite of oil	carbon	unsaturated	weight
	melting			
	Fatty acids * 1)	number	bonding	ratio %
	point *2)			
20				beta
	structure			
	a) saturated			
	lauric	12	: 0	0.2
25	myristic	14	: 0	1.1
	palmitic	16	: 0	41.7
	stearic	18	: 0	4.2
	archidic	20	: 0	0.4
30	b) unsaturated			
	palmitoleic	16	: 1	0.1
	oleic	18	: 1	39.2
				4.9 C

linolenic	18	: 2	10.0	-23.0 C
linolen	18	: 3	0.3	-

20.0 C

5 Remarks: *1) these are existent as triglyceride compounds of fatty acids
and

glycerine

*2) crude palm oil is semi-solid at room temperature of 25~30
C

10 is only homogeneous liquid at or above 60 C

3) Test of separation process of the separation agent from substance of different
affinity in the mixture of oil and the separation agent:

15 a) The auxiliary separation agent, the solution of 1g/l of arthrofactin as described
in 1) and desalted water (electric conductivity 5 / -cm) prepared by a reverse
osmotic membrane was used as a mother liquid to emulsify oil.

b) As the oil used for the test becomes semi-solid and loses fluidity when its
20 temperature is below 60 C, the oil container should be put in a hot bath to raise
its temperature so that the sample to be drawn for the test is homogeneous.

c) Prepare the separation agent as described in above 3) a) as much as required to
maintain a volume ratio of oil and it at 1 : 2 and put it in a mixing container
25 with a temperature control mechanism and keep its temperature around 50 C. The
mixing container is equipped with mechanism of supplying hot or chilled water
to its surrounding and of controlling the temperature of its content and is further
provided with an agitator to mix the content to homogeneity. Besides, it has an
outlet at its bottom to discharge the bottom layer when stationary separation after
30 agitation separates the content into upper oil layer and lower separation agent
layer.

The total space volume of the mixing container is 10 L. The running speed of the agitator is quite slow, not so fast as 150 rpm and its blades are of propeller type. It has a satisfactory mixing capacity even under a restricted condition to prevent oxidation of oil components due to agitation. Before agitation the separation agent and the oil are removed of oxygen by decompression. In the course of agitation, nitrogen gas is injected from the bottom to prevent oxidation of the contents.

d) Put 4 L of the separation agent prepared as described in 3) a) into the mixing container as described in above c) and start agitation. Next draw 2 L of oil that is homogeneous under the controlled temperature as described in 3) b) and add it onto the separation agent and start mixing operation. After 10 minutes of agitation at temperature of 50 C, start chilling the container gradually at the reduction speed of about 5 C per hour to ultimately reduce the temperature to 20 C which is attained after 5~6 hours of chilling.

Stop agitation when the temperature reaches 20 C and leave the mixture still for about three hours during which the surrounding temperature of the mixing container should be maintained at 20 C.

e) After the stationary process, about 3.6 L of the bottom layer of separation agent was discharged from the outlet. Oil block of extremely high concentration was not discharged but remained in the oil layer. In the liquid layer that was discharged, the micro components of oil of high hydrophobic character and of saturated fatty acid group is considered to have moved from emulsified oil to separation agent layer and to have moved gradually to the area close to water layer, removing the separation agent by natural compression to induce buoyancy of oil particles.

f) After removal of hydrophile solution, about 10~15 % of separation agent remains in the oil layer. To intensify removal of this residue, 50 CC each of oil as described in above 3) e) was centrifuged in centrifugal tubes at a speed that imparts acceleration G of 2500 to the tube. The upper portion of the layers in the

tube after 5 minutes of centrifugal operation shows a red liquid layer which is supposed to be consisting of glycerides made of unsaturated fatty acids containing vitric carotinoids.

- 5 The lower layer, on the other hand, is supposed to be consisting of separated triglycerides of saturated fatty acids with high viscosity. At the surface between the further lower layers there was a sticky film formed which is supposed to be so firmly coked resinoids of very high melting point that it remains in tact even after the upper fluid portion is removed.

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- g) After centrifugal process, the volume of the upper transparent fluid of unsaturated fatty acids is about 1.0 L, which is approximately 80 % of unsaturated fatty acids supposed to be in the original oil composition from the analysis data. As the rate of recovery in chilled filtration methods is said to be
15 about 60 %, the method of this invention is considered to have a remarkable improvement.

- h) The clouding points varies from 20 C to 5C in tests of several samples from the separated oil with high transparency. The results shows that the separation
20 is incomplete since the proposed cloud point is less than 5 C.

- The oil separated and cleansed in the first phase of operation is separated again with a fresh separation agent prepared in the same manners as described in 3) c) through e) and with the surrounding temperature in separation process at 10 C, repeating the process described in 3) f) and g). About 900 CC is separated in
25 the second separation and 5 samples are all of cloud point less than 5 C.

(Example 2)

- In a crude petroleum tank, the stationary bottom oil forms a solid and nonfluid substance by complex intermixture of solidifying metamorphosis of water and
30 sand accumulated for long years of use and oxidized compounds of petroleum components. Fluid petroleum tar loses its fluidity and solidifies when moisture and salt of sea water is mixed with repeated vibration.

Spray the separation agent as described in 3 b) heated to about 50 C onto this kind of solid substance and circulate the agent to keep spraying, then the solid substance recovers fluidity from the surface and separation becomes possible of fluid tar, sand and water. In this way fluid and uniform tar is recovered .

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Centrifugal operation of the recovered tar makes its salt content nearly nil and viscosity so low that the oil thus separated can be used for fuel.

A method to separate specific components of oil and fat

Scope of Claimed Patent :

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Claim No. 1 : Oil and fat are composed of mixture of various components, each different from the others in its chemical and physical properties attributable to different molecular structures. The method is to separate a specific and the similar group of components from the other components of different properties in oil and fat.

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Hydrophile groups have several independent chemical structures like aminoacids, mono-saccharides and poly-saccharides and the chemical structures of the hydrophile group interactingly form bonded circular structures or linear structures.

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The method is to separate a specific component of oil or fat which has a characteristic of forming layers of fractions comprising of each different structure depending on difference of reaction to a separation agent when oil or fat is mixed with solution containing of a compound that has a peculiar surface active feature and physical operation is applied to the mixture to separate the solution.

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Claim No. 2 : The compounds to be used as the separation agent as mentioned above in Claim No. 1 are lipo-peptides made of circular structural oligo-peptides as hydrophobic or hydrophile group, which has surface active property.

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Claim No. 3 : Oil or fat is a composite of ester compounds made of glycerine and saturated fatty acids with lighter gravity and higher melting point and ester compound of glycerine and unsaturated fatty acids with heavier gravity and lower melting point.

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The separation method using the separation agent described in Claim No.1 and Claim No. 2 is aimed to transform composition of oil or fat of above mentioned



structure into oil or fat of such structure as keeps its liquidity in a room temperature environment by separation of a specific components from the others in oil or fat.

- 5 Claim No. 4 : The volume ratio of separation agent as described in Claim No. 1 and Claim No. 2 and oil or fat should be in the range of 1:0.2~1:10. The separation method as above described in Claim No. 1, Claim No. 2 and Claim No.3 includes maintenance of homogeneity of the mixture of oil or fat and the separation agent for a certain period of time, control of temperature of the
- 10 mixture and stationary separation process and centrifugal separation.

