Title: SEPARATION OF SUNFLOWER OIL AND WAX

Abstract: There is disclosed a method for treating a feed stream comprising sunflower oil, sunflower wax, and water, involving the adjustment of the pH of the feed stream to form a lipophilic phase and an aqueous phase which phases are separated to form a lipophilic stream and an aqueous stream. Also disclosed are methods for treating a lipophilic stream comprising sunflower wax and sunflower oil to form a solid wax and an extract or a solution comprising oil.
For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.
SEPARATION OF SUNFLOWER OIL AND WAX

CROSS REFERENCE TO RELATED APPLICATIONS

This application claims priority to co-pending U.S. provisional patent application Serial No. 60/659,937 filed March 9, 2005, the entire contents of which are incorporated herein by reference.

FIELD OF THE DISCLOSURE

The present invention generally relates to a system and method for separating sunflower oil and wax. The present invention more particularly relates to separating sunflower oil and wax from a stream formed on dewaxing of sunflower oil.

BACKGROUND OF THE DISCLOSURE

It is generally known to treat sunflower oil for wax removal. The treatment forms a stream comprising sunflower oil, sunflower wax and water. The stream can be used in feed and in other application as such or after separation of the lipophilic compounds from the water. Typically, the aqueous solution, as such or after separation of lipophilic matter, is sent to wastewater treatment. In many cases, the aqueous phase still contains a relatively large amount of organic matter, which represents major losses of lipophilic matter, on one hand, and increased waste-water treatment cost due to high biological oxygen demand ("BOD"), on the other. In other words, the known treatment has several disadvantages including difficulties in separation of the lipophilic compounds, which forms an aqueous waste stream with high BOD, and losses of lipophilic fraction.

Accordingly, there is a need for a method that separates efficiently the lipophilic compounds from a stream formed on dewaxing of sunflower oil. There is also a need for separating sunflower wax from the separated lipophilic
compound. There is also a need for separating sunflower oil from the separated lipophilic compound. It would be advantageous to provide a method for separating sunflower oil and wax filling any one or more of these needs or having other advantageous features.

SUMMARY OF THE DISCLOSURE

The present disclosure relates to a method for separating a feed stream comprising sunflower oil, sunflower wax, and water into a lipophilic stream and an aqueous stream. In this method, the pH of the feed stream is adjusted such that a lipophilic phase comprising wax and oil is formed, and an aqueous phase is formed. The lipophilic phase and the aqueous phase are separated, thereby forming a lipophilic stream and an aqueous stream.

The present disclosure also relates to a method for treating a lipophilic stream comprising sunflower wax and sunflower oil, such that there are formed a solid wax and an oil. The treatment is carried out by at least one of extracting the lipophilic stream with a solvent; or by adjusting solvent content and crystallizing wax; or by adjusting the temperature of the lipophilic stream and crystallizing wax; or by dissolving the lipophilic stream comprising the wax and oil, in an organic solvent to form a solution comprising wax, oil, and solvent, and then crystallizing the wax, to form a solid wax and either an extract comprising oil or a solution comprising oil.

DETAILED DESCRIPTION OF THE DISCLOSURE

The present disclosure relates to a method for separating a feed stream comprising sunflower oil, sunflower wax, and water into a lipophilic stream and an aqueous stream. In this method, the pH of the feed stream is adjusted such that a lipophilic phase comprising wax and oil is formed, and an aqueous phase is formed. The lipophilic phase and the aqueous phase are separated, thereby forming a lipophilic stream and an aqueous stream.

The present disclosure also relates to a method for treating a lipophilic stream comprising sunflower wax and sunflower oil, such that there are formed a solid wax and an oil. The treatment is carried out by extracting the lipophilic
stream with an organic solvent; or by adjusting organic solvent content and crystallizing wax; or by adjusting the temperature of the lipophilic stream and crystallizing wax; or by dissolving the lipophilic stream comprising the wax and oil, in an organic solvent to form a solution comprising wax, oil and organic solvent and then crystallizing the wax, to form a solid wax and either an extract comprising oil or a solution comprising oil.

Separation of oil from wax in the separated lipophilic stream ends up with two separate phases:

the 1st is preferred by the wax more than it is for oil and

the 2nd is preferred by the oil more than it is for wax

A more quantitative way to describe it uses the wax/oil weight ratio in the lipophilic phase and in the two phases formed in the operation, referred to here as R, R1 (1st phase) and R2 (2nd phase). The ratio in the 1st phase (R1) is greater than in the lipophilic phase (R), while the ratio in the 2nd (R2) is smaller than that in the lipophilic phase (R). Differently put, R1 > R > R2.

Typically, the 1st phase is solid, made mostly of solid wax. Typically, the 2nd phase is liquid and is made mainly of a solution of oil in a solvent.

In one case (Case A), the lipophilic stream is solid or partially solid, e.g. a paste, or is converted to that form. Such conversion could be done e.g. by cooling to a temperature where at least part of the wax solidifies. That temperature is dependent on the composition of the lipophilic phase. Alternatively, conversion to solid form could be conducted by removing solvent from the lipophilic phase, if solvent exists there. Solvent removal could be done by, e.g. by distillation. The solid or partially solid lipophilic phase is extracted with a solvent, preferably in a counter-current mode. In the extraction step, oil is extracted into the solvent to form an oil solution in the solvent. The wax is not extracted or extracted only to a limited extent. The non-extracted wax is separated from the oil solution in the solvent, e.g. by decanting, filtration or centrifugation. A wax product is formed and optionally further treated. The oil solution in the solvent could be used as such, combined with other process streams, e.g. ones formed in sunflower oil extraction, or further treated, e.g. by solvent distillation to form an oil product.
In an alternative (Case B), a solution containing oil and wax is treated. Preferably, the solution contains also a solvent. That solution could be the lipophilic phase as obtained in the step of separating the lipophilic phase and the aqueous phase, as in the case where solvent is used in such separating operation (per Claim 4) and/or in the case where the temperature of the lipophilic phase is high enough to melt the components. Alternatively, the solution is formed from the lipophilic phase, e.g. by adding solvent to it, dissolving it in a solvent, or changing the composition of existing solvent. The solution is treated for crystallizing wax out of it. Any method that leads to wax crystallization is suitable, as long as the wax/oil weight ratio in the crystallized wax is greater than that in the lipophilic phase. An example for crystallization method is cooling to a temperature where wax crystals start to form. That temperature depends on the composition of the cooled solution. For example, typically, the higher is the solvent content of the solution, the lower would that temperature be. Alternatively, crystallization is induced by solvent removal from the solution, e.g. by distillation. The amount of solvent to be removed is such that wax starts to crystallize at the temperature of solvent removal or after cooling. According to another alternative, the composition of the solution is changed by adding to it another component, which component does not dissolve well wax and is referred to as anti-solvent. An anti-solvent is added in an amount sufficient to induce wax crystallization. Wax crystals are separated from the oil-containing solution by any suitable method, such as decantation, filtration and centrifugation. As in Case A, wax crystals, which are a wax-enriched phase, and an oil solution are formed.

In the method for separating a feed stream comprising sunflower wax, sunflower oil, and water, into a lipophilic stream and an aqueous stream, in one embodiment, the pH of the feed stream is adjusted to less than about 9. In another embodiment, the pH is adjusted to a range of from about 0.5 to about 7. Any suitable pH may be utilized.

In another embodiment, an organic solvent is present during the separation of the lipophilic phase and the aqueous phase to form a lipophilic stream and an aqueous stream. Any organic solvent, which enables separating a feed stream
comprising sunflower wax, sunflower oil, and water, into a lipophilic stream and an aqueous stream may be utilized. In one embodiment, the organic solvent may be a component of the feed stream. In another embodiment, the organic solvent may be added prior to adjusting the pH of the feed stream, or during the adjustment of the pH of the feed stream, or after the pH of the feed stream has been adjusted. In another embodiment, the organic solvent may be any organic solvent. In one embodiment, the organic solvent may be selected from ethanol or a hydrocarbon having about 4 to about 8 carbon atoms, such as hexane, iso-hexane, butane, pentane, heptane and octane.

In another embodiment of the method for separating a feed stream comprising sunflower oil, sunflower wax, and water into a lipophilic stream and an aqueous stream, the phase separation may be carried out at any temperature up to the boiling temperature of the aqueous phase. In one embodiment, the separation of the lipophilic phase and the aqueous phase is carried out at a temperature greater than 40 degree C., and in another embodiment, at a temperature greater than about 60 degree C.

In another embodiment of the present disclosure, there is provided a method for treating a lipophilic stream comprising sunflower wax and sunflower oil, having a wax/oil weight/weight ratio of from about 10/1 to about 1/50, to form a solid wax and either an extract comprising oil or a solution comprising oil. In this method, the lipophilic stream is extracted with an organic solvent that will allow the treatment to occur; or in case it already contains a solvent, adjusting the solvent content; or by adjusting the temperature of the lipophilic stream; or where the wax and oil of the lipophilic stream are dissolved in an organic solvent to form a solution comprising wax, oil and organic solvent, and thereafter crystallizing the wax from the solution, to form a solid wax and a solution comprising oil. If desired, the solid wax may be separated from the solution comprising the oil, using any known techniques.

In one embodiment, the lipophilic stream that is treated to form a solid wax and either an extract comprising oil or a solution comprising oil, is a lipophilic stream that is produced by reducing the pH of a feed stream comprising sunflower
oil, sunflower wax, and water to thereby form a lipophilic phase and an aqueous phase, and then separating the phases to form a lipophilic stream and an aqueous stream. In another embodiment, the lipophilic stream that is treated to form a solid wax and either an extract comprising oil or a solution comprising oil, is a lipophilic stream produced by membrane dewaxing.

Membrane dewaxing is a treatment wherein a wax-containing oil is treated on a membrane to form two products, a dewaxed oil where wax concentration is smaller than the original one and a lipophilic stream containing wax and some oil. The amount of oil in the lipophilic stream is small compared with the amount of oil in the dewaxed oil stream. For example, the wax-containing oil is filtered on the membrane. Typically, the membrane composition is such that the oil permeates through the membrane, while the wax-containing lipophilic stream is retained on the membrane. For further information on membrane dewaxing, attention is called to U.S. Patent Numbers 6,355,173; 5,482,633; 5,066,504; and 4,545,940.

In another embodiment, the lipophilic stream comprising sunflower wax and sunflower oil, that is treated to form a solid wax and either an extract comprising oil or a solution comprising oil, comprises a wax/oil weight/weight ratio in a range of from about 3:1 to about 1:3.

In a further embodiment, the organic solvent used in treating the lipophilic stream to form a solid wax and either an extract comprising oil or a solution comprising oil, may be any organic solvent that will allow the treatment to occur. In one embodiment, the solvent that may be used in the treatment of the lipophilic stream comprising the sunflower wax and the sunflower oil, to form a solid wax and an extract comprising oil or a solution comprising oil, is an organic solvent. In another embodiment, the organic solvent used in treating the lipophilic stream may be ethanol or a hydrocarbon having from about 4 to about 8 carbon atoms, such as hexane, iso-hexane and butane, pentane, heptane and octane.

In another embodiment, the organic solvent, if used in the treatment of the feed stream comprising sunflower oil, sunflower wax, and water, is the same or similar solvent, as the organic solvent utilized in the treatment of the lipophilic
stream comprising sunflower wax and sunflower oil. For example, the solvents may have the same or similar polarity.

In a further embodiment, the method for treating a lipophilic stream comprising sunflower wax and sunflower oil described herein to form a solid wax and either an extract comprising oil or a solution comprising oil results in the separation of at least about 70% of the wax from the lipophilic stream.

In another embodiment, the method for treating the lipophilic stream comprising sunflower wax and sunflower oil described herein, involves the solvent contacting the lipophilic stream in a counter-current mode.

In another embodiment, the method for treating the lipophilic stream comprising sunflower wax and sunflower oil described herein, involves contacting the solvent and the lipophilic stream at a temperature above the solvent crystallization temperature, and in another embodiment, at a temperature of lower than about 40°C.

In another embodiment, the lipophilic stream comprising sunflower wax and sunflower oil described herein, is treated by dissolving the wax and oil in an organic solvent to form a solution comprising the wax, oil, and solvent, and subsequently crystallizing the wax from the solution to form a solid wax and a solution comprising the oil. In still another embodiment, the lipophilic stream is already a solution comprising the wax, oil, and solvent, and is treated for crystallizing the wax from the solution to form a solid wax and a solution comprising the oil. In these embodiments, the crystallizing may be carried out by at least one of solvent removal, addition of a non-solvent, cooling, and combinations thereof.

In another embodiment, the method for treating the lipophilic stream comprising sunflower wax and sunflower oil is as follows. A first part of the lipophilic stream comprising the sunflower wax and the sunflower oil wherein the wax/oil weight/weight ratio is from about 10:1 to about 1:50, is extracted with solvent to form a solid wax and an extract comprising oil. A second part of the lipophilic stream comprising the sunflower wax and the sunflower oil is then treated to form a solid wax and a solution comprising oil by dissolving the wax and
oil of the lipophilic stream in organic solvent, which is the extract comprising oil resulting from the extraction of the first part of the lipophilic stream. Thereafter the solution comprising wax and oil dissolved in the extract is treated to form a solid wax and a solution comprising oil by crystallizing wax from the solution.

BRIEF DESCRIPTION OF THE DRAWINGS

FIGURE 1 is a schematic diagram of an exemplary embodiment of the present disclosure.

FIGURE 2 is a schematic diagram of an alternative embodiment of the present disclosure.

DETAILED DESCRIPTION OF THE PREFERRED AND OTHER EXEMPLARY EMBODIMENTS

FIGURE 1 presents an exemplary embodiment of the disclosure. In general, such embodiment is directed to separation of oil and wax, preferably in the presence of an organic solvent and/or at elevated-temperature. Referring to FIGURE 1, a stream (44) comprising sunflower oil, sunflower wax, fatty acid salts and water is treated for lipids separation in operation (50).

Some oils contain a relatively high proportion of fatty compounds with a melting point higher than that of the bulk oil. Such fatty compounds are referred to here as wax. An example of that are esters of fatty acids and fatty alcohols present in some vegetable oils at concentrations of up to about 2000 ppm. Their solubility is strongly temperature dependent, so that on refrigeration of the oil, haziness may appear. In order to avoid this phenomenon, some vegetable oils are dewaxed (typically after degumming and alkali treatment) by chilling (winterization) followed by the removal of insolubles. Such removal uses various means, e.g. filtration (optionally, using a filter aid such as a diatomaceous earth), centrifugation and various membrane separations. Separation of the insolubles is conducted at relatively low temperature in order to
avoid re-dissolution. At those temperatures, oil is viscous and fractions of it stay with the separated insolubles leading to significant oil losses with the wax.

In the exemplary embodiment of FIGURE 1, stream (44) is a product of dewaxing sunflower oil. Sunflower material (12) is extracted in operation (20). The sunflower material (12) is produced by dehulling, flaking and pressing out part of the oil. The rest of the oil is extracted from the pressed flakes in (20) with hexane (32) to form flakes (24) and a miscella (22) comprising hexane, sunflower oil (triglycerides) and other extracted compounds, such as sunflower wax, phospholipids and fatty acids. The flakes are typically desolventized and used in animal feed. The miscella is desolventized in (30) by distillation of the hexane, to form a hexane stream, which is recycled (32) to the extraction stage (20), and a crude oil stream (34). The crude oil can be combined with the oil obtained by pressing the flakes and the combined stream is refined by known methods such as degumming and alkali treatment (not shown in FIGURE 1). A typical operation in the refining of sunflower oil is dewaxing (40). According to the embodiment of FIGURE 1, the crude oil is mixed with an alkali, e.g. a hydroxide, carbonate or bicarbonate of sodium, potassium or ammonium (35) and water or an aqueous solution (37) (or an aqueous solution of the alkali) and the mixture is cooled to facilitate crystallization of sunflower wax. The crystallized wax is separated from the oil, e.g. by centrifugation, to form dewaxed oil (42) and a wax-containing stream (44). Alternatively, the crystallized wax is separated from the oil by filtration on a filter aid so that the wax is obtained with a filter aid. In other methods, dewaxing and bleaching are combined and wax is obtained with a bleaching agent, e.g. bleaching clay. In those cases, stream (44) is formed by separation of filter aid and/or bleaching agent, e.g. solvent wash. According to still another alternative, the crystallized wax is separated by membrane filtration. The solvent (e.g. hexane) may be partially or fully removed (e.g. by distillation) before the improved solid-liquid separation, or left there for the following steps. In other cases, dewaxing is combined with alkali treatment so that waxes and oil are present in the soapstock separated from the oil. Also feasible is a combination of degumming and dewaxing so that wax and oil are present in the gums. In such
cases there might be a preference to first separate the oil and the wax from the soapstock or the gums to form stream (44).

Stream (44) is typically a non-homogeneous phase and comprises typically about 70% to about 98% water, depending on the composition of stream (34) and the ratio between stream (34) and stream (37). It typically contains sunflower oil (triglycerides) and sunflower wax in wax/oil weight/weight ratio in the range between about 10:1 and about 1:50, more typically about 3:1 and 1:10. It also contains fatty acid salts (soap), and possibly excess alkali, resulting from the reaction of the alkali with free fatty acids in the treated crude oil. The weight/weight ratio between the wax and the soap is typically in the range between about 10:1 and about 1:10.

Stream (44) is treated in (50) for separation of the lipids from the water. Separation is facilitated by adjusting the pH of the stream. According to one preferred embodiment, the pH of the stream is adjusted to less than about 9, more suitably to a range between about 0.5 and about 7. Since the stream is non-homogenous and contains dispersed organic matter, the pH is referred to the aqueous part.

The pH adjustment can be carried out by removal of a base. Thus, when ammonia is used as the alkali (35) in dewaxing, pH adjustment in (50) may involve distillation of ammonia. In another embodiment, pH adjustment of the stream is conducted by adding an acid (45) or an acidic compound (or by contacting with an acidic cation exchanger). Any acidic compound is suitable, if the acid is strong enough to reach the desired pH. In one embodiment, the acid is a strong mineral one, such as sulfuric acid.

Adjustment of pH facilitates separation of lipids from the water in the treated stream. After pH adjustment, the phases can be separated, e.g. by centrifugation, into a lipophilic phase (52) and an aqueous solution (54). The aqueous solution (54) may contain the salt of the alkali used in dewaxing and the acid used in pH adjustment and other soluble compounds.

According to another embodiment, phase separation is improved by the presence of an organic solvent. The organic solvent might be present in the
treated stream (44) prior to pH adjustment. According to an alternative embodiment, the solvent is added (47) to the stream prior to, during, or after pH adjustment. According to another embodiment, the organic solvent is one of ethanol and a hydrocarbon having about 4 to about 8 carbon atoms. According to another embodiment, the organic solvent comprises hexane or is hexane. The amount of organic solvent to be added is determined by the composition of the treated stream and by the nature of the solvent. For example, in an embodiment where the organic solvent is hexane, the water content of the treated stream (44) is about 95% and the contents of oil, fatty acid and wax are similar to each other, the amount of the solvent is in the range of from about 1% to about 10% of the treated stream. According to another embodiment, the range is from about 2% to about 7%.

According to another embodiment, pH adjustment and/or the following phase separation is conducted at a temperature lower than about 40°C. According to another embodiment the temperature is lower than about 25°C.

Conducting the phase separation in the presence of an organic solvent results in the formation of a lipophilic phase, typically solid, that contains the added solvent and floats on the aqueous phase and is easy to separate. In most cases, separation can be done by decantation with no need for centrifugation. The separated aqueous phase (54) is quite clear and low in dispersed organic matter. Losses of lipophilic compounds are minimized and so is the cost of wastewater treatment. The separated aqueous phase or part of it can also be used in the process, including recycle to the oil refining and/or to the dewaxing (40).

According to an alternative embodiment, pH adjustment and/or phase separation is conducted at an elevated temperature of in one embodiment at least about 40°C, in another embodiment at least about 60°C, and in another embodiment at least about 80°C. The inventors have found that at these elevated temperature a liquid lipophilic phase and an aqueous phase are formed after pH adjustment. The phases separate well and the separated aqueous phase is relatively clear and low in organics.
The lipophilic composition separated from the aqueous phase according to the above methods (52) consists mainly of wax, oil and fatty acids separated from the sunflower oil in the dewaxing operation (40). Similar compositions are formed by other processes, e.g. membrane dewaxing of sunflower oil or solvent wash of wax from filter aid or bleaching agent. Typically, in such compositions the weight/weight ratio between the oil (triglyceride) and the wax is in the range between about 10:1 and about 1:50, more typically between about 3:1 and 1:10. In some applications, the lipophilic composition is used as such or after pre-treatment, such as desolventization. More value, however, could be gained by separating, at least partially wax and/or oil from the other components. Such separation forms a valuable, concentrated and purified sunflower wax product. The separated oil, if relatively low in wax, can be combined with the crude oil, so that oil losses due to dewaxing are practically eliminated. The inventors of the present invention have discovered methods that achieve the required separation of wax and oil from lipophilic compositions containing them. Those are described in the following embodiments and in FIGURES 1 and 2. The following describes an embodiment where the wax is a sunflower wax and the oil is sunflower oil. The methods are suitable for lipophilic compounds containing sunflower oil and wax of any source, but is described in the following for the specific case where the lipophilic compound is formed by pH adjustment of an aqueous stream from sunflower dewaxing.

According to an embodiment of the invention shown in FIGURE 1, sunflower oil (and optionally also fatty acids) is extracted in operation (60) from the lipophilic composition (52) into an organic solvent (53) at a temperature wherein the sunflower wax is in a solid form. According to another embodiment, extraction is conducted in a counter-current mode. Optionally, the extraction is conducted in a vertical column where the lipophilic compound and the organic solvent are flowing counter-currently. According to another embodiment, the organic solvent is of lower specific weight than the lipophilic compound and is flowing upwards. According to another embodiment, the organic solvent is pumped with a low-turbulence pump, e.g. a Moyner pump. The extraction forms a
concentrated, solid sunflower wax, which is separated (62) from the extract comprising sunflower oil (64).

An alternative embodiment of the invention is shown in FIGURE 2, where operations (20), (30), (40) and (50) are similar to those in FIGURE 1. According to this alternative embodiment, wax is separated from the oil (and optionally also from fatty acids) by crystallization. The lipophilic composition (52) is liquefied by means of heating, adding a reagent or dissolution. In the following step, crystallization is induced, e.g. by cooling, adding a component (soap, another surfactant, urea, thiourea, acetamide, a non-solvent) or removing a component to crystallize wax. FIGURE 2 presents another embodiment where the lipophilic compound is dissolved in an organic solvent (68) in operation (70) to form an organic solution (72) comprising the solvent, the wax, the oil and fatty acids, if present in the lipophilic composition. In some process options, the lipophilic composition (52) also consist of water, e.g. due to incomplete separation of the lipophilic composition from the aqueous phase in (40). In those cases, an aqueous phase is formed during dissolution in the organic solvent and is separated (see aqueous phase 64). In the following step, the organic solution (72) is treated for the crystallization of wax in operation (80). Wax crystallization is facilitated by removal of solvent from the solution, by cooling, by the addition of a non-solvent or by a combination of those. Optionally crystallization seeds are added (not shown in the FIGURE). A concentrated, solid sunflower wax is formed and separated (82) from a solution containing the organic solvent and the oil (84). Separation may use means such as decantation, centrifugation, filtration and membrane filtration.

According to an exemplary embodiment the organic solvent for extracting the oil (53) and/or for dissolution of the lipophilic compound (68) is selected from a group consisting of ethanol and a hydrocarbon having about 4 to about 8 carbon atoms. According to another embodiment, the organic solvent comprises hexane or is hexane. According to another embodiment, the lipophilic composition (52) is formed by dewaxing sunflower oil and the organic solvent
used in (63) and/or (68) is the same solvent used for the extraction of sunflower oil (32) in operation (20).

The formed solid wax (62) and/or (82) is suitable for use in various applications, as such, or after further treatment. Such treatment may involve at least one of desolventization, recrystallization, wash with a solvent, wash with water and other means of purification.

According to another embodiment, oil separated from the wax, for example oil in (64) of FIGURE 1 and/or oil in (84) of FIGURE 2, is combined with extracted oil and ends up in refined and dewaxed oil. According to another embodiment, the solvent stream containing that oil ((64) and/or (84)) is combined with the miscella of oil extraction from sunflower material (22) and desolventized together so that oil separated from the wax ends up in (34). According to an alternative embodiment, the solvent stream containing that oil ((64) and/or (84)) is first desolventized and then the oil is combined with (34). The combined oil may contain small amounts of wax extracted with the oil in (60) or left dissolved in (80). Such small amounts present no problem since it is removed in the dewaxing operation (40) of the combined stream. Yet, if desired, the wax content of stream (64) and/or (84) can be minimized, and wax recovery in (60) and/or (80) can be increased by conducting those operations at reduced temperature – less than about 25 according to another mode and less than about 15 according to another mode.

According to an alternative embodiment not shown in the FIGURE, the lipophilic compound, e.g. (52) is divided into at least two streams. One of those is treated by oil extraction, as in FIGURE 1 and the other by crystallization, as in FIGURE 2. The wax products of the two treatments may serve different needs. Such two-line operation enables synergism and cost saving. For example, the extract (64) is used as an organic solvent in (68).

EXAMPLES

While the invention will now be described in connection with certain embodiments in the following examples so that aspects thereof may be more fully
understood and appreciated, the examples are not intended to limit the invention to these particular examples.

**Comparative Example A**

An aqueous suspension, obtained on industrial sunflower oil dewaxing (ADW), was examined here and in the following examples and comparative examples. The ADW contained sunflower oil, sunflower wax and other components. No major phase separation was observed when ADW was kept with no treatment at room temperature for 100 days.

**Example 1**

0.223 gr concentrated H₂SO₄ was added drop-wise, while stirring, to 106gr ADW to reach pH of 3.2. Fast phase separation was observed.

**Example 2**

0.48gr concentrated H₂SO₄ was added drop-wise, while stirring, to 106gr ADW to reach pH of 1.06. Fast phase separation was observed. The heavier phase was aqueous and quite clear.

**Example 3**

0.45gr concentrated HCl was added drop-wise, while stirring, to 109gr ADW to reach pH of 1.0. Fast phase separation was observed. The heavier phase was aqueous and quite clear.

ADW is a stable suspension of lipophilic compounds in an aqueous solution, as demonstrated by Comparative Example A. Adjustment of pH with H₂SO₄ or HCl facilitates phase separation, as shown in Examples 1-3.
Comparative Example B
The light phases, formed in Examples 2 and 3, were separated from the heavy phases and each of the light phases was dissolved in hot hexane. Two liquid phases were formed in both cases. After phase separation the newly formed aqueous phases were weighed. The weights of the newly formed aqueous phases were 15.2gr and 16.1gr for the light phases formed in Experiment 2 and Experiment 3, respectively.

Example 4
0.48gr concentrated H₂SO₄ and 106gr solution of ADW were introduced into a vial, as in Example 2, to form an aqueous phase with pH of 1.06 and a semi-solid lipophilic phase. 11.24gr hexane was added into a vial leading to immediate coagulation.
The aqueous phase was removed and the remaining light phase was dissolved in hot hexane. Two liquid phases were formed.
The newly formed heavy phase was weighed. Its weight was 3gr.

Example 5
0.45gr concentrated HCl and 109gr solution of ADW were introduced into a vial, as in Example 3, to form an aqueous phase with pH of 1.0 and a semi-solid lipophilic phase.
4gr hexane was added into a vial with gentle stirring, leading to immediate coagulation. After 30 seconds, particles of light phase were floating on top of the aqueous phase. No liquid hexane phase was observed.
The light phase was separated and dissolved in hot hexane. Two liquid phases were formed. The newly formed heavy phase was weighed. Its weight was 3.2gr.
Example 6

0.40gr concentrated H₂SO₄ and 110gr solution of ADW were introduced into a vial to form an aqueous phase with pH of 1.46 and a semi-solid lipophilic phase. The vial was heated to 90°C. The light phase liquefied to get a thin layer of liquid phase floating on top of the aqueous phase. The aqueous phase was removed, leaving behind the liquid light phase. 20gr of hot hexane were added to the vial. Few droplets of newly formed aqueous phase were observed.

Example 7

The experiment in Example 6 was repeated up to the point of separation of the aqueous phase and leaving a liquid lipophilic phase in the vial. That liquid lipophilic phase was contacted with cold water. Solid particles were formed, floating on top of the water. Those particles are easily separated on a 100 micron screen.

Comparative Example B shows that, while pH adjustment facilitates phase separation, the formed semi-solid light lipophilic phase still contains significant amounts of water (which separates on dissolution in hot hexane). Examples 4-5 demonstrate that addition of hexane to the system leads to a better phase separation (without forming hexane solution), so that the formed light phase contains much less water. Similar improvement in phase separation is achieved by conducting the separation at an elevated temperature, as demonstrated in Example 6. The light phase is in liquid form (rather than semi-solid in Examples 1-5) and separates well from the aqueous phase, carrying practically no water with it. The liquid lipophilic phase could be cooled in contact with cold water to form easy-to-separate solid particles.
Comparative Example C
A sample of the light phase particles obtained in Example 4 was dried and analyzed. It contained about 25% wax, about 35% Oil and about 40% fatty acids.

Example 8
Another sample of the light phase particles obtained in Example 4 was contacted with hexane at 1 (solids) to 4 (hexane) w/w ratio. The mixture was shaken at 20°C for 30min. The suspension was centrifuged and the liquid phase was removed. Samples of both phases were analyzed and the results are presented as 8A, in Table 1 on solvent-free basis.

The remaining solids were contacted again with fresh hexane at the same w/w ratio and temperature and for similar time. Then the phases were separated by centrifugation and analyzed. The results are presented as 8B in Table 1 on solvent-free basis.

<table>
<thead>
<tr>
<th></th>
<th>Fatty acids (Wt%)</th>
<th>Oil (Wt%)</th>
<th>Wax (Wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Particles prior to contact with hexane</td>
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<td>35</td>
<td>25</td>
</tr>
<tr>
<td>Particle of 8A</td>
<td>19.7</td>
<td>4.5</td>
<td>75.8</td>
</tr>
<tr>
<td>Liquid phase of 8A</td>
<td>55</td>
<td>27</td>
<td>17</td>
</tr>
<tr>
<td>Particle of 8B</td>
<td>10.13</td>
<td>2.31</td>
<td>87.6</td>
</tr>
<tr>
<td>Liquid phase of 8A</td>
<td>55.5</td>
<td>26.5</td>
<td>17</td>
</tr>
</tbody>
</table>

Example 8 demonstrates the efficiency of hexane wash in purification of wax in the pH adjustment-separated lipophilic phase. Wax concentration increases from 25% to about 76% and about 88% after the first and the second hexane wash, respectively.
**Example 9 re-crystallization**

0.42gr concentrated H₂SO₄ and 105gr solution of WPX were introduced into a vial to reach pH of 1.2 in the aqueous phase. 3.0gr hexane were added, leading to coagulation and formation of large light phase particles. Those were separated from the aqueous phase by filtration on a coarse screen and a sample of the particles was analyzed.

The separated particles were mixed with 20gr of hot hexane to form a solution, which was then filtered. The clear solution was cooled to 8°C, whereby solid particles and liquid were formed. The phases were separated and analyzed. The results of the analyses are presented in Table 2 on a solvent-free basis.

<table>
<thead>
<tr>
<th></th>
<th>Fatty acids (Wt%)</th>
<th>Oil (Wt%)</th>
<th>Wax (Wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Particles formed on acidulation</td>
<td>40</td>
<td>35</td>
<td>25</td>
</tr>
<tr>
<td>Composition of re-crystallized solids</td>
<td>4.6</td>
<td>6.0</td>
<td>89.1</td>
</tr>
<tr>
<td>Composition of the solutes in the hexane phase</td>
<td>54.0</td>
<td>41.5</td>
<td>4.7</td>
</tr>
</tbody>
</table>

Example 9 demonstrates wax purification via dissolution of the pH adjusted formed particles followed by recrystallization. Wax concentration increases from about 25% to about 89%.

**Comparative Example D**

ADW was pH adjusted and heated to 90°C and the phases were separated as in Example 6. The lipophilic phase was analyzed and the results are presented in Table 3 on a solvent-free basis.
Example 10
A liquid light phase is formed in a procedure similar to that in Comparative Example D. 6.4gr hexane at 25°C was added to the hot lipid phase obtained from 16.4gr ADW and cooled to about 70°C. The mixture was shaken, using a vortex, and centrifuged. The solid phase was analyzed and the results are presented in Table 3 on a solvent-free basis.

Example 11
0.45gr of the solids obtained in Example 10 and 5gr hexane were shaken at 25°C for 30 min. The solids were filtered, dried and analyzed. The results are presented in Table 3 on a solvent-free basis.

Table 3

<table>
<thead>
<tr>
<th></th>
<th>Wax (Wt%)</th>
<th>Fatty acids (Wt%)</th>
<th>Oil (Wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>D</td>
<td>25</td>
<td>45</td>
<td>35</td>
</tr>
<tr>
<td>Example 10</td>
<td>68</td>
<td>25</td>
<td>6.5</td>
</tr>
<tr>
<td>Example 11</td>
<td>91</td>
<td>7</td>
<td>2</td>
</tr>
</tbody>
</table>

Examples 10 and 11 demonstrate purification of wax in a lipophilic phase formed by pH adjustment of ADW and phase separation at an elevated temperature. Wax purity increased from 25% to 68% in the first stage and to 91% in the second.

While several exemplary embodiments described in this disclosure are presently shown, it should be understood that these embodiments are offered by way of example only. The invention is not limited to a particular embodiment, but extends to various modifications, combinations, and permutations.
CLAIMS

What is claimed is:

1. A method for treating a feed stream comprising sunflower oil, sunflower wax, and water comprising:
   a. adjusting the pH of the feed stream to form a lipophilic phase comprising the wax and the oil, and an aqueous phase; and
   b. separating the lipophilic phase and the aqueous phase to form a lipophilic stream and an aqueous stream.

2. The method of claim 1 wherein the pH of the feed stream is adjusted to a pH of less than about 9.

3. The method of claim 2 wherein the pH is from about 0.5 to about 7.

4. The method of claim 1 wherein the lipophilic phase is separated from the aqueous phase in the presence of an organic solvent, that enables separating the feed stream into a lipophilic stream and an aqueous stream.

5. The method of claim 4 wherein the organic solvent is a component of the feed stream.

6. The method of claim 4 wherein the organic solvent is added prior to adjusting the pH of the feed stream, during the adjustment of the pH of the feed stream, or after the pH of the feed stream has been adjusted.

7. The method of claim 4 wherein the organic solvent is selected from the group consisting of ethanol and a hydrocarbon having about 4 to about 8 carbon atoms.

8. The method of claim 7 wherein the organic solvent comprises hexane.
9. The method of claim 1 wherein the separation of the lipophilic phase and
the aqueous phase is conducted at a temperature up to the boiling temperature of
the aqueous phase.

10. The method of claim 9 wherein the temperature is greater than about 60°C.

11. A method for treating a lipophilic stream comprising sunflower wax and
sunflower oil in a wax/oil weight/weight ratio of from about 10:1 to about 1:50, to
form a solid wax and an extract comprising oil or a first solution comprising oil,
comprising at least one, or more, process selected from the group consisting of:
   a. extracting the lipophilic stream with an organic solvent;
   b. adjusting the content of the organic solvent and crystallizing wax;
   c. adjusting the temperature of the lipophilic stream and crystallizing wax;
and
   d. dissolving the wax and oil of the lipophilic stream in an organic solvent
to form a second solution comprising the wax, oil, and solvent, and crystallizing
wax from the second solution.

12. The method of claim 11 wherein the organic solvent is an organic solvent
that will allow the treatment to occur.

13. The method of claim 11 further comprising separating the solid wax from
the extract comprising oil or the solution comprising oil.

14. The method of claim 11 wherein the lipophilic stream comprising sunflower
wax and sunflower oil is formed by a method selected from the group consisting of
the method according to claim 1 and membrane dewaxing.

15. The method of claim 11 wherein the wax/oil weight/weight ratio is from
about 3:1 to about 1:10.

16. The method of claim 12 wherein the organic solvent is selected from the
group consisting of ethanol and a hydrocarbon having from about 4 to about 8
carbon atoms.
17. The method of claim 16 wherein the organic solvent comprises hexane.

18. The method of claim 11 wherein at least about 70% of the wax is separated from the lipophilic stream.

19. The method of claim 11 wherein contact with the organic solvent is conducted in a counter-current mode.

20. The method of claim 11 wherein contact with the organic solvent is conducted at a temperature above the solvent crystallization temperature.

21. The method of claim 20 wherein the temperature is lower than about 40°C.

22. The method of claim 11 wherein crystallization is conducted by at least one, or more, methods selected from the group consisting of solvent removal, addition of a non-solvent, and cooling.

23. The method of claim 11 wherein a part of the lipophilic stream is treated with a solvent to form the extract comprising oil, which extract is then utilized as the organic solvent in treating another part of the lipophilic stream wherein the treatment comprises dissolving the wax and oil in the organic solvent to form a solution comprising wax, oil and solvent, and then crystallizing the wax from the solution.