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PROCESSES OF PRODUCING HIGH MELTING
POINT WAXES

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The present application is a continuation in part of my co-pending United States Patent application, Serial No. 412,597, filed on September 27, 1941, and which is now abandoned, for "Processes of producing high melting point waxes."

The present invention relates to processes of producing high melting point, preferably micro-crystalline waxes from crude oil containing a high percentage of wax bearing substances, and more particularly to processes of producing such waxes from crude oil tank bottoms.

It is one of the objects of my present invention to obtain in an efficient and easy way, by simple processes, high melting point waxes directly from dehydrated crude oil, tank bottoms, crude oil emulsions, and other wax-bearing substances which contain not only high melting waxes and heavy oils but also asphaltic ingredients and distillable hydrocarbons which are liquid at ordinary temperatures, as light oils and gasoline.

It is a further object of my present invention to avoid the usual solvent treatments for separation of the high melting point waxes from heavy oils which treatments are not only cumbersome and expensive but also require very special equipment for the handling of the solvents in order to eliminate losses.

It is still a further object of my present invention to recover high melting point micro-crystalline waxes from deposits which settle in the form of emulsions when crude oils containing wax bearing substances and asphaltic ingredients are stored.

It is another object of my present invention to greatly simplify and shorten the time needed for obtaining micro-crystalline waxes from tank bottoms and similar crude oils by a process which eliminates the necessity of a separate solvent separation step after distillation.

Still another object of my present invention is a process which enables separation of high melting point amorphous micro-crystalline waxes from heavy oils present in the crude oil before distillation.

It is well-known that oil-free high melting point waxes cannot be obtained from crude oil, i. e., oil containing not only wax-bearing ingredients but also heavy oils, by distillation alone because these heavy oils cannot be removed by distillation without cracking. Thus, by distillation alone only a base stock containing also heavy oils can be produced from which base stock high melting point waxes might then be derived.

Thus, such wax base stocks produced by reducing tank bottoms, i. e., by the removal of the

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light oils and lower melting point waxes by distillation, still contain heavy oils besides high melting point waxes; such heavy oils can be removed after distillation only by cumbersome, lengthy and expensive solvent treatments.

In order to avoid the presence of asphaltic bodies during distillation, it is known to separate such asphaltic ingredients already before distillation by proper treatment, e. g., with sulphuric acid. However, this does not affect the necessity of solvent treatment after distillation, since the obtained wax base stock still contains heavy oils which have to be removed by separate solvent treatment. Thus, in the usual processes for producing high melting point waxes, the wax-bearing crude oil is first subjected to an acid treatment to remove the asphaltic ingredients, then reduced to remove the light oils, and finally subjected to a complicated treatment to remove the heavy oils; this latter treatment consists usually in dissolving the wax base stock obtained by reduction in an appropriate solvent at an elevated temperature, thereafter cooling or chilling the thus obtained solution to a temperature sufficiently low to effect precipitation of the wax, removal of the precipitated wax by low pressure or vacuum filtration, washing of the thus removed wax with cold solvent, and finally removal of this wash solvent by distillation. It is often necessary to repeat such dissolving, cooling and removal of the wax several times in order to obtain high melting point waxes absolutely free of heavy oils.

During all solvent treatments, for instance those described above, it is important that the wax precipitated from the solvent is not too voluminous or porous, does not retain large amounts of solvents and dissolved oils, and shows good washing qualities. With many types of oils or solvents, it is necessary to add so-called crystallization modifiers to the waxy oil solution before chilling in order to obtain a slurry which can be filtered without substantial losses. The weight of the solvent present in the so-called dry cake after filtering is sometimes two to four times that of the wax and this solvent, of course, contains a high percentage of heavy oil; this heavy oil cannot be removed otherwise than by repeated separate washing operation. It is therefore of utmost importance in all solvent treatments to obtain the wax cake in a compact form showing good washing qualities.

In this connection, it should be mentioned that the presence of asphaltic bodies in the solution during solvent treatments greatly reduces the congealing tendencies of the amorphous waxes in

the solution and that such asphaltic bodies are very useful as so-called wax separations aids. Therefore, in case de-asphaltized wax base stocks are subjected to solvent treatments, such solvents have to be chosen with particular care so as not to swell or solvate the wax crystals, since as mentioned above the structure assumed by the wax when precipitated from the solvent is of utmost importance. Because the so-called liquid petroleum solvents, i. e., the liquid members of the aliphatic hydrocarbon series exercise a marked swelling and solvating effect on refined, i. e., de-asphaltized micro-crystalline waxes, it has been found advisable not to use such liquid petroleum solvents for the removal of heavy oils from waxes but it is customary to use very specific rather expensive solvents as ethylene dichloride, methyl-ethyl-ketone and similar solvents which have only a slight swelling or solvating effect on refined waxes. Since, as stated above, such solvents are expensive, complicated processes have to be used to recover them after the treatment so as to eliminate losses of the same.

With the objects listed in the first paragraphs of this description and also with the additional object of avoiding all disadvantages of the above described known processes, my present invention mainly consists in processes of producing high melting point waxes from crude oil having a gravity A. P. I. of at least 30 to 40 degrees and containing dissolved wax-bearing substances and asphaltic ingredients by cooling this crude oil in the presence of the asphaltic ingredients contained in the same from a temperature at which all the waxy ingredients of said oil, including the high melting point waxes contained in the same, are in solution to a temperature at which mainly the high melting point waxes are precipitated from the oil, while substantially all lower melting point waxes remain in solution, removing the thus formed precipitate and separating the high melting point waxes from this precipitate by distillation.

In this connection, I wish to emphasize that it is of great importance that the above described wax separation is carried out before acid treatment, i. e., before removal of the asphaltic ingredients, since the same greatly reduce the congealing tendencies of the precipitated high melting point waxes and are very useful as wax separation aids. Therefore, the removed wax precipitate occludes almost no light oils and only a small percentage of low melting point waxes and thus the amount of heavy oils present in the removed wax precipitate is negligible.

In this connection, it should also be stressed that the light oils forming the main part of the crude oil are excellent selective solvents for the separation of high melting point waxes from lower melting point waxes and heavy oils when used as described above, i. e., when the precipitation is carried out in the presence of asphaltic ingredients.

I have found that from heated solutions of amorphous waxes in crude oil or crude oil naphtha mixtures still containing asphaltic bodies substantially only the high melting point waxes precipitate together with some asphaltic bodies when these solutions are cooled as described above. I found also that under such conditions the wax precipitate can be easily separated from the crude oil and when filtered, a filter cake is formed which retains only a relatively small amount of solvent, i. e., crude oil, and almost no heavy oils. This filter cake can easily be washed with fresh gasoline

to render it absolutely oil free as the asphaltic ingredients still present in the filter cake reduce the congealing tendency of the wax to such a degree that no swelling or dissolution of the high melting point waxes takes place while in contact with the cold wash gasoline.

I found that best results for precipitating the wax in filtrable form in accordance with my above described process are obtained when the crude oil shows a gravity A. P. I. of at least 30 to 40 degrees or is brought to such a gravity by dilution with higher gravity oils, e. g., light oils or gasoline.

In this connection, it should be stressed that there is a definite interrelation between the asphalt contents of the crude oil or the crude oil solution subjected to precipitation and the required gravity, namely the higher the asphalt contents, the lower the gravity required; this is due to the reduction of the congealing tendencies of the amorphous waxes by the asphalt content of the crude oil. In general, the quantity of sulphuric acid and clay required for removal of the asphaltic bodies and bleaching of the obtained wax precipitate to comparable colors may be taken as a guide or even as a measure for determination of the necessary gravity of the oil, i. e., for the amount of light oil or gasoline to be added as solvent. Thus, it is evident that if crude oils having a low gravity, e. g., crude oil tank bottoms are used as initial materials, the same have to be diluted with light oils, naphtha or gasoline until the obtained solution has a gravity A. P. I. of at least 30 to 40 degrees.

Crude oil tank bottoms consist of emulsions of water and crude oil and contain a relatively high percentage of high melting point waxes, i. e., usually from 5% to 10%, and among other impurities also asphaltic ingredients. I have found that if such low gravity crude oil emulsions, i. e., crude oil tank bottoms, are dissolved in crude oil or crude oil naphtha mixtures, heated and thereafter cooled as described above, the high melting point waxes contained in the dissolved crude oil emulsions would not form a substantially oil-free precipitate; since the emulsions are very stable, such precipitation would have the result that emulsified crude oil and water together with the high melting point waxes would be precipitated as described above and thus the high melting point waxes could not be obtained free from heavy oil.

Therefore, in case crude oil emulsions are used, it is of utmost importance that the emulsions be first broken and the water separated before the bottoms are dissolved in crude oil of higher gravity or crude oil naphtha mixtures; after the water is separated from the crude oil tank bottoms, i. e., after the crude oil tank bottoms are dehydrated, these dehydrated tank bottoms are diluted with oil of higher gravity, heated and cooled; as result of such cooling, all the high melting point waxes together with a small percentage of lower melting point waxes and some occluded crude oil will be precipitated; this precipitate will be almost free of heavy oils, as explained above.

It is known to produce waxes from tank bottoms, crude oil emulsions and the like. However, all processes for these purposes contain as main process steps dehydrating and cleaning of the tank bottoms followed by immediate distillation by which a still bottom is obtained which contains no light oils but still contains all asphaltic bodies and heavy oils besides the waxes. In the usual practice, this still bottom is then subjected

to an acid treatment and filtered through clay in order to produce a base stock from which high melting point waxes may be derived. This base stock is thereafter subjected to repeated known solvent treatments to remove the heavy oils. My processes described in the preceding paragraphs do away with any solvent treatments after distillation and are thus not only much simpler but also substantially less expensive and cumbersome. In connection with above, it should be mentioned that substantially less sulphuric acid and bleaching clay are needed for my new processes than for those known at present, since in the known processes a great percentage of the acid and clay is used up by the heavy oils and soluble asphaltic ingredients not present in the precipitate obtained by my processes.

I have found that in order to remove all crude oil from the wax precipitate, it is in some cases advantageous to wash the same with cold gasoline so as to render the wax entirely oil free. This can be done without any danger of dissolution or swelling of the wax particles, since enough asphaltic bodies are co-precipitated with the high melting point waxes to reduce the coagulating tendencies of the wax while it is in contact with the cold wash gasoline.

I have furthermore found that it is advisable to de-asphaltize and bleach the precipitate before distillation. In accordance with a preferred embodiment of my invention, this de-asphaltization by acid treatment or similar means is preferably carried out before removal of the wash gasoline; in this way, an acid sludge containing the asphaltic ingredients is formed which settles very fast and can be removed easily. It is also possible to bleach the thus obtained wax-gasoline solution without removal of the gasoline, e. g., by percolating it through a clay tower or contacting it with activated clay. However, I wish to stress that in accordance with another embodiment of my present invention it is also possible to combine the bleaching step with the distillation step as will be described below in detail.

Although my above described processes can be carried out in various ways, I prefer to include into them the following process steps:

First of all, the crude oil containing wax-bearing substances, asphaltic ingredients and water is freed of dirt and water by any suitable process. Then, the thus obtained dehydrated crude oil is brought to a gravity A. P. I. of at least 30 to 40 degrees by addition of gasoline or light oils, if it had a lower gravity; in case it has the required gravity, no gasoline, light oils or other solvents have to be added.

This crude oil is then heated in a tank with steam coils or by piping it through steam heated pipes to a temperature of about 160° to 180° F., i. e., until a clear solution is obtained. This solution might, however, contain small particles of undissolved asphaltic ingredients in suspension; the presence of such asphalt particles in no way impairs the results of my process.

Thereafter, the temperature of the crude oil or crude oil solution is lowered by well-known means to about 10° to 20° F. above that temperature at which the high melting point waxes contained in the heated crude oil are precipitated; during such cooling, it is advisable to keep the crude oil in motion so as to keep the fine particles of undissolved asphaltic ingredients in suspension and prevent precipitation of the same, as they are useful as nuclei for precipitation of the wax.

As next step, the fine granular precipitate of

high melting point waxes and asphaltic bodies formed by this cooling is separated by cold settling, centrifuging or filtering during which separation the crude oil is cooled to the precipitation temperature of the high melting point waxes. I prefer to remove the high melting point waxes by means of continuous rotary drum type vacuum filters which allow continuous and automatic filtering and washing of the wax cake.

Thereafter, if lighter colors are required, it is advisable to treat the filtered and washed precipitate with about 5% to 10% of concentrated sulphuric acid and to remove the formed acid sludge together with the asphaltic ingredients. Finally, the wax-gasoline solution can be bleached in well-known way and the wash gasoline removed by distillation.

The thus obtained wax composition—if not washed with gasoline—consists mainly of high melting point waxes, a small percentage of low melting point waxes, light oils, asphaltic ingredients and only traces of heavy oils; if washed with gasoline, it is absolutely oil free. This wax composition might then be distilled so as to remove all low melting point waxes and light oils and to obtain pure high melting point waxes by distillation in a vacuum and/or by steam.

As mentioned above, I may use as initial materials for my processes the most different types of crude oils; the only requirement is that besides high melting point waxes they also contain asphaltic ingredients and preferably distillable hydrocarbons. Thus, the term "crude oil" as used above and in the following description and claims is intended to define not only crude oils of usual type but to comprehend also tank bottoms, crude oil solutions and emulsions, waxy sediments, and other wax-bearing substances which contain, besides high melting point waxes, asphaltic ingredients and preferably also distillable hydrocarbons which are liquid at ordinary temperatures, as light oils and gasoline.

Despite the fact that as described above the main part of my present invention resides in characteristic features of my new wax precipitation processes, my present invention also includes certain features of the distillation itself and after-treatment of the waxes obtained by such distillation.

Thus, I propose to carry out the distillation of the precipitate by mixing it with superheated steam, thereafter heating this mixture above the boiling point of the lower melting point waxes contained in it and introducing the thus heated mixture into a distillation chamber in which vacuum is maintained, whereby the high melting point waxes are separated from the precipitate-steam mixture and accumulate in said chamber in liquid state, while the lower melting point waxes and light oils escape from said chamber in gaseous state.

In accordance with my present invention, it is of particular importance that the precipitate to be distilled be intimately mixed before actual distillation, i. e. before introduction into the distillation chamber with the preferably superheated steam. This can, in accordance with a preferred embodiment of my present invention, be obtained by passing the precipitate through a heating device, e. g., a heating coil, and mixing it directly before entering the heating coil with preferably superheated steam. Thus, the precipitate and the steam are intimately mixed. Thereafter, the intimately mixed precipitate and steam are heated above the boiling point of the light oils

and the lower melting point waxes. The thus heated mixture enters the distillation chamber in which preferably vacuum is maintained. As all lower melting point waxes and all light oils are heated above their boiling point, they will, immediately upon entrance into the distillation chamber, vaporize and thus escape from the distillation chamber in gaseous state.

In accordance with the different compositions of the crude oils and crude oil tank bottoms used as initial materials and the precipitates obtained from the same, it is possible in some cases to separate the high melting point waxes in one operation, i. e., to obtain on the bottom of the distillation chamber, after the first distillation of the precipitate, high melting point waxes, while in other cases repeated distillation is required. This repeated distillation can be carried out by treating the liquid bottom accumulating in the distillation chamber and rich in high melting point waxes in the same way as the precipitate used as initial material for the distillation step: this still bottom is removed from the distillation chamber, intimately mixed with steam, heated to a temperature at which all light oils and lower melting waxes to be eliminated are in gaseous state, and again introduced into a distillation chamber; thereby the waxy still bottom is further distilled and becomes richer in high melting point waxes. If any low melting point waxes or light oils are still left in the thus treated still bottom and the same does not consist only or mainly of high melting point waxes, the treatment may be repeated as often as required. It should be noted that I may introduce the still bottom, after mixing it with steam and heating this mixture, into the same distillation chamber from which it has been withdrawn, thus circulating the waxy still bottom through the same distillation chamber and heater as often as required for the production of high melting point waxes of the desired purity.

I have found that discoloration of the waxes at the high temperatures which are used for distillation is substantially completely avoided if the precipitate and the re-distilled waxy still bottoms are mixed with the absorbent agents before heating. Therefore, in accordance with a preferred embodiment of my invention, I mix the precipitate, before distillation, with the absorbent agents, thus combining the two steps of distilling and bleaching to a high temperature.

The following examples will clearly disclose the most important characteristic features of my present invention. I wish to stress that these examples are not intended to limit my present invention to the features disclosed in the same. The scope of my present invention is defined exclusively by the appended claims.

Example I

Crude oil tank bottom separated by settling from East Texas crude oil stored in tanks is separated from water and sand in the usual way by heating to about 160° F. and keeping the oil at this temperature over a period of several days. The water oil emulsion of the crude oil tank bottom is broken by this treatment and the water is then removed.

The dehydrated tank bottom has a gravity A. P. I. of 32.5 degrees and consists of 56% of ingredients distillable in a vacuum at 4 mm. pressure and a temperature of 650° F., and of 43% of a black residue. This black residue consists of about 23% of ingredients removable by acid

treatment, about 40% of dark brown high melting point waxes and about 37% of a mixture of heavy oils and lower melting point waxes.

This dehydrated crude oil tank bottom is then diluted by adding thereto about the double amount of gasoline having a gravity A. P. I. of 55 degrees, thus obtaining a crude oil-gasoline solution having a gravity A. P. I. of 45.4 degrees. Addition of this gasoline is combined with heating of the dehydrated tank bottom. This is done by pumping the gasoline directly into a steam heated pipe through which the tank bottom is pumped so that a very thorough mixture, i. e., crude oil-gasoline solution having a temperature of about 175° F., is obtained; in this solution, the tank bottom and also the high melting point waxes contained in the same, are completely dissolved.

This heated solution is then pumped through pipes provided with jackets for cooling water, until its temperature is reduced to about 90° to 95° F. At this temperature, the cooled crude oil-gasoline solution is discharged into a slurry tank and further cooled in the same during several hours until a final temperature of about 80° F. is reached. The slurry formed in the tank flows by gravity to a rotary drum type continuous vacuum filter.

The wax cake on the filter is washed with fresh cold gasoline. The thus washed dry cake discharged from the filter is stripped of gasoline by distillation. The final residue obtained after distillation is a black wax having a melting point A. S. T. M. of 191/192° F., a penetration at 77° and 100 grams of 4 and an oil content of less than 1%.

Example II

The dry filter cake obtained as described in Example I is then heated and pumped in molten state to a treating tank and treated with about 4% of sulphuric acid at 170° to 180° F. The precipitated acid sludge is removed and the solution distilled as described above. The obtained residue is a brown wax having a melting point A. S. T. M. of 189/190° F., a penetration at 77° and 100 grams of 5 to 6 and an oil content of less than 1%.

Example III

The dry filter cake obtained in the way described in Example I is transferred to a distillation chamber, where its temperature is raised first to 550° F. under atmospheric pressure and then in a vacuum to 680° F. until a still bottom having a flash point of above 550° F. is produced.

This still bottom, namely the mixture of high melting point waxes accumulating in liquid state at the bottom of the still, has a melting point of 190/192° F., penetration 5. By this process a black wax is obtained.

Example IV

A dry filter cake obtained as described in Example I is treated in molten state with concentrated sulphuric acid; this treatment is repeated several times, if necessary. The excess of sulphuric acid is neutralized by hydrated lime and the cleaned wax-gasoline solution is transferred to the still through a coil heater. Steam is blown into the pipe carrying the waxy mixture at a point before this substance enters the heater. In this heater the wax-gasoline-steam mixture is heated to above that temperature at which all light oils and lower melting point waxes to be eliminated are in gaseous state.

Thereafter, the thus heated mixture enters the still. In the still, high melting point waxes are accumulating at the bottom in liquid state, while the lower melting point waxes and light oils are driven off from the still in gaseous state.

A pump circulates the waxy still bottom through the heater and back to the still; during this circulation the temperature of the waxy still bottom is raised by blowing steam into the pipe carrying the circulating still bottom.

It is important that steam be added, i. e., blown into the pipe in such a manner that the wax containing substance—which might be either the original precipitate or the circulating waxy still bottom—enters the heater always in mixture with steam, thus avoiding cracking of the high melting point waxes contained in the same.

When a temperature of about 700° F. is reached, a still bottom is produced which after cooling consists of a hard brown wax having a melting point of about 189/190° F.

Example V

A dry filter cake of the type obtained by the process described in Example I is first subjected to a sulphuric acid treatment as described in Example II; thereafter, fuller's earth or about 12% by weight of activated clay is mixed with the acid treated filter cake and the thus obtained mixture is then passed to a distillation chamber, i. e., a still and there distilled as described in Example III.

Example VI

A dry filter cake obtained as described in Example I is treated in molten state with concentrated sulphuric acid; this treatment is repeated several times, if necessary. The excess of sulphuric acid is neutralized by hydrated lime. The thus de-asphaltized wax-gasoline solution is percolated through a clay tower, then treated with fuller's earth or activated clay and distilled in one of the ways described in the previous examples.

It will be understood that each of the elements described above, or two or more together, may also find a useful application in other processes of producing waxes differing from the processes described above.

While I have described the invention as embodied in processes of producing high melting point waxes, I do not intend to be limited to the details shown, since various modifications and changes may be made without departing in any way from the spirit of my invention.

Without further analysis, the foregoing will so fully reveal the gist of my invention that others can, by applying current knowledge, readily adapt it for various applications without omitting features that, from the standpoint of prior art, fairly constitute essential characteristics of the generic or specific aspects of this invention and therefore such adaptations should and are intended to be comprehended within the meaning and range of equivalence of the following claims.

What I claim as new and desire to secure by Letters Patent is:

1. Process of producing high-melting point waxes from crude oil containing wax-bearing substances, asphaltic ingredients and oil insoluble impurities as water and sand, said process comprising separating from said crude oil said oil insoluble impurities, diluting the thus obtained dehydrated crude oil in a solvent, heating the thus obtained solution to a temperature at which

also the high melting point waxes contained in said solution are dissolved in the same, thereafter cooling said solution containing said dissolved wax-bearing substances and said asphaltic ingredients to a temperature at which substantially all said high melting point waxes are precipitated from said solution while substantially all the lower melting point waxes contained in said solution remain dissolved, removing the thus formed precipitate, and separating the high melting point waxes from the same by distillation.

2. Process of producing high melting point waxes from dehydrated crude oil tank bottom containing a very high percentage of wax-bearing substances, including high melting point waxes, and containing also asphaltic ingredients, said process comprising diluting said dehydrated crude oil tank bottom with a liquid petroleum solvent, heating said diluted dehydrated crude oil tank bottom to about 180° F., cooling the thus heated diluted dehydrated crude oil tank bottom while keeping it in motion to a temperature at which substantially only said high melting point waxes are precipitated from said diluted dehydrated crude oil tank bottom while a high percentage of the lower melting point waxes contained in said diluted dehydrated crude oil tank bottom remain in solution, removing the thus formed precipitate containing only a small percentage of lower melting point waxes while being substantially free of heavy oils, and separating the high melting point waxes from the thus formed precipitate by distillation.

3. Process of producing high melting point waxes from crude oil tank bottom containing a very high percentage of wax-bearing substances, including high melting point waxes, and containing among other impurities also water, and asphaltic ingredients, said process comprising dehydrating said crude oil tank bottom, diluting said dehydrated crude oil tank bottom with a liquid petroleum solvent, heating said diluted dehydrated crude oil tank bottom to at least 180° F., cooling the thus heated diluted dehydrated crude oil tank bottom while keeping it in motion to between 80 and 90° F. at which temperature substantially only said high melting point waxes are precipitated from said diluted dehydrated crude oil tank bottom while a high percentage of the lower melting point waxes contained in said diluted dehydrated crude oil tank bottom remain in solution, removing the thus formed precipitate consisting mainly of high melting point waxes and containing only a small percentage of lower melting point waxes while being substantially free of heavy oils, and separating the high melting point waxes by distillation from the thus formed precipitate.

4. Process of producing high melting point waxes from crude oil tank bottom containing a very high percentage of wax-bearing substances, including high melting point waxes, and containing among other impurities also water and asphaltic ingredients, said process comprising removing said water from said crude oil tank bottom, diluting said water-free crude oil tank bottom with a liquid petroleum solvent in such a manner as to obtain a water-free tank bottom solution having a gravity, A. P. I. of at least 35 to 40 degrees, heating said water-free tank bottom solution to about 180° F., cooling the thus heated water-free tank bottom solution while keeping it in motion to a temperature being between 10 and 20° F. higher than the precipitation temperature

at which substantially only said high melting point waxes are precipitated from said water-free tank bottom solution, further cooling said pre-cooled water-free tank bottom solution to said precipitation temperature, separating from it the formed precipitate containing only a small percentage of lower melting point waxes while being substantially free of heavy oils, and subjecting the thus formed precipitate to a distillation process, thereby separating the high melting point waxes from the same.

5. Process of producing high melting point waxes from crude oil tank bottom containing a very high percentage of wax-bearing substances, including high melting point waxes, and containing also water and asphaltic ingredients, said process comprising removing said water from said tank bottom, diluting the thus obtained dehydrated tank bottom with a liquid petroleum solvent, heating said dehydrated tank bottom solution to at least 170° F., cooling the thus heated dehydrated tank bottom solution while keeping it in motion to between 90 and 100° F. at which temperature substantially only said high melting point waxes are precipitated from said dehydrated tank bottom solution while a high percentage of the lower melting point waxes contained in said dehydrated tank bottom solution remains in solution, and separating from the thus treated dehydrated tank bottom solution the formed precipitate containing only a small percentage of lower melting point waxes while being substantially free of heavy oils, and subjecting the thus formed precipitate to a distillation process, thereby separating the high melting point waxes from the same.

6. Process of producing high melting point waxes from substantially water-free paraffin base crude oil containing wax-bearing substances including high melting point paraffin waxes and containing also impurities including asphaltic ingredients comprising heating said crude oil to a temperature at which also said high melting point paraffin waxes contained in said crude oil are dissolved in the same, thereafter cooling said crude oil containing said dissolved wax-bearing substances including said high melting point paraffin waxes in dissolved state and said impurities including said asphaltic ingredients to about 85° F. at which temperature substantially only said high melting point paraffin waxes are precipitated from said crude oil while a high percentage of the lower melting point waxes contained in said crude oil remain in solution, removing the thus formed precipitate being at least substantially free of heavy oils, treating said precipitate while in liquid state with concentrated sulphuric acid, removing the resulting sludge, mixing the thus obtained substantially sludge-free precipitate with superheated steam, heating said mixture to above the boiling point of the lower melting point waxes contained in said precipitate, and introducing the thus heated mixture into a distillation chamber whereby the high melting point waxes contained in said precipitate are separated from the same.

7. Process of producing high melting point waxes from crude oil containing a high percentage of wax-bearing substances including high melting point waxes and containing also among other impurities water and asphaltic ingredients, said process comprising removing said water from said crude oil, diluting the thus obtained dehydrated crude oil in a solvent, heating the thus obtained solution to a temperature at which also the high

melting point waxes contained in said solution are dissolved in the same, thereafter cooling said solution containing said dissolved wax-bearing substances and said asphaltic ingredients to a temperature at which substantially only said high melting point waxes are precipitated from said solution, removing the thus formed precipitate, removing the coprecipitated asphaltic ingredients from said precipitate, mixing the thus de-asphaltized precipitate with a bleaching absorbent agent and steam, heating said mixture to above the boiling point of the lower melting point waxes contained in said precipitate, introducing said heated mixture into a distillation chamber in which low pressure is maintained, whereby the high melting point waxes contained in said precipitate are separated from the same in bleached state and are accumulating in said chamber in liquid state, and the lower melting point waxes are escaping from said chamber in gaseous state.

8. Process of producing high melting point waxes from crude oil tank bottom containing a very high percentage of wax-bearing substances, including high melting point waxes, and containing also among other impurities water and asphaltic ingredients, said process comprising removing said water from said crude oil tank bottom, diluting thereafter said substantially water-free crude oil tank bottom with a liquid petroleum solvent, heating said diluted crude oil tank bottom to above 180° F., cooling the thus heated diluted crude oil tank bottom while keeping it in motion to a temperature at which substantially only said high melting point waxes are precipitated from said diluted crude oil tank bottom while a high percentage of the lower melting point waxes contained in said diluted crude oil tank bottom remain in solution, removing the thus formed precipitate being substantially free of heavy oils, treating said precipitate while in liquid state with concentrated sulphuric acid, removing the resulting sludge, mixing the thus obtained substantially sludge-free precipitate with a bleaching absorbent agent and steam, heating said mixture to above the boiling point of the precipitated lower melting point waxes contained in said precipitate, introducing said heated mixture into a distillation chamber in which low pressure is maintained, whereby the high melting point waxes contained in said precipitate are separated from the same and are accumulating in said chamber in liquid state, and the lower melting point waxes are escaping from said chamber in gaseous state.

9. Process of producing high melting point waxes from crude oil tank bottom containing a very high percentage of wax-bearing substances, including high melting point waxes, and containing also among other impurities water and asphaltic ingredients, said process comprising removing said water from said crude oil tank bottom, diluting thereafter said at least substantially water-free crude oil tank bottom with a liquid petroleum solvent, heating said diluted crude oil tank bottom to above 180° F., cooling the thus heated diluted crude oil tank bottom while keeping it in motion to between 80 and 90° F. at which temperature substantially only said high melting point waxes are precipitated from said diluted crude oil tank bottom while a high percentage of the lower melting point waxes contained in said diluted crude oil tank bottom remain in solution, removing the thus formed precipitate containing only a small percentage of lower melting

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point waxes and being substantially free of heavy oils, treating said precipitate while in liquid state with concentrated sulphuric acid, removing the resulting sludge, mixing the thus obtained substantially sludge-free precipitate with a bleaching absorbent agent and steam, heating said mixture to above the boiling point of the precipitated lower melting point waxes in said precipitate, introducing said heated mixture into a distillation chamber in which very low pressure is maintained, thus forming in said distillation chamber a liquid bottom rich in high melting point waxes while substantially all the lower melting point waxes are escaping from said distillation chamber in gaseous state, removing thereafter said wax-bearing liquid bottom from said distillation chamber, intimately mixing it with steam, heating said mixture above the boiling point of the lower melting point waxes left in said liquid bottom, and again introducing said mixture into a distillation chamber in which very low pressure is maintained, whereby said wax-bearing bottom is further distilled and its contents of high melting point waxes further increased.

10. Process of producing high melting point waxes from crude oil tank bottom containing a very high percentage of wax-bearing substances, including high melting point waxes, and containing also among other impurities water and asphaltic ingredients, said process comprising removing said water from said crude oil tank bottom, diluting the thus obtained substantially water-free crude oil tank bottom with a liquid petroleum solvent, heating said diluted crude oil tank bottom to at least 170° F., cooling the thus heated diluted crude oil tank bottom while keeping it in motion to a temperature being between 10 and 20° F. higher than the precipitation temperature at which substantially only said high melting point waxes are precipitated from said diluted crude oil tank bottom while a high percentage of the lower melting point waxes contained in said diluted crude oil tank bottom remain in solution, further cooling said pre-cooled diluted crude oil tank bottom to said precipitation temperature while simultaneously separating from it the formed precipitate containing only a small percentage of lower melting point waxes while being substantially free of heavy oils, treating said precipitate while in liquid state with concentrated sulphuric acid, removing the resulting sludge, mixing the thus obtained substantially sludge-free precipitate with a bleaching absorbent agent and steam, heating said mixture above the boiling point of the lower melting point waxes contained in said precipitate, introducing said heated mixture into a distillation chamber in which very low pressure is maintained, thus forming in said distillation chamber a liquid bottom rich in high melting point waxes, while substantially all the lower melting point waxes are escaping from said distillation chamber in gaseous state, removing thereafter said wax-bearing liquid bottom from said distillation chamber, mixing said wax-bearing liquid bottom again with steam and heating it above the boiling point of the low melting point waxes left in said liquid bottom, re-introducing this mixture into the same distillation chamber from which said wax-bearing liquid bottom has been removed and repeating this removing of the liquid bottom, mixing of said removed liquid bottom with steam, heating of the same and re-introducing it into the same distillation chamber until substantially all low melting point waxes are removed in gaseous state

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and the liquid bottom obtained consists substantially of high melting point waxes only.

11. Process of producing high melting point waxes from crude oil tank bottom containing a very high percentage of wax-bearing substances, including high melting point waxes, and containing among other impurities water and asphaltic ingredients, said process comprising separating said water from said crude oil tank bottom, diluting thereafter the thus dehydrated crude oil tank bottom with a liquid petroleum solvent, heating said diluted dehydrated crude oil tank bottom to above 180° F., cooling the thus heated diluted dehydrated crude oil tank bottom while keeping it in motion to between 80 and 90° F. at which temperature substantially only said high melting point waxes are precipitated from said diluted dehydrated crude oil tank bottom while a high percentage of the lower melting point waxes contained in said diluted crude oil tank bottom remain in solution, separating from the thus treated diluted dehydrated crude oil tank bottom during cooling of the same the formed precipitate consisting mainly of high melting point waxes and containing only a small percentage of lower melting point waxes while being substantially free of heavy oils, mixing said removed precipitate with steam, heating said mixture under pressure and introducing the thus heated mixture into a distillation chamber where the high melting point waxes contained in said precipitate are separated from the same.

12. In the process of producing high melting point waxes from crude oil having a gravity A.P.I. of less than 35° and containing not only a very high percentage of wax bearing substances but also, among other impurities, water and asphaltic ingredients, comprising the steps of separating said water from said crude oil, diluting the thus obtained substantially water-free crude oil in a solvent until a crude oil solution having a gravity A. P. I. of at least 35° is obtained, heating the thus obtained crude oil solution to a temperature of above 180° F., cooling it thereafter to between 70° and 80° F., and separating from the thus cooled crude oil solution the formed precipitate, thus obtaining a wax composition consisting of a high percentage of high melting point waxes and a low percentage of low melting point waxes while being at least substantially free from heavy oils.

13. In the process of producing high melting point waxes from crude oil having a gravity A. P. I. of less than 35° and containing not only a very high percentage of wax bearing substances but also, among other impurities, water and asphaltic ingredients, comprising the steps of separating said water from said crude oil, diluting the thus obtained substantially water-free crude oil in a solvent until a crude oil solution having a gravity A. P. I. of at least 35° is obtained, heating the thus obtained crude oil solution to a temperature at which also the high melting point waxes contained in said crude oil solution are dissolved, cooling it thereafter to a temperature at which substantially only said high melting point waxes are precipitated, and separating from the thus cooled crude oil solution the formed precipitate, thus obtaining a wax composition consisting of a high percentage of high melting point waxes and a low percentage of low melting point waxes while being at least substantially free from heavy oils.

14. In the process of producing high melting point waxes from crude oil having a gravity A. P. I. of less than 30° and containing not only

a very high percentage of wax bearing substances but also, among other impurities, water and asphaltic ingredients, comprising the steps of separating said water from said crude oil, diluting the thus obtained substantially water-free crude oil in a solvent until a crude oil solution having a gravity A. P. I. of at least 30° is obtained, heating the thus obtained crude oil solution to a temperature at which also the high melting point waxes contained in said crude oil solution are dissolved, cooling it thereafter to a temperature at which substantially only said high melting point waxes are precipitated, separating from the thus cooled crude oil solution the formed precipitate, and treating the same with sulphuric acid and a bleaching agent, thus obtaining a bleached light colored wax composition consisting of a high percentage of high melting point waxes and a low percentage of low melting point waxes while being at least substantially free from asphaltic ingredients and heavy oils.

15. In the process of producing high melting point waxes from crude oil having a gravity A. P. I. of less than 30° and containing not only a very high percentage of wax bearing substances but also, among other impurities, water and asphaltic ingredients, comprising the steps of separating said water from said crude oil, diluting the thus obtained substantially water-free crude oil in a solvent until a crude oil solution having a gravity A. P. I. of at least 30° is obtained, heating the thus obtained crude oil solution to a temperature at which also the high melting point waxes contained in said crude oil solution are dissolved, cooling it thereafter to a temperature at which substantially only said high melting point waxes are precipitated, separating from the thus cooled crude oil solution the formed precipitate, washing this precipitate with gasoline by mixing it with the same, adding to the thus obtained mixture sulphuric acid, removing the sludge formed by this acid addition, and finally bleaching and simultaneously distilling the thus treated mixture, thus obtaining bleached high melting point waxes being free of low melting point waxes, asphaltic ingredients and heavy oils.

16. In the process of producing high melting

point waxes from crude oil having a gravity A. P. I. of less than 30° and containing not only a very high percentage of wax bearing substances but also, among other impurities, water and asphaltic ingredients, comprising the steps of separating said water from said crude oil, diluting the thus obtained substantially water-free crude oil in a solvent until a crude oil solution having a gravity A. P. I. of at least 30° is obtained, heating the thus obtained crude oil solution to a temperature at which also the high melting point waxes contained in said crude oil solution are dissolved, cooling it thereafter to a temperature at which substantially only said high melting point waxes are precipitated, separating from the thus cooled crude oil solution the formed precipitate, and treating the same with sulphuric acid and a bleaching agent, thus obtaining a bleached light colored wax composition consisting of a high percentage of high melting point waxes and a low percentage of low melting point waxes while being at least substantially free from asphaltic ingredients and heavy oils.

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