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PROCESS FOR DEWAXING OIL

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7 Claims. (Cl. 204—24)

This invention relates to the electrical separation of wax from wax-bearing oil and particularly to aids and accelerators to dewaxing wax-bearing oil by electrodeposition or electroplating, and is a division of my copending application Serial No. 745,167, filed September 22, 1934.

In the processes for dewaxing oil electrically the wax-bearing oil is mixed with a suitable diluent, chilled to a temperature at which wax is precipitated out of solution as a finely divided wax suspension and the wax suspension removed from the mixture by electrodeposition upon electrically charged electrodes. In most cases the wax precipitates formed in unrefined oils are comprised of negatively charged wax crystals or particles and are deposited or plated in a solid cake or layer upon the positive electrode under the influence of an electric field.

Objects of this invention are to increase the rate and efficiency of dewaxing wax-bearing oil by electrodeposition methods.

It has been found that the rate of electrodeposition can be increased by the addition to the oil or oil diluent mixture prior to chilling and electrical treatment, certain extraneous substances which act as promoters or accelerators. It is believed that these substances, upon chilling, may be co-precipitated with the wax to be removed from the oil, and that the effect of the co-precipitated substance upon the wax is favorable to electrodeposition.

For example, a number of dewaxing tests were run on Santa Fe Springs raw lubricating oil distillate with added extraneous aids to electrodeposition as follows. The wax-bearing lubricating oil distillate was first diluted with 50 volume percent of light hydrocarbon distillate having a boiling point range of approximately 326° to 395° F., then a small quantity of an accelerator was added to the oil-diluent solution and the mixture chilled to a temperature at which wax precipitated from solution in the form of a fine suspension. This mixture was subsequently subjected to a unidirectional electric field between oppositely charged, flat plate, immersed electrodes having areas of 8 square inches and spaced ¾ inch apart from face to face. Under the influence of the unidirectional electric field the precipitated wax was removed from the wax-bearing mixture as a deposit or plated layer upon the positive electrode. Blank tests were simultaneously made in the same manner and under identical conditions but without the accelerators being added, in order to determine the relative rates of electrodeposition with and without the said accelerator. Table I indicates the conditions of the hereinbefore described tests.

It was discovered that the accelerators when added to the waxy oil solution at temperatures at or below the cloud point of the solution, were ineffective to increase the rate of wax deposition while those same accelerators when added at a temperature above the cloud point were effective in increasing the rate of electrodeposition. The electrodeposition accelerators listed hereinafter in Table I were therefore added to the waxy oil-diluent solutions at temperatures of 120° F. or higher, which were temperatures well above the cloud point of the oil solutions.

Table I

Test No.	Duration of electrodeposition-minutes	Temp. °F.	Voltage between electrodes	Electrodeposition accelerator	Deposit	
					Grams	Percent increase
1	7	52	34,500	None.	70	
				0.5% I. G. S. wax	210	300
				0.5% I. G. E. wax	186	266
2	5	50	30,600	None.	78	
				0.5% beeswax (purified).	200	256
3	7	52	34,500	None.	70	
				0.5% Montan wax (crude).	165	236
4	7	50	31,500	None.	89	
				0.5% Carnauba wax	202	
5	5	51	34,200	None.	88	
				0.5% I. G. O. P. wax	180	205
6	10	51	34,350	None.	93	
				0.5% stearine pitch	171	184
7	5	49	30,600	None.	78	
				0.5% I. G. B. wax	139	179
8	10	50	34,200	None.	113	
				0.4% calcium stearate	174	154
9	10	50	33,900	None.	114	
				0.5% A-grade asphalt	181	156
10	10	48	33,900	None.	121	
				0.2% formic acid	177	146

Additional substances not included in Table I which were found to act as accelerators to electrodeposition of wax by the same test methods are

as follows: acetic acid, alpha bromonaphthalene, p-dichlordiphenyl, palmitic acid, naphthalene, pyrogallol, oleic acid, cresol, trioxymethylene p-aminophenol, pyridine, albino asphalt, nitrobenzene, sodium phenoxide, benzoic acid, aniline and calcium naphthenate.

It is not known certainly what the chemical compositions of the waxes are, but they are believed to contain montanic melissic, carnaubic, cerotic and other fatty acids of the general formula $C_nH_{2n}O_2$, high molecular weight alcohols and esters of these acids and alcohols.

The I. G. O. P., I. G. B., I. G. E. and I. G. S. waxes are standard products of the I. G. Farbenindustrie Aktiengesellschaft. Albino asphalt comprises an acid treated naphtha extract of a 300° F. melting point cracked residuum. It has a melting point of approximately 120° F.

In Table II is found a list of the physical characteristics of the accelerators appearing in Table I, and the class of chemical compositions to which they are each believed to be related.

Table II

Electrodeposition accelerator	(a) Acid No.	(b) Sap. No.	(c) M. P. °F. (Ubbel.)	Chemical composition
I. G. O. P. wax	13.8	113	219	Esters of montanic acid.
I. G. B. wax	57.8	148	177.5	Esters of montanic acid.
I. G. E. wax	14.3	142	166	Esters of montanic acid.
I. G. S. wax	143.0	170	181	Esters of montanic acid.
Montan wax (refined)	64.0	67	180	Contains montanic acid.
Montan wax (crude)	26.0	92	183	Contains montanic acid.
Carnauba wax	4.0	71	184	Esters of carnaubic acid.
Beeswax	20.0	91	147	Contains melissic and cerotic acid.
A-grade asphalt (d)	------	------	209–260	Petroleum pitch or residue.
Calcium stearate (e)	------	------	----------	Insoluble metallic soap.
Formic acid (f)	------	------	47.5	Organic acid—40% aq. sol.
Stearine pitch	------	------	----------	Fatty acid.

(a) Acid number (neutralization number) by the A. S. T. M. test D-188-27T.
(b) Sap. No.—Saponification number by the A. S. T. M. test D-94-28.
(c) M. P.—Melting point, Ubbelohde.
(d) 23-30% fixed carbon 75-95% sol. in CCl_3 ductility 0. sp. gr. 1.09.
(e) Insoluble in water. Sol. in alcohol. Forms colloidal suspension in oil.
(f) B. P. 100.8° C. sp. gr. 1.218.

Substances which have been found to produce the most pronounced promotion or acceleration of the rate of the electrodeposition of waxes from suspension in wax-bearing oils are therefore believed to be of the following classes:

1. Waxes which contain high molecular weight alcohols, montanic acid, carnaubic acid, melissic acid and esters thereof.
2. Petroleum asphalts, residues or pitches or extracts thereof.
3. Fatty acid bottoms or pitch.
4. Metallic soaps such as calcium stearate.
5. Organic acids.
6. Aromatic compounds such as naphthalene, a-bromonaphthalene and p-dichlordiphenyl.
7. Aromatic nitrogen compounds such as nitrobenzene, pyridine and aniline.
8. Phenolic compounds such as cresol, sodium phenoxide and p-aminophenol.
9. Petroleum residues, pitches or extracts thereof such as A-grade asphalt or albino asphalt.

This invention resides in a process for increasing the rate of electrodeposition of precipitated wax from wax-bearing oil wherein extraneous electrodeposition aids are added to the wax-bearing oil prior to chilling and the subsequently precipitated wax resulting from chilling the mixture or solution removed from the oil by electrodeposition under the influence of an electric current or electric field.

This invention also resides in the hereinbefore described electrodeposition dewaxing aids and the employment thereof as aids to the process of electrodeposition of wax from wax-bearing oil.

The accelerators or promoters to electrodeposition have been found effective when used in conjunction with various oil solvents and diluents such as liquid propane, gasoline, benzene, kerosene, carbon tetrachloride, and lubricating oil fractions.

An example of the practical application of the invention is as follows:

The wax-bearing oil to be dewaxed is mixed under pressure with a suitable diluent such as liquid propane in the approximate proportion of one part oil to two parts liquid propane by volume at a temperature well above the cloud point of the solution which may be approximately 100° F. An electrodeposition accelerator such as I. G. S. wax is added to the warm oil-propane solution in a small quantity ranging between 0.2% to 0.5% of the volume of the oil. The solution containing the accelerator is self-chilled by reduction of pressure and evaporation of propane to a temperature of −40° F. at which temperature wax precipitates in the form of a fine suspension. The chilled mixture containing the precipitate is then passed between metallic plate electrodes which are spaced approximately ¾ inch apart, face to face, and which are charged to a unidirectional electropotential difference of 34,500 volts. The precipitated wax, which is in most cases charged negatively, moves under the influence of the electric field between the electrodes and is deposited in a solid layer or coating on the surface of the positively charged electrode.

The positive electrode plates, after receiving a coating of wax may be removed from the oil solution and the wax subsequently removed therefrom by suitable mechanical means.

The quantity of wax deposited upon the electrode in a given period of time from the oil solution containing an accelerator is found to be increased to as much as 300% over that which is deposited upon the electrode under similar conditions but without the presence of the added accelerator.

The oil-propane solution from which the wax has been electrically deposited is subsequently freed from the dissolved propane diluent by distillation to produce a dewaxed depropanized oil.

It is to be understood that the foregoing is

merely illustrative of one method of operation and that the invention is not limited thereby but may include other methods to accomplish the same within the scope of the invention.

I claim:

1. A process for dewaxing oil comprising adding to the wax-bearing oil a compound having a benzene ring nucleus adapted to accelerate the rate of electrodeposition of wax, chilling the mixture to precipitate wax and removing the wax from the mixture by electrodeposition.

2. A process for dewaxing oil comprising adding to the wax-bearing oil an electrodeposition accelerator belonging to the group of aromatic compounds containing a plurality of benzene nuclei consisting of naphthalene, alpha bromonaphthalene and p-dichlordiphenyl, chilling the oil to precipitate wax and removing the wax from the mixture by electrodeposition.

3. A process for dewaxing oil comprising adding p-dichlordiphenyl to the wax-bearing oil, chilling the oil to precipitate the wax and removing the wax from the mixture by electrodeposition.

4. A process for dewaxing oil comprising adding to the wax-bearing oil an electrodeposition accelerator belonging to the group of phenolic compounds consisting of cresol, phenol, sodium phenoxide, and p-aminophenol, chilling the oil to precipitate wax and removing the wax from the mixture by electrodeposition.

5. A process for dewaxing oil comprising adding cresol to the wax-bearing oil, chilling the oil to precipitate the wax and removing the wax from the mixture by electrodeposition.

6. A process for dewaxing oil comprising adding to the wax-bearing oil an electrodeposition accelerator belonging to the group of aromatic nitrogen compounds consisting of nitrobenzene, and aniline, chilling the oil to precipitate wax and removing the wax from the mixture by electrodeposition.

7. A process for dewaxing oil comprising adding nitrobenzene to the wax-bearing oil, chilling the oil to precipitate the wax and removing the wax from the mixture by electrodeposition.

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