

March 30, 1943.

R. F. PFENNIG ET AL

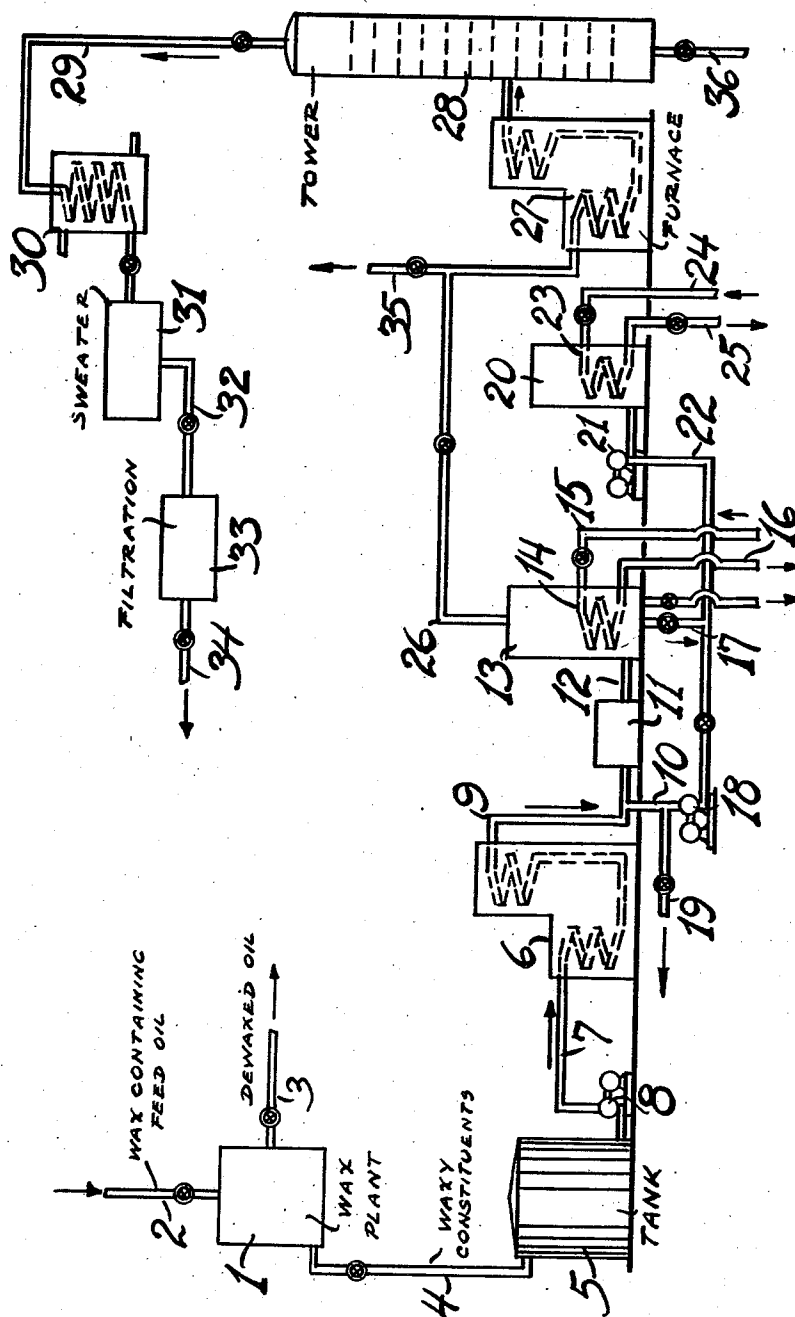
2,315,077

METHOD OF TREATING SLACK WAX

Filed Aug. 24, 1940

2 Sheets-Sheet 1

FIG.-I



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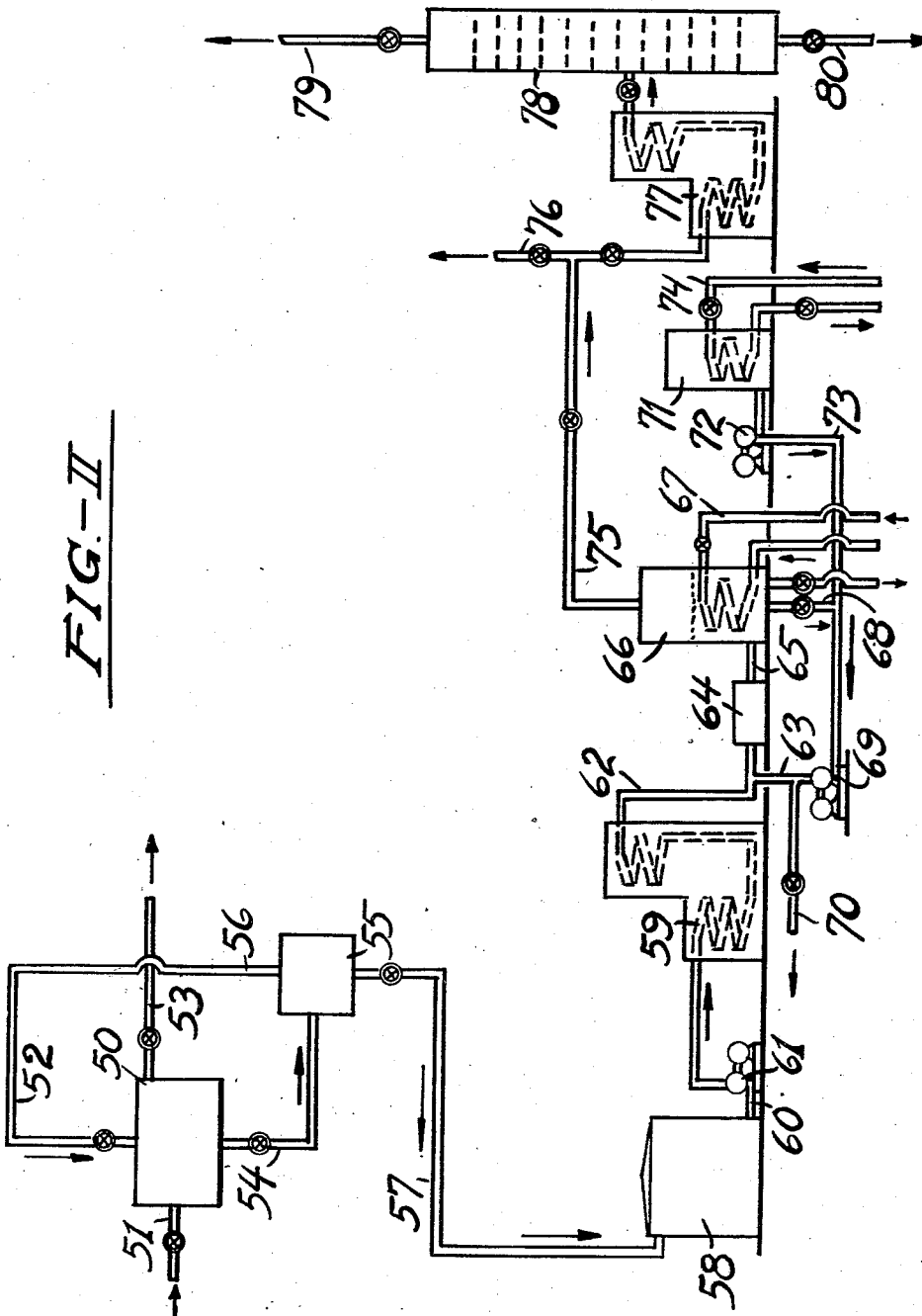
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2 Sheets-Sheet 2

FIG.-II



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UNITED STATES PATENT OFFICE

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METHOD OF TREATING SLACK WAX

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Application August 24, 1940, Serial No. 354,066

10 Claims. (Cl. 196—21)

This invention relates to a refining operation and is particularly concerned with an improved process for the refining of petroleum hydrocarbons. In accordance with the present process petroleum hydrocarbons or fractions derived therefrom are treated with an anhydrous molten alkali under conditions to produce high yields of products of improved quality. The present application contains subject matter in common with the subject matter of application 331,698, filed April 26, 1940, for Pfennig and Davis, and is a continuation in part of said application.

It is well known in the art to refine and process mineral oils and petroleum hydrocarbons by various procedures in order to produce satisfactory refined products. In these operations it is usually essential that the refined product be neutral or non-acid, that it be relatively stable in that deterioration of the product will not occur over a relatively long period of time, and that its color, as well as its color stability, be satisfactory. Thus various processes are directed specifically to the production of neutral, stable, and color satisfactory products in the refining of mineral oils such as in the processing of low boiling petroleum oils, motor fuels, kerosenes, cracked naphtha fractions, heating oil fractions, lubricating oils, wax fractions, as well as solvent extracts. For example, in the processing of lubricating oil stocks derived from naphthenic crudes, it is known that certain constituents such as naphthenic acids and the like must be removed for the production of neutral distillates. A conventional procedure for accomplishing this result is to inject aqueous solutions of sodium hydroxide or equivalent solutions directly into the still, preheater, or fractionating tower and to allow the distillation to proceed in the presence of this alkali. Various modifications of this process are also employed, one of which is to inject dry lime into the preheater or still in order to neutralize acidic compounds, particularly naturally occurring acidic oxygen-containing compounds, which results in an improvement in the color and quality of the refined oil. It is also known to pass mixtures of petroleum vapors and steam through strong aqueous alkali solutions at elevated temperatures. Another practice is to contact petroleum vapors with fused caustic in a distillation tower by circulating the liquid caustic over several plates near the bottom of the tower. By operating in this manner, the vapors from the still are passed countercurrently through the liquid caustic, thus neutralizing acidic materials and effecting a color stability improvement in the distillate. Hydrocarbon oils have also been extracted with aqueous solutions of sodium hydroxide at relatively high temperatures, for example, in the range from about 300° F. to 400° F. and at elevated pressures such as

300 pounds to 400 pounds per square inch. However, while accomplishing satisfactory results in general, these processes possess certain disadvantages which increase operating difficulties and render the operations relatively expensive. A distillation operation conducted in the presence of a solution of sodium hydroxide tends to cause deposition of the caustic on the walls of the equipment and heating means, which materially decreases heat transfer rates and increases operating costs. Furthermore, it is necessary to frequently clean the equipment, thus substantially decreasing its yearly throughput. Other disadvantages encountered in current processes are the extensive pressure drops which are incurred, particularly in vacuum distillation operations, as well as in the formation of emulsions which are difficult to break.

In order to produce light naphtha products having a satisfactory stability, color, and other desirable characteristics, it is the conventional practice to process these materials by various modifications of acid treating, doctor sweetening and rerunning operations. Heating oil distillates, as well as related oils boiling in this boiling range are similarly refined utilizing modifications of acid treatment, water washing, alkali washing, and distillation operations depending to some extent upon the character of the feed oil. Commercial and highly refined waxes having a satisfactory color and stability are produced by various operations comprising acid treating, caustic washing, rerunning, percolation, through bone char or other similar adsorbent, and related stages. Solvent extracts produced by the utilization of solvents of the class which have a preferential selectivity for the relatively more aromatic type constituents as compared to the relatively more paraffinic type constituents, as for example phenol, furfural, sulfur dioxide, cresol, nitro benzene, aniline, beta beta dichlor-diethyl ether, and the like, are also further refined using conventional acid treating and various distillation and rerunning stages.

We have now discovered an improved process for the production of high yields of improved quality products which have a better color and color stability than has heretofore been secured by known processes. Furthermore, in the case of relatively low molecular weight distillates such as cracked naphthas, kerosenes, and heating oils, our process substantially completely removes the mercaptan sulphur compounds. Our process comprises contacting mineral oils, particularly petroleum hydrocarbons, in the liquid state with an anhydrous fused alkali at a temperature above the fusing point of the alkali. A preferred modification of our invention comprises contacting the feed material in an initial stage with an anhydrous fused alkali at a tem-

perature above the fusion point of the alkali and then in a secondary stage distilling the feed oil under controlled conditions. Our process produces finished products which have a low neutralization value and which have exceptionally good cast and high color stability. In accordance with our process the feed material is contacted in the liquid phase at approximately atmospheric pressure with an anhydrous caustic molten alkali which may or may not be mixed with another anhydrous material capable of lowering its fusion point, but which does not change its chemical activity.

Our invention may be readily understood by reference to the attached drawings illustrating modifications of the same. Figure 1 illustrates the adaptation of our invention in the manufacture of high quality refined waxes, while Figure 2 illustrates the adaptation for the production of high quality products from feed oils employing a solvent extraction stage. Referring specifically to Figure 1, a feed oil, which for the purpose of illustration is assumed to be a wax-containing lubricating oil fraction, is introduced into dewaxing plant 1 by means of feed line 2. Wax plant 1 may comprise any suitable number of units and be operated in any conventional manner for the removal and segregation of a waxy constituent fraction and for the production of a satisfactory dewaxed oil. The dewaxed oil is withdrawn from plant 1 by means of line 3, while the waxy constituents are removed by means of line 4 and introduced into storage tank 5. The waxy stock is withdrawn from storage tank 5 and introduced into furnace 6 for heating by means of line 7 and pump 8. By means of line 9, the heated oil is withdrawn from furnace 6 and mixed with a fused alkali, introduced by means of line 10, and the resultant mixture passed into mixer 11. For purposes of description the fused alkali is taken to be an anhydrous mixture of sodium hydroxide and sodium carbonate. The mixture comprising waxy constituents, fused sodium hydroxide, and sodium carbonate is withdrawn from mixer 11 by means of line 12 and introduced into settling zone 13. Settling zone 13 is so designed that the waxy constituents are in contact with a fused alkali for an optimum time period at the desired temperatures. The desired temperature in settling zone 13 is maintained by heating means 14 comprising lines 15 and 16. The fused alkali mixture is withdrawn from settling drum 13 by means of line 17 and recycled to the mixer as described by means of recycle pump 18. Spent fused alkali may be withdrawn from the system by means of line 19, while fresh or make-up alkali may be introduced from make-up alkali drum 20 by means of pump 21 and line 22. The desired temperature is maintained in make-up drum 20 by means of heating coil 23 which comprises lines 24 and 25. The alkali-contacted waxy constituents are withdrawn from settling zone 13 by means of line 26, passed through furnace 27, and introduced into distillation tower 28. Under certain conditions it may be desirable to remove the waxy constituents from the system by means of line 35. These removed constituents may be handled in any manner desirable and further refined by sweating and filtration. However, in general it is preferred to distill the waxy constituents as described. Temperature and pressure conditions of distillation tower 28 are adapted to remove overhead by means of line 29 the desired products, and

to remove by means of line 36 a bottoms product. It is to be understood that distillation tower 28 may comprise any number of towers arranged in any desirable manner and that one or more side streams may be segregated. The overhead waxy constituents removed by means of line 29 are condensed in condenser 30 and passed to sweater 31. A sweated waxy fraction is removed from the unit 31 by means of line 32 and passed through filtration unit 33 in which the waxy constituents contact bone charcoal. A refined high quality wax fraction is withdrawn from unit 33 by means of line 34 and handled in any manner desirable.

Referring specifically to Figure 2, a feed oil, which for the purposes of description is taken to be a petroleum oil, is introduced into solvent treating plant 50 by means of line 51. In this plant the oil is contacted with a solvent of the class which has a preferential selectivity for the relatively more aromatic constituents as compared to the relatively more paraffinic constituents. For purposes of description it is assumed that the solvent comprises phenol, which is introduced by means of line 52. Temperature and pressure conditions are adjusted to secure the formation of a raffinate or relatively high paraffinic phase which is withdrawn from the system by means of line 53, and a highly aromatic solvent extract phase which is withdrawn by means of line 54. It is to be understood that solvent treating plant 50 may comprise any suitable number of stages or may comprise conventional solvent countercurrent treating towers. The operation may be conducted and operated in accordance with conventional methods. The solvent extract removed by means of line 54 is introduced into solvent recovery unit 55 in which the solvent is removed by means of line 56 and preferably recycled while a solvent free extract oil is removed by means of line 57 and passed to storage tank 58. The solvent-free extract is withdrawn from storage tank 58 and introduced into furnace 59 by means of line 60 and pump 61. The oil is heated in furnace 59 and withdrawn by means of line 62, and then mixed with an anhydrous fused alkali which is introduced by means of line 63. The resultant mixture is then introduced into mixer 64. For purposes of description the fused alkali is taken to be an anhydrous mixture of sodium hydroxide and sodium carbonate. The mixture comprising the solvent extract, the fused sodium hydroxide and sodium carbonate is withdrawn from mixer 64 by means of line 65 and introduced into settling zone 66. Settling zone 66 is designed so that the solvent extract is in contact with the fused alkali for the desired time period at the optimum temperature. The desired temperature in settling zone 66 is maintained by heating means 67. The fused alkali mixture is withdrawn from settling zone 66 by means of line 68 and, by means of pump 69, it is recycled to line 62 where it is mixed with the oil withdrawn from furnace 59. Spent fused alkali may be withdrawn from the system by means of line 70, while fresh or make-up alkali may be introduced from make-up alkali drum 71 by means of pump 72 and line 73. The desired temperature is maintained in make-up drum 71 by means of heating coil arrangement 74. The alkali-contacted solvent extract is withdrawn from settling zone 66 by means of line 75, and may be withdrawn from the system by means of line 76 and further refined in any desirable manner. However, it is preferred to pass the alkali-contacted solvent extract oil

through furnace 77 and then to distillation tower 78. Temperature and pressure conditions of distillation tower 78 are adapted to remove overhead by means of line 79 the desired products and to remove by means of line 80 a bottoms product. Distillation tower 78 may comprise any number and arrangement of fractionating or distillation means.

The process of the present invention may be widely varied. The invention essentially comprises contacting feed materials in the liquid state with an anhydrous fused alkali at a temperature above the fusion temperature of the alkali. In accordance with the preferred modification of the invention, the alkali-contacted feed material is then distilled in a secondary stage. The process may be adapted to the treatment of any feed oil. The invention, however, is particularly suited for the processing of petroleum hydrocarbons such as light naphthas, kerosene fractions, cracked naphthas, and heating oil fractions. The invention is especially suitable for the production of high quality refined waxes and for the production of high quality refined products derived from solvent extracts, particularly phenol extracts.

Although any anhydrous fused alkali may under certain conditions be employed, we prefer to utilize anhydrous alkali metal hydroxides, such as sodium hydroxide and potassium hydroxide. An especially desirable treating agent comprises a mixture of sodium hydroxide and potassium hydroxide, particularly when the concentration of the sodium hydroxide is in the range from about 25% to 85%. In general, the most satisfactory operation is secured when the sodium hydroxide, potassium hydroxide mixture comprises from 45% to 55% of sodium hydroxide. Other desirable alkali mixtures comprise sodium hydroxide admixed with various anhydrous inorganic salts which are capable of lowering the fusion point of the sodium hydroxide. Particularly desirable salts are sodium carbonate, sodium sulfide, sodium bromide, sodium iodide and sodium sulfate. An especially desirable alkali mixture utilizing sodium hydroxide comprises a mixture of sodium hydroxide and anhydrous sodium carbonate. When employing this mixture, it is desirable to use at least 75% sodium hydroxide, preferably from 85% to 95% sodium hydroxide. A particularly desirable mixture comprises 92% sodium hydroxide and 8% sodium carbonate. The alkali should be substantially anhydrous and in no instance is it desirable that the moisture content of the treating alkali or alkali mixture exceed about 2.0%. In general, the moisture content of the alkali treating agent should be below about 0.5% and should preferably be anhydrous.

The time of contact between the fused anhydrous alkali mixture and the oil will depend primarily upon the intimacy of contact attained between the oil and alkali mixture and to some extent upon the temperature at which the oil is treated. However, when treating a feed oil derived from a naphthenic crude and which boils in the range above about 500° F., it is preferred to contact the same for a period of from 5 to 25 minutes, preferably from 10 to 20 minutes, at a suitable temperature ranging between 370° F. and 700° F., preferably from about 500° F. to 550° F. When treating a kerosene fraction the time of contact should preferably be from 5 to 15 minutes at a temperature in the range from 370° F. to 450° F., whereas waxy constituents should be contacted for a time period from 10 to 20 minutes at a temperature in the range from

about 400° F. to 700° F., preferably at a temperature in the range from about 500° F. to 550° F. Solvent extracts, such as phenol extracts, are treated under conditions which depend to a large extent upon the character of the solvent extract. Relatively high boiling extracts are contacted for from 5 to 20 minutes at a temperature in the range from about 400° F. to 600° F. When very intimate mixing of the oil and alkali are obtainable, improved results may be obtained when employing times of contact considerably less than 5 minutes. The pressure employed should be at least sufficient to maintain the feed oil in the liquid state and in general the operation is conducted at about atmospheric pressure. However, when oils such as naphthas and kerosenes are being treated, pressures as high as about 10 to 15 atmospheres must be employed due to the high vapor pressure of the oil at the treating temperature.

The quantity of anhydrous fused alkali used per volume of feed material will vary considerably depending upon the feed oil being treated and the particular alkali or alkali mixture employed. Usually the quantity of fused alkali utilized will vary in the range from about 0.1 to 3.0 volumes of alkali per volume of feed material. In general it is preferred to employ from 1.0 to 1.5 volumes of anhydrous fused alkali per volume of feed material. The alkali treated feed material, after separation from the alkali in accordance with the preferred modification of the invention, is then heated and discharged preferably into a vacuum distillation unit in which temperature and pressure conditions are adjusted to remove substantially the entire quantity overhead.

In order to further illustrate our invention the following examples are given which should not be construed as limiting the same in any manner whatsoever.

EXAMPLE 1

A crude residuum derived from a Texas crude was dewaxed utilizing propane as a dewaxing solvent. The slack wax produced had the following inspections:

Melting point, ° F.	118
Expressible oil and moisture ¹ , per cent.	11.67
Gravity, ° A. P. I.	41.5
Initial boiling point, ° F. (10 mm. Hg)	390
Final boiling point, ° F. (10 mm. Hg)	572

¹ As determined by the A. S. T. M. test D308-29r.

One volume of this slack wax was then intimately contacted with about 0.8 volume of a molten caustic alkali reagent consisting of 92% NaOH and 8% Na₂CO₃ by weight (fusion point about 510° F.) for about 10 minutes at a temperature of 550° F. The apparatus consisted of a closed vessel provided with a mechanical stirrer and heated by means of a gas flame. After settling and separating the wax from the reagent the caustic treated wax was charged to a vacuum still and distilled into a cut equivalent (in volume) to the first 80% of the charge to the fusion pot. The distillate had the following properties:

Gravity, ° A. P. I.	41.9
Melting point, ° F.	112
Expressible oil and moisture, percent.	11.64
Color, Tag-Robinson	18 3/4

The distillate was subsequently sweated to produce a wax with the following characteristics:

Melting point, °F.....	122
Color, Saybolt.....	28
Colorhold, ¹ Saybolt (302° F. for 16 hrs. in presence of copper strip).....	17

¹ Colorhold test (16 hours at 302° F.).—60 cc. of molten wax is filtered through filter paper into a test tube. A clean, dry and polished copper strip one-half inch by two inches is immersed completely in the wax and the wax is then heated in a bath at 302° F. for 16 hours. At the end of this period the wax is filtered through paper and its color determined in a Saybolt colorimeter at 160° F.

In another operation, similar slack wax which had not been treated with fused alkali was vacuum distilled into a similar cut having the following properties:

Gravity, °A. P. I.....	41.5
Melting point, °F.....	114
Expressible oil and moisture, percent.....	14.1
Color, Tag-Robinson.....	12½

This distillate was subsequently treated with four pounds of sulphuric acid per barrel, and was then soda neutralized, water washed, dried and sweated. The following tests were obtained on the sweated wax:

Melting point, °F.....	126
Color, Saybolt.....	28
Colorhold, Saybolt (302° F. for 16 hrs. in the presence of copper strip).....	—14

It is to be noted that the stability of the waxes produced in accordance with the conventional methods of acid treatment, soda neutralization, sweating, and percolation through bone char, are definitely inferior with respect to the stability of the waxes produced in accordance with the present process.

EXAMPLE 2

A representative sample of a paraffin distillate derived from a Panhandle crude was dewaxed in a propane solution to yield a slack wax having the following properties:

Gravity, °A. P. I.....	41.9
Vacuum Engler (10 mm. Hg.).....	
IBP, °F.....	383
FBP, °F.....	586

This slack wax was treated with fused caustic as described in Example 1. The treated wax was redistilled into a 97% overhead cut; the distillate possessed the following characteristics:

Gravity, °A. P. I.....	42.3
Melting point, °F.....	117.5
Color, Tag-Robinson.....	19¼

This distillate was then sweated to produce a wax having the following characteristics:

Melting point, °F.....	130
Percent yield, based on slack wax.....	41.0
Color after percolation through bone charcoal, Saybolt.....	28
Colorhold, Saybolt (302° F.) for 16 hrs. in presence of copper strip.....	—14

In another operation the same distillate from the fused alkali treated wax was acid treated, soda neutralized, water washed and dried prior to sweating. The sweated wax had the following properties:

Color after percolation through bone charcoal, Saybolt.....	25
Colorhold, Saybolt (302° F. for 16 hrs. in presence of copper strip).....	10

A similar wax which had not been treated with fused alkali was vacuum distilled into one cut consisting of 97% of the slack wax. The distillate had the following characteristics:

Gravity, °A. P. I.....	42.5
Melting point, °F.....	113
Color, Tag-Robinson.....	15¼

After acid treatment, soda neutralization, water washing and drying, the wax was sweated to produce a crude scale wax having the following properties:

Melting point, °F.....	126
Percent scale, based on slack wax.....	36.2
Color after percolation through bone charcoal, Saybolt.....	28
Colorhold, Saybolt (302° F. for 16 hrs. in presence of copper strip).....	—19

It will be noted from the above data that the wax produced from the Panhandle wax distillate by either of the two caustic fusion methods was distinctly superior with respect to color stability to the wax produced by the acid treatment.

EXAMPLE 3

In an operation (A) a phenol extract was intimately contacted with about 1.5 volumes of molten caustic alkali consisting of 92% NaOH and 8% Na₂CO₃ by weight (fusion point about 510° F.) for about 10 minutes at a temperature of 550° F., the phenol extract having been secured by extracting a Coastal oil which had been redistilled over caustic in a battery of shell stills and which had a Saybolt viscosity of 160 at 100° F. and a neutralization value equivalent to 0.06 to 0.3 milligram of potassium hydroxide per gram of oil. The apparatus consisted of a closed vessel provided with a mechanical stirrer and heated by means of a gas flame. After settling and separating the oil from the reagent, the caustic treated oil was charged to a vacuum still and distilled into finished overhead fractions having the following properties:

Vis. at 100° F., S. S. U.....	67	112	269
Vis. at 210° F., S. S. U.....		37.6	43.4
Color, Tag-Robinson.....	17¼	17	14½
Colorhold ¹ (16 hrs. at 250° F.), Tag-Robinson.....	10½	12¾	9¾
Neutralization value.....	.01	.007	.02

¹ Colorhold test (16 hours at 250° F.).—75 cc. of oil is placed in a sample bottle. The unstoppered bottle is heated in an oven at 250° F. for 16 hours. After the sample is cooled to room temperature its color is determined in a Tag-Robinson colorimeter.

In another operation (B) a volume of extract secured by phenol extracting approximately 900 vis./100° F. Coastal oil and which had a neutralization value equivalent to 0.1 to 0.3 milligram of potassium hydroxide per gram of oil was intimately contacted with about 1.5 volumes of a molten caustic alkali reagent consisting of 51% NaOH and 49% KOH by weight (fusion point about 369° F.) for about 10 minutes at a temperature of 550° F. The oil was subsequently treated as in operation A, the finished fractions having the following properties:

Vis. at 100° F., S. S. U.....	285	836	1681	3280	5500	9480	15900	22240
Color, Tag-Robinson.....	15	14¼	12¾	11¾	11¾	9¾	10¼	9½
Colorhold (16 hrs. at 250° F.), Tag-Robinson.....	10½	10¼	10¾	7¾	9¾	7¾	9¾	3

In a further operation, (C), an extract having a neutralization value equivalent to 1.3 milligrams of potassium hydroxide per gram of oil,

which was obtained by treating gas oil free total Coastal rerun distillate equivalent to 45% of the crude with 200% of 97% phenol at 180° F. with 4% water injection, was treated by (1), conventional rerunning over caustic, and (2) counter-current aqueous caustic extraction at elevated temperatures followed by rerunning.

The results of these observations were as follows:

OPERATION I

Tests on overhead fractions from vacuum distillation of phenol extract rerun over caustic

Gravity, °A. P. I.	17.9	16.1	15.0	13.7	12.2
Vis. at 100° F., S. S. U.	136	323			
Vis. at 210° F., S. S. U.			56.1	76.7	121
Color, Tag-Robinson	13	11	9½	9	8
Colorhold (16 hrs. at 250° F.), Tag-Robinson	2½	3½	4½	2½	1½
Neutralization value	0.02	0.05	0.06	0.06	0.035

OPERATION II

Tests on overhead fractions from vacuum distillation of caustic extracted phenol extract

Gravity °A. P. I.	17.1	15.7	14.3	13.0	11.6
Vis. at 100° F., S. S. U.	201	467			
Vis. at 210° F., S. S. U.			61.8	87.1	128.9
Color, Tag-Robinson	11	10¼	9	9	5
Colorhold (16 hrs. at 250° F.), Tag-Robinson	2	3¼	2¼	1½	2
Neutralization value	0.03	0.05	0.02	0.05	0.02

In addition, tests are shown on the following oils obtained by treating an extract (blend of extract having a neutralization value equivalent to 0.08 milligram KOH per gram oil obtained from phenol treating Coastal pale oils of approximately 100, 250, and 900 viscosity at 100° F.) with 10 lbs. of 98% H₂SO₄ per barrel of oil, soda neutralizing and distilling in an iron pot still:

Vis. at 100° F., S. S. U.	112	388	734	1188
Color, Tag-Robinson	14¼	12¾	11¼	10¾
Colorhold (16 hrs. at 250° F.), Tag-Robinson	1.5	1.5	1.0	¾

From these operations it is apparent that extracts produced in accordance with the methods of aqueous caustic extraction at elevated temperatures and conventional distillation over an excess of caustic, or acid treatment followed by distillation would require additional chemical and physical treatment to render them color stable, whereas extracts produced in accordance with our method are color stable and require no further treatment.

EXAMPLE 4

The charge stock employed in this operation comprised a reduced cracked naphtha having the following properties:

Gravity, °A. P. I.	40.2
IBP, ° F.	289
FBP, ° F.	477
Copper No.	1.0
Sulfur (lamp), per cent.	0.05

One portion of this naphtha was treated with about 0.8 volume of molten caustic alkali reagent consisting of 51% NaOH and 49% KOH by weight (fusion point about 379° F.) for about 10 minutes at a temperature of 410° F. under pressure of about 10 atmospheres. The apparatus consisted of a closed vessel provided with a mechanical stirrer and heated by means of a gas flame. After settling and separating the naphtha from the reagent, the caustic treated naphtha

was charged to a vacuum still and distilled into an overhead cut amounting to 95% of the charge to the fusion pot. The properties of this distillate are tabulated in Table A below.

A second portion of the reduced cracked naphtha was treated with 3 pounds of 98% H₂SO₄ per barrel of naphtha. After separating and removing the sludge, the acid treated naphtha was subjected to a water wash, a caustic wash, then another water wash, and (finally) it was distilled into an overhead cut amounting to 95% of the charge to the acid treater. The properties of this finished distillate are also presented in Table A below.

TABLE A

	Distillate from naphtha treated with acid	Distillate from naphtha treated with fused caustic
Gravity, °A. P. I.	42.5	42.8
Distillation:		
I. B. P., ° F.	289	286
F. B. P., ° F.	400	388
10% off at, ° F.	289	297
50% off at, ° F.	316	312
95% off at, ° F.	376	366
Standard heat color ¹ (16 hrs. at 212° F.), Saybolt	+8	+20
Direct oxidation color, Saybolt	-4	+21
Sunlight stability color, Saybolt:		
2-hour exposure	+6	+17
4-hour exposure	-5	+16+
Octane number, A. S. T. M.	68.2	70.1
Sulfur (lamp), percent	0.017	0.015

¹Standard heat color (16 hours at 212° F.).—100 cc. of oil are placed in a bath at 210° F. During the first half hour of the test the stopper is left off the bottle and during the remaining 15½ hours of the test the bottle is kept stoppered. If any evaporation occurs the loss is made up by the addition of naphtha having a 30 color. The color is determined before and after the test by means of a Saybolt colorimeter.

It will be noted that, with respect to color stability and octane number, the fused-caustic treated product is superior to the acid treated product. Attention is also called to the fact that the fused caustic treatment effected about the same drop in sulfur content of the naphtha as did the acid treatment, the latter being considered a particularly good means of lowering the sulfur content of oils. The fused alkali treatment produced an oil that was sweet to the doctor test. This result is secured when treating naphthas boiling in the motor fuel boiling range.

EXAMPLE 5

A high-sulfur kerosene distillate derived from a West Texas crude was employed as the charge stock in this operation. After being caustic washed for removal of hydrogen sulfide, this distillate had the following properties:

Gravity, °A. P. I.	43.7
Distillation:	
IBP, ° F.	318
FBP, ° F.	547
10% off at, ° F.	367
50% off at, ° F.	423
95% off at, ° F.	531
Color, Saybolt	+15
Copper No.	33
Silver No.	27.0
Sulfur (lamp), per cent.	0.11

A portion of this kerosene distillate was treated with about 0.8 volume of molten caustic alkali reagent consisting of 92% NaOH and 8% Na₂CO₃ by weight (fusion point about 510° F.) for about 10 minutes at a temperature of 550° F. The ap-

paratus consisted of a closed vessel provided with a mechanical stirrer and heated by means of a gas flame. After settling and separating the kerosene from the reagent, the caustic treated kerosene was charged to a vacuum still and distilled into a 95% overhead cut based on the charge to the fusion pot. The properties of this distillate are tabulated in Table B below.

Another portion of the high-sulfur kerosene distillate was treated with 5.0 pounds of 98% H_2SO_4 per barrel of oil. After settling and removing the sludge from the oil, the latter was doctor sweetened and distilled into a 95% overhead cut based on the charge to the acid treater. This distillate was doctor sweetened again; after which, it was water washed and dried. The properties of the oil thus treated are tabulated in Table B which follows:

TABLE B

	Distillate from kerosene treated with acid	Distillate from kerosene treated with fused caustic
Gravity, °A. P. I.-----	44.2	43.9
Distillation:		
I. B. P., °F.-----	330	327
F. B. P., °F.-----	520	517
10% off at, °F.-----	367	362
50% off at, °F.-----	418	413
95% off at, °F.-----	506	504
Color, Saybolt.-----	+27	+27
Direct oxidation color, Saybolt.-----	-9	+26
Copper No. after redistilling oil, prior to final sweetening.-----		0.1
Silver No.-----	10.0	2.0
Sulfur (lamp), percent.-----	.04	.09

The above data show that fused caustic treatment of kerosene distillates produces an oil superior, as judged by color stability and silver number, to similar oils produced by the conventional method of acid treating, followed by doctor sweetening, distilling and then resweetening. It will be noted particularly that the fused caustic treatment followed by distillation substantially sweetened the oil, effecting a reduction in copper number of the oil from 33 to 0.1. The copper number is a measure of the amount of mercaptan sulfur present in the oil.

The process of the present invention is not to be limited by any theory or mode of operation but only by the following claims in which it is desired to claim all novelty insofar as the prior art permits.

We claim:

1. A continuous process for the production of color stable waxes from crude slack wax comprising the steps of subjecting the liquid slack wax to intimate mixing with a liquid anhydrous molten caustic alkali at a temperature above the fusion point of said alkali, separating and removing the liquid treated wax from the alkali, recycling the molten alkali to the wax-alkali mixer, reheating and distilling the liquid slack wax into

a suitable fraction under mild thermal conditions, sweating the resultant distillate to yield a scale wax of suitable melting point, and percolating the sweated wax through bone charcoal for the production of the required color.

2. A continuous process for the production of color stable waxes from slack wax comprising the step of subjecting liquid slack wax to intimate mixing with a liquid anhydrous molten caustic alkali at a temperature above the fusion point of said alkali, separating and removing the liquid treated wax from the alkali, recycling the molten alkali to the wax-alkali mixer, reheating and distilling the wax into suitable fractions under mild thermal conditions, acid treating, soda neutralizing, water washing, drying, and sweating the resulting distillate to a suitable melting point, and percolating the sweated wax through bone charcoal for the production of the required color.

3. A process in accordance with claim 1 in which the anhydrous molten alkali is a mixture comprising about 20% to 100% caustic soda and about 80% to 0% caustic potash.

4. A process in accordance with claim 2 in which the anhydrous molten alkali is a mixture comprising about 20% to 100% caustic soda and about 80% to 0% caustic potash.

5. A process in accordance with claim 1 in which the anhydrous molten alkali is a mixture comprising about 75% to 100% caustic soda and about 25% to 0% sodium carbonate.

6. A process in accordance with claim 2 in which the anhydrous molten alkali is a mixture comprising about 75% to 100% caustic soda and about 25% to 0% sodium carbonate.

7. A method of treating slack wax to obtain a purified wax comprising the steps of intimately contacting molten slack wax with anhydrous molten alkali at a temperature above the fusion point of said alkali but no higher than 550° F., subsequently removing the liquid wax from the alkali and vaporizing said wax to obtain a distillate fraction.

8. A method in accordance with claim 7 in which the molten wax is contacted with a mixture of sodium hydroxide and sodium carbonate for approximately 10 minutes.

9. A method of treating slack wax to obtain a purified wax comprising the steps of intimately contacting liquid slack wax with anhydrous molten alkali at a temperature above the fusion point of said alkali but no greater than 550° F., settling the mixture, separating and removing treated wax from the alkali and subsequently vacuum distilling said wax to obtain a distillate fraction.

10. A method in accordance with claim 9 in which the method is continuous and the alkali removed from the settling step is recycled to the contacting step.

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