

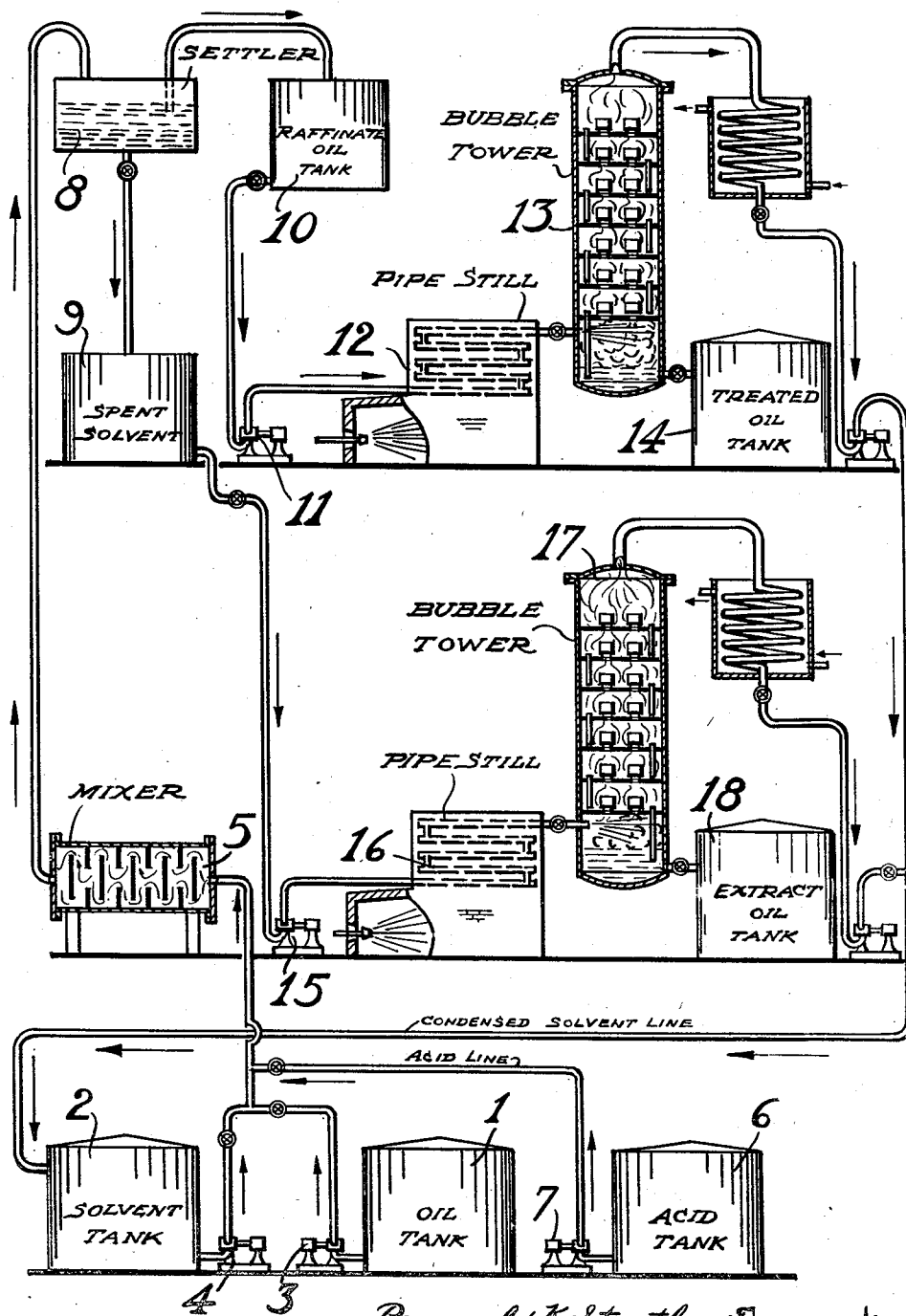
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TREATING RESIDUAL OILS WITH A SELECTIVE SOLVENT

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TREATING RESIDUAL OILS WITH A
SELECTIVE SOLVENT

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This invention relates to a process of treating petroleum oils, such as residual oils or dirty distillates, with a selective solvent for improving the color, viscosity index, Conradson carbon test and other characteristics of the oil. The invention will be fully understood from the following description when read in conjunction with the drawing, the sole figure of which is a diagrammatic side elevation partly in section of an apparatus appropriate for carrying out the invention.

Various processes have been developed in recent years in which an oil is treated with a selective solvent, such as for example, phenol, nitrobenzene, furfural, liquid SO_2 , etc. at a temperature below that at which the solvent becomes completely miscible with the oil. Under such conditions the mixture separates into a solvent layer and a raffinate layer, the former consisting of the largest portion of the selective solvent together with constituents extracted from the oil, and the latter consisting mostly of the oil having in solution a minor portion of the solvent. The solvent is then removed by distillation or by other means from both layers, whereby the raffinate layer yields a lubricating oil of greatly improved quality as regards gravity, color, viscosity index, Conradson carbon, etc.

In treating residual stocks with a selective solvent it has always been found difficult to obtain what could be considered a good color and in many cases the Conradson carbon of the oil is not reduced sufficiently by the selective solvent treatment. The same is true of dirty lubricating oil distillates which have been contaminated during the storage with inferior and/or asphalt containing stocks. We have discovered that the treatment of such stocks with a selective solvent gives much better results in improving color, Conradson carbon, etc. when the treatment of the oil is carried out in the presence of a small quantity of an acid. The amount of acid to be used is of the order of magnitude of 1% of the oil or less and in certain preferred cases it is $\frac{1}{2}\%$ or less. The acid is preferably an inorganic acid, such as hydrochloric acid or sulphuric acid, but organic acids such as picric, acetic, oxalic, citric, benzene sulfonic acids also give good results. The acid is either added to the oil before the treatment with the selective solvent or it may be added to the selective solvent or it may be added to the mixture of oil and selective solvent. The treatment with the selective solvent is, in any case, carried out in the presence of the acid.

When the stock is first treated with the small

amount of the acid, coagulation of the asphaltic material takes place, but there is no separation of sludge from the oil. If the oil is then treated with the selective solvent, separation takes place at once, leaving an oil of comparatively good color in the raffinate layer, the sludge being removed with the extract. In this invention it is sometimes desirable to dilute the oil to be treated with a light naphtha or benzol, toluol, etc. before the treating.

Referring now to the figure, oil and solvent from storage tanks 1 and 2 are passed by means of pumps 3 and 4 to a mixer 5. The proper amount of acid is proportioned to the oil and solvent from the acid supply 6 by means of pump 7. The oil, solvent, and acid mixture is allowed to separate into layers in settler 8. From the latter the spent solvent flows into tank 9 and the raffinate oil into tank 10. Pump 11 feeds the raffinate oil to the pipe still 12, 13, where solvent and acid are removed from the oil by fractionation. The treated oil passes to tank 14 and the condensed solvent from tower 13 is returned to tank 2. Similarly spent solvent from tank 9 is pumped by pump 15 to the pipe still 16, 17, the recovered solvent passing to solvent tank 2, and the extract oil to tank 18.

In the above described apparatus the oil may be given a single or successive treatment with solvent and acid.

The invention is most advantageously carried out in a batch method of operation, acid being added only in or before the first batch treatment, but acid may also be added in successive batch treatments or a continuous counter-current treatment may be employed.

When treating with phenol, hydrochloric acid is preferably used, since sulfuric acid causes some loss of phenol due to sulfonation.

The following example will illustrate our invention:

To an Oklahoma crude mix there is added 50% its volume of phenol and the temperature of the mixture is raised to 160° F. The mixture is then blown for thirty minutes with hydrogen chloride gas then thoroughly agitated and allowed to settle at the treating temperature of 160° F. The amount of hydrogen chloride gas absorbed is about 0.5% by weight of the oil. The settling takes place very rapidly. The extract is drawn off and the same amount of phenol is added again. The mixture is then agitated and settled. The procedure of adding phenol, agitating and settling is repeated twice more, making a total of four 50% batch treats with phenol.

Only the first treat is, however, blown with hydrogen chloride gas. After the solvent treat, the raffinate is treated with 4% of acid treated clay. The original Oklahoma crude mix has a gravity of 19.8° A. P. I. viscosity Saybolt of 144 seconds at 210° F. and a Conradson carbon test of 5.595%. The treated raffinate is a transparent red oil and has a gravity of 26.2° A. P. I., viscosity Saybolt of 99 seconds at 210° F. and a Conradson carbon test of 1.267%. The yield of raffinate is 55%. Treating the same stock with phenol only, the color obtained after contacting with clay is dark and the oil is non-transparent.

Our process may be modified in several ways. Thus, for example, the minute quantity of acid remaining in the raffinate may be removed by neutralization with alkali instead of by means of clay treatment. Our invention is not to be limited by the example given for illustration but only by the following claims in which it is our intention to claim all novelty inherent in the invention:

What we claim is:

1. The process of treating a petroleum stock containing asphaltic impurities which comprises agitating the stock with a selective solvent in the presence of not over, approximately, 1% of an acid, which does not react with the selective solvent under the conditions of working, and which is not present in the solvent, as such, as

an impurity below the temperature at which the stock becomes completely miscible with the selective solvent, separating the solvent layer from the raffinate layer and removing the selective solvent from the raffinate layer.

2. Process according to claim 1 in which the acid employed is an inorganic acid.

3. Process according to claim 1 in which the acid employed is hydrochloric acid.

4. Process of treating a petroleum stock containing asphaltic impurities which comprises adding to the stock an inorganic acid in an amount less than that which causes separation of sludge, and then treating the stock containing the acid with a selective solvent.

5. The process of treating a petroleum stock containing asphaltic impurities which comprises adding to the stock a selective solvent, introducing hydrogen chloride gas into the mixture in an amount less than that which causes separation of sludge, agitating the mixture, allowing layer formation to take place, separating the solvent layer from the raffinate layer and removing the selective solvent from the raffinate layer.

6. Process according to claim 5 in which the selective solvent is a phenolic compound.

7. Process according to claim 5 in which the selective solvent is undiluted phenol.

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