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WILLIAM T. MALONEY, OF OLEAN, NEW YORK.

PROCESS OF REFINING MINERAL OILS.

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To all whom it may concern:

Be it known that I, WILLIAM T. MALONEY, a citizen of the United States, residing at Olean, in the county of Cattaraugus and State of New York, have invented certain new and useful Improvements in Processes of Refining Mineral Oils, of which the following is a specification.

The present invention relates to the art of refining mineral oils, and more particularly to a process which contemplates the intimate association of air, oxygen, or ozone with the oil whereby those elements in the oil which readily combine with such oxidizing agents can be separated from the oil by treatment with alkali, the hypochlorites of the alkalis, or sodium or potassium bisulfite, thereby forming a precipitate, the reagent being recoverable with a minimum loss.

In the common process of refining oils by the addition of sulphuric acid and agitation of the mixture, a quantity of "sludge" is formed which is practically worthless, and from which a part of the original volume of acid can only be recovered at considerable expense. Further, a quantity of the oil combines with the acid in this process and is carried off in the form of "sludge," resulting in an appreciable decrease in the volume of refined oil as compared with the volume prior to treatment. The oil so made is, moreover, oxidizable and is therefore wholly unsuited for use in transformers until it has been subjected to other and expensive processes, or treated with such a large amount of acid as to cause an enormous loss of oil.

It is the purpose of the present invention to manufacture by a cheap and efficient process an oil which is suited to all conditions of lubrication, but which is more particularly adapted for use in electric transformers. An oil for this purpose must possess qualities not found in the ordinary lubricating oils. It must be incapable, or practically incapable of absorbing oxygen, and it must serve as an insulator as well as a cooling medium in the transformer. Therefore it is necessary that it be capable of withstanding electric currents of very high voltage. If it cannot resist oxidation there will be a "sludge" formed which may cake around the transformer coil, and, because of its thick and tarry consistency there is a tendency for the coil to become heated, and the oil likewise loses its insulating properties.

One of the objects of the present invention is to produce an oil suitable for use in transformers which will not "sludge" when subjected to the tests for such an oil. Any oil will "sludge" in a transformer in time, but if an oil passes the usual tests it is called "non-sludging."

Another object is to produce an oil the volume of which is not appreciably diminished by treatment in the process of refining.

A further object is to so treat an oil with alkali the hypochlorites of the alkalis, or sodium or potassium bisulfite that the reagent can be recovered for re-use with a minimum decrease in quantity and quality.

A still further object is to provide a process for refining mineral oils which is cheap and efficient, and which does not necessitate a change in the present refinery equipment.

In attaining these objects with this process an oil is produced which shows a marked improvement in color, demulsibility and oxidation tests, and, in the case of many oils, in viscosity and cold tests. The cost of producing such oil is cheapened through a saving of oil, and also from the actual cost of treatment.

Oil is first introduced into an agitator of the conventional type commonly used in the refining industry. It is then agitated, or blown, with air, oxygen, or ozone, the oil being either hot or cold. If air or oxygen is used for blowing it is preferable that the oil be heated to a temperature not to exceed 50° Fahrenheit below its flash point, but when ozone is used the oil may be cold, because the unusual activity of ozone will produce the same effect with equal efficiency and speed. This particular treatment is not essential, but is desirable inasmuch as it hastens the absorption and combination of air, oxygen, or ozone, as the case may be, with the oxidizable substances in the oil. The time of agitation varies with the quality and amount of oil being treated, and the temperature to which the oil is heated may be any temperature up to 50° Fahrenheit below its flash point. This limit is set for the sake of safety, and also to prevent excessive decomposition.

Except in those cases where ozone is used, it is preferable to use temperatures of 180° to 300° F. during the oxidation treatment, a certain amount of heat being required to produce satisfactory results.

Next, the oil is similarly treated by agitation with a strong solution of alkali or a solution giving an alkali reaction. By alkali is meant caustic soda, caustic potash, the carbonates of soda or potassium, calcium oxide or hydrate. Hypochlorites of the alkalis, sodium or potassium bisulfite may also be advantageously used, or, in fact, any compound which, on hydration, gives an alkaline reaction. The selection of a reagent depends largely on its cost. The solvent used, in the case of any of the various reagents, is water.

When treated in this manner a flocculent precipitate is formed and such precipitation can be facilitated by heating the oil as it is agitated, although this step of the process can also be performed if the oil is cold. If hot, however, the reagent settles out easily and may then be drawn off. The treatment with alkali after oxidation is preferably carried out at temperatures of 180° to 200° F. It is advisable to continue this step of the process until there is no further evidence of precipitation. In settling out the alkaline solution sinks to the bottom of the agitator with very little diminution of its original volume. Above this solution there is a well-defined band of precipitate which increases in thickness as the solution is cooled, either by artificial means or by being allowed to stand. The alkaline solution may then be drawn off and is immediately ready for reuse. To hasten precipitation it has been found advisable, in some cases, to add a little clay or similar material in the form of a fine powder and agitated with the oil.

After having been cooled sufficiently, the oil is separated from the precipitate by means of a continuous filter, or by any other means of filtering, to remove the precipitate.

A variation of the process described above is accomplished with equal efficiency in adding a small quantity of alkali, the hypochlorites of the alkalis, or sodium or potassium bisulfite before or during the blowing with air, oxygen, or ozone. It is best to introduce the reagent in the form of a solution in order to insure a more intimate contact with the entire body of oil, the solvent for the reagent being water.

A still further variation, and one which is preliminary to both of the above is as follows:—

The oil is well agitated with alkali, hypochlorites of the alkalis, or sodium or potassium bisulfite, preferably in solution, and the excess reagent allowed to settle to the bottom of the agitator where it is drawn off, or, if desired, allowed to remain in the oil. A 10% solution of sodium carbonate, or soda ash, is very satisfactory and is cheaper than caustic soda. At this point it might be added that an efficient solution for causing heavy precipitation is a 35% to 40% solution of caustic soda. These solutions are not especially recommended, however, inasmuch as the variations in quality of the oil treated are considerable, and the particular strength of solution must of necessity depend on the particular oil being treated, and the conditions under which this treatment is taking place.

The mixture, or solution, of alkali, hypochlorites of the alkalis, or sodium or potassium bisulfite, and oil is next exposed to air, oxygen, or ozone either in shallow pans or in the ordinary storage tanks. The use of pans shortens the necessary time of exposure inasmuch as a greater surface of liquid is presented thereby hastening the action. If done in storage tanks the time of exposure can be shortened by putting a slight pressure of air, oxygen, or ozone on the surface of the liquid.

In the course of time, varying from one to several days depending on the qualities of the oil being treated, a precipitate will form, and the oil will be brighter and frequently of lighter color than the same oil which has been exposed to air, oxygen, or ozone, but which has not been treated with the reagent. In some cases the oil may then be filtered free of the precipitate when it will be found to pass the desired tests as to color and demulsibility. Often it is advantageous, after filtering, to again agitate the oil with a strong solution of alkaline reagent in the manner first described. By strong solution I mean a 25 to 50% solution.

The color of the oil is dependent upon the amount of oxygen absorbed by it, and this, in turn, is dependent upon the time of exposure and the pressure of the oxygen-containing gas used. Test can be readily made to determine when this stage has been reached.

If too high a temperature is reached in the precipitation with alkali, the hypochlorites of the alkalis, or sodium or potassium bisulfite there is a tendency for the oil to temporarily darken in color, but upon standing and cooling the good color is restored. Or, if desired, the oil may be washed with a hot, dilute solution of the reagent until there is no further improvement in its color, and then washed with water. By dilute solution I mean a 5% solution, or even weaker.

It is then necessary to heat the oil and blow the moisture out with air, or by any other approved method. The judicious use of strong alkaline solution shortens the time consumed in this step of the process by eliminating the necessity for such drying or brightening.

The use of ozone in the process of purification is particularly advantageous in the treatment of gasoline and transformer oil. Because of its volatility and extremely inflammable qualities gasoline cannot be safely

heated for treatment. The use of ozone enables the process to be carried on efficiently without heat, thereby eliminating the danger from fire or explosion. In the making of transformer oil the best method is to use ozone with warm, or even hot oil, with or without an addition of alkali. By using ozone the higher temperature required with air or oxygen is not necessary. Excessive heat is to be avoided because of decomposition. In the case of some oils a preliminary light treatment with acid is advantageous before this process is used.

No claim is made to particular apparatus for performing the process above described for the reason that the ordinary refinery apparatus now in use is sufficient for that purpose, except in the case of ozone the use of which requires the installation of ozonizing equipment.

What is claimed is:—

1. The process of making transformer oil which consists of exposing mineral oil to the action of an oxygen containing gas to oxidize the sludging components, and adding an alkaline reagent to precipitate the oxidized substances.

2. The process of making transformer oil which consists in exposing mineral oil to the action of an oxygen containing gas while heating, to oxidize the sludging components, and adding an alkaline reagent to precipitate the oxidized substances.

3. The process of making transformer oil which consists in exposing mineral oil to the action of an oxygen containing gas while heating, to oxidize the sludging components, and adding an alkaline reagent while heating to precipitate the oxidized substances.

4. The process of making transformer oil which consists in exposing mineral oil to the action of an oxygen containing gas to oxidize the sludging components, and adding an alkaline reagent while heating, to precipitate the oxidized substances.

5. The process of making transformer oil which consists in exposing mineral oil to agitation with an alkaline solution, settling, drawing off the excess of alkaline solution, exposing the treated oil to the action of an oxygen containing gas to oxidize sludging components, and filtering off any precipitate that is formed.

6. The process of producing transformer oil which consists in adding a small quantity of alkaline reagent to the body of the oil and at the same time agitating the oil with a gas containing oxygen, in next agitating the oil with a concentrated alkaline solution, allowing the solution to settle, and finally filtering the body of oil free from the precipitate formed.

7. The process of making transformer oil which consists in exposing mineral oil to the action of a gas containing ozone to oxidize the sludging components, and adding an alkaline reagent to precipitate the oxidized substances.

8. The process of producing transformer oils which consists in agitating mineral oil with ozone, treating the oil with an alkaline reagent, and separating the precipitate formed by the reaction from the body of oil by filtration.

9. The process of producing transformer oils which consists in adding a small quantity of alkali to mineral oil and simultaneously agitating the oil with ozone, then in agitating the oil with a strong alkaline solution, in allowing the solution so formed to settle, drawing off the excess of alkaline solution, and finally filtering off any precipitate that has formed.

10. The process of producing transformer oils which consists in adding a small quantity of alkali to mineral oil, while agitating the oil with ozone, then washing the oil with a dilute alkaline solution, in allowing the solution to settle, and drawing off the excess of alkaline solution, and finally filtering off any precipitate that has formed.

In testimony whereof I affix my signature.

WILLIAM T. MALONEY.