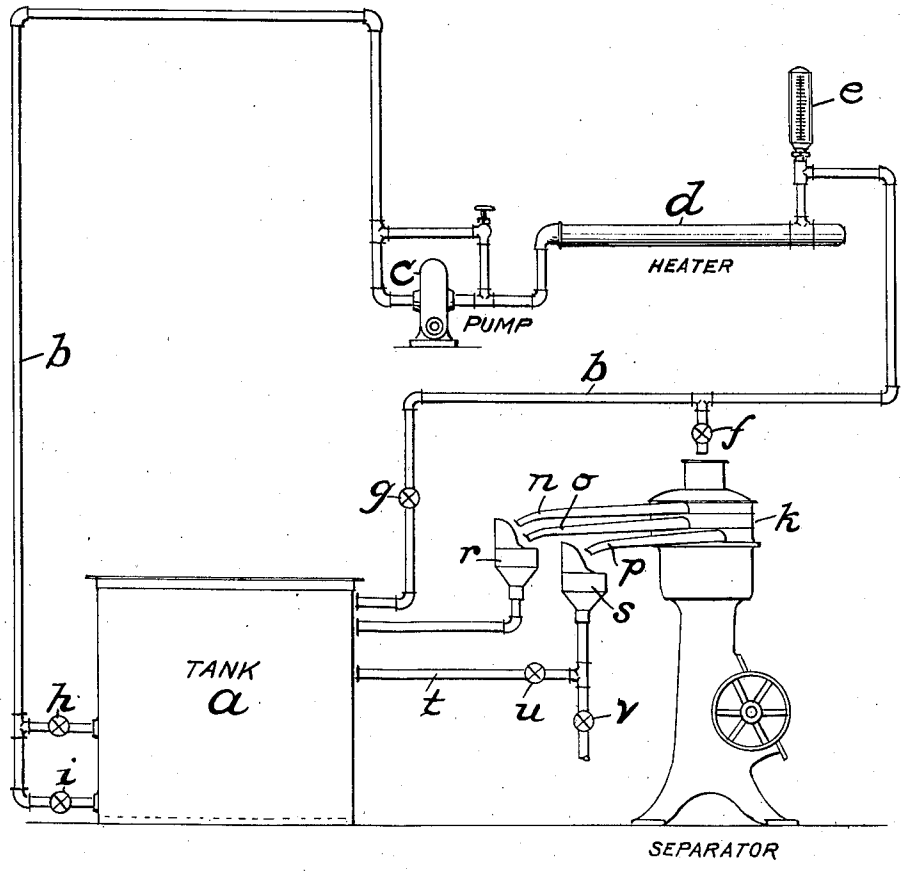
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F. H. McBERTY

PROCESS OF EFFECTING REACTION BETWEEN LIQUIDS TENDING TO FORM TIGHT EMULSIONS

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WITNESS:

Robt. R. Ritchel.

INVENTOR

Ford H. McBerty

BY

Bussey and Harding

ATTORNEYS.

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FORD H. McBERTY, OF POUGHKEEPSIE, NEW YORK, ASSIGNOR TO THE DE LAVAL SEPARATOR COMPANY, OF NEW YORK, N. Y., A CORPORATION OF NEW JERSEY.

PROCESS OF EFFECTING REACTION BETWEEN LIQUIDS TENDING TO FORM TIGHT EMULSIONS.

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In the process of purifying used lubricating oil, it is known to add to and mix with the oil a counter-colloid, such as oleic acid and the soda ash solution, which will agglomerate the carbon, and then separate from the oil, by centrifugal force, the counter-colloid and the carbon agglomerated therewith. It is known to mix other liquids which are relatively or absolutely insoluble one in another, in order to cause similar or other reactions between them, with a view, usually, to the recovery of one of the liquids in a pure condition by subsequent separation. In such cases, vigorous stirring or agitation of the liquids is necessary in order to obtain the large contact surface required to secure a thorough reaction. Under these conditions, a so-called emulsion is often formed, which is so tight that subsequent separation, even by centrifugal force, becomes difficult or even impossible.

The object of my invention is to secure the desired reaction with a minimum of agitation and with avoidance of a tight emulsion, so that subsequent separation will be facilitated.

I have discovered this object can be accomplished by mixing the liquids with such avoidance or limitation of agitation that excessive dispersion of the internal phase is prevented, then separating the mixture into its components, and then re-mixing the components; the operation being continued until the desired maximum action of the components on each other has been secured. The preferred mode of carrying out the process will vary with the liquids which are being treated. One separated component may be used to treat further quantities of the other component or may be used to re-treat the quantity of the other component that has been already treated; and the relative proportions of the two components may be regulated and may, if desired, be varied during the process. One liquid may be added to a quiescent body of another liquid or gradually to a flowing stream of such other liquid.

In order that my invention may be carried out by those skilled in the art, I will describe specifically one preferred way of practicing the process in its application to a specific purpose; namely, the removal of finely divided carbon from used automobile crank case oil. It is known that a water solution of trisodium phosphate is particularly effective to agglomerate the carbon, but its use has been hitherto restricted by reason of its strong tendency to form emulsions. A convenient apparatus in which the process can be practiced is shown in the drawing, which is a diagrammatic view in elevation of such apparatus.

The apparatus comprises a tank *a* and a pipe line *b* establishing a closed circuit from and back to the tank. In the pipe line is interposed a pump *c*, a heater *d*, a thermometer *e*, valve *f* and beyond valve *f* a valve *g*. Valves *h* and *i*, located at different levels, control the passage of liquid from tank *a* to pipe line *b*. A centrifugal separator *k* communicates with pipe line *b* through valve *f*. The discharge spouts *n*, *o*, from the separator communicate with a funnel *r*, which discharges into tank *a*. Spout *o* is for the discharge of the lighter component. If the material within the separator should overflow, the outflow will discharge through pipe *n*. The discharge spout *p*, through which the heavier component is discharged from the separator, discharges into a funnel *s*, which communicates with a pipe *t* (controlled by a valve *u*) leading back to the tank, and with a valved outlet *v* leading to a sewer or other suitable discharge.

The contaminated oil is placed in tank *a* and circulated through pipe line *b* back into the tank, being heated en route, by heater *d*, to a temperature of (say) 180° F. A water solution of trisodium phosphate is then added to the oil in tank *a*. The valves *f* and *g* are then adjusted so that part or all of the stream from the heater *d* is run through the centrifuge. The valve *u* is opened and the valve *v* closed so that both components discharged from the centrifuge return to tank *a*. If necessary or desirable, the ratio of oil to water solution in the mixture that is circulated through pipe line *b* is controlled by means of valves *h* and *i*. During the described continuous circulation, centrifugal separation and remixture, there is a gradual agglomeration of the carbon by the trisodium phosphate solution, part of the carbon coagulating and depositing on the walls of the centrifugal bowl and accumulating there, the remainder being taken up by the trisodium phosphate solution. At this stage, the clear oil and the

water solution separately discharging from the centrifuge may be maintained separate. The water solution and the carbon transferred to it may be released from the circuit by opening the valve *v* and closing the valve *u*. The purified oil may be released from the circuit by turning the oil spout *o* of the centrifuge around and allowing it to discharge into a barrel or any other desired receptacle.

A characteristic feature of the described process is that the process involves, not only the mixing of two liquids adapted to form an emulsion and tending to react one on the other and a separation of the constituent liquids of the emulsion; but a re-mixing of the separated constituents (or a mixing of one of the separated liquids with a part of the other liquid not previously mixed with the first liquid), followed by another separation: the operation being repeated as often as is necessary until a final satisfactory separation. Such a process would be without utility as applied to an emulsion wherein the reaction is substantially complete while the emulsion is still comparatively weak; because it would be possible to separate the constituent in a single separating operation. With many emulsions, however, such as that produced by adding a carbon agglomerating element to used lubricating oil, the more effective the agglomerating agent, the tighter the emulsion, and the more difficult the separation. It is obvious that if the most effective reagent could be used and if, then, the difficulty of effecting a complete separation could be overcome, a pronounced advance in the art would be made.

It is known, with typical emulsions, that if incompletely formed, as for instance by agitation for but a short time, which, for convenience, we will designate a single time unit, the emulsion will not become tight and the constituents may be separated easily and quickly, while if the agitation is continued for multiple time units, the emulsion will become tight and will be permanent, or will be separable into its constituents only with difficulty. An emulsion of trisodium phosphate and oil is an example of such an emulsion. That is, if said liquid and reagent are mixed for but one unit of time with a minimum of agitation, they can readily be separated, but the reaction whereby the reagent agglomerates the carbon of the oil will be incomplete. In order that the reaction should be complete, the liquid and reagent would require mixing for multiple units of time, which, as above stated, would produce a tight emulsion. I have found that if, instead of securing a complete reaction by maintaining the mixture for multiple units of time, or by agitation to expedite the reaction, I first mix for a single unit of time, with avoidance of unnecessary agitation, and then separate, and repeat the operation until the aggregate units of time about equal those necessary to complete the reaction in a single mixing, the reaction will be completed; that is, all the carbon will be agglomerated, but after each mixing, the separation can be readily effected because the immediately preceding mixing has not been of such a character as to create a tight or stable emulsion. As a result, after the final operation, the agglomeration of the carbon is substantially completed and no difficulty is encountered in thus effecting, fractionally, the separation from the oil of substantially all the carbon.

It is obvious that the success of the process is not dependent on the particular liquids that are emulsified with each other or on the particular reagent that is emulsified with any particular liquid that it is desired to purify. It is characteristic of emulsions, in general, that the longer they stand, or the more violently they are agitated, the more nearly inseparable they become. My process is therefore workable with all emulsions and is useful in all cases where reagents are added to liquids to produce emulsions whose subsequent separation presents difficulties.

Examples of other uses to which the process may be applied are:

The removal of oil-soluble saponifiable compounds from liquid petroleum products, as in the treatment of automobile engine, Diesel engine, turbine and similar oils, dry cleaners' solvents, transformer oils, switch oil, etc., with alkaline aqueous solutions to reduce their so-called "neutralization values".

The removal of saponifiable oils, fats or waxes from high-boiling solvents used for extraction of oil seeds, slaughter house scrap, garbage, etc., by the so-called wet process involving treatment with alkaline aqueous solutions.

The removal of metallic salts, soaps, etc. (such as anhydrous halides of aluminum, iron, sulfur, tin, zinc; anhydrous heavy metal soaps, such as ferric oleate; hydrated acid soaps of the alkali and alkali earth metals, such as dry cleaners' benzine soaps), from their solutions in liquid hydrocarbons by double composition with suitable compound or compounds in aqueous solution. Examples of such reactions are: the removal of residual stannic, zinc or other chlorides from oils or solvents by treatment with suitable alkaline aqueous solutions, as when these chlorides are added to the hydrocarbon liquid to agglomerate colloidally dispersed asphaltic material; the removal of heavy metal soaps from transformer oils, dry cleaners' solvent, turbine oils, etc. by treatment with aqueous solutions of basic alkali metal phosphate, silicate, hydroxide, etc.; the removal of residual benzine soap from dry cleaners' solvent by the use of alkaline aqueous solutions of alkali metal compound, in order to facilitate agglomeration of suspended dirt, etc.

The removal of chemically active organic

compounds from their solutions by reaction with a second compund in a solution only slightly miscible with the first, under such conditions that the formation of solids may occur at the interface between the solutions. For instance, the removal of aniline from its solution in nitrobenzol, solvent naphtha or similar solvent by the use of an aqueous solution of hydrochloric acid saturated with, or approaching saturation with, aniline hydrochloride.

Having now fully described my invention, what I claim and desire to protect by Letters Patent is:

1. In the process of purifying a liquid by causing it to react with another liquid tending to agglomerate the impurities in the first liquid but which also tends to form so tight an emulsion with the first liquid as to obstruct the separation of the liquids; the process of preventing the formation of a tight emulsion before final separation which comprises mixing the liquids, separating them after the reaction between them is only partially completed and before a tight emulsion is formed, remixing them, and continuing the operation until the reaction is substantially completed.

2. In the process of purifying a liquid by causing it to react with another liquid tending to agglomerate the impurities in the first liquid but which also tends to form so tight an emulsion with the first liquid as to obstruct the separation of the liquids; the process of preventing the formation of a tight emulsion before final separation, which comprises adding one liquid to the other, establishing a flowing stream of the mixed liquids, effecting a continuous separation of the liquids in the flowing stream after said reaction is only partially completed and before a tight emulsion is formed, remixing the liquids, and continuing the operation until said reaction is substantially completed.

3. In the process of purifying a liquid by causing it to react with another liquid tending to agglomerate the impurities in the first liquid but which also tends to form so tight an emulsion with the first liquid as to obstruct the separation of the liquids; the process of preventing the formation of a tight emulsion before final separation, which comprises establishing a continuous flow of a mixture of the two liquids, effecting a continuous separation of the liquids after said reaction is only partially completed and before a tight emulsion is formed, conveying the separated liquids to a liquid body containing both liquids, and from said liquid body maintaining seid continuous flow toward the locus of separation.

4. In the process of purifying a liquid by causing it to react with another liquid tending to agglomerate the impurities in the first liquid but which also tends to form so tight an emulsion with the first liquid as to obstruct the separation of the liquids; the process of preventing the formation of a tight emulsion before final separation, which comprises mixing and establishing a flowing stream of the two liquids, effecting a separation of the two liquids of the flowing stream after said reaction is only partially completed and before a tight emulsion is formed, remixing the two liquids and conveying them to the flowing stream, and regulating the proportions of the two liquids in the flowing stream.

5. In the process of purifying a liquid by causing it to react with another liquid tending to agglomerate the impurities in the first liquid but which also tends to form so tight an emulsion with the first liquid as to obstruct the separation of the liquids; the process of preventing the formation of a tight emulsion before final separation, which comprises mixing the two liquids, separating them after said reaction is only partially completed and before a tight emulsion is formed, mixing one of the separated liquids with the other liquid, separating the mixture, and continuing the operation until said reaction is substantially completed.

6. In the process of purifying a liquid by causing it to react with another liquid tending to agglomerate the impurities in the first liquid but which also tends to form so tight an emulsion with the first liquid as to obstruct the separation of the liquids; the process of preventing the formation of a tight emulsion before final separation, which comprises adding one liquid to the other, establishing a flowing stream of the mixed liquids, effecting a continuous separation of the mixed liquids in the flowing stream after said reaction is only partially completed and before a tight emulsion is formed, adding one of the separated liquids to the other liquid and separating them, and continuing the operation until said reaction is substantially completed.

7. In the process of purifying a liquid by causing it to react with another liquid tending to agglomerate the impurities in the first liquid but which also tends to form so tight an emulsion with the first liquid as to obstruct the separation of the liquids; the process of preventing the formation of a tight emulsion before final separation, which comprises flowing one of the liquids in an endless circuit and in the course of its flow subjecting it to heat, then adding the other liquid to the liquid in circuit, diverting a mixture of the liquids and effecting their separation after said reaction is only partially completed and before a tight emulsion is formed, restoring both liquids to the circuit, and continuing the operation until said reaction is substantially completed.

8. In the process of purifying a liquid by causing it to react with another liquid tend-

ing to agglomerate the impurities in the first liquid but which also tends to form so tight an emulsion with the first liquid as to obstruct the separation of the liquids; the process of preventing the formation of a tight emulsion before final separation, which comprises mixing the liquids, separating them after said reaction is only partially completed and before a tight emulsion is formed, remixing them, continuing the operation until said reaction is substantially completed, and then finally separating the two liquids and maintaining them separated.

9. The process of eliminating colloidal carbon from used lubricating oil; which comprises adding to the oil a solution adapted to agglomerate the carbon, and, before the oil and the solution forms a tight emulsion, centrifugally separating from the oil the solution and also any carbon that separates out, and remixing the separated oil and the solution and again separating as before, the two stage operation of mixing and separation being repeated until substantially all the carbon is separated from the oil.

10. In the purification of a liquid by means of another liquid substantially immiscible therewith but adapted to react therewith to loosen the bond between the first liquid and its contaminating ingredient while tending to form a tight emulsion of the liquids; the process which consists in mixing the liquids to the limited degree required to produce a relatively loose emulsion and a partial reaction of the character specified, separating the two liquids, thereby causing the second liquid to carry off such portion of the contaminating ingredient as has been freed from the first liquid, remixing the two liquids, and continuing the operation until the specified reaction is completed.

In testimony of which invention, I have hereunto set my hand, at Poughkeepsie, New York, on this eleventh day of August, 1924.

FORD H. McBERTY.