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PROCESS FOR RECLAIMING OIL
CONTAINING CARBON

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My invention relates to the reclaiming of used oils containing considerable quantities of finely divided carbon. Examples of such oils are those from internal combustion engines, particularly Diesel engines, and insulating oils from trans-
formers and high capacity switches.

The object of the invention is to provide a process that may be operated either on batches or (preferably) continuously to remove such carbon and any accompanying acid bodies from the oil at small expense and particularly with minimum loss of the reclaiming agent.

Reclaiming of such oils, involving removal of carbon, by treatment with alkalis, is old and well known. Typical known processes effect an admixture or agglomeration of the carbon and other impurities with an aqueous solution of a reagent. These, with the carbon, are subsequently gravitationally or centrifugally separated from the oil and thrown away. To minimize expense a minimum quantity of the chemical causing agglomeration of the carbon is used (the solution usually having a gravity between 1.05 and 1.1). Nevertheless, the cost is still high, because the solution can be used only once.

My invention involves the initial conception of the possibility of effecting a separation not only of the reagent and carbon from the oil but the separation of the reagent from the carbon either in the oil separation process or subsequent thereto or partly concurrently with and subsequent thereto. In research work involved in the development of the invention I discovered that the specific gravity of the carbonaceous material whose removal from the oil is to be effected lies within narrow limits, usually between 10% and 15% heavier than the oil from which it is formed. I have also discovered that the specific gravity of such carbonaceous material ranges distinctly above the specific gravities of the different aqueous solutions of the reagents that are employed in commercial processes for removing carbonaceous materials from used oil. I have discovered that if the oil is properly treated with an alkaline reagent solution having a specific gravity substantially in excess of that of the carbonaceous material, it is practicable, by centrifugal force, to separate the carbonaceous material from both the oil and the reagent solution so that it will occupy in a centrifugal separator bowl a zone between them. This annular zone of carbonaceous material will, as the centrifugal operation progresses, sometimes build outward into the reagent solution space and then escape with the solution through the heavy liquid outlet. In other cases

it is necessary to, at intervals, admit some water and dilute the reagent so as to permit the escape of carbonaceous material. The feed of regular solution may then be immediately resumed and the separation continued. For quick change of solution density, a bowl of the type shown in Hall Patent No. 1,411,792 is particularly adapted.

In the tank into which the reagent solution and carbonaceous material are conducted, the carbon, by reason of its lighter specific gravity, rises to the surface and may be readily removed, leaving the reagent solution in condition for re-use. The separated carbonaceous material is usually a tar-like, substantially oil-free and substantially reagent-free body that, on exposure to air, dries to a stone-like consistency and may be easily burned like coal.

The process, so far as it is above described, is therefore one in which the reagent is used in a solution so concentrated as to be presumptively prohibitively expensive; but since in such solution the reagent is largely recovered in condition for re-use, with no appreciable expense involved in the recovery, it will be understood that the process is far more economical than the processes in general use, and that such economy is obtained without sacrifice of efficiency.

In order to practice the process efficiently, I have found it necessary to employ, as my reclaiming agent, an alkaline solution having a specific gravity more than 30% higher than that of the oil as contrasted with ordinary alkaline solutions having a specific gravity less than 15% higher, and usually considerably less than 15% higher, than that of the oil. There is no necessary upper limit to the specific gravity of the alkaline solution so long as it is sufficiently free flowing to freely discharge from the usual centrifuge. In the case of most oils the specific gravity of the alkaline solution should not be less than 1.2 and it is preferred that it should not exceed 1.4, although a higher specific gravity is permissible.

In the practice of my process I have been able to continuously reclaim carbon-containing oils with solutions of caustic soda, with solutions of sodium metasilicate, and with mixtures of them. These reagents are given merely as examples, since it should be understood that my invention does not involve the use of any particular reagent; the invention being in fact one permitting the use of any efficient alkaline solution or equivalent.

The addition of small quantities of certain dispersing or emulsifying agents to the solution accelerates the coagulation of the carbon. I have

found that triethanolamine is a very active reagent for this purpose. Apparently there is a reaction between the triethanolamine and the alkaline solution, since analysis of the combined solution finds only a small fraction of the nitrogen that was in the triethanolamine originally used. It is the resultant material that accelerates the agglomeration.

The use, as an oil reclaiming agent, of a solution of caustic soda or sodium metasilicate, to which has been added a relatively small proportion of triethanolamine, has been found to substantially improve the efficiency of the process regardless of the specific gravity of the treating solution, although, if the gravity of the solution is of ordinary gravity, it has no advantage over known reclaiming agents with respect to economy. In claiming this specific oil reclaiming solution, it is to be understood that sodium metasilicate is an equivalent of the caustic soda specified in the claims.

As an example of reclamation of used oil by my process, I treated 500 gallons of carbonized Diesel engine oil with two gallons of solution containing about 5 pounds of caustic soda or an equal amount of sodium metasilicate and had about 1 gallon of solution left. That is, I used only about one-half pound of chemical per 100 gallons of oil.

While, if triethanolamine be added, its permissive proportion may vary within a considerable range, say from about somewhat less than one percent to somewhat over five percent, the use of about 2½% of triethanolamine with a solution of caustic soda having a specific gravity of 1.4 (specifically, three gallons of such a caustic soda solution to which is added 270 cc. of triethanolamine) is found to be a very effective oil reclaiming agent.

Although my process may be carried on with other apparatus, I have found that shown on the accompanying drawing, which is a side elevation thereof, somewhat diagrammatic, to be particularly adapted for its efficient execution.

a is a reagent tank and *b* a reagent pump. *c* is a pipe from a dirty oil supply tank *d* to the dirty oil pump *e*. *f* is a heat exchanger, *g* a heater and *h* a centrifugal purifier with a heavy liquid outlet *i* and a light liquid outlet *j*. *k* is a cleaned oil pipe, *x* a cleaned oil pump and *m* a cleaned oil receiving tank. The pipe from the heater to the cleaned oil pump is in the form of a siphon *o* with a small hole *p* at the top connected by a pipe *q* with the cleaned oil space in the heat exchanger. *r* is a float entirely submerged in the reagent solution, *s* a valve, operated by the float, in a water supply pipe *t*, and *u* a tumble weight on the valve.

In operation the pump *b* draws reagent from the tank *a* and forces it into the pipe *c*. There it meets the stream of dirty oil from the oil supply tank *d* and with it flows to the dirty oil pump *e*, where the reagent and oil are thoroughly mixed. The mixture then flows through the heat exchanger *f* (wherein it takes up heat from the escaping treated oil), through the heater *g* (wherein its temperature is raised to between 170° F. and the boiling point) and thence into the centrifugal purifier *h*.

In the purifier the mixture is separated into three concentric annular layers, namely: the heavy reagent layer next to the bowl shell, the cleaned oil layer around the center, and a layer of carbonaceous material between them.

The reagent escapes through the heavy liquid outlet and returns to the reagent tank *a* for use with another quantity of oil. The cleaned oil

escapes from the light liquid outlet and flows through the heat exchanger *f*, wherein it gives up a large part of its heat to the incoming dirty oil and is then pumped into the cleaned oil receiving tank *m*.

The carbonaceous material will in some cases collect within the separator bowl until its outer face reaches the outlet for heavy liquid, after which any further collection causes the escape, with the reagent, of an equal quantity of carbon. When this does not occur normally, it is possible, by flowing a small quantity of water into the bowl, to temporarily reduce the specific gravity of the reagent solution therein until a large part of the carbon will pass out and to then resume the flow of standard strength reagent and continue the process.

In either case, when the carbonaceous material reaches the tank *a* it quickly separates from the reagent and rises to the top, whence it can be readily skimmed and dried for burning.

In order that there shall be efficient transfer of heat it is necessary that the oil shall flow rapidly over the heating surfaces. This requires passages of small cross section, resulting in a considerable pressure drop through the heater, which has to be overcome by the pump *x*. For best results a heat exchanger should be arranged for counterflow with the cold liquid entering at the bottom and hot liquid entering at the top, as shown in the drawing, and the heating surfaces must be kept covered. To prevent danger of hot cleaned oil overflowing, the pump *x* must have some excess capacity, which would tend to draw all oil out of the cleaned oil space of the exchanger. The arrangement shown prevents this, since as long as the hole *p* is open, air will flow in through it and break the pump suction so that the oil flow will be only that due to gravity and it will rise in the heat exchanger. When the oil reaches the pipe *q*, some oil will flow therewith and cover the hole *p* and shut out the air, so that the full suction of the pump will then act to cause rapid flow until the oil drops and permits air to enter the hole *p* and again break the suction. The flow through the heat exchanger is thus maintained at a rapid rate during a major part of the time, and the surfaces are always covered and there is no danger of overflow.

Because of the high temperature at which it is circulating, there is a continual evaporation of water from the reagent solution, which causes its density to increase. An increase of density causes the float *r* to rise and open the valve *s* to permit water from the pipe *t* to flow into the tank and reduce the density to normal, whereupon the float will fall and shut off the water. By use of the tumble weight *u* there is caused, when the float is down, a resistance to upward movement that prevents its rise until there has been a considerable increase in gravity. When the float does rise it will stay up until the gravity is reduced to the point where carbon will be discharged, thus automatically causing intermittent discharge of the carbon. It is practicable to use other means, not involving the rise of a tumble weight, to intermittently admit water direct to the machine, as, for example, a motor-operated valve.

What I claim and desire to protect by Letters Patent is:

1. The process of reclaiming used lubricating oil containing dispersed carbon which comprises coagulating dispersed carbon out of the oil phase and preventing its dispersal in the aqueous phase

by mixing and heating with an alkaline solution having a specific gravity more than 30% greater than that of the oil, separating the oil from the alkaline solution and the carbon and the carbon from the alkaline solution, treating successive quantities of used oil with the alkaline solution and adding water to compensate for evaporation and thus maintain the gravity of the solution.

2. The process of reclaiming oils containing dispersed carbon which comprises mixing therewith a reagent having an alkaline reaction and including an agglomerating agent and a non-miscible solution and having a specific gravity greater than that of the carbon, passing the mixture in heat exchange relation with previously reclaimed oil, heating the mixture to not less than 170° F. and not above the boiling point, centrifugally separating the solution and carbon from the oil, and gravitationally separating the carbon from the solution.

3. The process of reclaiming oils containing dispersed carbon which comprises mixing therewith a reagent having an alkaline reaction and including an agglomerating agent and a non-miscible solution and having a specific gravity greater than that of the carbon, passing the mixture in heat exchange relation with previously reclaimed oil, heating the mixture to not less than 170° F. and not above the boiling point, centrifugally separating the solution and carbon from the oil, gravitationally separating the carbon from the solution, and compensating for evaporation of the solvent by adding solvent to the solution.

4. The process of reclaiming used lubricating oil containing dispersed carbon which comprises mixing with the oil an aqueous solution of caustic soda to which is added a relatively small proportion of triethanolamine, heating the mixture and centrifugally separating the solution and carbon from the oil.

5. The process of reclaiming used lubricating oil containing dispersed carbon which comprises mixing with the oil an aqueous solution of caustic soda to which is added a relatively small proportion of triethanolamine, heating the mixture, centrifugally separating the solution and carbon from the oil and separating the carbon from the solution.

6. The process of reclaiming used lubricating oil containing disbursed carbon which comprises establishing a continuously flowing stream of oil to be reclaimed and, from a supply tank, a continuously flowing stream of a reagent having an alkaline reaction and having a specific gravity greater than and adapted to coagulate the carbon, uniting and mixing the streams, maintaining a continuous flow of the mixture and in the course of its flow first heating it to a temperature below its boiling point, thereby coagulating the carbon

out of the oil phase, then separating the oil from the reagent and impurities, and then continuously discharging the separated cleaned oil and the separated reagent, the latter to its said source of supply.

7. The process of reclaiming used lubricating oil containing disbursed carbon which comprises establishing a continuously flowing stream of oil to be reclaimed and, from a supply tank, a continuously flowing stream of a reagent having an alkaline reaction and having a specific gravity greater than and adapted to coagulate the carbon, uniting and mixing the streams, maintaining a continuous flow of the mixture and in the course of its flow first heating it to a temperature below its boiling point, thereby coagulating the carbon out of the oil phase, then separating the oil from the reagent and carbon and the reagent from the carbon, and continuously uniting and mixing the separated reagent with the stream of oil to be reclaimed.

8. The process of reclaiming used lubricating oil containing disbursed carbon which comprises establishing a continuously flowing stream of oil to be reclaimed and, from a supply tank, a continuously flowing stream of a reagent having an alkaline reaction and having a specific gravity greater than and adapted to coagulate the carbon, uniting and mixing the streams, maintaining a continuous flow of the mixture and in the course of its flow first heating it to a temperature below its boiling point, thereby coagulating the carbon out of the oil phase, then separating the oil from the reagent and impurities, and then continuously discharging the separated cleaned oil and the separated reagent, the latter to its said source of supply, adding water to the reagent to compensate for evaporation and controlling the addition of water in accordance with variations in the specific gravity of the reagent.

9. The process of reclaiming used lubricating oil containing disbursed carbon which comprises establishing a continuously flowing stream of oil to be reclaimed and, from a supply tank, a continuously flowing stream of a reagent having an alkaline reaction and having a specific gravity greater than and adapted to coagulate the carbon, uniting and mixing the streams, maintaining a continuous flow of the mixture and in the course of its flow heating it in two stages, then separating the oil from the reagent and impurities, then continuously discharging the separated cleaned oil and the separated reagent, returning the separated reagent to the supply tank and flowing the separated cleaned oil in heat exchange relation with the flowing mixture of oil and reagent to effect the first stage heating of said mixture.

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