D. M. ALLAN, Jr.

REFINING PETROLEUM.

APPLICATION FILED JUNE 25, 1913.

1,187,797.

Patented June 20, 1916.

Fig. 1.

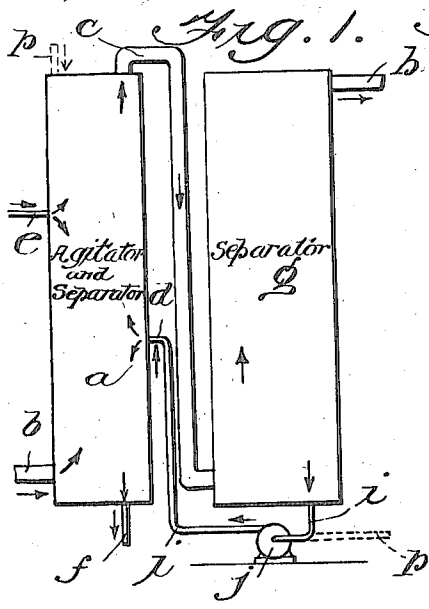


Fig. 2.

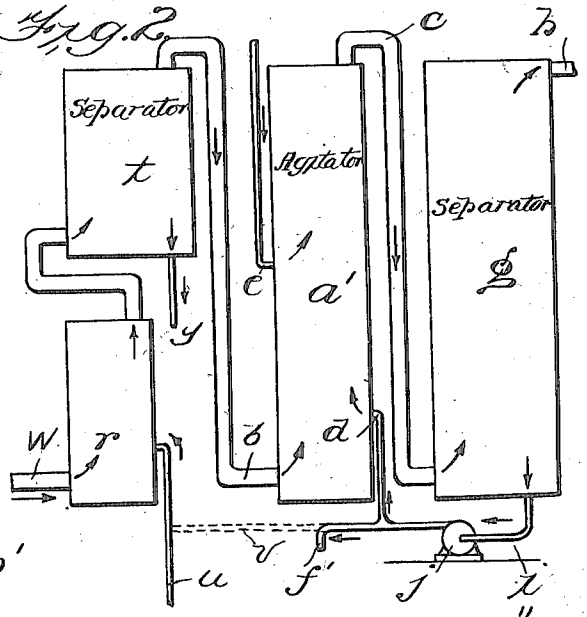


Fig. 3.

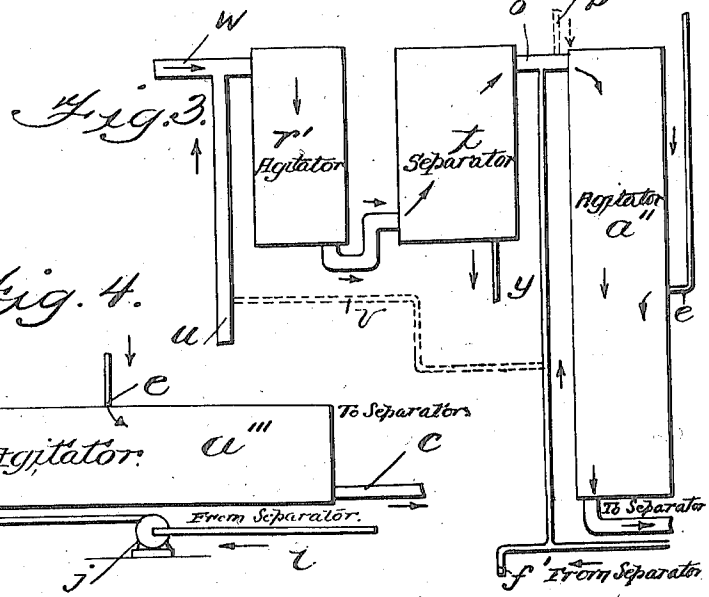


Fig. 4.

Fig. 5.

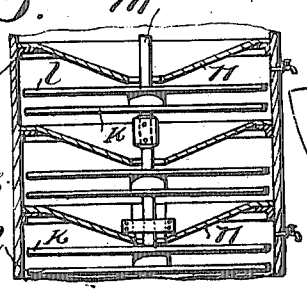
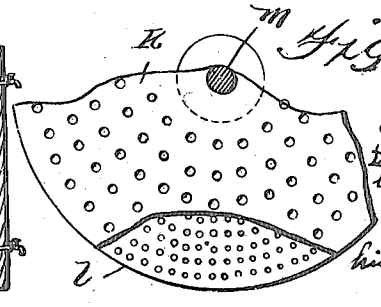


Fig. 6.



Witnesses
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UNITED STATES PATENT OFFICE.

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REFINING PETROLEUM.

1,187,797. Specification of Letters Patent. Patented June 20, 1916.

Application filed June 25, 1913. Serial No. 775,710.

To all whom it may concern:

Be it known that I, DAVID M. ALLAN, JR., a citizen of the United States, residing at Bayonne, in the county of Hudson and State of New Jersey, have invented certain new and useful Improvements in Refining Petroleum, of which the following is a specification.

This invention relates primarily to refining petroleum by bringing the same, in the form of naphtha or of burning oil distillate, or of other known or suitable product, distillate or undistilled, or even in its natural crude form, into contact either with sulfuric acid of 66° B. (or other strength sufficient to attack hydrocarbons of objectionable odor and color in the petroleum and to form with such hydrocarbons a heavy viscous liquid of blackish or brownish coloration) or with an aqueous solution of alkali (caustic soda) of sufficient strength to unite with sulfonic acids remaining in the petroleum as the result of a prior treatment with sulfuric acid followed by washing with water: but the invention is believed not to be limited in respect of novelty and usefulness to such refining. It extends to the refining of other oily liquids with sulfuric acid or with alkaline lye and to the refining of petroleum and other oily liquids by means of liquids other than sulfuric acid and alkaline lye. And in its yet broader aspects it extends generally to the treatment of one liquid with another which is only imperfectly (if at all) miscible with the former. The treatment may be, for example, in order to remove constituents considered objectionable in the liquid under treatment, or in order to obtain constituents considered desirable as separate products.

In accordance with the invention the treating is carried on in a continuous manner; that is to say, applying the treating menstruum (sulfuric acid or an aqueous solution of alkali as aforesaid) to a constantly changing body (stream) of the liquid under treatment (petroleum) in contradistinction to a substantially constant batch of the same.

Heretofore continuous processes and continuously operating apparatus have been devised for treating one liquid with another not perfectly miscible therewith, including the treating of petroleum with sulfuric acid or with an aqueous solution of alkali; but all of these prior processes and apparatus are believed to have disadvantages which are (to a useful extent at least) obviated by means of the present invention.

In accordance with this invention, streams of the two liquids (primarily petroleum and sulfuric acid or an aqueous solution of alkali as aforesaid) are brought into contact with each other under such conditions of agitation and flow that a large part at least of the treating liquid (sulfuric acid or alkaline lye) accompanies the liquid under treatment (petroleum) for a considerable distance during which the liquids are repeatedly agitated; the resulting mixture is allowed (or caused) to separate; and a part at least of the so separated partly spent treating liquid (sulfuric acid or alkaline lye) is returned to the stream of liquid under treatment (petroleum), best in advance of the place of original introduction of the treating liquid, and is again carried to the place of separation as aforesaid in admixture with the stream of liquid under treatment, thus constituting a species of endless circulation of treating liquid.

All the so returned liquid might be carried again to said place of separation; but it is considered an advantage and special improvement to carry thither a part only of the same and to withdraw another part after it has acted on the liquid under treatment.

The fresh and the partly spent treating liquids can be introduced at the same place, but more advantageously at different places (best the partly spent liquid in advance of the fresh, as already mentioned); and each of them can be introduced at one or more places.

Both processes and apparatus are included in the invention. Modifications by omission or otherwise can be made indefinitely without exceeding the limits of the invention; so long as the substance is taken of any one or more of the claims at the close of this specification.

In the accompanying drawings: Figure 1 is a diagram illustrating a petroleum treating apparatus in accordance with the invention in what is considered the best form of such apparatus; Figs. 2, 3 and 4 are diagrams illustrating other forms of such apparatus; and Figs. 5 and 6 are detail views of agitating appliances, not of my invention and not essential to be used in carrying my invention into effect.

The agitator and separator *a* (Fig. 1) is provided with inlets *b e* and outlets *c f;* and inside of same are agitating appliances best (Figs. 5 and 6) in the form of pairs of perforated plates *k l* fast on a vertical revolving shaft *m* between open centered ingathering plates *n* of a coned shape. The plates *k l* fill the interior of the agitator except for sufficient clearance to allow of easy revolution.

The separator *g* is connected below with the outlet *c* of the agitator and separator *a*. It has outlets *h i*. Outlet *i* is connected through pump *j* with inlet *d* of tank *a*. This last is termed a separator as well as an agitator because it is so designed that treating liquid in a spent condition (acid sludge or alkali sludge) shall separate therein from the liquid (petroleum) with which it is brought in contact in said tank *a*.

The relative proportions in which petroleum and the refining menstruum shall be introduced, the former at inlet *b* and the latter at inlet *e*, will depend in part on the quality of the petroleum and in part on the strength of the sulfuric acid or the alkaline lye. As a basis of starting, those proportions may be taken which would give satisfactory results in any known way for the quality of petroleum which it is desired to treat and the strength of sulfuric acid or alkaline lye which it is desired to employ as the treating liquid or menstruum. The revolutions of the shaft *m* per minute and the volume of petroleum introduced in the same period of time would best be so adjusted that a large volume of menstruum (not fully exhausted) will be carried over with the petroleum through the outlet *c* and that the menstruum (quite spent or in a greater or less degree of exhaustion) will escape by outlet *f*, eventually at least in amount corresponding with that in which said menstruum is introduced by the inlet *e*. Increased speed of rotation tends to emulsify the petroleum more completely with the menstruum and so favors the passage over of the menstruum. Increased supply of petroleum creates a larger current upward through the agitator and thus tends to carry over more of the menstruum. With shaft *m* making thirty eight turns per minute in a tank twenty five feet high by seven feet in diameter with ten pairs of plates *k l* on said shaft and with an inflow of three hundred barrels of burning oil (kerosene) distillate per hour (forty gallons to the barrel) and twenty five pounds of sulfuric acid of 66° B. (92 and ½% of sulfuric monohydrate) per minute, the mixed liquids escaping at outlet *c* carried spent and unspent sulfuric acid (sulfonic acids, and so forth) equivalent to five to ten pounds of sulfuric acid per minute. It is, of course, possible, and may be desirable, for the so escaping liquids to carry over more of the unspent acid and acid residues (or spent acid) per minute than is equivalent to the sulfuric acid supplied per minute by the inlet *e;* because the conditions of agitation and flow which tend to carry over fresh liquid from inlet *e* also tend to carry over partly spent liquid returned by pump *j* from separating.

The speed of shaft *m* and the inflow of petroleum should not be so great as to carry refining menstruum away by the outlet *h* (unless other settlers should be provided) or to obtain an unsatisfactory petroleum at said outlet *h* (unless further treatment is to be performed). The proportion of sulfuric acid of given strength per barrel of oil will naturally be minimized consistently with a proper quality of the treated petroleum from outlet *h;* but it may on occasion at least be desirable to sacrifice economy of acid to a more rapid treatment.

The streams of petroleum (introduced by inlet *b*) and sulfuric acid or alkaline lye (introduced by inlets *e* and *d*) are brought into contact with each other in agitator and separator *a* under such conditions of agitation and flow that a part at least of the treating liquid accomplishes the stream of petroleum to the separator *g*. The liquids thus flow together are agitated repeatedly during their course of considerable extent between said inlets and the outlet *c*. The mixed liquids from this outlet are separated from each other in tank *g* by difference of specific gravity. The so separated partly spent treating liquid is returned by pump *j* and connected pipes to the stream of liquid (petroleum) under treatment in the agitator and separator *a*. And a part of the so returned partly spent liquid is again carried by the stream of petroleum to the separator *g*, thus establishing an endless circulation of treating liquid. The partly spent liquid which is returned at inlet *d* is thereby returned to the stream of petroleum in tank *a* in advance of the fresh treating liquid; which is supplied as shown at inlet *e*.

At *p* and *p'* are shown in dotted lines inlets which can be used instead of or in addition to the inlet *e;* although not with advantage. Were *p'* to be used alone, the fresh and the returning partly spent menstruum would both enter tank *a* by inlet *d;* and the special advantage of returning the partly spent sulfuric acid or alkaline lye to the liquid under treatment in advance of the fresh acid or fresh alkaline lye would not be secured.

Referring to Fig. 2, the streams of liquid introduced into agitator *a'*, the petroleum to be treated at inlet *b* and the sulfuric acid or the aqueous solution of alkali at inlets *e d*, are brought into contact with each other under such conditions of agitation and flow that all the treating liquid from both inlets

e d accompanies the stream of petroleum over into the separator *g*. The liquids thus flowing together are agitated repeatedly during the course of considerable extent between inlets *d e* and outlet *e*. The liquids are separated from each other in separator *g* by difference of specific gravity. A part of the so separated partly spent treating liquid is returned to the agitator *a'* by pump *j*. A part only of said liquid is so returned; because were all of it to be carried again to the separator *g* the accumulation would eventually fill the tank *g*. Another part is withdrawn at outlet *f'*; or, if preferred, it also can be returned by dotted pipe *v* to the stream of petroleum in agitator *r* on its way through separator *t* to agitator *a'*. It would be withdrawn at outlet *y* after acting in tanks *r t* on the liquid under treatment. In Fig. 2, as in Fig. 1, the partly spent treating liquid which is endlessly circulated is returned to the stream of petroleum at inlet *d* before the fresh treating liquid is brought at inlet *e* into contact with the stream of liquid under treatment.

It is considered undesirable to mix acid sludge with a large body of petroleum containing moisture, lest objectionable hydrocarbons be so liberated. To avoid this fresh acid sufficient in strength and amount to remove the moisture can be introduced by pipe *u*, dotted pipe *v* being closed or omitted.

The apparatus of Fig. 3 differs from that of Fig. 2 in the travel of the liquids downward in agitators *a'' r'* instead of upward, as in agitators *a' r* of Fig. 2. The separator and pump arrangements for the agitator *a''* may be identical with those of Fig. 2. The dotted inlet *p''* corresponds with dotted inlet *p'* of Fig. 1.

In Fig. 4 the agitator *a'''* is placed on its side so that the liquids travel horizontally; and a combined agitator and separator *r''* (corresponding with that portion of agitator and separator *a* which lies below the inlet *d*) is employed in place of an agitator *r* or *r'* and a separator *t* (as in Figs. 2 and 3.) The agitator and separator *r''* could be used with any of the agitators *a*, *a'* or *a''*; and either of the agitators *r r'* (with separator *t*) could be used with any of the other agitators *a*, *a'*, *a''*, *a'''* as well as with that in connection with which it is shown.

In specifying in hereinafter written claims that the liquid under treatment is primarily petroleum and the treating liquid primarily sulfuric acid or an aqueous solution of alkali, it will be understood that treatments involving some other liquid or liquids are not thereby to be excluded from the scope of said claims.

I claim as my invention or discovery:

1. The process of treating one liquid with another not perfectly miscible therewith, primarily petroleum with sulfuric acid or an aqueous solution of alkali, consisting in bringing streams of said liquids into contact with each other under such conditions of agitation and flow that part at least of the treating liquid accompanies the stream of liquid under treatment, agitating the liquids thus flowing together repeatedly during a course of considerable extent, separating the liquids from each other by difference of specific gravity, returning part at least of the so separated treating liquid in partly spent condition to the stream of liquid under treatment before this latter reaches such place of separation, and causing a part at least of the so returned liquid to be again carried by the stream of liquid under treatment to the said place of separation, substantially as described.

2. The process of treating one liquid with another not perfectly miscible therewith, primarily petroleum with sulfuric acid or an aqueous solution of alkali, consisting in bringing streams of said liquids into contact with each other under such conditions of agitation and flow that part at least of the treating liquid accompanies the stream of liquid under treatment, agitating the liquids thus flowing together repeatedly during a course of considerable extent, separating the liquids from each other by difference of specific gravity, returning part at least of the so separated treating liquid in partly spent condition to the stream of liquid under treatment before this latter reaches such place of separation and also before the fresh treating liquid is brought into contact with the stream of liquid under treatment, and causing a part at least of the so returned liquid to be again carried by the stream of liquid under treatment to the said place of separation, substantially as described.

3. The process of treating one liquid with another not perfectly miscible therewith, primarily petroleum with sulfuric acid or an aqueous solution of alkali, consisting in bringing streams of said liquids into contact with each other under such conditions of agitation and flow that part at least of the treating liquid accompanies the stream of liquid under treatment, agitating the liquids thus flowing together repeatedly during a course of considerable extent, separating the liquids from each other by difference of specific gravity, returning part at least of the so separated treating liquid in partly spent condition to the stream of liquid under treatment before this latter reaches such place of separation, causing part only of the so returned liquid to be again carried by the stream of liquid under treatment to said place of separation, and withdrawing another part of the so returned liquid after it has acted on the liquid under treatment, substantially as described.

4. Apparatus for treating one liquid with another not perfectly miscible therewith, primarily petroleum with sulfuric acid or an aqueous solution of alkali, comprising an agitator arranged for bringing streams of said liquids into contact with each other under such conditions of agitation and flow that part at least of the treating liquid accompanies the stream of liquid under treatment and for agitating the liquids thus flowing together repeatedly during a course of considerable extent, a separator receiving the mixed liquids from said agitator and arranged to separate them from each other by difference of specific gravity, and means for returning part at least of the so separated treating liquid in partly spent condition to said agitator, substantially as described.

5. Apparatus for treating one liquid with another not perfectly miscible therewith, primarily petroleum with sulfuric acid or an aqueous solution of alkali, comprising an agitator arranged for bringing streams of said liquids into contact with each other under such conditions of agitation and flow that part at least of the treating liquid accompanies the stream of liquid under treatment and for agitating the liquids thus flowing together repeatedly during a course of considerable extent, said agitator having an inlet for fresh treating liquid nearer to and an inlet for partly spent treating liquid farther from the outlet for said mixed liquids, a separator receiving the mixed liquids from said agitator and arranged to separate them from each other by difference of specific gravity, and means for returning part at least of the so separated treating liquid in partly spent condition to said agitator at said inlet for partly spent treating liquid, substantially as described.

6. Apparatus for treating one liquid with another not perfectly miscible therewith, primarily petroleum with sulfuric acid or an aqueous solution of alkali, comprising an agitator arranged for bringing streams of said liquids into contact with each other under such conditions of agitation and flow that part at least of the treating liquid accompanies the stream of liquid under treatment and for agitating the liquids thus flowing together repeatedly during a course of considerable extent, a separator receiving the mixed liquids from said agitator and arranged to separate them from each other by difference of specific gravity, and means for returning part at least of the so separated treating liquid in partly spent condition to said agitator and for withdrawing part of the so returned liquid after it has acted on the liquid under treatment, another part of the so returned liquid being carried over from the agitator into the separator, substantially as described.

In testimony whereof I affix my signature in presence of two witnesses.

DAVID M. ALLAN, JR.

Witnesses:
A. W. FORMAN,
G. W. LOUGH.